

# BRITISH CHEMICAL AND PHYSIOLOGICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

OCTOBER, 1938.

### I.—GENERAL; PLANT; MACHINERY.

**Trend of chemical engineering.** W. M. CUMMING and F. RUMFORD (Chem. and Ind., 1938, 851—859).

**Modern equipment for a thermochemical laboratory.** G. BEAUSEIGNEUR (Chaleur et Ind., 1938, 19, 366—377).—Various types of calorimeter, thermostats, and apparatus for measuring temp. and heats of reaction are illustrated. R. B. C.

**Piston ring and cylinder wear.** M. O. TEEFOR (J. Soc. Auto. Eng., 1938, 42, 137—140, 156r).—Data given for cast Fe and nitrided steel show that piston ring and cylinder wear are related to the ease with which the material comes apart and the size and properties of the particles which break loose from the surface. The particles should be small, giving a polishing action and little surface disturbance. The breaking away of particles depends on speed and loading and on the structure of the material. R. B. C.

**Permissible load in high-speed lubricated bearings.** H. THOMA (Forsch. Geb. Ingenieurw., 1938, 9, 149—158).—The bearing temp. permissible in service are restricted by the low  $\eta$  of the oil, and by the gas discharge and evaporation phenomena within the oil film. An analytical investigation of the thermal stability of oil films confirms the established practice with regard to the max. permissible temp. rise in high-speed bearings, viz., that this should not exceed 50°. Calculations of the pressure in the bearings indicate 50 kg./sq. cm. as the max. permissible val. which is independent of the dimensions of the bearing, the surface (peripheral) speed, or the oil  $\eta$ . An expression for calculating the bearing play required under various conditions is given. R. B. C.

**Application of catalysis in industry.** E. B. MAXTED (Chem. and Ind., 1938, 759—766).—Jubilee Memorial Lecture.

**Rapid reaction agglomerating furnace.** ANON. (Ind. Chem., 1938, 14, 268—271).—The design of the St. Jacques furnace for agglomerating dry dust collected on a blast-furnace plant is described. The dust, in an air-stream, is injected tangentially into the top of a vertical cylindrical furnace so that it travels spirally downwards along the walls, while hot combustion gases from gaseous, liquid, or solid fuel enter the base tangentially in the same direction. The two spirals merge above the combustion zone and travel up the centre of the furnace, the dust particles being thus rapidly heated (5—6 sec.) from the walls and flame. The product is removed from

the base of the furnace, and the waste heat of the gases is used for heating the air for combustion and, if necessary, for drying the charge. The furnace is also suitable for, e.g., flash-roasting sulphide ores or calcining  $\text{CaCO}_3$  or phosphate rock. I. C. R.

**Effect of an accumulator on the drying efficiency of steam-heated dryers.** W. MÜLLER (Z. Ver. deut. Ing., 1938, 82, 591—592).—A Cu spiral through which a flow of  $\text{H}_2\text{O}$  was maintained was placed inside a heat-insulated Fe container provided with steam and air inlets, and the heat transfer to the  $\text{H}_2\text{O}$  from various steam-air mixtures was measured. The heat transfer decreased with increasing % of air in the steam. R. B. C.

**Prevention of dissolved gases in boiler feed-water.** L. B. ROURKE (Eng. Boiler House Rev., 1938, 52, 24, 26, 28, 98, 100, 102, 104).—Practical hints are given. R. B. C.

**Regulation of [boiler feed-water] degasifiers on the steam side.** R. WISSEL (Arch. Wärmewirts., 1938, 19, 137).—A discussion. R. B. C.

**Steam temperature as a function of the output of a steam boiler and the carbon dioxide content of the flue gases.** F. W. J. WESTENDORP (Wärme, 1938, 61, 456—458).—An example shows the method of calculating the temp. of the superheated steam. R. B. C.

**Recent designs of mercury steam-power plant.** F. MÜNZINGER (Z. Ver. deut. Ing., 1938, 82, 99—101).—An illustrated review. Zn, Zr, or Na is added to Hg to improve heat transfer. R. B. C.

**Operating experience with a hydrogen-permutit water-softening plant.** S. SCHUBERT (Arch. Wärmewirts., 1938, 19, 129—131).—A plant for reducing the total hardness of boiler feed- $\text{H}_2\text{O}$  from 24.5—35° to 0.01—0.07° is diagrammatically described. The  $\text{H}_2\text{O}$ , after being filtered through a bed of mixed H- and Na-permutit, is mixed with steam condensate containing  $\text{Na}_3\text{PO}_4$  and then degasified. The bed is regenerated by treatment with HCl and NaCl and washing with  $\text{H}_2\text{O}$ . A boiler using the softened  $\text{H}_2\text{O}$  showed no signs of corrosion after a long period of service. R. B. C.

**Behaviour of sodium sulphite in high-pressure steam boilers.** R. M. HITCHENS and J. W. PURSELL (Trans. Amer. Soc. Mech. Eng., 1938, 60, 469—473).—By means of the highly sensitive tests described for sulphide and sulphite in condensed steam and for sulphide in boiler- $\text{H}_2\text{O}$  it was found that hydrolysis of  $\text{Na}_2\text{SO}_3$  in boiler- $\text{H}_2\text{O}$  does not occur up to a boiler pressure of 1775 lb./sq. in. Tests on  $\text{H}_2\text{O}$  in boilers operating at 675 and 1400 lb./sq. in. showed no

autoxidation and reduction of  $\text{Na}_2\text{SO}_3$ ; slight traces of such a reaction, however, were found when operating at 1775 lb./sq. in. The slowness of  $\text{Na}_2\text{SO}_3$  consumption in the three boilers precluded any significant autoxidation and reduction at the metal surfaces. Of the small amounts of  $\text{Na}_2\text{SO}_3$  carried into the steam mechanically, a portion decomposed to  $\text{Na}_2\text{S}$  and  $\text{Na}_2\text{SO}_4$  as the  $\text{H}_2\text{O}$  spray became conc. or dried. The amounts present, however, were too small to be of significance.  $\text{Na}_2\text{SO}_3$  can be employed in boilers with no harmful effects. A continuous feed to maintain 10–20 p.p.m. of  $\text{Na}_2\text{SO}_3$  in the  $\text{H}_2\text{O}$  is recommended. R. B. C.

**Chlorination of condenser cooling water.** R. B. MARTIN (Trans. Amer. Soc. Mech. Eng., 1938, 60, 475–483).—An illustrated review. R. B. C.

**Slag-bottom [boiler] furnaces [fired with pulverised coal].** W. E. CALDWELL (Combustion, 1938, 10, No. 1, 18–21).—Their advantages are discussed. Typical designs are illustrated. R. B. C.

**Boiler firing with coke breeze.** G. WOLTMANN and W. LASS (Wärme, 1938, 61, 399–402).—Data for various types of boiler fired with coke breeze up to 10-mm. size are given. Easier operation and lower initial and maintenance costs are obtained with a  $\text{H}_2\text{O}$ -tube boiler fitted with a compartmented travelling grate than with a flame-tube boiler either hand-fired or fitted with a travelling grate. R. B. C.

**[Prevention of emission of] smoke and fly ash from spreader stokers [of boiler furnaces].** J. F. BARKLEY (Combustion, 1938, 9, No. 12, 37–38).—Preventive measures, *e.g.*, control of air-fuel ratio, are summarised. R. B. C.

**Selection of economisers and air preheaters [for steam-boiler plant].** J. H. SENGSTAKEN (Combustion, 1938, 10, No. 1, 22–24).—A practical discussion. R. B. C.

**Influence of air content of steam on heat transfer in heat interchangers.** H. CLAASSEN (Wärme, 1938, 61, 403–405).—A curve shows that the heat-transfer coeff. decreases as the % of air in the steam increases. Possible methods of improving the efficiency of the interchanger, *e.g.*, by use of increased steam velocities, are discussed. R. B. C.

**Heat-transfer coefficients in staggered tube banks.** C. C. WINDING (Ind. Eng. Chem., 1938, 30, 942–947).—Tests on similar banks of round, oval, and streamline tubes when heating air show that the heat-transfer coeffs. for streamline tubes are > those for round or oval tubes for 1 or 2 rows. For 3 or more rows the figures for round tubes are the highest, whilst those for streamline tubes are lower and those for oval tubes are the lowest. F. J. B.

**Suggestions for research on heat transfer to boiling liquids.** H. CLAASSEN (Z. Ver. deut. Ing. Verfahrenstech., 1938, 30–31).—A discussion. R. B. C.

**Determination of condensate exit temperature in steam-heated apparatus with vertical heating surface.** E. KIRSCHBAUM (Z. Ver. deut. Ing. Verfahrenstech., 1938, 14–16).—An expression for

calculating the cooling of the condensate as a function of the condensation temp. of the liquid and the wall temp. is derived. Good agreement between experimental and calc. results is obtained. R. B. C.

**Properties of the Fourier solution in complex systems; application to the distribution of a flux between substances in contact.** P. VERNOTTE (Compt. rend., 1938, 206, 1286–1288).—Theoretical. The distribution between two media of a heat flux originating at their point of contact is considered. A. J. E. W.

**Partition of a heat flux occurring at the interface of two limited media in imperfect contact. Paradox according to the propagation of heat.** P. VERNOTTE (Compt. rend., 1938, 207, 124–126).—Theoretical (cf. preceding abstract). W. R. A.

**Thermal conductivity of solidified gases and liquids.** A. EUCKEN and H. ENGLERT (Z. ges. Kälte-Ind., 1938, 45, 109–118).—An apparatus similar to that of Schleiermacher, in which observations are made during the variable state, the conductivity being calc. from the rate of temp. rise of the central wire when carrying a steady current, was employed. Data for  $\text{C}_6\text{H}_6$  and glycerin at 0° and –78.5°, for  $\text{CO}_2$  at –78.5° and –103.9°, and for  $\text{NH}_3$  at –103.9° are tabulated. R. B. C.

**Errors in measuring temperature in gas streams due to conduction and radiation of heat from the point at which the temperature is measured.** W. TEWES (Arch. Wärmewirts., 1938, 19, 189–191).—The mean and true errors are calc. R. B. C.

**Refrigerating installations. III. Evaporator and condenser types. IV. Other systems of refrigeration.** J. J. PIQUÉ (Dairy Ind., 1938, 3, 250–253, 296–299; cf. B., 1938, 991).—III. The efficiency of various types is discussed.

IV. Adsorption, absorption, resorption, and vac. systems are described. W. L. D.

**Operation of completely automatic cooling plants.** S. JUNG (Z. ges. Kälte-Ind., 1938, 45, 81–88).—Factors involved in the operation of small refrigerators and brine coolers used for air-conditioning are discussed and various types of control equipment described. R. B. C.

**Requirements of insulating materials in cooling chambers.** I. S. CAMMERER (Z. ges. Kälte-Ind., 1938, 45, 88–91).—Data showing the rate at which  $\text{H}_2\text{O}$  is absorbed by various insulating materials, *e.g.*, cork and bituminised slag wool, under cold-storage conditions are tabulated. The harmful effects of such absorption are discussed. R. B. C.

**Cleaning and separating materials by froth flotation.** K. WINNACKER (Z. Ver. deut. Ing. Verfahrenstech., 1938, 35–41).—A review. R. B. C.

**Fine crushers.** K. MEISTER (Z. Ver. deut. Ing. Verfahrenstech., 1938, 54–59).—Roll crushers, ball mills, and hammer mills are illustrated, and their suitabilities for crushing various materials discussed. R. B. C.

**Pulverisers with air separation and air drying.** W. A. KOREN (Ind. Eng. Chem., 1938, 30, 909–915).

—Mechanical air separators or "whizzers" are claimed to be more efficient and to give greater output of grinding plant than stationary types of cyclone separator. Drying, while grinding, by the introduction of hot gases into the stream of air used for classification is recommended where the moisture content of the raw feed is not too great. F. J. B.

**Grinding machinery for production of [coal, coke, cement, etc.] dust.** H. J. HERMISSON (Z. Ver. deut. Ing. Verfahrenstech., 1938, 75—80).—An illustrated review. R. B. C.

**Ball, rod, and tube mills.** W. H. WHITINGTON (Ind. Eng. Chem., 1938, 30, 897—904).—A general account is given of ball, pebble, and rod mills in which guidance is given in the selection of suitable grinding and auxiliary plant for sp. cases. F. J. B.

**Multiple use of pebble and ball mills.** E. M. UNDERWOOD (Ind. Eng. Chem., 1938, 30, 905—908).—With the advent of new materials of construction and the jacketing of ball and pebble mills they may be used for general process work where controlled heating or cooling under pressure or vac. together with mixing are required. The extent of the grinding taking place at the same time may be varied by varying the ball size, wt., and load. F. J. B.

**Testing the fineness of air-blown materials.** J. HEYD (Keram. Rund., 1938, 46, 234—236, 247—248, 269—270).—Sieve-analysis, air-flotation, elutriation, and microscopical methods of determination are described and a qual. "feel test" by smearing is outlined. J. A. S.

**Determination of the specific surface of powders.** I. P. C. CARMAN (J.S.C.I., 1938, 57, 225—234).—It is shown that the sp. surface,  $S_0$ , is inversely  $\propto$  the square root of the permeability,  $K$ . An equation relating the two includes terms for porosity,  $\epsilon$ , and for  $\eta$  of the liquid used, both of which are readily measured. Thus, by determining  $K$ ,  $S_0$  can be calc. A simple experimental arrangement for measuring  $K$  is described. The equation is tested over a wide variety of conditions, including mixed sizes and irregular shapes of particle. The smallest particle size tested is  $\sim 0.1$  mm. The method is more suitable for rapid routine work, and more accurate, than are the indirect methods of calculating  $S_0$  from size-distribution analyses.

**Comparison of the specific surfaces of finely-divided solids.** P. J. ASKEY and C. G. P. FEACHEM (J.S.C.I., 1938, 57, 272—276).—Areas are compared by measuring amounts of A adsorbed at liquid air temp., from which saturation vals. of van der Waals adsorption are obtained by a simple graphical construction. The method and apparatus used are described in detail.

**Mechanical separation of liquids [from solids].** E. HEGELMANN (Z. Ver. deut. Ing. Verfahrenstech., 1938, 97—103).—An illustrated review. R. B. C.

**Filtration through compressible homogeneous sludges.** T. SHAW (J. Chem. Eng. China, 1936, 3, 47—52).—Data for the rate of flow through filter cake, made up with No. 7 Pb shot, as a function of

compression, free vol., and capillary diameter are recorded. The rate decreases very rapidly at high compressions. The Poiseuille equation does not hold. CH. ABS. (e)

**Determination of the utility of filter-cloths.** E. TROJE (Z. Wirts. Zuckerind., 1938, 88, 383—384).—The required properties of filter-cloths and the possibility of their attainment with natural and artificial fibres are discussed. I. A. P.

**Outflow measurements with sharp-edged orifices.** W. SCHULTES, K. JAROSCHEK, and H. WERKMEISTER (Forsch. Geb. Ingenieurw., 1938, 9, 126—138).—Whilst standards have been established for nozzles and orifices for measuring fluid flow through closed pipelines, none is available for outflow measurements in which the discharged vol. is determined from the head prevailing in the container. The head and the effect of the discharge nozzles or orifices are influenced by the flow conditions within the container, which so far have not been sufficiently elucidated. The most important of the prevailing phenomena are discussed with a view of formulating a standardised method of outflow measurement. R. B. C.

**New nozzle shapes for low and medium Reynolds numbers.** W. KOENNECKE (Forsch. Geb. Ingenieurw., 1938, 9, 103—125).—The nozzles for low Reynolds nos. ( $R$ ) described in the German specifications do not permit reliable measurement of fluid flow at vals. of  $R < 10^5$ , when the flow coeffs. are appreciably affected by the orifice ratio. Their field of application is therefore restricted to the measurement of large discharge quantities and to media of low  $\eta$ , e.g.,  $H_2O$  and steam. Experiments leading to the development of nozzle shapes possessing const. flow coeffs. over a wide range of  $R$  and orifice ratios are described. R. B. C.

**Fractional distillation—a gas-scrubbing process.** H. M. LOWE (Gas World, 1938, 109, Coking Sect., 91—95).—The application to fractional distillation of principles derived from the closely parallel process of scrubbing is discussed and laboratory fractionating apparatus utilising these principles is described. A. R. PE.

**Condensation of mixed vapours.** J. L. WALLACE and A. W. DAVISON (Ind. Eng. Chem., 1938, 30, 948—953).—The heat-transfer coeffs. for a range of EtOH- $H_2O$  mixtures have been explored, using a single  $H_2O$ -cooled tube provided with inserted thermocouples and calibrated in terms of pure vapours,  $H_2O$ ,  $C_6H_6$ , and PhMe. Discrepancies which appear in the figures are accounted for by dropwise condensation occasioned by the high rate of condensation. F. J. B.

**Chemical dehumidification of air.** A. WEISSELBERG (Chem. Met. Eng., 1938, 45, 418—421).—Methods are reviewed with special reference to the use of LiCl and  $CaCl_2$ , and the advantage of using dehumidified air for drying materials is discussed. A. R. PE.

**Loss of pressure due to flow of compressed air in mains.** A. S. FORD (Colliery Eng., 1938, 15, 262—263).—A nomogram is given. R. B. C.

**Conception of density, physical quantities relating thereto, and their use in ceramics.** H. CASSAN (Chaleur et Ind., 1937, 18, 511—516; 1938, 19, 216—222).—Technical terms relating to  $d$  are defined, and methods for determining  $d$ , including Le Chatelier's volumometer, the pycnometer, and the hydrostatic balance, are diagrammatically described.

R. B. C.

**Definition of consistency and theoretical considerations.** M. MOONEY (Amer. Soc. Test. Mat. Symp. on Consistency, 1937, 9—14).

R. B. C.

**Reproduction of daylight for examination of colours.** J. PINTE and R. TOUSSAINT (Rev. Gén. Mat. Col., 1938, 42, 282—284).—Light of different  $\lambda$  varies greatly in intensity with the source (grey and blue skies, direct sunlight, and incandescence lamps). The importance of reproducing average daylight is discussed, and a suitable lamp employing three dichromatic filters with a single source of light is described.

R. J. W. R.

**Porosity of rocks.** **Brown-coal coke for power station.**—See II. **Circulation apparatus for pulp digesters.**—See V. **Sublimation of Dryice.**—See VII. **Welding pressure vessels.** Pb.—See X. **Protecting steam-power plant.**—See XI. **Soap-base dryer.** **Conveyor technique.**—See XII. **Paint mills.**—See XIII. **Steam-jet evacuators in sugar industry.**—See XVII. **Chemical engineering in fermentation etc. industries.**—See XVIII. **Determining hardness of H<sub>2</sub>O.** **Water-pipe corrosion control.**—See XXIII.

See also A., I, 470, **Mixed indicator to replace Me-orange.** 479, **Measuring  $\gamma$  of liquids, and  $\eta$ .**

## PATENTS.

**Furnace and its method of operation.** K. M. SIMPSON (U.S.P. 2,078,348, 27.4.37. Appl., 21.2.36).—In an open-hearth furnace, all the fuel (oil) and a minor part of the air are injected downward through the roof to impinge on the charge, the major part of the air flowing in horizontally.

B. M. V.

**Furnace construction.** J. K. MANN and M. H. DETRICK CO., LTD. (B.P. 487,435, 20.2.37).—A furnace for heating oil or the like in tubes by both radiation and convection is described.

B. M. V.

**Furnaces.** S. L. BENGSTON. From L. LEE (B.P. 488,233, 14.5.37).—A method of operating a walking-beam bed is described.

B. M. V.

**Retorts for low-temp. carbonisation, distilling acetate of lime, and the like.** P. HADAMOVSKY (B.P. 488,313, 18.3.37).—Arrangements of tubes carrying heat-transmitting medium around the walls and forming partitions in the retort are claimed, no direct heat being supplied.

B. M. V.

**[Blast]-roasting of cement, lime, or similar material.** METALGES A.-G. (B.P. 488,320, 20.4.37. Ger., 20.5.36).—Methods of building up nearly dry granules of cement slurry (and fuel) without using returned sinter, and the roasting of them in a blast roaster, are claimed.

B. M. V.

**Apparatus for drying materials, particularly materials in the form of slurry.** E. ALLEN & CO.,

LTD., and T. H. VARAH (B.P. 487,496, 21.11.36).—A rotary drum, combined with or separate from a rotary kiln, is divided internally into outer annular sectors and inner sectors, the division between the two sets being perforated so as to retain tumbling bodies in the outer sectors and to permit material to pass. Central baffles are provided to cause the hot gases to pass alternately inwards and outwards; chains may also be provided.

B. M. V.

**Heat-treatment of materials enclosed in cans or similar containers.** THERMAL ENG. CORP., Assocs. of H. L. SMITH, jun., and L. N. JONES (B.P. 488,230, 14.4.37. U.S., 25.4.36).—Cylindrical containers are partly filled and are conveyed in such a position that the centre of gravity of the contents is not on the axis; during the heat-treatment they are rolled or oscillated.

B. M. V.

**Heat-radiating combustion flues for furnaces.** SURFACE COMBUSTION CORP. (B.P. 487,764, 1.3.38. U.S., 1.3.37).—The units comprise 3 horizontal tubes all connected together at the end inside the hot chamber; the other end of the lowest tube is provided with a burner and the other two tubes are provided with exhausting means, all these ends being outside the heated chamber.

B. M. V.

**Heat-radiating tubes.** SURFACE COMBUSTION CORP. (B.P. 488,101, 30.12.36. U.S., 30.12.35).—In a hairpin form of combustion tube the cross-section is varied along the length.

B. M. V.

**Plate heat exchangers for fluids.** W. W. TRIGGS. From E. AHLBORN A.-G. (B.P. 487,840, 24.12.36).—Thin plates (forming the heat-transmitting surfaces) are separated by sinuous ducts alternately formed of metal and of rubber or other yielding material. The outer and other thick metal parts may have rubber gaskets inserted in their edges.

B. M. V.

**Plate heat exchangers for fluids.** A. SWAN and A. A. GRIFFITH (B.P. 488,571, 9.1.37).—Alternate corrugated and flat sheets are assembled with the former alternately at 90° (4 sheets to a cycle); all points of contact are joined in metallic union to give great rigidity.

B. M. V.

**Heat exchangers for fluids applicable to the conditioning of air.** G. G. BELL (B.P. 488,591, 23.3.37).—The claims relate to the casting, by means of wax patterns, of an exchanger comprising cast walls from which protrude wires or strips of good-conducting metal.

B. M. V.

**Fluid-circulating cleaning composition.** E. W. ADAMS and T. H. ROGERS, Assrs. to STANDARD OIL Co. (U.S.P. 2,063,972, 15.12.36. Appl., 25.5.32).—The removal of grease and incrustation deposits from fluid-circulating systems, e.g., engine or steam radiators, can be effected *in situ* by circulating therethrough a cleaning agent compounded of H<sub>2</sub>O 24, a light mineral oil 0.5—2, and inhibited 25—30% H<sub>2</sub>SO<sub>4</sub> 0.3—1 vol., such acid being recovered from petroleum oil acid sludge.

H. C. M.

**Insulating bodies adapted to stand high temperatures.** GEN. ELECTRIC Co., LTD. From PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN

M.B.H. (B.P. 487,957, 19.1. and 16.9.37).—Pure  $\text{Al}_2\text{O}_3$  is sintered at approx.  $1500^\circ$  with 1—20% of  $\text{CaO}$  and 0.1—1.5% of  $\text{SiO}_2$ . B. M. V.

**Cooling mixtures.** H. STRATMANN and F. G. WERNER (KÜHLSOLE-WERK STRATMANN & WERNER) (B.P. 473,808, 31.10.36).—A cooling mixture for radiators, heat-exchange apparatus, etc. is composed of an emulsified solution of one or more salts, stabilised so that a durable protective film is formed on metal surfaces, and the corrosive activity of the solution is neutralised; e.g., 5 pts. of an emulsion of asphalt 100 pts. and  $\text{C}_6\text{H}_6$  5 in  $\text{H}_2\text{O}$  100 and  $\text{C}_{10}\text{H}_7\text{SO}_3\text{H}$  (I) 2 are emulsified with a cooling mixture consisting of  $\text{MgCl}_2$  25 and  $\text{CaCl}_2$  8 in  $\text{H}_2\text{O}$  100 pts. by wt. (I) acts both as stabiliser and emulsifier, but separate substances may be used for each function.

I. C. R.

**Catalysis.** [Treatment of contact mass.] HOUDRY PROCESS CORP., Assrs. of E. J. HOUDRY (B.P. 478,592, 20.7.36. U.S., 27.8.35. Cf. B.P. 416,025; B., 1934, 1010).—In a cyclic catalytic process where different chemical operations are effected in sequence in presence of the same contact mass containing a component unfavourable to one reaction but favourable to the other, the component is poisoned prior to the reaction for which it is unfavourable and regenerated between that reaction and the one for which it is favourable. The component may be a metal or metallic compound of Fe, Mn, Ni, Cu, or Co, which may be poisoned, e.g., by  $\text{H}_2\text{S}$ , before the one reaction and reconverted into the metal before the other reaction, e.g., by blowing with a gas containing  $\text{O}_2$ , with or without steam. Its use in the catalytic conversion or cracking of mineral oil is indicated. D. M. M.

**Softening of water and dissolving calcium salts with thiotetraphosphates.** A. H. FISKE and C. S. BRYAN, Assrs. to RUMFORD CHEM. WORKS (U.S.P. 2,067,628, 12.1.37. Appl., 15.5.36).—By addition of alkali thiotetraphosphates, e.g.,  $\text{Na}_6\text{P}_4\text{O}_{12}\text{S}$ ,  $\text{Na}_6\text{P}_4\text{O}_{10}\text{S}_3$ , to hard  $\text{H}_2\text{O}$ , pptn. of Ca salts is prevented. F. M. L.

**Treatment of water so as to prevent effects of hardness.** A. G. FREEBORN (B.P. 487,595, 18.11.36).—Unidirectional current at  $>12$  mv. is applied to suitable submerged electrodes. B. M. V.

**Apparatus for testing the hardness of water.** PERMUTIT CO., LTD., and H. S. LAWRENCE (B.P. 486,810, 23.3.37).—Soap is admixed with a flowing sample and the foam (if formed) causes the  $\text{H}_2\text{O}$  to take a different path from the hard  $\text{H}_2\text{O}$ , e.g., by attaching itself to a plate or rod inclined rather above the surface of the hard  $\text{H}_2\text{O}$  and sending the frothy liquid to the other side of a rocking trough. B. M. V.

**Cleaning of steam.** W. T. SHERMAN, Assr. to SOCONY-VACUUM OIL CO., INC. (U.S.P. 2,078,779, 27.4.37. Appl., 23.6.34).—Saturated steam, withdrawn from the place of formation, makes contact with another body of  $\text{H}_2\text{O}$  low in solids; entrained liquid is separated and returned to the main body of scrubbing liquid, but a small proportion is bled off from the main body and replaced by a still smaller proportion of feed- $\text{H}_2\text{O}$ , the temp. of which is below

that of the steam so that the make up is largely pure condensate, whereby the content of solids is maintained low. B. M. V.

**Flotation apparatus for concentrating ores.** E. H. HOAG (B.P. 488,588, 5.3.37).—The cell is divided longitudinally into agitation and quiescent chambers, the latter being provided with longitudinal aëration means at different levels and the former (which is preferably central) with mechanical agitators and transverse baffles. B. M. V.

**Flotation of minerals.** E. GAYFORD, J. H. HEGINBOTHAM, and P. D. P. HAMILTON, Assrs. to GEN. ENG. CO. (U.S.P. 2,073,148, 9.3.37. Appl., 14.6.34).—Air under pressure is introduced into the bottom of a body of mineral pulp through a no. of small apertures and made to pass upward against the wet bottom of a disc, which is revolved at sufficient speed to disperse the air in minutely disseminated form throughout the body of the pulp without unduly agitating the latter. D. M. M.

**Ore-reducing machine.** A. D. HADSEL (U.S.P. 2,078,708, 27.4.37. Appl., 14.10.33).—The material is elevated by buckets on a wheel and dropped on a "breaker medium." Means for screening are embodied, the process being a wet one. B. M. V.

**Crushing, grinding, and pulverising machines.** E. ALLEN & CO., LTD., and G. N. NICHOLSON (B.P. 487,943, 28.12.36).—Protector plates for the supporting discs of swing hammers are described. B. M. V.

**Triturating machines.** M. ROCA and E. GUIX (B.P. 487,638, 31.8.37. Spain, 17.11.36).—Feeding from a hopper is effected by a roll with claws, and shredding by the interaction of stationary and rotating knives. B. M. V.

**Sifting apparatus for materials in a finely-divided state.** W. E. COTTON & SONS (BRASS-FOUNDERS), LTD., and W. E. COTTON (B.P. 488,301, 18.2.37).—A sieve is supported in a frame over a funnel; the frame, supported on enclosed rollers, is reciprocated by a crank of variable stroke. B. M. V.

[Manufacture of] dry powdered [dye] materials. G. LORD and G. REEVES (B.P. 488,258, 30.12.36).—Claim is made for an intimate mixture of an insol. or slightly sol. "substance" with 20—30% of the K salt of a saturated, soap-forming acid  $\leq \text{C}_{14}$ , crystallisable in  $\text{H}_2\text{O}$  and free from glycerin. The "substance" described is a  $\text{H}_2\text{O}$ -insol. dye having an affinity for cellulose acetate. B. M. V.

**Filter-presses.** MANOR ENG. CO., LTD., and E. W. MALKIN (B.P. 488,280, 30.11.37).—In a plate-and-frame press for filtering and drying clay or the like, the plates are hollow for heating steam, vac. is applied to the filtrate passages during filtration, to cause de-aëration, and drying is started by compressed air and finished under vac., with heat. B. M. V.

**Filter-presses and extrusion presses for producing foils or sheets.** A. KAMPFER (B.P. 488,040, 23.1.37. Ger., 10.6., 30.6., and 22.7.36).—For the filtration (actually extrusion of filaments which recombine) and subsequent extrusion in foil form of

highly polymerised substances that are quite fluid at 100—145° but are poor conductors of heat and are transformed at excessively high temp., the press chamber is divided into a no. of cells by hollow walls through which heating medium is passed and the plunger is correspondingly formed. B. M. V.

**Centrifugal strainer.** J. E. GAGE and J. K. ROEDEL (U.S.P. 2,078,604, 27.4.37. Appl., 10.12.35).—A cylindrical screen is rotated by the motor, and a feeding and cleaning device comprising a slotted axial tube and satellite brushes is rotated by friction from the screen hub, *i.e.*, at a different speed. B. M. V.

**Centrifugal machine.** J. S. PECKER, Assr. to AMER. CENTRIFUGAL CORP. (U.S.P. 2,078,273, 27.4.37. Appl., 30.1.35).—For a basket-type centrifuge, combined scraper-discharger and feeding means are described (59 claims). B. M. V.

**Separation of liquids from solids, and the washing of solids in fluid suspension.** V. HARMS, and DORR-OLIVER N.V. (B.P. 488,287, 8.1.37).—Series of thickeners are superposed in one tank and are in hydraulic communication at the axis, but those passages through the decks are shielded so as not, where possible, to be used for direct transfer of thick pulp. This pulp is pumped out from an annular sump at each deck and mixed with clear overflow from two stages down (or with fresh wash liquid as the case may be), and the mixture pumped back to a central feed collar one stage down or up. B. M. V.

**Separation of a lighter fluid from a heavier fluid in thermal elements and other apparatus.** A. R. J. RAMSEY. From AUTOMATIC CONTROL CORP. (B.P. 488,598, 13.9.37).—Liquid-filled thermostatic elements are so shaped that air may be eliminated by spinning them (in pairs) in a centrifuge before closing. B. M. V.

**Automatically regulating the concentration or viscosity of liquids and suspensions.** K. T. KÄLLE (B.P. 487,656, 18.11.37. Swed., 18.12.36).—The pulp is admitted [under const. head] tangentially to a vertical cylindrical vessel having both ends tapering to restricted openings, the whirling causing the surface of the pulp to become paraboloid at each opening to an extent depending on the consistency of the pulp; the variations in shape are caused to regulate the diluting liquid. B. M. V.

**Purification of liquids.** T. W. BYRD (U.S.P. 2,075,175, 30.3.37. Appl., 14.2.35).—Liquids such as dairy products, *e.g.*, cream, are filtered, sterilised, and deodorised in a centrifugal filter in which the filtering means is of a non-metallic, flexible, preferably fibrous, nature and the cream, after filtering, is atomised into a chamber where it is acted on by steam or hot air. D. M. M.

**Distribution of washing liquid in tower scrubbers.** GAS LIGHT & COKE CO., and L. SILVER (B.P. 487,946, 30.12.36).—At least two layers of troughs at 90° are provided with drip eills over the troughs below, or over the vertical plate-scrubbing surfaces as the case may be. B. M. V.

**High-vacuum distillation process.** EASTMAN KODAK CO., Asses. of K. C. D. HICKMAN and J. C.

HECKER (B.P. 487,697, 29.1.37. U.S., 31.1.36).—In a short-path, high-vac. still the active principle (vitamins, sterols, hormones) is condensed in a flushing liquid, which may be previously obtained distillate or hexyl phthalate and is recirculated over the condensing surface until of sufficient concn. B. M. V.

**Apparatus for evaporating and distilling.** B. FOX and C. E. ERICSON, Assrs. to BUENA VISTA IRON CO. (U.S.P. 2,078,377, 27.4.37. Appl., 11.12.31. Renewed 4.5.35).—The preheating of the feed, double-effect evaporation, and condensation are all effected within one shell. B. M. V.

**Facilitating the flow of fluids in pipes or conduits.** SOC. ANON. D'ÉTUDES DES BREVETS ET PROC. COANDA—SOC. COANDA (B.P. 488,321, 26.4.37. Fr., 25.5.36).—Tapered, flared, or conical elements are disposed along the axis of the pipe to form central pockets which open towards the outlet end of the pipe. B. M. V.

**Atomising or dispersing devices.** G. SCHLAGINTWEIT (B.P. 488,192, 29.12.36. Ger., 30.12.35).—Pulverulent, liquid, or gaseous material is forced or blown through apertures in an inner wall and then through apertures of different form in an outer wall, the latter being adjustable; *e.g.*, the outer wall may comprise a spiral spring subjected to less or more compression. B. M. V.

**Apparatus for physical and chemical treatment of gases with liquids.** F. GARNIER (B.P. 487,518, 11.2.37. Fr., 12.2.36).—While the gases flow upwards in a flue, atomised liquid is injected in the same direction. In a higher part impact devices acting both as further mixers and entrainment separators are provided. B. M. V.

**Means for bringing gas and liquid into intimate contact.** H. A. BRASSERT & CO., LTD. From G. DILL (B.P. 487,704, 25.2.37).—The gas flowing through a Venturi throat is subjected to a diverging spray of liquid injected in the same direction; on emergence the mixture is caused to be spread radially by smoothly curved deflectors. B. M. V.

**Apparatus for dosing liquids and gases.** G. ORNSTEIN (B.P. 487,500, 21.12.36).—A rotary meter in the main conduit for fluid under pressure turns a rotary valve which directs the main fluid to either side of a diaphragm piston (and to exhaust), and that piston works a smaller pump for the dosing fluid. The claims relate mainly to a frictionless form of rotary valve. B. M. V.

**Apparatus for reactions between liquids and gases or vapours.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 486,298, 25.9.36).—Apparatus for the process described in B.P. 483,823 (B., 1938, 885) comprises a vertical tube with a lower constricted portion in which is situated the upwardly directed capillary inlet for halogen vapour; at the top of both the wide and constricted portions there are "overflows" (co-axial traps) to cause the descending parent liquid to assume a film form. B. M. V.

**Dust-collecting apparatus.** VISCO ENG. CO., LTD., and L. C. ELLIS (B.P. 488,129, 21.5.37).—Filter bags adapted to be cleaned by agitation and/or reverse current are suspended from the closed top, and to

the open bottom is attached a comparatively heavy neck which is also attached to the fixed casing by a bellows attachment.

B. M. V.

**Extraction of dust from air and gases by spraying with liquids.** T. G. NYBORG, and MINING ENG. CO., LTD. (B.P. 487,672, 21.11.36, 13.2. and 16.7.37).—Wetting of fine dust is claimed to be complete when the fine liquid spray is introduced in the general direction of gas travel, and the gas-borne dust moves vortically through a "vena contracta" so that the solid particles tend toward the axis. In practice the quantity of the spray liquid is reduced so that stiff sludge is formed, and this is found to build up on the walls of the mixing tube in the form of a natural vena contracta from which lumps drop off at intervals, separation being then effected by simple reversal of direction of the clean gas stream.

B. M. V.

**Methods for sealing off vessels filled with gas to pressures higher than atmospheric.** GEN. ELECTRIC CO., LTD. From PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 487,891, 19.1.37).—A vitreous filling tube is attached to the main vessel and in the tube, resting against a shoulder or the like, is a blob of solder (Sn-Pb) of low m.p. and of such shape that it does not completely obstruct the tube. After filling with gas to the desired final pressure the blob is melted by external heat and allowed approx. to seal the tube on cooling. The source of pressure is then removed and the vitreous tube melted to itself at a point outwards of the blob.

B. M. V.

**Means for generating and regulating pressure [pneumatic power] gas from chemical reactions.** W. KOCHMANN (B.P. 487,400, 18.9.36).—The solid mass, which on reaction with a liquid will produce a gas, is shaped in such form that the reaction will proceed at a rate in agreement with the rate of demand of the pneumatic engine when operating at reasonable efficiency.

B. M. V.

**Psychrometric apparatus for direct reading of humidity.** J. CRAPEZ (B.P. 487,654, 5.11.37. Fr., 5.11.36).—A dry-bulb thermometer can be tilted, and a horizontal line adjusted to coincide with the wet-bulb temp. shown on a vertical stem; scales are calibrated to read R.H. ("hygrometric state") and proportion of H<sub>2</sub>O: air without calculation.

B. M. V.

**Friction element and bond therefor.** R. E. SPOKES, Assr. to AMER. BRAKEBLOK CORP. (U.S.P. 2,078,617, 27.4.37. Appl., 8.2.34).—A bond for asbestos or the like comprises a dispersion of rubber, a vulcanising agent, a condensation product of cashew nutshell liquid, and an aldehyde and a retarder of condensation (cresylic acid, PhOH, resorcinol), the product of reaction having a long chain of unsaturated linkings combining with the unsaturated linking of the rubber to prevent oxidation of the latter.

B. M. V.

**Hinges for gastight doors.** SOC. CHEM. IND. IN BASLE (B.P. 487,579, 2.12.37. Switz., 9.12.36).

**Distilling apparatus. Heat-exchange apparatus. Separating sludge from liquid.**—See II. Removing salts from solutions.—See VII. Heat

insulation.—See VIII. Electrical purification of gas.—See XI. Amine resins.—See XIII. Rectifying fermentation vapours.—See XVIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Fermentation of cellulose, cellulose humic acid, lignin, and lignin humic acid.** E. BERL and W. KOERBER (J. Amer. Chem. Soc., 1938, 60, 1596—1598).—An aerobic bacillus from horse dung (cf. Snieszko, A., 1933, 1333) or the anaerobic *Amylobacter navicula* ferments cellulose at  $p_H$  7.4 or, less well, 6, but neither has any effect on lignin or humic acids derived from lignin, cellulose, or peat. The rôle of bacteria in the formation of coal and oil is thus limited to the first stage.

R. S. C.

**Humic acid curves of coal: coalification.** D. J. W. KREULEN, C. KRUGSMAN, and D. T. J. TER HORST (Fuel, 1938, 17, 243—253; cf. B., 1934, 353).—The rates of production of humic acids (I) on treatment with O<sub>2</sub> at 200—260° have been determined for a range of coals. Not only the yields of (I) but also the O content of the residual coal (after extraction of the humic acids) increased with time of oxidation. The rate of production of (I) on re-oxidation of the residual coal was > that from the original coal. It is concluded that the oxidation of coal to (I) proceeds in stages through one or more intermediate products. On this assumption an equation representing the rate of production of (I) has been deduced; calc. rates agree well with observed rates. Relations between the rate of production under standard conditions, the energy of activation of the reaction, and the rank of the coals are discussed.

A. B. M.

**Recovery of iron pyrites from German bituminous coal.** A. DÖRING and G. ERBERICH (Glückauf, 1938 74, 537—540).—350—400 tons/day of pyrites concentrates (typical analysis given) containing 30—35% of S were obtainable from coal-cleaning plants in the Ruhr. By direct roasting or wet settling processes it is possible to obtain a material containing 40% of S.

R. B. C.

**Possibilities of recovering iron pyrites at bituminous coal-cleaning plants.** F. L. KÜHLWEIN and G. LOHMANN (Glückauf, 1938, 74, 540—546).—Experiments have shown that recovery is practicable if a cleaning process suited to the particular coal is employed. Data illustrating coal treatment for pyrites recovery by wet, dry, and flotation cleaning processes are tabulated and discussed.

R. B. C.

**Representation of results of the separation process, and investigation of accuracy of separation in coal-cleaning processes, with special reference to the operation of [float-and-sink] settling machines.** H. PAUL (Glückauf, 1938, 74, 277—284).—In cleaning coal by the float-and-sink process the efficiency of cleaning is governed by the accuracy of separation in conjunction with the separation  $d$  at which the machine operates.

R. B. C.

**Cleaning coal by the Tromp heavy-liquid process.** O. SCHÄFER (Glückauf, 1938, 74, 581—

586).—The Tromp float-and-sink process in which finely-ground magnetite in  $H_2O$  is employed as the separating liquid is diagrammatically described. Data obtained when employing the Tromp process and a wet jigging process for cleaning coal are compared. Capital and operating costs for the Tromp process are tabulated. R. B. C.

Progress in coal preparation in Belgium, Holland, and France. WÜSTER (Glückauf, 1938, 74, 546—549).—An illustrated review. R. B. C.

Slurry treatment at Bolsover Colliery. ANON. (Iron & Coal Tr. Rev., 1938, 137, 48—49).—The effluent from the coal washery, containing 10—12% of solids, is treated with 0.5 lb. of  $Ca(OH)_2$  and 0.4 lb. of Unifloc reagent per 1000 gals., and the pptd. material is filtered off and burned under boilers. R. B. C.

Solid smokeless fuel; its production, preparation, utilisation, and characteristics. M. BARASH (Gas World, 1938, 109, 129—136).—A review. A. R. PE.

Ball-mill grindability indices of some American coals. H. F. YANCEY and M. R. GEER (U.S. Bur. Mines, 1938, Rept. Invest. 3409, 9 pp.).—Tabulated data on the grindability indices (as determined by the Bureau of Mines ball-mill method) of coals of widely varying rank are presented. With bituminous coals an increase in grindability index with increase in fixed C content was observed. H. C. M.

Pulverised coal for [ore-roasting] furnaces. C. CAMPBELL (Colliery Guard., 1938, 157, 312—313).—A review. R. B. C.

Application of protective gases in production and use of brown-coal dust. H. STIMMEL (Z. Ver. deut. Ing. Verfahrenstech., 1938, 26—29).—By grinding brown coal and brown-coal coke, or by conveying these materials, in a gas poor in  $O_2$  and containing 5% of  $CO_2$ , the danger of explosion and spontaneous ignition is prevented. An apparatus for effecting these operations is illustrated. R. B. C.

Preparation of thin sections of coal. R. THIESSEN, G. C. SPRUNK, and H. J. O'DONNELL (U.S. Bur. Mines, 1938, Inf. Circ. 7021, 8 pp.).—The procedure used by the U.S. Bureau of Mines for (1) taking a carbonisation sample in the mine; (2) removing a column of coal from the mine for subsequent macroscopic examination and microscopic analysis; (3) preparing, for macroscopic study, relatively large polished blocks representing the entire column of coal, and (4) preparing thin sections of the coal column for microscopic analysis, is described in detail. H. C. M.

Composition of the ashes of some Indian coals and lignites. J. M. MAJUMDAR (Fuel, 1938, 17, 230—235).—The compositions of the ashes of 49 Indian coals are recorded. The ashes of the Tertiary coals differ from those of the other coals in possessing low % of  $SiO_2$ ,  $Al_2O_3$ , and  $TiO_2$  (absent in most cases), and high % of Fe, CaO, S, and alkalis. A. B. M.

Relation between volatile matter and the hydrogen-carbon ratio of coal and its banded

constituents. C. H. FISHER (Ind. Eng. Chem. [Anal.], 1938, 10, 374—378).—A plot of volatile matter against H:C ratio gives two curves, approximated by three straight lines. Vitrains and clarains lie on one curve, and fusains, attrital matter, durains, and spores on the other. A more useful relation results when volatile matter is plotted against  $(100H:C)^2$ . Vitrains and clarains fall on the upper (shorter) linear curve, and the other constituents on the lower. Applications of these relations are discussed. L. S. T.

Behaviour of activated bituminous coals and semi-cokes on carbonisation and gasification in a current of steam. C. KRÖGER and G. MELHORN (Brennstoff-Chem., 1938, 19, 257—261; cf. A., 1938, I, 364).—A semi-coke activated by addition of  $Li_2CO_3$  gave a slightly higher steam decomp. at 500—700° than did a similarly activated graphite (cf. *loc. cit.*).  $K_2CO_3-CuO$  or  $K_2CO_3-Co$  oxide was less effective in activating semi-coke than in activating graphite; e.g., a semi-coke treated with  $K_2CO_3-Co$  oxide gave a 24% conversion of steam into gas at 600°, whereas a similarly treated graphite gave a 39% conversion. No increased activation of the semi-coke was obtained by adding the oxides etc. to the coal and subsequently carbonising it in a current of steam. A. B. M.

Electrical carbonisation of bituminous coal by the Stevens process. H. GRAHN (Kohle u. Erz, 1938, 35, No. 3, 54—58).—The process is diagrammatically described. R. B. C.

Determination of minimum temperature of sustained combustion of solid fuels. H. W. NELSON, O. P. BRYSCHE, and J. H. LUM (Penna. State Coll. Min. Ind. Exp. Stat., Bull. 22, 1938, 33 pp.).—50 samples of anthracite representative of producing seams in Pennsylvania were studied by the method proposed by Wheeler (cf. B., 1924, 896).  $O_2$  was passed (200 c.c./min.) across the upper surface of a standard 5-g. sample contained in a Pyrex tube at the centre of an electric furnace, the temp. of which was raised 3.8°/min. The crossing point of the anthracite and furnace-temp. curves was taken as the min. temp. of sustained combustion. Comparison of the temp. of sustained combustion with certain properties, e.g., *d* and calorific val., of the anthracites showed that no fixed relation existed, and that a no. of other variables complicated the oxidation reaction. When the volatile matter content of each sample was plotted against the H content and the points thus defined were grouped into regions of equal crossing temp. by drawing boundary lines, a series of iso-ignition bands resulted. The peculiar curvature and trend of these bands permitted a separation of the anthracites into two groups corresponding with the free- and hard-burning anthracites differentiated in combustion practice. Meyer's method for determining the ignition temp. of anthracites (cf. B., 1932, 823), in which the change in electrical resistance with temp. is measured, was found to be unreliable. R. B. C.

Report by Director of Fuel Research on a demonstration of the Freeman assay for coal



and carbonaceous materials. DEPT. SCI. & IND. RES. (H.M. Stat. Off., 1938, 5 pp.).—The coal used was Top Hard coal similar to that employed in the official test of the Freeman low-temp. carbonising retort in 1925 (Rept. of Tests by Director of Fuel Res., Dept. Sci. Ind. Res., on the Freeman multiple retort). The data show that it is possible to calculate the correlation factor connecting the yields of tar obtainable in the Freeman assay and the Freeman retort. In the case of Top Hard coal the factor is 113%, the yield in the assay being taken as 100.

R. B. C.

Determination of calorific value of solid and liquid fuels. Calorimetric correction equation. M. VÉRON (Chaleur et Ind., 1938, 19, 282—290).—The determination of (a) the upper calorific val. at const. vol., (b) the  $H_2O$  equiv. of the bomb, and (c) the temp. correction to account for external heat exchange are discussed mathematically. R. B. C.

Decomposition temperatures of South Wales coals. T. EVANS and T. H. C. PHELPS (Proc. S. Wales Inst. Eng., 1938, 54, 169—180).—A simple apparatus and method for the rapid determination of the active decomp. points (as defined by the temp. at which there is a sudden acceleration in the rate of gas evolution) of such coals are described. The decomp. temp. rises progressively with increase of C content and of C:H ratio and with decrease of H content and of volatile matter content. H. C. M.

Heating of coke ovens by producer gas. P. G. DRURY (Gas World, 1938, 109, Coking Sect., 89—90).—The design and operation of a battery which can be heated by producer gas or rich gas, or any combination thereof, are described. A. R. PE.

Laboratory coke oven. C. C. HSIAO (J. Chem. Eng. China, 1938, 5, 3—9).—A coking chamber,  $9 \times 5 \times 1\frac{3}{4}$  in., at  $1000^\circ$  is charged with 2 lb. of graded coal. The coke compares closely with that from 500-lb. test charges as regards proximate analysis, but has higher porosity. A. R. PE.

Production and marketing of coke. W. L. BOON and G. E. FOXWELL (Gas World, 1938, 109, Coking Sect., 100—106).—Co-operation among collieries operating coke ovens to build up markets outside the Fe and steel industry is urged; possible directions of such developments and types of coke required thereby are surveyed. A. R. PE.

Grinding of brown-coal coke at Böhlen power station. K. VOIGT (Z. Ver. deut. Ing. Verfahrenstechn., 1938, 23—26).—Power requirements and costs for grinding coke to be burned in pulverised form in the boiler furnaces are given. R. B. C.

Activated carbon from bagasse. A. N. RAO and S. N. G. RAO (Current Sci., 1938, 6, 616—617).—An active form of C is made by treating bagasse with aq.  $ZnCl_2$ , drying, igniting at an optimum temp., and treating with HCl to remove and recover the  $ZnCl_2$ .

L. S. T.

Activated carbon from paddy husk. N. G. CHOKKANNA and B. T. NARAYANAN (Current Sci., 1938, 6, 617).—A method for the large-scale production of active C from paddy husk by treatment with

aq.  $ZnCl_2$ , drying, heating to an optimum temp., and finally washing with dil. HCl is outlined. L. S. T.

Formation and properties of activated carbon. E. BERL (Trans. Faraday Soc., 1938, 34, 1040—1052).—Formation of active C from carbonaceous material by different activation processes is considered. Carbons made by heating with Na compounds are less active than those obtained with the corresponding K compounds. Unlike Na, K enters the graphite lattice, forms a compound,  $C_8K$  or  $C_{16}K$ , and also forms a compound  $C_6O_6K_6$  with CO. X-Ray examination and electrical conductivity show that amorphous C is not responsible for the activity. Activity depends on the surface structure of the graphite crystals. The effect of temp. on active carbons has been studied. Hydrogenation, *i.e.*, saturation of unsaturated C atoms, renders active C inactive. The mechanism of activation by K compounds is discussed. Activation by  $O_2$  and O-containing gases (*e.g.*,  $CO_2$  or  $H_2O$ ) and with  $ZnCl_2$  and  $H_3PO_4$  is reviewed. Uses of active carbons in gas adsorption and for decolorisation and medical purposes are discussed. W. R. A.

Carbonising conditions and carbon reactivity. H. L. RILEY (Trans. Faraday Soc., 1938, 34, 1011—1016).—The physical and chemical nature of "black" C is determined by the conditions under which it has been prepared. The variation in reactivity of C towards a solution of  $CrO_3$  in  $H_3PO_4$  with the temp. of carbonisation is explained by a theory which involves the bonding of H atoms between the hexagon layer planes of the graphite crystal lattice. Evidence in support of the theory is summarised. The reactivity of C is then largely determined by the accessibility of the hexagon layer surfaces of the graphite crystallites, and small amounts of  $H_2$  or residual hydrocarbons reduce the accessibility. W. R. A.

Alteration of charcoal on storage in air. H. BERGSTRÖM (Jernkont. Ann., 1938, 122, 126—138).—Wood charcoal absorbs  $O_2$  and evolves a smaller amount of  $CO_2$  during storage, thus increasing in wt. On heating, stored charcoal evolves more  $CO_2$ , CO, and  $H_2O$  and less  $H_2$  and  $CH_4$  than fresh charcoal. M. H. M. A.

Mechanics of adsorption by means of activated carbons. M. M. BRAIDICH (J. Amer. Water Works Assoc., 1938, 30, 1299—1319).—Activated carbons that serve as gas adsorbers belong usually to a distinct class from decolorisers, and their respective properties appear to depend largely on the structural dimension of the pores in the cell walls, and not to different forms of material. The production of activated carbons, the theories of their method of adsorption, and the evaluation of their adsorptive capacity are discussed. O. M.

Pott-Broche process for pressure-extraction and hydrogenation of coal. A. SANDER (Teer u. Bitumen, 1938, 36, 269—271).—A review.

R. B. C.

Determination of forms of sulphur in insoluble residues from hydrogenated coal. R. F. ABERNETHY, H. M. COOPER, and E. C. TAPLEY (Ind. Eng. Chem. [Anal.], 1938, 10, 389—390).—(i)

Sulphide S is determined by heating the residue with 1:1 HCl, absorbing the  $H_2S$  in aq.  $NH_3-CdCl_2$  solution, and titrating the acidified solution with I. (ii) Sulphate S is determined as  $BaSO_4$  in the filtrate from the distillation residue from (i) after removal of Fe by aq.  $NH_3$ . (iii) Pyritic S, found in the residue from (ii), is measured by the usual method after a  $HNO_3$  attack, and (iv) org. S determined by Eshka's method on the residue from (iii). Total S is found by the standard Eshka procedure. L. S. T.

**Permanganate oxidation of some Indian coals.** B. B. NIYOGI (Fuel, 1938, 17, 228—229).—The O used and solubility produced on treatment of 17 Indian coals with alkaline  $KMnO_4$  under standard conditions (cf. B., 1933, 256, 451) have been determined. There is some relation between the results and the rank of the coal as measured by the fuel ratio (i.e., the ratio of fixed C to volatile matter), although there are some discrepancies. A. B. M.

**Oxidation of coal by nitric acid and determination of cellulose in lignite.** I. UBALDINI (Annali Chim. Appl., 1938, 28, 199—209).—The action of a mixture of 80% AcOH and  $HNO_3$  (10:1) on various types of lignite and coal was investigated. With more recently formed lignites (e.g., humic lignite), the lignite is almost completely dissolved, leaving a residue of mineral and cellulosic material. Cellulose is determined by conversion into glucose, which is determined by means of Fehling's solution and iodometric titration. Brown lignites examined contained 0.29—1.93% of cellulose. Data for the content of  $CO_2H$  and phenolic OH groups in lignites are tabulated. F. O. H.

**Oxidation of anthracite: effect of time of contact on concentration of oxygen in the effluent gases.** G. S. SCOTT and G. W. JONES (U.S. Bur. Mines, 1938, Rept. Invest. 3405, 7 pp.; cf. B., 1937, 200, 999).—Experiments on the oxidation of anthracite in air at temp. below its ignition point have shown that the % of  $O_2$  in the effluent gases is inversely  $\propto$  the time of contact with the heated coal at any given temp., and that the oxidation is equiv. to a first-order chemical reaction. It is concluded that the  $O_2$  content of the effluent gases from heated areas in anthracite mines is of little, if any, val. in predicting the temp. or activity of the fire. H. C. M.

**New apparatus for examining firedamp and mine gases.** R. KATTWINKEL (Glückauf, 1938, 74, 482—484).—A modified Schondorff-Broockmann apparatus is diagrammatically described.  $CH_4$  is burned over an electrically-heated Pt spiral in an improved type of combustion capillary tube. R. B. C.

**High-output gas producer with rotating and side grates for brown coal.** H. WINKELMANN (Feuerungstech., 1938, 26, 112—113).—The producer described (no illustrations given) has a central rotary grate surrounded by an annular fixed grate of tube elements through which air is supplied at the sides, thus providing two independent combustion zones. The fuel throughput can be varied from 7 to 45 tons/24 hr. without appreciably affecting the gas composition. R. B. C.

**Heating of gasworks chamber ovens with rich gas.** A. STEDING (Gas- u. Wasserfach, 1938, 81, 638—644).—A comparison is made, for several types of coal, of the costs of coal gas and producer gas as fuels, regard being had to the prices of coal and coke; certain technical points concerning the use of the richer fuel are discussed. A. R. PE.

**Refractory screen carburettor process [for production of carburetted water-gas].** N. R. JONES (Amer. Gas Assoc. Month., 1938, 20, 156—157).—A carburetted water-gas plant, in which oils high in C are used, is diagrammatically described. The screen retains C and coke dust and aids in transferring heat to the back-run steam and reversed air. R. B. C.

**Formation of emulsions in gasholder oil and water.** W. ZWIEG (Gas- u. Wasserfach, 1938, 81, 581—583).—The resinous particles deposited from town's gas containing NO may form soaps which favour emulsification. A. R. PE.

**Recovery of sulphur from fuel gases and its utilisation.** A. THAU (Z. Ver. deut. Ing. Verfahrenstech., 1938, 81—86).—Various processes for obtaining free S, and the Katasulf and Ges. für Kohlentechnik processes, are described. R. B. C.

**Developments in gas manufacture.** H. MÜLLER (Gas- u. Wasserfach, 1938, 81, 590—599).—In the detoxification process at Nordhausen the gas is heated to 350° by the combustion, over a catalyst containing Cu or Mo, of its residual  $O_2$  from S purification, and the reaction  $CO + H_2O = CO_2 + H_2$  then keeps the main catalyst at 400°. In spite of a reduced  $\alpha$  the combustion characteristics of the gas remain satisfactory, and the benzol yield is improved in both quality and quantity since org. S compounds and unsaturated hydrocarbons are hydrogenated and acid purification is rendered unnecessary. In this effect the process resembles the Rostin method of catalytic vapour-phase purification of benzol and brown-coal oils, for which, in conjunction with the Lurgi gasification, considerable development is foreseen. A. R. PE.

**Gas purification by the I.G. Alkacid process and sulphur recovery by the I.G.-Claus process.** H. BAEHR (Refiner, 1938, 17, 237—244).—A review. R. B. C.

**Recovery of ammonia from gas by the aid of gas-sulphur.** F. ROSENDAHL (Montan. Runds., 1938, 30, No. 14, 5—7).—The Katasulf and Ges. für Kohlentechnik desulphurisation processes are described. R. B. C.

**Consistency measurements in the coal-tar industry.** E. O. RHODES, E. W. VOLKMANN, and C. T. BARKER (Amer. Soc. Test. Mat. Symp. on Consistency, 1937, 30—46).—A comprehensive review. R. B. C.

**Flow of asphalts measured in absolute units.** R. N. TRAXLER (Amer. Soc. Test. Mat. Symp. on Consistency, 1937, 23—28).—A review. R. B. C.

**Chemistry of petroleum—past and present.** J. VON BRAUN (J. Inst. Petroleum Tech., 1938, 24, 393—406).—A lecture. T. C. G. T.

**Chemical trends in the petroleum industry.** P. K. FROLICH (Ind. Eng. Chem., 1938, 30, 916—922).—Increasing knowledge of the reactions of petroleum constituents is being applied primarily to the production of improved fuels and lubricants. The methods, such as catalytic hydrogenation and polymerisation, by which the mol. structure of products is being controlled are outlined. The vast potential field of development in the org. chemical industry is surveyed and illustrated by a review of the resins and plastics already commercially available from this source. T. C. G. T.

**Application of physico-chemical principles to investigation of the properties of rocks.** I. Porosity—origin, significance, and measurement. A. H. NISSAN. II. Apparatus and technique for porosity measurement. A. H. NISSAN, C. E. WOOD, L. V. W. CLARK, and A. W. NASH (J. Inst. Petroleum Tech., 1938, 24, 351—369, 370—392).—I. The theories and methods employed in the investigation of rock porosity are summarised, with particular reference to the oil industry.

II. Methods and technique for the accurate determination of porosity have been developed and are detailed. T. C. G. T.

**Distillation of Caçapava and Marahú schists.** R. C. ROQUETTE (Rev. Chim. Ind., 1938, 7, 184—186).—Brazilian schists from Caçapava and Marahú give on distillation with steam 3.1% and 20.9%, respectively, of crude oil, together with coke and  $(\text{NH}_4)_2\text{SO}_4$ . The fractions obtained by distillation of the crude oils are detailed. F. R. G.

**[Petroleum] refinery and natural hydrocarbon gases.** G. AUER (Refiner, 1938, 17, 395—398).—The analyses and properties, e.g.,  $\eta$ , of typical gases are reviewed. R. B. C.

**Cathodic protection [of metals] in petroleum refineries.** D. S. SNEIGR (Refiner, 1938, 17, 345—348).—A review. R. B. C.

**Cathodic protection of a large [natural] gas [steel] pipe passing through a steel casing filled with salt water and mud.** G. CORFIELD (Petrol. Eng., 1938, 9, No. 8, 78, 80, 83).—The technique adopted to prevent corrosion of a pipe laid in the vicinity of an electric track is described. R. B. C.

**Laboratory control of corrosion of [petroleum-] distillation equipment and of [petroleum-] desalting processes.** L. L. DAVIS, J. M. JONES, and C. A. NEILSON (Refiner, 1938, 17, 271—279).—A review. R. B. C.

**Special steels for the petroleum industry.** ANON. (Petrol. Times, 1938, 40, 183—185).—Developments are reviewed. R. B. C.

**Fractionation of mineral oils by means of gas-saturated propane.** M. GODLEWICZ (Petroleum, 1938, 34, No. 28, 1—12; cf. B., 1935, 131).—By subjecting a mixture of a crude heavy mineral oil with liquid  $\text{C}_3\text{H}_8$  to  $\text{CH}_4$  under pressure (40—130 atm.) successive fractions of the oil (beginning with the asphalt and dark-coloured substances) separate as the pressure of the  $\text{CH}_4$  is increased. The pro-  
4 G (B.)

erties ( $d$ ,  $\eta$ ,  $\eta$  index, behaviour on vac. distillation, etc.) of a no. of such fractions from various crudes are tabulated. The fractions have better  $\eta$  indices and lower  $d$  than fractions obtained from the same crude by distillation. Some experiments on the  $\text{CH}_4$  fractionation of oils dissolved in liquid  $\text{C}_3\text{H}_8$  in presence of cresol are also recorded; in this case a system consisting of three immiscible liquid phases is observed. A. B. M.

**Approximate mol. wts. of higher hydrocarbon fractions.** F. A. LUCY (Ind. Eng. Chem., 1938, 30, 959).—The b.p. of three hydrocarbon oils in the range  $\text{C}_{18-21}$  have been determined and found to conform to a Walker-type equation,  $T = 111.3 \times n^{0.4395}$ , where  $T$  is the uncorr. distilling temp. in  $^\circ\text{K}$ . at 1 mm., and  $n$  is the no. of C atoms per mol. Mol. wt. =  $(T/35)^{2.276}$  and is approx. correct down to  $\text{C}_{15}$ . T. C. G. T.

**Recent improvements in natural gasoline manufacture.** H. H. BEESON (Refiner, 1938, 17, 330—334; Nat. Petrol. News, 1938, 30, 241—244R).—A flow-sheet is given of a vapour-rectification process for recovering gasoline and  $\text{C}_4\text{H}_{10}$ — $\text{C}_3\text{H}_8$  from gas containing  $\text{CH}_4$  40.96,  $\text{C}_2\text{H}_6$  15.97,  $\text{C}_3\text{H}_8$  22.29,  $n\text{-C}_4\text{H}_{10}$  10.05, *iso*- $\text{C}_4\text{H}_{10}$  4.01,  $n\text{-C}_5\text{H}_{12}$  1.96, *iso*- $\text{C}_5\text{H}_{12}$  2.67, hexanes and heptanes 2.09. R. B. C.

**Filtering with bauxite produces economies in lubricant processing.** ANON. (Nat. Petrol. News, 1938, 30, No. 16, 195—196R).—The yield of filtered oil was 40% greater when bauxite was used in place of fuller's earth. R. B. C.

**Polymerisation and its economics.** W. A. KOEHLER (Chem. Met. Eng., 1938, 45, 412—415).—The  $\text{C}_3$  and  $\text{C}_4$  members of the gases separated in stabilising cracking-plant motor spirit can be polymerised to yield spirit of high  $\text{C}_8\text{H}_{18}$  no. The potential supply is important and two processes are described, one of which depends on heat and pressure only, whilst the other uses catalysts. A. R. PE.

**Economic aspects of polymerisation [of petroleum-refinery by-product gases].** M. B. COOKE (Refiner, 1938, 17, 325—329).—A discussion. R. B. C.

**Electrolytic plumbate process for treating gasoline.** A. S. JAMES (Nat. Petrol. News, 1938, 30, No. 12, 139—140R).—When  $\text{Na}_4\text{PbO}_4$ , obtained by electrolysis of  $\text{Na}_2\text{PbO}_2$  (I), is added to the crude gasoline, mercaptans are oxidised to disulphides without affecting the  $\text{C}_8\text{H}_{18}$  no. The (I) formed is reoxidised by electrolysis. 0.024—0.071 kw.-hr. per barrel of gasoline is required. R. B. C.

**Production of substitute motor fuels in Switzerland.** P. SCHLÄPFER (Monats-Bull. Schweiz. Ver. Gas- u. Wasserfachm., 1938, 18, 49—60).—The possibilities of producing motor fuel by coal hydrogenation, the Fischer-Tropsch process, carbonisation of wood, low-temp. carbonisation of peat and coal, gasification of wood in vehicle producers, etc. are discussed. R. B. C.

**Properties of spirits produced by hydrogenation-cracking of low-temperature tar.** J. H. CARLILE, C. M. CAWLEY, and C. C. HALL (J.S.C.I.,

1938, 57, 240—242).—The properties of the spirit vary greatly according to the nature of the catalyst used. The spirits produced in presence of a Mo sulphide catalyst supported on  $Al_2O_3$  gel and pelleted  $MoS_2$  have the following respective properties:  $d_{4}^{15}$  0.79—0.81, 0.73; proportion boiling below  $100^\circ$ , 19—27, 51—52 vol.-%; content of aromatic hydrocarbons 22—26, <2 wt.-%;  $C_8H_{18}$  no. 67—69, 69—71 (84 after addition of 4 ml. of  $PbEt_4$ /gal.). By complete conversion of the tar the yield of these spirits amounts to 101—105 vol.-%.

**Catalytic pyrolysis of vegetable seeds. Mustard seeds.** K. PING and Y. C. SUN (J. Chem. Eng. China, 1938, 5, 10—18).—The crude oil distilled from a mixture of white and black mustard seeds and  $CaO$  through a cracking chamber containing various catalysts at about  $500^\circ$  yielded on fractionation 12—17 c.c. up to  $200^\circ$ , and 3—14 c.c. at  $200$ — $300^\circ$ , per 100 g. of seeds. The yield was greatly reduced after refining with  $H_2SO_4$ , but further light oils could be obtained by cracking the distillation residue. The yield of  $(NH_4)_2SO_4$  was 3.3%. A. R. PE.

**Examination [and determination of] alcohol in alcohol-gasoline mixtures.** K. HONDA (J. Fuel Soc. Japan, 1938, 17, 63—64).—The mixture is saturated with  $EtOH$ -sol. nigrosine and the resulting tint compared with those of standards. A. R. PE.

**Stability of alcohol fuels towards cold and water.** R. HEINZE, M. MARDER, and G. ELSNER (Angew. Chem., 1938, 51, 524—526).—Data for the clouding temp. of various  $EtOH$ - $MeOH$ -petroleum spirit mixtures containing  $H_2O$  have been obtained and are presented in the form of triangular diagrams. C. R. H.

**Determination of spontaneous ignition points [of solid and liquid fuels].** H. WINTER and H. MÖNNIG (Glückauf, 1938, 74, 335—336).—Data obtained for petrol,  $C_6H_6$ , creosote oil, brown coal, cokes, etc., when using the apparatus of Jentzsch and of Feddeler, are compared. R. B. C.

**Evaporation and knock-rating [of liquid fuels].** O. HERSTAD (Oel u. Kohle, 1938, 14, 579—580; cf. B., 1938, 16).—A benzine was separated into two fractions of b.p. < and  $>128^\circ$ , respectively, and each of these was further separated into two fractions by extraction with a suitable solvent. The observed  $C_8H_{18}$  nos. of the various fractions agreed (with one exception of low  $C_8H_{18}$  no.) with those deduced by the "transformation" method, i.e., from the max. evaporation time (cf. B., 1937, 868). A relation also exists between the spontaneous ignition temp. and the  $C_8H_{18}$  nos. of the fractions. A. B. M.

**Effect of test conditions on [motor] fuel rating.** A. E. BECKER (J. Soc. Auto. Eng., 1938, 42, 63—72T).—The Co-operative Fuel Research Committee of the Society of Automotive Engineers have obtained data on the effects of spark advance, engine speed, and fuel temp. on the  $C_8H_{18}$  nos. of winter gasolines which are to be used as a basis for a laboratory engine method for rating motor fuels. R. B. C.

**Combination oil and gas burners.** O. F. CAMPBELL (Refiner, 1938, 17, 315—324, 329). R. B. C.

**Use of ammonia as a fuel for explosion engines.** P. MONTAGNE (Compt. rend., 1938, 206, 1623—1625).—Calc. data comparing  $NH_3$  with other fuels are given. The work available per cycle in a 4-stroke engine using  $NH_3$  is < that obtained with liquid fuels and  $CO$ , but slightly > that obtained when using water-gas or  $H_2$ . The work done per unit wt. of fuel is << that for liquid fuels and  $H_2$ , and > that for  $CO$  and water-gas. A. J. E. W.

**Motion pictures of engine flames correlated with pressure cards.** G. M. RASSWEILER and L. WITHROW (J. Soc. Auto. Eng., 1938, 42, 185—204T).—Further work with the camera (cf. B., 1936, 775) is described. The experimental observations consist of pictures showing successive positions of the flame at intervals of 2.4 crankshaft degrees during single explosions and pressure-time records of the same explosions. A method is described for sorting out the pressure changes due to combustion from an observed pressure card. When the pressure changes resulting from combustion are summed and put on a % basis the % of pressure rise due to combustion is  $\approx$  the % of charge burned (by wt.) at the corresponding instants in the combustion period. R. B. C.

**Projection of flame in gaseous mixtures and the phenomenon of knock in explosion engines.** P. LAFFITTE (Compt. rend., 1938, 206, 1814—1816).—The formation of "projected" flames of high velocity of propagation in regions containing no combustible (cf. A., 1938, I, 255) is illustrated by photographs, which confirm that the projection is preceded by a displacement of the gaseous mixture, followed in some cases by auto-inflammation. Knock in internal-combustion engines probably involves production of projected flames, and not of true explosion waves. A. J. E. W.

**Calculation of instantaneous temperature and pressure maxima in explosion engines.** L. REINGOLD (Compt. rend., 1938, 206, 1792—1794).—The method of calculation outlined is based on the consideration of limiting conditions in which no dissociation of the products or reactants occurs on combustion. A. J. E. W.

**Combustion process in the compression-ignition engine.** J. W. DRINKWATER and A. C. EGERTON (Proc. Inst. Mech. Eng., 1938, 138, 415—493).—The composition of the gases in the combustion chamber and cylinder of a Diesel engine running at half rated speed has been investigated by methods based on those developed previously for petrol engines (cf. B., 1934, 175; 1936, 7) and curves are given relating their pressure and content of  $O_2$  and  $CO_2$  to the crank angle under various running conditions. Only traces of aldehyde were found, even when the air intake was throttled so that combustion was very incomplete.  $NO_2$  increased during the combustion period to a max. mol. fraction of  $\sim 0.003$  and then diminished through dissociation. A. R. PE.

**Surface finish related to wear in internal-combustion engines.** K. W. CONNOR (J. Soc. Auto. Eng., 1938, 43, 305—312T; Auto. Eng., 1938, 28, 253—256).—The use of the portable profilometer

(Mech. Eng., 1938, 60, 205) for studying wear in engines and for checking and maintaining surface finish on engine parts is discussed. R. B. C.

**Artificial ageing of hydrocarbon oils.** E. H. KADMER (Chem.-Ztg., 1938, 62, 611—613).—Accelerated methods of testing the ageing properties of lubricating oils are discussed. Many differ considerably from the conditions encountered, *e.g.*, in internal-combustion engines. The method of Barnard (J. Soc. Auto. Eng., 1934, 167) has been applied to comparison of the ageing properties of fresh and regenerated mineral oils and synthetic oils. Mineral oils of low *d* are most stable, showing least increase in  $\eta$ , acid val., and sap. val. No retardation of the ageing of a heavy oil was observed in presence of 1% of Cr oleate. Rust and colloidal graphite have little effect on ageing properties. J. W. S.

**Laboratory methods of estimating the ageing tendency of mineral oils.** M. MARDER and V. TOLKMITT (Oel u. Kohle, 1938, 14, 615—619, 635—637).—Existing tests require the oil to be heated usually in contact with  $O_2$ , the ageing being measured by the change in some property such as asphalt content,  $\eta$ , acidity, or sap. val. Little or no correlation is found among the indications of these criteria and the results depend on temp., duration of heating, and partial  $O_2$  pressure. These and other factors vary under working conditions and it is suggested that test conditions must be chosen to imitate either the severest conditions of practice, or the average conditions, as indicated by correlation with engine tests. A. R. PE.

**Kinetics of oxidation of naphthenic oils by oxygen.** K. I. IVANOV (Petroleum, 1938, 34, No. 32, 1—7; No. 33, 1—7; No. 34, 1—6).—Naphthenic and paraffinic petroleum distillates have been treated with  $O_2$  at  $150^\circ/15$  atm. and the products separated into unsaponifiable matter, fatty acids, and OH-acids insol. in light petroleum. The susceptibility to oxidation increased with the severity of the prior refining with  $H_2SO_4$ . The chief products were carboxylic acids, the total yield of which approaches a limit after about 4 hr. under the above conditions, whilst that of the  $Et_2O$ -sol. fatty acids then begins to fall. The rate of oxidation rises with the partial pressure of  $O_2$ , but the course of the reaction, which is believed to occur homogeneously in the oil phase, is little affected by pressure. A. R. PE.

**Graphical representation of the viscosity-temperature function [of lubricating oils].** F. JOSTES (Oel u. Kohle, 1938, 14, 559—564).—Graphical methods of determining the val. of the “ $\eta$  pole height” and of *m* in Walther's equation,  $\log \log (\eta + 0.8) = m(\log T - \log T_1) + W_1$ , are illustrated. The pole height is of more fundamental significance than the  $\eta$  index and is preferable to the latter for characterising lubricating oils. A. B. M.

**Lubrication. I. Theory of thick-film lubrication of a complete journal bearing of finite length.** M. MUSKAT and F. MORGAN (J. Appl. Physics, 1938, 9, 393—409).—Mathematical. The hydrodynamic theory of lubrication for such a bearing is developed by successive approximation. J. A. D.

**Film lubrication of finite curved surfaces.** S. M. SKINNER (J. Appl. Physics, 1938, 9, 409—421).

**Lubrication of Diesel and Otto engines with special reference to the effect of various fuels.** W. RICHTER (Brennstoff u. Wärmewirts., 1938, 20, 67—76).—The deterioration of the lubricant in use, possible methods of improving the oils by addition agents, the causes of the sticking of piston rings, and mechanical aspects of lubrication are discussed in relation to experimental data. R. B. C.

**Composition of the fatty acid mixture recovered from oxidation of synthetic paraffin wax.** E. JANTZEN, W. RHEINHEIMER, and W. ASCHÉ (Fette u. Seifen, 1938, 45, 388—393).—The fatty acids (I val. 8.8, sap. val. 261.8) recovered from the oxidation of paraffin wax obtained by the Fischer-Tropsch process (and consisting of hydrocarbons:  $C_{16-19}$ , 27.4;  $C_{19-22}$ , 31.0;  $C_{22-25}$ , 23.7;  $C_{25-27}$ , 11.7;  $C_{28}$  1.7%) consisted almost entirely of saturated *n*-fatty acids of both the odd and even series, among which acids from  $C_8$  to  $C_{18}$  were separated and identified by fractionation of the Me esters. Acids  $< C_8$  and  $> C_{18}$  formed 7.4 and 18 wt.-%, respectively, of the total recovered (some losses are inevitable), the balance consisting of approx. equal mol. proportions of  $C_{9-15}$  acids, with only slightly smaller amounts of  $C_8$  and  $C_{16-18}$  acids. Small amounts of unsaturated and OH-acids were present. It is concluded, therefore, that all the  $CH_2$  groups of the paraffin chain are approx. equally susceptible to oxidative attack, the central groups being rather more labile than those nearer the ends of the chain. E. L.

**Erratum:** On p. 876, col. 1, line 11 from bottom, for STEVENS read STREVENS.

**Boiler firing with coke breeze. Grinding machinery for coal etc. Gas-scrubbing process. High-speed lubricated bearings.**—See I. Prep. of  $H_2$ . Purifying inert gases and  $H_2$ . Determining CO and  $O_2$  in gases.—See VII. Bituminous road materials and roof coatings.—See IX. Metallurgy of Zn and coal distillation. Austrian mining industry.—See X. Petroleum solvents.—See XIII. Mineral oils as insecticides.—See XVI. Lubricating greases.—See XVII. Activated carbons. Filter-beds for effluents.—See XXIII.

#### PATENTS.

**Coal-cleaning apparatus.** K. PRINS (U.S.P. 2,078,275, 27.4.37. Appl., 6.2.33).—A dry and non-pneumatic table having vertical and longitudinal shake is described. B. M. V.

**Drying of [combustible] material.** R. L. SUTHERLAND, Assr. to NORTHERN COAL PRODUCTS Co. (U.S.P. 2,078,515, 27.4.37. Appl., 29.6.33).—For the drying of coal, lignite, etc. an inert medium is maintained at a temp.  $<$  that causing risk of degradation or ignition of the material, and the humidity of the medium is reduced so that at that dry-bulb temp. the temp. of evaporation will be lower by a desired margin. The v.-p. differential between medium and material is controlled throughout the process by varying the quantity of medium, and that

is adjusted by varying the thickness (and resistance) of the bed acted on, the gas pressure remaining const.

B. M. V.

**Treatment of solid fuels.** C. ELLIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 2,076,497, 6.4.37. Appl., 29.12.31).—Coal, coke, etc. is rendered non-dusting by spraying it with a heated heavy hydrocarbon oil, *e.g.*, melted petroleum jelly, in the form of a fine mist, sufficient being used to coat the fuel with a thin film of the oil and the fuel preferably being agitated during spraying.

D. M. M.

**Coating [solid] fuel to prevent dusting.** P. K. FROLICH, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 2,078,755, 27.4.37. Appl., 12.12.31).—Solid fuels, *e.g.*, coal or coke, are coated with a hydrocarbon sulphonic acid compound (I) derived from acid-treated petroleum; the (I) may be neutralised and derived from the acid sludge, or may be oil-sol. and extracted from the oil with an org. solvent, *e.g.*, Pr<sup>n</sup>OH.

D. M. M.

**[Underfired] coke ovens.** SOC. GÉN. DE FOURS À COKE, SYSTÈMES LECOCQ SOC. ANON. (B.P. 487,919, 23.7.37. Belg., 24.7.36).—The supply of heating gas is led to the flues in the heating walls by means of continuous vertical pipes introduced through the brickwork of the regenerators in such a way that annular spaces are formed between the pipes and the brickwork; these spaces open into a horizontal channel communicating with the exterior in such a way that the spaces form chimneys through which a supply of fresh air suitably controlled is automatically drawn, cooling the pipes in its upward passage.

D. M. M.

**Rotary ovens for heat-treatment of coal.** SOC. FINANCIÈRE DE TRANSPORTS ET D'ENTREPRISES INDUSTRI. (SOFINA) SOC. ANON. (B.P. 488,852, 14.11.36. Belg., 12.12.35. Cf. B.P. 356,236; B., 1931, 1035).—Coal is heated to the globulation temp. in a rotary oven in such a way that a rapid rise in temp. is ensured in the globulation zone while the coal is continuously stirred, but so as to avoid any excessive release of volatile matters in this zone; this part of the oven is separated from the rest in a gastight manner and is so arranged as to prevent the dilution of the atm. by air, steam, or other gases evolved from coal at low temp. The rotary oven is externally heated by a ring-like chamber covering its full length, and the temp. is regulated by mixing heating and combustion gases.

D. M. M.

**Heat-treatment of solid carbonaceous fuels.** P. M. SCHUFTAN (B.P. 488,567, 8.1. and 22.12.37).—A continuous vertical gas-making retort with an automatic coke extractor at the bottom, but without the ordinary external heating flues, is used, and the column of fuel is heated by the direct sensible heat of the gaseous products of combustion of either a gaseous or liquid fuel with O<sub>2</sub>, or a gas of O<sub>2</sub> content > that of air, which pass upward through the column of fuel without any sensible combustion taking place between the O<sub>2</sub> and the solid fuel in the retort. Water-gas may be used as the fuel, being generated by the action of steam in the coke in the lower part of the retort, and burned with O<sub>2</sub> a little higher up it. Alternatively, an annular combustion chamber is

provided around a portion of the retort and in it gaseous or liquid fuels are burned, the products passing directly through open ports to the interior of the retort.

D. M. M.

**Conversion and coking of carbonaceous materials.** A. FISHER, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 2,073,367, 9.3.37. Appl., 2.6.33).—A mixture of finely-divided carbonaceous material with a hydrocarbon oil is passed through a coil still at moderate temp. (455—480°) and pressure (100—500 lb./sq. in.). It is then passed to a coking chamber having a refractory floor, *e.g.*, of carborundum or fused Al<sub>2</sub>O<sub>3</sub>, where coke is allowed to build up on the floor to about 6 in., being removed by a ram at intervals. The pressure in the coking chamber is preferably sub-atm. and volatile products are withdrawn and passed through a fractionating tower. Undesired fractions may be recycled.

D. M. M.

**Low-temperature carbonisation.** E. RAHM, Assr. to A. M. BARR (U.S.P. 2,072,721, 2.3.37. Appl., 1.2.35).—Coal is carbonised by passage through a horizontal cylindrical retort, externally heated and containing a ribbon-blade screw conveyor. The retort is slowly rotated in the same direction as required, by the screw propeller, to move the coal forward in the retort. The propeller is revolved at a speed > that of the retort and at frequent and uniform intervals, interspersed with equally frequent and uniform periods of idleness.

D. M. M.

**Destructive distillation at low temperature.** H. E. G. ROWLEY. From SOC. CHIM. DE LA GRANDE-PAROISSE (AZOTE & PROD. CHIM.) (B.P. 487,983, 12.7.37).—Coal, lignite, shale, etc. is passed vertically downwards through a series of chambers, in the first of which it is dried by direct contact with flue gases, and in the second acted on by a horizontal stream of inert gas, *e.g.*, superheated steam, which is recycled after removal of the products of distillation. In the third chamber the material being distilled is acted on by a gas containing O<sub>2</sub>, to burn the C and volatile substances and raise the residue to a high temp., when it is used, in the fourth chamber, for reheating the distillation gas prior to recycling it.

D. M. M.

**Destructive-distillation process for manufacture of motor fuels.** NON-POISONOUS GAS HOLDING Co., LTD. (B.P. 473,722, 14.8.36. Ger., 15.5.36).—Predried wood, peat, etc. is distilled at <480°, and the steam obtained from the drying operation is used for converting the distillation residue at the same temp. into water-gas consisting mainly of CO, CO<sub>2</sub>, and H<sub>2</sub>. This is converted chiefly into CH<sub>4</sub> and H<sub>2</sub> by heating (250°; 15 atm.; Ni catalyst). Wood impregnated with alkali carbonates from previous distillations gives a distillation residue more readily converted into gas. The resultant gaseous fuel has a heating val. of 7000—7500 g.-cal.

H. C. M.

**Distilling apparatus.** B. O. DAUBERT (U.S.P. 2,078,914, 27.4.37. Appl., 28.10.33).—A no. of horizontally-disposed, externally-heated retorts are arranged in four vertical series, the outlet of each retort except the last leading into the inlet of the retort below, and the material to be carbonised, *e.g.*, lignite, passing in succession through each retort of a

vertical series. The heating of each retort is independently controlled and the temp. rises from 370 to 430° in the top retort to 455° in the second, 510° in the third, and 580° in the fourth. Means are provided for continuously moving the material along each retort. D. M. M.

**Production of hydrocarbon products of low b.p. by treatment of carbonaceous materials at elevated temperatures.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 488,651, 7.12.36 and 10.11.37).—Liquid or fusible hydrocarbons, *e.g.*, benzines, solvents, Diesel or fuel oils, lubricants and waxes, or hydrocarbon gases, are produced by thermal treatment of carbonaceous materials containing  $> C_1$  involving rupture of C-C or C-X linkings, where X is another element, and removal of asphalts, resins, etc. in presence of catalysts comprising a component (a) consisting of preformed S compounds, *e.g.*, sulphides, and/or P compounds, *e.g.*, phosphates, of Mn, Fe, Cu, or Zn (the phosphate of Zn being always the neutral one and this being used only in presence of  $H_2$  from an extraneous source), and a component (b) consisting of one or more of the metals Mo, Sn, W, Re, U, V, Cr, Ni, or Co, or their compounds. These components are mixed and/or chemically combined and the wt. of (a) is always  $>$  that of (b), the ratio (a) : (b) varying between 51 : 49 and 99.5 : 0.5. A quoted example is FeS 85—70 and  $WO_2$  15—30 pts. D. M. M.

**Production of high-boiling products by treatment of solid carbonaceous materials with hydrogenating gases.** H. E. POTTS. From INTERNAT. HYDROGENATION PATENTS Co., LTD. (B.P. 488,856, 9.1.37).—The materials are pasted with oil and treated with  $H_2$ , or gases containing it, in amounts of  $>1000$  (1500—7000) cu.m. per ton of coal paste, at 100—1000 atm. while passing over stationary lumps of catalysts, preferably of regular form, comprising sulphides of group V or VI or of the Fe group. The temp. should be  $<415^\circ$  (330—400°) during the first quarter of the reaction time, and may be raised by steps thereafter to 430°, 450°, or 480°; ground catalysts also may be added to the paste. D. M. M.

(A) Treatment of carbonaceous materials. (B) De-aëration of granules of active carbon. H. A. INGOLS and P. F. PIE, jun., Assrs. to DARCO CORP. (U.S.P. 2,076,645—6, 13.4.37. Appl., 20.12.35).—(A) In the manufacture of activated C, for use in aq. media, the hot C is cooled in an atm. of steam until the block *d* is  $>1.0$  g./c.c. (B) Non-sinkable, granular activated C for use in aq. medium having a block *d*  $<1.0$  g./c.c. is rendered sinkable and its *d* is raised to  $>1.0$  g./c.c. by either placing it in  $H_2O$  and boiling the mixture, or moistening the granules with  $H_2O$ , heating them to 100° until the  $H_2O$  boils, and mixing the hot C granules with  $H_2O$ . D. M. M.

**Revivification of charcoal.** B. M. JOHNSON, Assr. to CARBORUNDUM Co. (U.S.P. 2,076,647, 13.4.37. Appl., 20.8.34).—Spent bone black or char which has been used for decolorising sugar solutions is revived by heating in vertical cast-Fe retorts which are mounted in rows along the sides of a heat distributor which consists of a high central arch and two lower arches, one arch being at either end of it; the relative

heights of the arches are adapted to give the hot gases a large horizontal component of velocity toward the retorts. D. M. M.

**Gas producers.** V. CHAMPY (B.P. 488,359, 17.1.38. Fr., 16.1.37).—A down-draught, ash-melting gas producer suitable for lean or anthracitic coals is provided with top air-inlet profiled orifices discharging flush with the gasification chamber and with bottom gas-outlet passages on the opposite side, disposed in front of a converging-diverging nozzle. Forms of air inlet and gas outlet are described. The ratio between the cross-section of the neck of the former to that of the latter should be between 1 : 4 and 1 : 3. D. M. M.

**Gas pyrolysis.** W. W. GARY, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 2,072,801, 2.3.37. Appl., 30.11.34).—Tubes, especially those of high-Cr steel, used in pyrolysis of gaseous hydrocarbons are treated, after burning out deposited C, with a solution containing 10 wt.-% of  $H_2SO_4$  and 20 wt.-% of NaCl for 1—2 hr., washed with  $H_2O$ , and preferably treated with conc.  $HNO_3$ , to remove oxide films, before being put into service again. D. M. M.

**Apparatus for producing water-gas.** H. J. CARSON (U.S.P. 2,072,357, 2.3.37. Appl., 14.3.34. Cf. U.S.P. 1,952,654; B., 1935, 213).—A water-gas generator designed with two air-inlet levels will permit small amounts of air to be admitted to the lower layers of the fuel bed during gas-making periods so as to maintain the temp. above that necessary to keep the ash- and clinker-forming material in liquid form. A refractory bottom is provided so that materials may be introduced to flux the clinker. D. M. M.

**Water-gas set.** G. W. PARKER (U.S.P. 2,077,579, 20.4.37. Appl., 27.7.34).—Forms of, and methods of supporting, chequer brickwork for the carburettor of a water-gas set are claimed. D. M. M.

**Gas purification.** A. L. WARD and C. W. JORDAN, Assrs. to UNITED GAS IMPROVEMENT Co. (U.S.P. 2,073,083, 9.3.37. Appl., 19.1.33. Cf. U.S.P. 1,976,704; B., 1935, 936).—Gas containing  $H_2S$ ,  $O_2$ , and N oxides is purified by first scrubbing with an alkaline solution and then passing it through solid purifying agent, *e.g.*, Fe oxide, enough  $H_2S$  being left in from the alkaline scrubbing plant to keep the metallic sulphides in the solid material in sufficient quantity to remove the N oxides. Part of the foul gas may by-pass the liquid scrubber and go straight to the solid purifiers, or one of the units of the latter may be switched from the outlet to the inlet side of the liquid scrubber. D. M. M.

**Removal of organically-combined sulphur from gases.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 478,877, 21.7.36).—The gases are treated in presence of  $O_2$  and under alkaline conditions, *e.g.*, in presence of  $NH_3$ , with an active charcoal prepared by treating mineral coal in a shaft furnace with hot combustion gases which are blown through the coal with sufficient pressure to cause strong agitation of the coal. D. M. M.

**Purification of combustible gases by washing with oils.** W. C. HOLMES & Co., LTD., D. M.

HENSHAW, and C. COOPER (B.P. 488,812, 23.6.37. Cf. B.P. 430,989; B., 1935, 891).—Gases are scrubbed with wash oil, using 30 gals. per 1000 cu. ft. to remove most of the low-boiling volatile substances, and the used oil is then stripped by distillation of these substances, which are separated by redistillation into light and heavy fractions, some or all of the heavy fraction being returned to the wash oil before re-circulation.

D. M. M.

**Treatment or regeneration of gas-scrubbing liquids.** GAS LIGHT & COKE CO., W. K. HUTCHISON, and I. G. C. DRYDEN (B.P. 488,951, 18.1.37).—Unconc. spent gas-washing liquor containing  $(\text{NH}_4)_2\text{CO}_3$  and  $\text{NH}_4\text{HS}$  is treated, prior to removal of  $\text{H}_2\text{S}$ , in concurrent flow with  $\text{CO}_2$  and  $\text{H}_2\text{S}$  removed from a pretreated portion of spent liquid, in a bubble-cap column, thus converting it into a mixture of  $\text{NH}_4\text{HCO}_3$  and  $\text{NH}_4\text{HS}$ , the surplus gases ( $\text{CO}_2$  58,  $\text{H}_2\text{S}$  42,  $\text{NH}_3$  0.03%) being available for conversion into  $\text{S}$  in a Claus kiln. In the second stage, the liquid is treated in a bubble-cap column in countercurrent with  $\text{CO}_2$ , derived from the third stage, thus removing  $\text{H}_2\text{S}$  and leaving a solution of  $\text{NH}_4\text{HCO}_3$ . In the third stage the liquid is heated to  $102^\circ$  to drive off a large proportion of the  $\text{CO}_2$  together with steam and some  $\text{NH}_3$ , the  $\text{NH}_3$  being recovered in 3 more bubble-cap sections.

D. M. M.

(A) Electrically treating tar emulsions. (B) Introducing high potentials into a dehydrator [for emulsions]. W. WOELFLIN, Assr. to PETROLEUM RECTIFYING CO. OF CALIFORNIA (U.S.P. 2,072,917-8, 9.3.37. Appl., [A] 12.2.34, [B] 26.3.34).—(A) Emulsions of the  $\text{H}_2\text{O}$ -in-tar type are separated by the action of an electric field in a separator where the  $\text{H}_2\text{O}$  may be drawn off from the top. (B) A "boot" is provided in the electric treater extending through the  $\text{H}_2\text{O}$  layer to protect the insulator carrying the conductor from the  $\text{H}_2\text{O}$ .

D. M. M.

**Treatment of tar.** A. H. RADASCH and W. M. BYWATER, Assrs. to BARRETT Co. (U.S.P. 2,075,866, 6.4.47. Appl., 21.3.34. Can., 15.7.33).—Tar is heated in a pipe-still to above its initial b.p. and then flashed, to vaporise the tar acids. These are scrubbed from the vapours in the vapour phase by reaction with alkaline reagent without condensing the neutral oil vapours, which are removed at  $165$ – $175^\circ$  and used as part of an inert gas stream to remove more tar acids from the residue from the flashing operation, in a secondary distillation. The tar acids so removed are also extracted by means of an alkaline reagent, but in a different apparatus from the first alkaline washer and with a different reagent.

D. M. M.

**Preparation of high-grade asphalts.** A. P. ANDERSON and W. K. NELSON, Assrs. to SHELL DEVELOPMENT Co. (U.S.P. 2,073,088, 9.3.37. Appl., 3.4.35).—Asphalt of low susceptibility can be produced by blending asphaltic bituminous material containing  $\approx 20\%$  of asphaltenes with an easy-flowing hydrocarbon oil of low  $\eta$  index, blowing the mixture with an oxidising gas, and blending the blown material with a highly-viscous hydrocarbon oil of low  $\eta$  index.

D. M. M.

**Production of hydrocarbons by treatment with hydrogenating gases of high-boiling carbon-**

**aceous materials which contain high-molecular substances, such as asphalts and resins.** H. E. POTTS. From INTERNAT. HYDROGENATION PATENTS Co., LTD. (B.P. 488,513, 12.4.37).—The high-boiling ( $325$ – $360^\circ$ ) oils from the hydrogenation of normally liquid or fusible carbonaceous materials are mixed with fresh stock for recycling, but pptd. asphalts etc. are removed from the mixture by sedimentation, filtration, or centrifuging before hydrogenating it.

D. M. M.

**Emulsions.** P. R. SMITH, Assr. to BARBER Co., INC. (U.S.P. 2,077,905, 20.4.37. Appl., 11.2.29).—The breaking of bituminous emulsions with a  $\text{H}_2\text{O}$  external phase, during admixture with an aggregate, e.g., trap rock, limestone, etc., is prevented by adding sufficient of a  $\text{H}_2\text{O}$ -sol. salt which will ppt. all the  $\text{H}_2\text{O}$ -sol. alkaline-earth salt of the aggregate adjacent to the surface of the aggregate in a  $\text{H}_2\text{O}$ -insol. form.

D. M. M.

[Oil-well] drilling fluids. R. and M. F. CROSS (U.S.P. 2,073,413, 9.3.37. Appl., 25.11.36).—As weighting agent the fluid contains a substantial quantity of  $\text{ZnCl}_2$ .

**Fractional separation of crude hydrocarbon oil.** F. J. CLEVELAND. From J. PINTSCH A.-G. (B.P. 476,610, 18.6.36).—A no. of carrier substances of known b.p., e.g.,  $\text{C}_6\text{H}_{14}$ ,  $\text{C}_{10}\text{H}_{22}$ ,  $\text{C}_{10}\text{H}_2$ ,  $\text{C}_{10}\text{H}_7\text{Br}$ ,  $\text{COPh}_2$ , are added to the crude oil in order of magnitude of the b.p. These substances are selected so that each one has a b.p.  $<$  the temp. at which the fraction to be collected distils in absence of the carrier, and a v.p. at the desired distillation temp. sufficient to bring the total v.p. at that temp. up to atm. pressure in the still. The mixture is distilled after addition of each carrier until the b.p. of the latter is reached, when the next carrier is added. The latter may be added as vapour or liquid.

C. C.

**Cracking of hydrocarbon mixtures.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 480,442, 21.8.36).—Coke formed during cracking of hydrocarbon mixtures, prepared from  $\text{CO}$  and  $\text{H}_2$ , is reduced in amount if the mixtures are dewaxed and refined before cracking. Refining may be with conc.  $\text{H}_2\text{SO}_4$ , metal halides, solvents, or by hydrogenation.

T. C. G. T.

**Conversion of hydrocarbon oils.** A. L. MOND. From UNIVERSAL OIL PRODUCTS Co. (B.P. 476,026, 10.5.37).—A process for the fractional distillation of hydrocarbon oils of relatively wide boiling range, e.g., crude petroleum, accompanied by the pyrolytic conversion, under independently controlled conditions, of relatively low- and high-boiling fractions of the charging stock from the topping still as well as selective further conversion, within the same system, of the relatively low- and high-boiling intermediate products of the process, whereby high yields of good quality motor fuel are produced, is claimed. Apparatus is described and figured.

H. C. M.

**Conversion of hydrocarbons.** (A) P. H. SULIVAN, (B) J. C. BLACK, Assrs. to GASOLINE PRODUCTS, Co., INC. (U.S.P. 2,072,805 and 2,075,164, [A] 2.3.37, [B] 30.3.37. Appl., [A] 31.10.34, [B] 6.11.22).—(A) Residual oils from a petroleum distillation are



mildly cracked and the vapours mixed with those from the still and fractionated to produce a clean condensate. This is more drastically cracked, to give a cracked gasoline fraction, gas, and an intermediate fraction which is polymerised at raised temp. and increased pressure to produce a motor fuel fraction and a heavy reflux, which latter is recycled. (B) The container for cracking petroleum oils is made of an Fe-Cr or Fe-Cr-Ni alloy which is not corrodible by S. (Cf. U.S.P. 1,426,813; B., 1923, 741 A.) D. M. M.

**Catalytic conversion of higher-boiling hydrocarbons into lower-boiling hydrocarbons.** E. J. HOUDRY, Assr. to HOUDRY PROCESS CORP. (U.S.P. 2,078,247, 27.4.37. Appl., 11.5.32).—High-boiling hydrocarbons are continuously vaporised at  $>430^{\circ}$  ( $455^{\circ}$ ) and passed at  $>530^{\circ}$  over a catalyst consisting of activated Al hydrosilicate in moulded form. The products are cooled and fractionated to separate out the low-boiling constituents, which are raised by heat exchange to  $>260^{\circ}$  and stabilised at  $260-370^{\circ}$  by contact with a selective adsorbent or the above catalyst. D. M. M.

**Production of gas from oil.** T. NAGEL (B.P. 476,275, 12.1.37).—Hydrocarbon oil is heated under controlled temp. and pressure conditions, e.g., at  $370-480^{\circ}/10$  atm., such that the oil is maintained as a liquid, and the pressure on the oil is then released by injecting the oil along with hot lean gas, e.g., a CO-H<sub>2</sub> mixture, into a higher-temp. zone, e.g., at  $650-870^{\circ}$ , whereby the oil is converted almost instantly into gas and the complex hydrocarbons are decomposed into the more simple aliphatic gaseous hydrocarbons of lower mol. wt. Cooling is then immediately commenced before any substantial degree of polymerisation can occur and is continued to below polymerising temp., e.g.,  $540^{\circ}$ , under conditions substantially precluding polymerisation during cooling. H. C. M.

**Recovery of hydrocarbons.** J. K. ROBERTS and P. C. KEITH, jun., Assrs. to M. W. KELLOGG Co. and STANDARD OIL Co. (U.S.P. 2,077,344, 13.4.37. Appl., 16.11.34).—A gas mixture containing non-condensable gases is treated with an absorbing oil at 50 lb./sq. in. to remove all the desired hydrocarbons. These are then vaporised and stripped from the absorbing oil at 45 lb./sq. in., cooled to a temp. a little  $>$  the normal in an initial cooling stage, and then further cooled to a temp.  $<$  normal by indirect contact with expanding gases, whereby the majority of the hydrocarbon gases are condensed, and the condensed vapours are fractionated at  $100-1000$  lb./sq. in. D. M. M.

**Breaking petroleum emulsions.** (A) M. DE GROOTE, B. KEISER, L. L. FAURE, and A. F. WIRTEL, (B, E, I, J) M. DE GROOTE, (C, F) M. DE GROOTE and A. F. WIRTEL, (D, G, H), M. DE GROOTE and B. KEISER, Assrs. to TRETOLITE Co. (U.S.P. 2,076,623-4, 2,077,229-30, 2,077,745-6, 2,078,652-5, [A-D] 13.4.37, [E-F] 20.4.37, [G-J] 27.4.37. Appl., [A] 16.3.36, [B] 16.11.36, [C] 18.9.36, [D] 6.11.36, [E] 21.8.36, [F] 18.9.36, [G, H] 6.6.36, [I] 8.6.36, [J] 13.6.36).—H<sub>2</sub>O-in-oil petroleum emulsions are separated by addition of demulsifying agents which

consist of: (A) a cyclohexylamine (I) salt of an alkylated naphthalenesulphonic acid (II) in which at least one alkyl contains C<sub>3-10</sub>, e.g., a (I) salt of a propylated or isopropylated (II); (B) the product obtained by sulphonating an alkali-sol. condensation material obtained by interaction of an alkylene oxide [e.g., (CH<sub>2</sub>)<sub>2</sub>O] and a resin derived in part from a phenol, e.g., a CH<sub>2</sub>O-type resin derived from PhOH; the demulsifier may be in the form of a salt; (C) a substituted amine salt of an alkylated (II) in which at least one alkyl substituted in the C<sub>10</sub>H<sub>8</sub> nucleus contains C<sub>3-10</sub>, the substituted amine being an alkyl, aralkyl, or (I) compound or their functional equivs., and the substituent introduced into this amine being derived from a polyhydric alcohol and having a residual OH; (D) an amino-fatty acid compound in which the NH<sub>2</sub>-N is attached to the long C chain containing  $<$  C<sub>8</sub>, e.g., the reaction product of partly oxidised castor oil and NH<sub>3</sub> or a primary or secondary amine, e.g., NH<sub>2</sub>·C<sub>2</sub>H<sub>4</sub>·OH, in which the C:C linking becomes saturated; (E) the product obtained by replacing at least one polyhydric alcohol OH in a polyhydric alcohol ester of a detergent-forming carboxylic acid by a combination of a SO<sub>3</sub> radical and an ionisable H atom equiv.; (F) a polyhydric alcohol ether-substituted alkylamine salt of an alkylated (II) in which at least one alkyl substituted in the C<sub>10</sub>H<sub>8</sub> nucleus contains C<sub>3-10</sub>; (G) a fatty acid amide in which a OH of the fatty acid acyl radical has been replaced by a polybasic carboxylic acid residue. The acyl radical of the amide may be derived from ricinoleic acid and the N group may be of the unsubstituted NH<sub>2</sub>-radical type, whilst the polybasic carboxylic acid residue may be oxalic or maleic acid residues; (H) a non-SO<sub>3</sub>H, non-resinoid amide in which the acyl is derived from a detergent-forming monocarboxylic acid, e.g., ricinoleamide; (I) a sulpho-aromatic compound of a higher aliphatic ketone derived from fatty acids having  $<$  C<sub>11-22</sub>; (J) an acid ester formed by interaction of a polybasic carboxylic acid of relatively high acidity and a single mol. of a monohydric alcohol, e.g., Pr or Bu H phthalate. D. M. M.

**Separation of sludge from a liquid [oil].** L. D. JONES (U.S.P. 2,077,799, 20.4.37. Appl., 16.5.34).—Oil is intimately mixed with a reagent, e.g., H<sub>2</sub>SO<sub>4</sub>, and the resulting suspension of sludge is continuously impelled alternately at timed intervals through two interconnected parallel conduits to parallel subsidence zones or to a battery of centrifugal separators. D. M. M.

**Oil separation.** E. R. WILES, Assr. to BARNSDALL REFINING CORP. (U.S.P. 2,078,186, 20.4.37. Appl., 12.2.31).—Paraffinic and naphthenic constituents of an oil are separated by the use of C<sub>2</sub>H<sub>4</sub>Cl<sub>2</sub> in the proportion of 15-30% of oil to 85-70% of C<sub>2</sub>H<sub>4</sub>Cl<sub>2</sub>. The mixture is kept at 5° long enough to produce max. dissolution and then cooled to  $<1.5^{\circ}$  ( $-15^{\circ}$ ) to produce stratification, the layers being subsequently separated. D. M. M.

**Dehydration of oils.** R. C. SULLIVAN (B.P. 475,663, 2.6.36).—Wet oil, e.g., oil fuel from ships' bunkers, is heated at  $>100^{\circ}$  at a depth such as to overcome "puking" and is then passed through a

series of tall narrow towers. Steam and oil vapours are drawn off at the top of the towers and dehydrated oil is removed from the end tower. Apparatus is claimed. C. C.

**Dehazing furnace oil.** R. Z. WILLIAMS, Assr. to PAN AMERICAN PETROLEUM CORP. (U.S.P. 2,076,392, 6.4.37. Appl., 8.1.36).—The oil is treated with an aq. alkali solution, *e.g.*, NaOH, at 77—100° (82—93°).

D. M. M.

**Reagent and process for treating crude oils.** P. R. HERSHMAN, Assr. to PETRO CHEM. CO. (U.S.P. 2,074,183, 16.3.37. Appl., 7.6.34).—Oils are desalted and demulsified by treatment in continuous phase with a mixture of caustic alkali (NaOH) and a sol. alkali acetate (NaOAc) with EtOH and glycerin. An after-treatment with a HCO<sub>3</sub>' solution may be desirable. A preferred composition of the reagent is NaOH 25, NaOAc 10, EtOH 20, glycerin 10, and H<sub>2</sub>O up to 35—45%.

D. M. M.

**Process and apparatus for treatment or catalysis of hydrocarbons or other fluids and for heat exchange.** HOUDRY PROCESS CORP., Assees. of E. J. HOUDRY (B.P. 477,846, 6.7.36. U.S., 19.7.35).—A catalytic process for refining hydrocarbons and a special apparatus therefor, whereby the hydrocarbons are passed through a confined elongated annular space containing a contact mass, are claimed. The temp. of the space and contact mass is maintained as desired by circulating a heat-exchange fluid at a controlled temp. in intimate heat-transfer relation with the inner and outer surface of the annular space. C. C.

**Treatment of hydrocarbons.** B. ORMONT, Assr. to B. ORMONT ASSOCIATES, INC. (U.S.P. 2,078,407, 27.4.37. Appl., 21.9.33).—Oil is heated in a pipe-still to 260° and then comes in contact with nascent steam in the same still, where it is completely vaporised without rise of temp. The mixture of oil vapours and steam is cooled to recover the heavier fractions and the remaining oil vapours are heated to >480° (590—650°) to crack them, after which the temp. is lowered to 315—370° and the vapours are brought in contact with a film of moving H<sub>2</sub>O to maintain them in the vapour phase; the lighter fractions are then recovered and instantaneously flashed at 180—205° to ensure high antiknock vals.

D. M. M.

**Treatment of hydrocarbons of low b.p.** EDELEANU GES. M.B.H. (B.P. 475,255, 14.5.36. U.S., 15.5.35).—Light oils (benzines) are extracted with liquid SO<sub>2</sub> in presence of an auxiliary solvent (*e.g.*, mono- or di-ethylene glycol, trimethylene glycol, CH<sub>2</sub>Ph·OH, diacetone alcohol) which is miscible with SO<sub>2</sub> and has a selective solvent action on saturated hydrocarbons. This action of SO<sub>2</sub> for unsaturated and aromatic compounds is thereby increased. (Cf. B.P. 467,048; B., 1937, 754.)

C. C.

**Production of light hydrocarbons.** E. J. HOUDRY, Assr. to HOUDRY PROCESS CORP. (U.S.P. 2,072,108, 2.3.37. Appl., 16.9.33).—Motor fuel is produced from the middle and bottom cuts of the distillation of crude oil by first treating the bottom cut or the mixed middle and bottom cuts by solvent

extraction to remove the more refractory fractions and then cracking the remainder. D. M. M.

**Hydrocarbon oil treatment.** S. W. FERRIS, Assr. to ATLANTIC REFINING CO. (U.S.P. 2,072,104, 2.3.37. Appl., 1.4.33).—Viscous hydrocarbon oils containing both paraffinic (I) and naphthenic constituents (II) are intimately mixed with an aliphatic chlorohydrin or mixture of chlorohydrins, *e.g.*, ethylene or propylene chlorohydrin, epichlorohydrin, whereby the (II) are dissolved in the solvent separated with it and an oil rich in (I) is produced.

D. M. M.

**Treating hydrocarbon oils.** W. M. STRATFORD, Assr. to TEXAS CO. (U.S.P. 2,078,468, 27.4.37. Appl., 16.11.32).—Cracked naphtha is treated with an alkali or alkaline-earth metal (Na) at 430—480° under pressure, *e.g.*, 2000 lb./sq. in., in order to polymerise undesired unsaturated compounds and decompose S compounds. The process may be continuous and the naphtha is separated from the residue by distillation.

D. M. M.

**Refining of hydrocarbon oils.** EDELEANU GES. M.B.H. (B.P. 475,723 and 477,086, 21.5.36. U.S., 23.5.35).—(A) The oils are extracted with liquefied, normally gaseous aliphatic F compounds (other than CCl<sub>2</sub>F<sub>2</sub>), *e.g.*, F compounds of chlorinated or brominated aliphatic hydrocarbons in which the no. of F atoms is <, =, or > the no. of other halogen atoms. After removal of the asphalt in this way, wax may be separated by cooling the solution, and the oil further refined by solvent extraction, *e.g.*, with PhOH, furfuraldehyde, or liquid SO<sub>2</sub>. The original extraction may be made in presence of an auxiliary solvent. (B) Oil is treated with a F compound (I) (inorg., aromatic, or liquid aliphatic) which does not react chemically with the oil. The (I) may completely dissolve the oil, leaving asphaltic material insol. After separation of the latter, the wax may be pptd. by cooling the solution. The (I) may be a liquid aliphatic fluorohalogen compound in which the no. of F atoms is < the no. of atoms of other halogens, or aromatic F derivatives. Alternatively, the oil may be extracted selectively by a liquid aliphatic F derivative (F atoms > or = the no. of other halogens), or by inorg. F compounds of Sb, Sn, As, B, S, Te, or Se, *e.g.*, SF<sub>6</sub> or SO<sub>2</sub>F<sub>2</sub>. Auxiliary solvents miscible or partly miscible with the (I) and miscible or immiscible with the oil may be present, or selective solvents [as in (A)] may be used in addition to oil-miscible (I). C. C.

**Solvent refining of mineral oil.** L. A. CLARKE, Assr. to TEXAS CO. (U.S.P. 2,078,442, 27.4.37. Appl., 29.8.35).—Constituents of high and low  $\eta$  index of hydrocarbon oils are separated by the use of a selective solvent consisting of compounds of the class of methoxy- or ethoxy-methyl or -ethyl acetates, preferably the  $\beta$ -compounds. D. M. M.

**Refining of mineral oil.** W. H. VOLCK, Assr. to CALIFORNIA SPRAY-CHEM. CORP. (U.S.P. 2,076,105, 6.4.37. Appl., 13.6.33).—Crude petroleum oils or distillates therefrom are refined by treatment at room temp. or slightly above, *e.g.*, at 50—75°, with a selective solvent consisting of 2—20% of N(C<sub>2</sub>H<sub>4</sub>·OH)<sub>3</sub> and 98—80% of COMe<sub>2</sub>. The amount of solvent

may vary from slightly < the vol. of the oil to 3 or 4 times its vol. The result is to remove a large proportion of the lighter unsaturateds, especially those that are phytocidal. D. M. M.

**Refining of mineral oils.** ROHM & HAAS Co. (B.P. 477,888, 5.6.36. U.S., 13.6.35).—Hydrocarbon mixtures, *e.g.*, lubricating oil, kerosene, gasoline, are refined by treatment with a solvent, *e.g.*,  $\text{NH}_2\text{Me}$ , in amount at least sufficient to form two phases. These are separated and the solvent is removed from each phase, the whole process being carried out in a closed system. An auxiliary liquid miscible with  $\text{NH}_2\text{Me}$  may also be used, *e.g.*,  $\text{NH}_3$ ,  $\text{H}_2\text{O}$ ,  $\text{MeOH}$ . Wax may be separated from lubricating oil by cooling the mixture. C. C.

**Apparatus for refining and purification of mineral oils or tar oils.** R. POKORNY and E. K. MAULER (B.P. 480,676, 22.7.36).—To permit continuous refinement of crude oils, or to regenerate waste oil, apparatus is claimed embodying a device for heating the oil and automatically injecting into it a proportionate amount of purifying agent (*e.g.*, acid) and later of absorbing or neutralising agent (*e.g.*, earth). Mixing is effected by three horizontally axled propeller agitators, two being alined below and being driven faster than, and in opposite direction to that of, the third. The settling device consists of a series of decanting tubes. The filtering device has special heating arrangements and forces the oil along a tortuous path. T. C. G. T.

**Treatment of petroleum distillates.** F. GARDNER (U.S.P. 2,073,147, 9.3.37. Appl., 11.11.35).—Mercaptans are converted into disulphides in petroleum distillates by treatment with an alkali (Na) plumbate, without addition of S. D. M. M.

**Refining of hydrocarbon distillates.** M. H. GWYNN (U.S.P. 2,073,578, 9.3.37. Appl., 10.1.33).—Hydrocarbon distillates are refined and S compounds and gum-formers converted into less troublesome compounds by treatment with  $\text{H}_2$  in presence of catalysts, *e.g.*, Ni or Cu, in a series of stages at 75—290°/1—100 atm. D. M. M.

**Desulphurisation of hydrocarbons.** A. E. BUELL and W. A. SCHULZE, Assrs. to PHILLIPS PETROLEUM Co. (U.S.P. 2,075,171—4, 30.3.37. Appl., 21.5.35).—Straight-run and cracked gasolines are partly desulphurised by catalytic treatment in the vapour phase at 260—425° for 2—10 sec., using as catalysts natural minerals or ores consisting of sulphides of metals the oxides of which are grouped as dehydration and dehydrogenation catalysts; such metals are V, W, Mo, or Cd, and preferred ores are vanadinite, wolframite, molybdenite, molybdate, and greenockite. D. M. M.

**Reclaiming spent "doctor" solution.** E. T. PUMMILL, Assr. to SOCONY-VACUUM OIL Co., INC. (U.S.P. 2,078,773, 27.4.37. Appl., 5.10.35).—Spent "doctor" solution containing PbS in suspension is treated with  $\text{Ca}(\text{OH})_2$  or  $\text{Mg}(\text{OH})_2$ , preferably the former, before blowing with air at elevated temp. (38—60°). This results in full re-use of Pb and Na

present in the "doctor" solution. Preferably > ½ mol. of  $\text{Ca}(\text{OH})_2$  is added per mol. of PbS present.

D. M. M.

**Production of antiknock gasoline.** L. H. FITCH, jun., Assr. to PHILLIPS PETROLEUM Co. (U.S.P. 2,072,745, 2.3.37. Appl., 8.10.34).—A stream of light aliphatic hydrocarbons, b.p. < that of gasoline but > that of  $\text{CH}_4$ , is cracked at high temp. (465°) and pressure (2000 lb./sq. in.) and the products are fractionated under pressure (325 lb./sq. in.) into three fractions, the intermediate one of which is re-cracked at low pressure (200 lb./sq. in.) and temp. (760—815°). The products of this second cracking are cooled, freed from tar, compressed, and used as scrubbing agent in the fractionator at a point below the exit of the intermediate fraction. The liquid drawn off from the bottom of the fractionator is then of gasoline boiling range with high antiknock val. D. M. M.

**Solvent fractionation of gasoline.** O. F. SIMONS and W. H. BAHLKE, Assrs. to STANDARD OIL Co. (U.S.P. 2,073,517, 9.3.37. Appl., 14.6.33).—Cracked petroleum naphtha is separated into high- and low-antiknock fractions by selective solvent action in two stages at < -15°. In the first stage the naphtha is mixed with  $\text{SO}_2$  and  $\text{C}_3\text{H}_8$  and the phases are allowed to separate, whilst in the second the lower phase containing  $\text{SO}_2$  and the high-antiknock components is mixed, also at < -15°, with cracked naphtha containing  $\text{C}_3\text{H}_8$ . The lower phase from the second stage is separated,  $\text{SO}_2$  evaporated off, and high-antiknock components remain. D. M. M.

**Refining of gasoline.** M. S. KHARASCH, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,076,581, 13.4.37. Appl., 18.2.33).—The gum and colour-forming constituents of gasoline are polymerised by treatment with 0.1—5% (1—2%) anhyd. HF for long enough to polymerise the more easily polymerisable constituents. The HF is then washed out with alkali, the gasoline distilled, and an inhibitor, *e.g.*, pyrocatechol, added if desired. D. M. M.

**Stabilisation of gasoline.** N. V. NIEUWE OCTROOI MAATS., Asses. of W. W. HOLLAND (B.P. 477,745, 17.4.37. U.S., 25.4.36).—Raw gasoline, without pretreatment, is treated with doctor solution, washed with  $\text{H}_2\text{O}$ , and the last traces of alkali are removed by treatment with a weak org. acid or a mildly acidic salt, *e.g.*,  $\text{NaHSO}_3$  or  $\text{Al}_2(\text{SO}_4)_3$ . Turbidity may be removed by cold filtration through clay, and any of the usual inhibitors then added. D. M. M.

**Combined stabilising and gas-conversion process.** M. P. YOUKER, Assr. to PHILLIPS PETROLEUM Co. (U.S.P. 2,076,013, 6.4.37. Appl., 5.7.33).—Gasoline containing substantial quantities of hydrocarbons too unstable to be incorporated in motor fuels is converted into a motor fuel with at least a part of the unstable constituents converted into stable constituents of high antiknock val. while the stable portion of the original gasoline is reformed to higher antiknock val. by treating the whole material at 400—620°/500—3000 lb. per sq. in. The product is rectified at 100—500 lb./sq. in. D. M. M.

**Motor fuels.** STANDARD OIL DEVELOPMENT Co. (B.P. 487,986, 30.7.37. U.S., 15.8.36).—The temp.

variation of the antiknock rating of a fuel is smoothed out by the use of at least two antiknock agents of different volatility, e.g.,  $\text{PbEt}_4$ ,  $\text{PbMe}_4$ ,  $\text{PbMe}_3\text{Et}$ , etc., so proportioned that no separate fraction of the fuel between the 10% and 95% points has an  $\text{C}_9\text{H}_{18}$  no. of >5 to 10 points below the C.F.R. val. of the total fuel. The total antiknock agent need not exceed the customary amount. D. M. M.

**Motor fuels.** TEXACO DEVELOPMENT CORP. (B.P. 488,625, 30.1.37. U.S., 6.2.36).—A primer, to be added to a Diesel-engine fuel, consists of 1—5 (3—4)% of a peroxide of an aliphatic ketone of  $> \text{C}_4$  in the unimol. form. Examples are peroxides of  $\text{COMeEt}$ ,  $\text{COMePr}$ , or  $\text{COEt}_2$ . D. M. M.

**Motor fuel.** H. B. J. SCHURINK, Assr. to SHELL DEVELOPMENT Co. (U.S.P. 2,078,736, 27.4.37. Appl., 26.11.35. Holl., 13.12.34).—Aq.  $\text{Bu}^o\text{OH}$  containing 12—35 wt.-% of  $\text{H}_2\text{O}$  may be used as an antiknock motor fuel either alone or mixed with petrol, benzol, or other motor fuel in amounts not exceeding the miscibility limits of the fuel components. D. M. M.

**Improving the knock rating of motor fuel by distillation.** J. M. SMITH, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 2,076,519, 6.4.37. Appl., 13.12.35).—Naphtha containing aliphatic and aromatic constituents is distilled in two portions, viz., one portion in presence of an added substance containing an aromatic nucleus and boiling substantially above the b.p. of the highest-boiling constituent of the naphtha, e.g., *o*-tolyl phosphate, *o*-toluidine, and the second in presence of an open-chain org. compound also of higher b.p. than that of the highest-boiling constituent of the naphtha. The last overheads of the first distillation are blended with the first overheads of the last distillation to produce a motor fuel. D. M. M.

**Treatment of motor fuel.** F. B. BEHRENS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 2,076,524, 13.4.37. Appl., 22.5.33. Renewed 14.9.36).—Deteriorated cracked gasoline containing org. peroxides formed during deterioration is restored and stabilised against further peroxidation by treatment with  $\text{MgO}$ , e.g., burned magnesite, after which an inhibitor is added. D. M. M.

**Producing an improved motor fuel and solvent oil.** J. C. BLACK, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 2,076,695, 13.4.37. Appl., 5.7.29).—Higher-boiling hydrocarbon oils are converted into lower-boiling ones by cracking, separating naphtha from the former, and then fractionally separating the higher-boiling portion of the naphtha and converting this by passage through a heating coil into a product of improved antiknock val., which is then fractionated to separate the gasoline fraction from the higher-boiling oil, the latter being then further cracked. D. M. M.

**Preparation of fuel oil.** R. L. SAVAGE, Assr. to TEXAS Co. (U.S.P. 2,078,882, 27.4.37. Appl., 13.6.34).—Acid oil sludge is heated by direct contact with a hot diluent oil which decomposes and vaporises the acidic S compounds, the heating being controlled to avoid carbonisation. The resultant vapours are withdrawn, and purified oil sludge and diluent oil are

collected together, the latter being then fractionated or flashed off and re-used. D. M. M.

**Manufacture of [light-weight, viscous] fuel.** M. GAERTNER (U.S.P. 2,077,406, 20.4.37. Appl., 21.4.28).—A mass of fuel oil is emulsified with a small amount of  $\text{H}_2\text{O}$  containing a stabiliser, e.g., dextrin, rosin, soaps,  $\text{PhOH}$ , etc., and air or other gas containing  $\text{O}_2$  is intimately mixed with the emulsion to produce a stable foam filled with air bubbles not under pressure. Such fuel, it is claimed, may be used in burners with an additional air supply and develops high thermal efficiency. D. M. M.

**Production of saturated hydrocarbons, in particular of the gasoline range.** H. E. POTTS. From INTERNAT. HYDROGENATION PATENTS Co., LTD. (B.P. 475,911, 16.6.36).—The production of such hydrocarbons by catalytic hydrogenation with an excess of free  $\text{H}_2$  in presence of a stationary catalyst of unsaturated hydrocarbons having a b.-p. range similar to that of gasoline and obtained by polymerising unsaturated aliphatic hydrocarbons can be effected with high quant. yields and with the min. splitting, depolymerising, and/or formation of high-mol. products, if the presence of  $\text{H}_2\text{O}$  vapour be maintained in the reaction space during hydrogenation. Hydrogenation may be effected at 200—300°/50—300 atm., using oxides or sulphides of W and/or Mo as catalyst, the amount of  $\text{H}_2\text{O}$  vapour supplied being 1—5 wt.-% of the material to be hydrogenated. H. C. M.

**Production of liquid hydrocarbon products by destructive hydrogenation of distillable solid carbonaceous substances or products obtained therefrom.** INTERNAT. HYDROGENATION PATENTS Co., LTD., Assces. of I. G. FARBENIND. A.-G. (B.P. 487,892, 21.1.37. Ger., 1.2.36).—The materials are hydrogenated at 300—500°/20—500 atm. in presence of Zn salts of monobasic carboxylic acids of  $< \text{C}_5$ , e.g.,  $(\text{HCO}_2)_2\text{Zn}$  or  $\text{Zn}(\text{OAc})_2$ . A halogen, H halide, or compounds furnishing either of them or metalloidal halides, e.g., halogen compounds of C or S, may be applied together with the Zn salts. D. M. M.

**Treatment of hydrocarbon oils.** A. M. MUCKENFUSS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,073,973, 16.3.37. Appl., 16.10.35. Cf. U.S.P. 2,021,567; B., 1936, 1195).—Hydrocarbon oils containing unsaturated hydrocarbons are hydrogenated in presence of a catalyst consisting of an alkali-metal compound of an aromatic hydrocarbon, preferably one with  $> \text{one C}_6\text{H}_6$  nucleus in the mol. Pressures of 300—500 lb./sq. in. and temp.  $> 250^\circ$  are used. D. M. M.

**Manufacture of hydrocarbons suitable for use in motor fuel blends.** UNIVERSAL OIL PRODUCTS Co. (B.P. 477,128, 8.4.37. U.S., 16.10.36).—*iso*-Heptenes are obtained by mixed polymerisation of *isobutene* and propene at 93—163°/34—41 (37.5) atm. in presence of a catalyst consisting of a precalcined mixture of an acid of P with a minor proportion of siliceous absorbent. 1—5% of steam may be present. The liquid products, b.p.  $< 150^\circ$ , are recovered. *iso*-Heptanes are prepared by hydrogenating liquid products of b.p.  $< 100^\circ$ . C. C.

**Production of hydrocarbon products, in particular motor fuels such as benzine, Diesel oil, and the like, by treatment of liquid or fusible carbonaceous material with hydrogenating gases.** L. E. JONES. From INTERNAT. HYDROGENATION PATENTS CO., LTD. (B.P. 477,944, 18.5.37).—Liquid or fusible carbonaceous materials containing 2—50 vol.-% of asphalt (prepared by the  $C_3H_8$  method) are hydrogenated by passing over a stationary catalyst of  $\leq$  standard hydrogenating activity, *e.g.*, sulphides or oxides of W or Mo, at 300—500° while maintaining a  $H_2$  partial pressure of 250—1000 atm., to produce motor fuels etc.

D. M. M.

**Preparation of polybasic acid derivatives of hydro-formed naphthas.** STANDARD OIL DEVELOPMENT Co. (B.P. 479,017, 19.6.36. U.S., 16.10.35).—Hydro-formed naphthas are obtained by treating a petroleum distillate, *e.g.*, burning oil or gas oil, with a selective solvent, *e.g.*,  $SO_2$ , and then passing the less saturated fraction or a portion thereof in the vapour phase and mixed with  $H_2$  over a catalyst consisting of oxides and/or sulphides of group VI metals, *e.g.*, Mo, at 220—820°/50—200 atm. The hydro-formed naphthas so obtained are converted into polybasic acids by treatment in either vapour or liquid phase with air in presence of a suitable catalyst such as an oxide of a metal of group V or VI, *e.g.*,  $V_2O_5$  (in the vapour phase) or Mn naphthenate (liquid phase). The reaction in the vapour phase is carried out at  $>200^\circ$  and in the liquid phase at 140—200°. D. M. M.

**Dehydrogenated fuel.** C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 2,077,994, 20.4.37. Appl., 14.9.33).—A motor fuel containing substantial amounts of aromatics is produced by dehydrogenating a pressure-cracked petroleum, in presence of air, at 205—315° and under superatm. pressure  $<$  that used in cracking, and separating a fraction of end b.p.  $\geq 460^\circ$ . D. M. M.

**Treatment of petroleum residuum.** R. R. THURSTON, Assr. to TEXAS Co. (U.S.P. 2,076,799, 13.4.37. Appl., 3.2.33).—Semi-paraffinic-type petroleum oils are distilled, preferably under vac., to produce a residuum from which the lubricating oil has been removed. This residuum is then treated with a selective solvent, *e.g.*, furfuraldehyde, the dissolved and undissolved fractions being thereby separated; then the sol. fraction has the solvent removed and is reduced by air-blowing at 450° to a desired m.p. and penetration. The insol. portion is also air-blown and converted into a rubber-like asphaltic material of high m.p. D. M. M.

**Manufacture of hydrocarbon mixtures containing a high percentage of alkenes of high mol. wt.** N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 478,841, 19.10.36. Holl., 1.11.35).—Hydrocarbons containing alkenes ( $< C_{10}$ ) are prepared by vapour-phase cracking of hydrocarbon material with a high paraffin wax content ( $>50\%$ ) in presence of an inert gas, *e.g.*, steam,  $N_2$ , etc., non-vapour-phase constituents being first removed and the total time of heating up to cracking temp. and cracking being  $\geq 5$  sec., computed according to a formula quoted. Pressures are preferably 2—5 atm. D. M. M.

**Manufacture of oxidation products from paraffin hydrocarbons of high mol. wt.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 478,317, 14.7.36. Cf. B.P. 369,852; B., 1932, 539).—Products produced by passing an oxidising gas, *e.g.*, air, at  $\geq 115^\circ$  through the mass containing the paraffin to be oxidised in presence of 0.025—0.50 wt.-% of a catalyst consisting of alkali compounds of Mn, in which the Mn is in the anion and the alkali the cation, *e.g.*,  $KMnO_4$ ,  $K_2MnO_4$  or  $K_2MnO_3$ , or a K Mn cyanide, are claimed. Preferably the hydrocarbons or a portion of them are pretreated in presence of the catalyst by prolonged heating at 100—200° without passing any gas, but in presence, if possible, of 10—50% of unsaponifiable constituents from another batch.

D. M. M.

**Propane dewaxing.** H. O. FORREST, Assr. to STANDARD OIL Co. (U.S.P. 2,076,141, 6.4.37. Appl., 8.3.35).—Waxy oil stock, mixed under pressure with a normally gaseous refrigerant, *e.g.*,  $C_3H_8$ , is heated if necessary to dissolve the wax, 2 vols. of  $C_3H_8$  being used per vol. of oil. The warm mixture is passed through a series of 6—12 (10) chilling stages with a pressure drop of 10—12 lb./stage, after which it is finally cooled by direct contact with chilled  $C_3H_8$ . Each chilling vessel has a cooling-coil provided in its upper part through which the chilled filtrate passes countercurrently to the oil-wax- $C_3H_8$  mixture.

D. M. M.

**De-asphalting and dewaxing of hydrocarbon oils.** E. PETTY and M. B. COOKE (B.P. 477,164, 30.6.36. Addn. to B.P. 466,731; B., 1937, 1012).—The oil-solvent phase, after removal of asphalt, is conc. and then chilled, whereby wax crystallisation is favoured. Before separating the wax, chilled diluent is added. C. C.

**Dewaxing of oil.** L. D. JONES, Assr. to SHARPLES SPECIALTY Co. (U.S.P. 2,076,147, 6.4.37. Appl., 25.6.31).—Oils containing amorphous wax are mixed with finely divided solids, *e.g.*, clay, and a solvent, warmed if necessary to dissolve the wax, cooled to 10—24°, filtered to remove the solids, again cooled, *e.g.*, to  $-34^\circ$  to  $-40^\circ$ , and then separated from wax by continuous centrifuging. D. M. M.

**Dewaxing oils.** W. W. TRIGGS. From MID-CONTINENT PETROLEUM CORP. (B.P. 488,063, 10.8.37).—The oil is dissolved in a liquefied, normally gaseous hydrocarbon, *e.g.*,  $C_3H_8$ , and the solution suddenly chilled by introduction of a stream of chilled  $C_3H_8$  into a stream of the solution, after which the wax is pptd. from the cooled oil while forcing a stream of the resultant material through an elongated passageway to provide a gradual decrease in pressure on the flowing stream and a gradual evaporation of a portion of the  $C_3H_8$ . The pptd. wax is filtered off. D. M. M.

**Dewaxing of motor oils.** J. K. ROBERTS and M. T. CARPENTER, Assrs. to STANDARD OIL Co. (U.S.P. 2,077,712, 20.4.37. Appl., 31.8.31).—Motor oils are continuously dewaxed by first mixing with a small amount of a mixed diluent (7:3  $C_4H_{10}$ - $C_3H_8$  mixture) at 65°, then cooling to room temp., and diluting with a large amount of pre-cooled diluent. The mixture is then passed to one of a series of chill chambers of which one is always being filled, another

cooled, and a third emptied. The wax slurry formed is mechanically removed and the cold filtrate used to pre-cool the diluent entering the chill chamber. The diluent is finally removed and returned to the system.

D. M. M.

**Dewaxing system [for viscous petroleum oil].** R. E. WILSON, Assr. to STANDARD OIL Co. (U.S.P. 2,077,656, 20.4.37. Appl., 31.8.31).—Oil is dewaxed by mixing with a diluent, *e.g.*,  $C_4H_{10}$ , at a relatively high temp. (60°), cooling the mixture, diluting with a liquefied, normally gaseous, hydrocarbon of lower boiling range than that of the first diluent or  $C_4H_{10}$ , *e.g.*,  $C_3H_8$ , chilling the mixture by vaporising a part of the second diluent, mechanically removing the separated wax, and then separately recovering and storing each diluent for re-use.

D. M. M.

**Forming wax and product therefrom.** J. F. WAIT (U.S.P. 2,075,151, 30.3.37. Appl., 29.4.33).—Lubricating oil or wax in fluid form is brought in contact with an alkali metal, *e.g.*, Na, at 200–450° to cause a rearrangement whereby both wax and oil are purified and the  $\eta$  index of the oil is improved. Subsequent treatment by absorbing agents may be necessary.

D. M. M.

**Wax crystallisation.** J. ROBINSON and V. VOORHEES, Assrs. to STANDARD OIL Co. (U.S.P. 2,076,161, 6.4.37. Appl., 22.7.35).—Waxy oil mixed under pressure with 3.5–4 vols. of normally gaseous solvent, *e.g.*,  $C_3H_8$ , is continuously crystallised by passage through a series of towers or stand-pipes of gradually increasing diameter in which the hydrostatic pressure is progressively diminished from one stage to the next. Hydrostatic pressure may be increased and the necessary tower height diminished by filling the tower with a heavy liquid, *e.g.*, brine, through which the  $C_3H_8$ -waxy oil mixture rises.

D. M. M.

**Bleaching of montan wax.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 488,381, 4.2.37).—The wax is treated above its m.p. (at 90–120°) for 1–6 hr., under pressure if desired, with aq.  $H_2O_2$ , preferably a 10–20% solution and containing  $H_2O_2$  in amount = 5% of the wax. As soon as the reaction is completed the mixture is treated with  $CrO_3$  and  $H_2SO_4$  until the former is used up.

D. M. M.

**Manufacture of [chlorinated] derivatives of paraffin wax.** D. W. F. HARDIE, C. OCKRENT, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 479,195, 1.7.36).—The unsaturated compounds obtained by treating a chlorinated paraffin wax ( $\approx 25\%$  Cl) with an excess of alcoholic alkali under pressure are again chlorinated, the final stages of the rechlorination being effected at elevated temp., *e.g.*, 80–90°. The unsaturated derivatives are purified by mol. distillation.

H. C. M.

**[Preparation of] pour-point depressants for lubricating oils.** STANDARD OIL DEVELOPMENT Co. (B.P. 479,479, 5.11.36. U.S., 2.1.36).—The efficiency of synthetic hydrocarbon oil pour-point depressants prepared by condensation of paraffinic with aromatic hydrocarbons, *e.g.*, by the Friedel-Crafts reaction, is greatly improved by subjecting the oil to mild treatment with a silent electric discharge, *e.g.*, using 1000–10,000 v. at a frequency of 500–

10,000 cycles/sec. under an abs. pressure of <20 (2–10) cm.

H. C. M.

**Production of high-boiling polymerides useful for blending [with lubricating oils] and other purposes.** S. C. FULTON and L. A. MIKESKA, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 2,072,107, 2.3.37. Appl., 6.6.33).—Lubricating oils are thickened and the slope of their  $\eta$ /temp. curve is improved by adding condensation products of an aliphatic halogen compound of  $\gamma C_6$ , *e.g.*,  $C_2H_4Cl_2$ , with an aromatic or hydroaromatic compound, *e.g.*,  $C_6H_6$ . The components are condensed at 20–50° in presence of a catalyst, *e.g.*,  $AlCl_3$ , in equimol. proportions and any  $CHPh_2Me$  formed is distilled off. Three types of products are formed: (a) oily liquids, mol. wt. <1200, sol. in hydrocarbon oils; (b) plastic solids, mol. wt. 1000–2500, sol. in org. solvents, sparingly sol. in hydrocarbon oils but more so after hydrogenation or alkylation; (c) insol. rubbery products of mol. wt. >2500. (a) and (b) are the desired products.

D. M. M.

**Manufacture of oily condensation products useful for imparting colour and fluorescence to lubricating oils.** C. OCKRENT, D. W. F. HARDIE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 477,928, 17.7.36).—An aromatic hydrocarbon, *e.g.*,  $C_6H_6$ , PhMe, or  $C_{10}H_8$ , is condensed with a chlorinated long-chain aliphatic hydrocarbon containing >35% (40%) Cl, *e.g.*, chlorinated paraffin wax, in presence of a Friedel-Crafts catalyst, *e.g.*,  $AlCl_3$ , until no more HCl is evolved, and the product is allowed to stratify. The lower layer is freed from catalyst by warming with dil. HCl and then heated at 300–400° until a homogeneous melt is obtained. Solid matter is removed by dissolving in a solvent, *e.g.*,  $C_6H_6$  or PhMe, filtering, and distilling off the solvent, or by dissolution in lubricating oil and filtering. The product when added in small proportions, *e.g.*, 1 to 10,000, to natural or synthetic lubricating oil imparts to the oil both colour and fluorescence.

D. M. M.

**Castor oil product [for lubricants].** K. T. STEIK, Assr. to NAT. OIL PRODUCTS Co. (U.S.P. 2,068,088, 19.1.37. Appl., 27.8.34).—The neutral product formed by interaction of naphthenic acids and (excess of) castor oil at >100° (~270°) is a mutual solvent for castor oil and mineral oil allowing the dissolution of the one in the other.

E. L.

**Voltolised oils.** STANDARD OIL DEVELOPMENT Co. (B.P. 488,026, 28.12.36. U.S., 31.1.36).—Fish oil consisting essentially of an unsaturated ester of a monocarboxylic acid and a monohydric aliphatic alcohol, *e.g.*, sperm, porpoise-jaw, or dolphin oil, or a mixture of them with or without petroleum or mineral oils, is subjected to high-tension, silent electric discharges at 4000–50,000 v., preferably a.c. of <60 cycles, and under a pressure of 0.001–0.8 atm. with suitable cooling arrangements. The oil may be fractionated or purified with clay before being voltolised in presence of S, a halogen, Se,  $H_2S$ , P,  $BF_3$ , or a ketone and in an atm. of  $H_2$ ,  $N_2$ , CO,  $CO_2$ ,  $O_2$ , or air. The products may be used directly or in compositions containing 0.01–50% of them, as lubricants, plasticisers, etc.

D. M. M.

**Lubricants.** CONTINENTAL OIL Co. (B.P. 475,787, 21.2.36. U.S., 21.2.35).—The film strength of a petroleum oil or hydrocarbon grease is greatly improved by addition thereto of a small amount, *e.g.*, 0.2–2 wt.-%, of one or more P esters (halogenated, if desired) containing an alkylated aryl group having  $\leq C_8$ , *e.g.*, tri-*p*-ethylphenyl phosphate. [Stat. ref.]

H. C. M.

**Lubricants.** W. V. GILBERT (B.P. 478,273, 17.7.36).—A Mn compound (I) or mixture of Mn compounds in colloidal form, *e.g.*, the chromate or a salt of an org. fatty acid, *e.g.*, oleic, is added to a lubricating medium to inhibit corrosion of the metallic surfaces being lubricated. The (I) employed must be free from  $SO_4^{''}$ ,  $NO_3'$ , and especially  $Cl'$  and may have a similar colour to that of the lubricant employed.

D. M. M.

**Lubricating compound.** C. E. LAUER, Assr. to TEXAS Co. (U.S.P. 2,077,762, 20.4.37. Appl., 6.7.36).—A lubricant suitable for saturating railway car-journal waste and for lubricating the journals is prepared by blending 20–50% (40%) of a dewaxed paraffin-base residuum ( $\eta > 700$  sec. Saybolt at 37.5°) with 80–50% (60%) of a distillate paraffin-base lubricating oil ( $\eta < 100$ ), the product having  $\eta$  150–350 and withstanding temp. of –43° without waste-grabbing or waste-rolling though giving adequate lubrication at temp. up to 500°. D. M. M.

**Extreme-pressure lubricating compositions.** R. L. HUMPHREYS and B. B. FARRINGTON, Assrs. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 2,073,841, 16.3.37. Appl., 8.12.34).—Such lubricants are prepared by adding to lubricating oils small amounts, *e.g.*, 0.5–5.0%, of org. derivatives of org. thioacids, especially their esters, and anhydrides; examples are Et or  $CH_2Ph$  thiobenzoate. D. M. M.

**Lubricating oil.** F. J. LICATA, Assr. to NAT. OIL PRODUCTS Co. (U.S.P. 2,076,153, 6.4.37. Appl., 1.3.33).—The pour point of lubricating oils is reduced by addition of 0.5% of a mixture consisting of 2–15% of diethylene glycol, 15% of castor oil fatty acids, and 70% of an Al soap, *e.g.*, Al stearate. D. M. M.

**Lubricating oils.** (A) LE R. G. STORY, (B) C. C. TOWNE, Assrs. to TEXAS Co. (U.S.P. 2,077,781 and 2,078,472, [A] 20.4.37, [B] 27.4.37. Appl., [A] 1.8.34, [B] 3.8.34).—(A) A waxy hydrocarbon consisting mainly of straight-chain hydrocarbons, *e.g.*, petroleum jelly, slack wax, or heavy oil containing wax, is mildly cracked at  $\geq 100$  lb./sq. in. and 400–590° and a heavy distillate oil separated from the products. This is chlorinated at slightly elevated temp. and the chlorination product condensed in presence of a metallic halide catalyst, *e.g.*,  $AlCl_3$ , at 10–52° for 18–24 hr. The condensation product may be used alone, or, preferably, blended with a petroleum lubricating oil. Improved  $\eta$  index and lower pour point are claimed. (B) The  $\eta$  index of naphthene- or paraffin-base oils is improved by adding small amounts, *e.g.*, 0.5–5%, of a synthetic resin consisting of a reaction product of rubber with a halide or halogen acid of Sn or B, *e.g.*,  $SnCl_4$ ,  $BCl_3$ ,  $BF_3$ , chlorostannic acid,  $HBF_4$ , etc. Alternatively, 0.5–40% of a synthetic resin consisting of a reaction product of rubber and  $SnCl_4$  may be employed. D. M. M.

**Lubricating oils.** L. LIBERTHSON, Assr. to L. SONNEBORN SONS, INC. (U.S.P. 2,073,641, 16.3.37. Appl., 2.10.34).—The pour point of the oils is reduced without reducing their demulsibility by adding  $\geq 10\%$  of the unsaponifiable constituent of a polymerised oil prepared by the action of a silent electrical discharge. D. M. M.

**Production of lubricating oils.** RUHRCHEMIE A.-G. (B.P. 473,935, 19.12.36. Ger., 20.12.35. Addn. to B.P. 470,534; B., 1938, 34).—In the production of lubricating oils by condensation of synthetic liquid hydrocarbon mixtures, previously cracked, in presence of  $AlCl_3$  catalyst, the catalyst, exhausted after repeated condensation, is completely revived by addition of 20% of fresh  $AlCl_3$ . The spent catalyst can be satisfactorily used for cracked products of different composition, *e.g.*, of increasing olefine content. H. G. M.

**Production of high-grade mineral lubricating oils.** C. CREANGA (B.P. 474,057, 27.4.36).—Petroleum residues are treated with porous adsorption agents, *e.g.*, activated hydrosilicates, to form homogeneous powders. The mineral oils are dissolved out by solvents, *e.g.*, gasoline, which do not dissolve the adsorbed impurities. No vac.-distillation or acid-refining is used and cracking is avoided. In examples, oils of high  $\eta$  index (60–80 Dean-Davis no.) and high chemical stability are obtained from Rumanian crude residues. The yield is 60–70%. H. G. M.

**Lowering the pour points of mineral [lubricating] oils.** A. J. VAN PESKI, Assr. to SHELL DEVELOPMENT Co. (U.S.P. 2,073,080, 9.3.37. Appl., 2.8.33. Holl., 5.8.32).— $< 1\%$  of an alkylated, highly condensed cyclic hydrocarbon having high-mol. paraffinic side chains and nuclei containing  $> 3$  aromatic or hydrogenated rings is added. An example is the condensation product of picene with chlorinated paraffin wax. D. M. M.

**Reclaiming lubricating oils.** V. VOORHEES, Assr. to STANDARD OIL Co. (U.S.P. 2,073,506, 9.3.37. Appl., 9.9.35).—Viscoresins, *i.e.*, hydrocarbon resins of mol. wt. 1000–10,000 and C:H ratio 85:15, prepared by polymerisation of olefines, may be recovered from deteriorated lubricating oils by the use of selective solvents, *e.g.*, liquid  $C_2H_6-C_4H_{10}$ . The oils are dissolved in the solvents and heated, under pressure, to a temp. near the crit. temp. of the solvent, when the resin is pptd. and may be mechanically separated. D. M. M.

**Solvent refining of lubricating oil.** (A) R. J. DEARBORN and W. P. GEE, (B) L. A. CLARKE, (C) L. A. CLARKE and C. C. TOWNE, Assrs. to TEXAS Co. (U.S.P. 2,074,254 and 2,075,268–9, [A] 16.3.37. [B, C] 30.3.37. Appl., [A] 10.5.34, [B, C] 17.4.35).—(A) Lubricating oil of low pour test and high  $\eta$  index is produced from viscous, wax-bearing oil by first diluting with a solvent consisting of  $C_6H_6$  or  $C_4H_8$ , or their homologues, and then extracting at 65–150° with furfuraldehyde (I), and separating raffinate and extract phases. The raffinate phase is cooled to 32° to separate (I) and the remaining mixture of oil, wax, and diluent is mixed with an aliphatic ketone, *e.g.*,  $COMe_2$ , in proportions to act as a selective

solvent between the oil and wax at  $-18^{\circ}$ . The whole is then cooled to  $-18^{\circ}$  to ppt. the wax, which is later removed. (B, C) Mineral lubricating oils are refined, their paraffinic and non-paraffinic constituents separated, and their  $\eta$  index is improved by treatment with selective solvents consisting of (B) aryl esters of halogenated fatty acids, e.g.,  $\text{CH}_2\text{Cl}\cdot\text{CO}_2\text{X}$  (where X is  $\text{CH}_2\text{Ph}$  or  $\text{C}_2\text{H}_4\text{Ph}$ ), a modifying solvent, e.g.,  $\text{C}_6\text{H}_6$ , being used if desired; or (C) halogen derivatives of aryl alkyl ethers, especially those in which the halogen is attached to the side chain of the compound, e.g.,  $\text{COPh}\cdot\text{CH}_2\text{Cl}$  or  $\text{COPh}\cdot\text{C}_2\text{H}_4\text{Cl}$ .

D. M. M.

**Apparatus for filtering of lubricating oil.** A. F. BURGESS. From DE LUXE PRODUCTS CORP. (B.P. 488,705, 5.10.36).—A form of filter cartridge made of fibrous adsorptive material (I) and mechanically strengthened is claimed. (I) is such as to adsorb all the sol. asphaltic materials which may be formed in the oil in use and chemically to remove any acids formed in an internal-combustion engine.

D. M. M.

**Production of lubricating oils, insulating oils, and similar viscous oils.** N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 476,984, 5.3.37. Holl., 13.3.36).—The oxidation stability of synthetic hydrocarbon oils obtained by polymerisation or condensation of unsaturated or aromatic hydrocarbons is increased by addition of 1–10% of non-paraffinic extracts obtained from natural hydrocarbons, b.p.  $>300^{\circ}$ , by means of selective solvents. The extracts are first refined by selective hydrogenation. C. C.

**Film-forming means in vacuum system for removing impurities from [insulating] oils.** J. FARWELL, Assr. to S. F. BOWSER & Co., INC. (U.S.P. 2,076,498, 6.4.37. Appl., 10.9.28).—Electric insulating oils, e.g., transformer oils, have their dielectric strength restored by passing through a vac. chamber in which they are made to flow down in thin films over concentric cylinders of porous material, e.g., cloth stretched between two plates in this chamber.

D. M. M.

**Sealing of coke chambers in coal-distillation apparatus.** T. KRETZ and E. KUHL (B.P. 488,535, 13.1.38. Ger., 18.1.37).

**Manufacture of [lubricant] petroleum products.** SOCONY-VACUUM OIL CO., INC., Assees. of R. C. MORAN, W. L. EVERS, and E. W. FULLER (B.P. 475,445, 29.9.36. U.S., 3.10., 26.10., and 16.11.35).—See U.S.P. 2,058,342–4; B., 1937, 1306.

**Treatment of contact mass. Flotation of minerals. Cleaning composition.**—See I. Phenols. Chlorinated aralkyl ketones.—See III. Alkali thiocyanates.—See VII. Waterproof concrete. Bituminous pavements. Bituminous wood-fibre composition. Preserving wood. Toxic agent for use with fibrous materials.—See IX. Materials resistant to liquid fuels. Heat-polymerising solvent naphtha.—See XIII. Rubber derivative.—See XIV. Insecticide sprays etc. Removing phenols from waste aq. liquids.—See XXIII.

### III.—ORGANIC INTERMEDIATES.

**Chlorinated solvents.** J. D. CONVERSE (Canad. Chem., 1938, 22, 361–364).—A brief review of manufacture, stabilisation, toxicity, and applications.

**Conductometric analysis of mixed sulphuric and butylsulphuric acids.** M. A. CHOCHLOVKIN and A. V. KALATSCHEVA (Sintet. Kautschuk, 1936, No. 3, 8–9).—The curve shows a slight break when all free  $\text{H}_2\text{SO}_4$  is titrated and only  $\text{BuHSO}_4$  remains. 0.1N-Ba(OH) $_2$  is used. CH. ABS. (e)

**Synthesis of fatty acids by oxidation of hydrocarbons.** G. WIETZEL (Angew. Chem., 1938, 51, 531–537).—A lecture.

**Preparation of oxalic acid from sawdust.** B. I. LEONOV (Prom. Org. Chim., 1938, 5, 489–492).— $\text{H}_2\text{C}_2\text{O}_4$  is obtained in 40–50% yield by heating 1:1 mixtures of  $\text{Ca(OH)}_2$  and sawdust at  $200^{\circ}/10$  atm., for 1.5 hr. R. T.

**Applications of formaldehyde. II. In the industrial field.** A. MONTEGAZZA (Mat. Plast., 1938, 5, 9–12; cf. B., 1937, 212).—A review.

**Determination of sodium formaldehydesulphoxylate and formaldehyde-sodium bisulphite in presence of each other.** L. SPITZER (Annali Chim. Appl., 1938, 28, 252–254).—With mixtures of the two compounds,  $\text{I}'\text{-IO}_3'$  and NaOH are added, followed, after 15 min., by HCl; I is then titrated with 0.1N- $\text{Na}_2\text{S}_2\text{O}_3$ . A further sample is treated with excess of  $\text{HgCl}_2$ , which is oxidised to  $\text{HgCl}$  only by  $\text{CH}_2\text{O}-\text{NaHSO}_3$ ; the  $\text{HgCl}$  is separated and determined iodometrically. The difference in I titres gives the equiv. of sulphoxylate. F. O. H.

**Improved technique for preparing *m*-nitroaniline from *m*-dinitrobenzene.** E. A. CALDERÓN (Anal. Asoc. Quím. Argentina, 1938, 26, 30–32).—25% *m*- $\text{C}_6\text{H}_4(\text{NO}_2)_2$  in boiling  $\text{H}_2\text{O}$  with a boiling solution containing 23% of  $\text{Na}_2\text{S}_9\text{H}_2\text{O}$  and 6% of S gives 60–87% of *m*- $\text{NO}_2\text{-C}_6\text{H}_4\text{-NH}_2$ . F. R. G.

**Analysis of technical  $\beta$ -naphthylamine-1-sulphonic acid.** K. D. SCHTSCHERBATSCHEV (Prom. Org. Chim., 1938, 5, 427–429).—7 g. of the product are dissolved in 100 c.c. of  $\text{H}_2\text{O}$  at  $80^{\circ}$ , the solution is made alkaline with aq.  $\text{NH}_3$  and filtered, and the filtrate + washings are diluted to 250 c.c. 25 c.c. of solution are titrated with 0.1N- $\text{NaNO}_2$  (A c.c., = the sum of 1-, 6-, and 8-sulphonic acids). 1 c.c. of conc. HCl is added to a second 25-c.c. portion of the solution, which is filtered after 30 min.; the  $\text{SO}_4''$  content of the filtrate + washings is determined by adding 25 c.c. of 0.2M- $\text{BaCl}_2$ , excess of which is titrated with 0.05M- $\text{K}_2\text{Cr}_2\text{O}_7$  (B c.c.). 25 c.c. of 40% NaOAc are added to a third 25-c.c. portion of solution, and AcOH is added to give a faintly acid reaction, followed by 25 c.c. of 0.2N-diazobenzene-2:5-disulphonic acid. 20 c.c. of 7% aq.  $\text{NH}_3$  are added after 90 min., and  $\text{SO}_4''$  is titrated as before (C c.c. of 0.05M- $\text{K}_2\text{Cr}_2\text{O}_7$ ). The % content of  $\beta$ -naphthylamine-1-sulphonic acid is given by  $100(1 - A + C - B)/A$ . R. T.

**Condensation of mixed vapours.**—See I. Steels in manufacture of org. acids.—See X. Naphthen-



ate driers.—See XIII. Synthetic tanning agents.—See XV. EtOH from potatoes.—See XVIII. Determining saccharin.—See XIX.

See also A., I, 465, Anodic oxidation of acid-ester salts. II, 345, Synthesis of  $n\text{-C}_6\text{H}_{14}$ . 346, Catalytic prep. of isoamyl acetate. 347, Electrolytic prep. of Ca gluconate and other salts of aldonic acids. 354, Prep. of cumulenes. 376, Electrolytic reduction of glutarimide and its derivatives. 377, Syntheses of pyrrole and indole derivatives. Prep. of isatin- $\beta$ -oxime. 379, Prep. of histidine. 386, Determining MeBr in air, EtOAc, and of  $\text{C}_5\text{H}_5\text{N}$ .

#### PATENTS.

Chlorination of methane or a homologue thereof. I. G. FARBENIND. A.-G. (B.P. 489,553, 29.1.37. Ger., 28.1.36).— $\text{CH}_4$  or a lower homologue is preheated and passed with  $\text{Cl}_2$  at high velocity through a thin porous layer or tissue of a contact material. This process strongly favours the production of  $\text{Cl}_1$ - and  $\text{Cl}_2$ -compounds. The chlorination of  $\text{CH}_4$  at  $450\text{--}480^\circ$  (preheated to  $250^\circ$ ) and of  $\text{C}_3\text{H}_8$  at  $350\text{--}370^\circ$  (preheated to  $220\text{--}240^\circ$ ) over a quartz filter is described. A. H. C.

Manufacture of higher chlorinated methanes. I. G. FARBENIND. A.-G. (B.P. 489,554, 28.1.37. Ger., 29.1.36).— $\text{MeCl}$  or  $\text{CH}_2\text{Cl}_2$  is further chlorinated in the liquid phase and with exposure to light and the resulting mixture continuously fractionated so that unaltered starting material is returned to the chlorination vessel. Apparatus and the production of  $\text{CH}_2\text{Cl}_2$  containing 2—10% of  $\text{CHCl}_3$  and  $\text{CHCl}_3$  containing 10% of  $\text{CCl}_4$  are described. A. H. C.

Manufacture and application [to textiles] of polymerised alkylene oxides. BRIT. CELANESE, LTD. (B.P. 487,652, 29.10.37. U.S., 7.11.36).—Polyalkylene oxides, useful as sizes and lubricants for textiles, are produced by treating an alkylene oxide, particularly ethylene, propylene, tetramethylene, and isobutylene oxides, and homologues and isomerides thereof, in liquid condition with an activated  $\text{Al}_2\text{O}_3$  earth at, e.g.,  $0^\circ$  for 2 to 7 days. The earth (fuller's earth) may be activated by heating at  $370\text{--}480^\circ$ . N. H. H.

Manufacture of monovinylacetylene. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 488,815, 16.7.37. Ger., 17.7.36).— $\text{CH}_2\text{C}=\text{CH}\cdot\text{CH}_2$  is obtained by passing  $\text{C}_2\text{H}_2$ , in the gas phase, over a catalyst consisting of a  $\text{Cu}^1$  salt ( $\text{CuCl}$ ), a sol. salt, preferably a halide, of another metal of groups I—III including the rare earths (e.g.,  $\text{MgCl}_2$ ), and either a polyhydric alcohol (butylene glycol, glycerol, mannitol, dulcitol) or an inorg. or an org. compound of N not derived from a N-acid ( $\text{NH}_4\text{Cl}$ ,  $\text{NH}_2\text{Ph}$ ,  $\text{HCl}$ , urea, acid amides, nitriles), at  $100\text{--}180^\circ$ . The  $\text{C}_2\text{H}_2$  may contain a little AcOH. S. C.

Production of oxidation products from aliphatic hydrocarbons. HENKEL & Co., G.M.B.H. (B.P. 489,443, 26.1.37. Ger., 31.3.36).—Higher aliphatic hydrocarbons are continuously oxidised (at a raised temp. and in presence of a catalyst) and a portion of the product is continuously saponified

with alkali and unsaponified material returned to the oxidising chamber. Suitable apparatus is claimed, and the oxidation of paraffins, b.p.  $270\text{--}360^\circ$ , at  $105\text{--}115^\circ$  in presence of Mn palmitate and saponification with soda is described. [Stat. ref.]

A. H. C.

Manufacture of surface-active agents. G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 487,669, 20.10.36).—The products of halogenation of saturated aliphatic or cycloaliphatic hydrocarbons of  $\leq \text{C}_8$  are condensed with aromatic OH-compounds or their derivatives in presence of Zn dust; the condensation products are optionally hydrogenated, and converted into  $\text{H}_2\text{O}$ -sol. derivatives. The use of Zn in place of  $\text{AlCl}_3$  as condensing agent is claimed to give colourless products. E.g., a hydrocarbon mixture (I) of mean mol. wt. 153 obtained by hydrogenation of brown coal (80% has b.p.  $70\text{--}130^\circ/20$  mm.) is chlorinated at  $95^\circ$  (wt. increase 13%) and the product is heated with PhOH and Zn dust at  $65\text{--}90^\circ$ ; the condensation product is freed from unchanged (I) and  $\text{ZnCl}_2$ , distilled in a vac., and condensed with twice its wt. of  $(\text{CH}_2)_2\text{O}$  (II) at  $140\text{--}160^\circ$ , to give a soap substitute. Other starting materials exemplified are petroleum distillates (b.p.  $180\text{--}290^\circ$ ), mixed cresols, and  $\text{OH}\cdot[\text{CH}_2]_2\text{OPh}$ , and  $\text{H}_2\text{O}$ -sol. products are obtained by condensing with (II) and sulphonating, condensing with epichlorohydrin followed by  $\text{NMe}_3$ , and condensing with  $\text{CHO}\cdot\text{CH}(\text{SO}_3\text{H})_2$ . [Stat. ref.] H. A. P.

Production of glycols. C. BARBIERI (B.P. 489,576, 29.1.37).—Olefines which may contain inert diluents (saturated hydrocarbons) are injected in a fine state with  $\text{Cl}_2$  into a stream of cold  $\text{H}_2\text{O}$  and the chlorohydrin is hydrolysed to glycerol. The chlorination of cracking gas (32.3% of olefines), treatment of the (distilled) chlorohydrin mixture with alkaline-earth hydroxide or carbonate [ $\text{Ca}(\text{OH})_2$ ], and hydrolysis of the resulting olefine oxide with dil.  $\text{H}_2\text{SO}_4$  is described. A. H. C.

Concentration of aliphatic acids. BRIT. CELANESE, LTD. (B.P. 489,259, 9.11.37. U.S., 14.11.36).—Aq. aliphatic acids (AcOH) free from mineral acid are conc. by extracting with  $\text{EtOAc}\text{-C}_6\text{H}_6$  (70—80 : 30—20) after adding 0.05—4% of a conc. aq. solution of an alkali salt (e.g.,  $\text{Na}_2\text{SO}_3$ ) to inhibit hydrolysis of  $\text{EtOAc}$ . Corrosion is inhibited by coating the interior of the extraction apparatus with urea- $\text{CH}_2\text{O}$  resin. A. H. C.

Production of fatty acids [by fermentation]. C. WEIZMANN (B.P. 489,170, 18.1.37).—The spent wash derived from  $\text{COMe}_2\text{-BuOH}$  fermentation of cereal (rice, maize) or sugary (molasses) mash is further fermented with a similar form of bacillus at  $35\text{--}37^\circ$  to yield fatty acids (chiefly  $\text{PrCO}_2\text{H}$ , 34 pts. from 275 pts. of molasses) (cf. B.P. 4845 of 1915 and 164,366; B., 1919, 301; 1921, 598).

A. H. C.

Manufacture of esters of  $\beta$ -acetylaminobutyric acid. F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 488,702, 29.3.38. Ger., 29.5.37).—*Me*, b.p.  $150\text{--}151^\circ/14$  mm., m.p.  $53\text{--}54^\circ$ , *Et*, b.p.  $152\text{--}153^\circ/14$  mm., *Pr*<sup>a</sup>, b.p.  $164\text{--}165^\circ/14$  mm., and *Pr*<sup>b</sup>, b.p.  $155\text{--}156^\circ/14$  mm.,  $\beta$ -acetamidobutyrate are obtained by

hydrogenation of the corresponding  $\beta$ -acetamidocrotonates in presence of Ni, Pd, etc. S. C.

**Manufacture of higher halogenated ketones.** I. G. FARBENIND. A.-G. (B.P. 489,552, 28.1.37. Ger., 28.1.36).—Monohalogenated aliphatic ketones (made by injecting halogen in countercurrent to the vapourised ketone at  $<$  the b.p. of the halogenated product) are further directly halogenated at  $<$  the b.p. of the monohalogen compound. The bromination of  $\text{COMe}\cdot\text{CH}_2\text{Br}$  to a mixture of  $\text{CO}(\text{CH}_2\text{Br})_2$  and  $\text{COMe}\cdot\text{CHBr}_2$  and the chlorination of  $\text{COMe}\cdot\text{CH}_2\text{Cl}$  to a mixture of  $\text{CH}_2\text{Cl}\cdot\text{CO}\cdot\text{CHCl}_2$  and  $\text{COMe}\cdot\text{CCl}_3$  is described. A. H. C.

**Counteracting polymerisation of methyl vinyl ketone during distillation or storage.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 489,634, 29.1.37).—Polymerisation of  $\text{COMe}\cdot\text{CH}:\text{CH}_2$  during storage or distillation (e.g., from mixtures with  $\text{CH}_2:\text{CH}\cdot\text{C}:\text{CH}$ ) is minimised by adding a small amount of an unsulphonated org. acid sol. in  $\text{H}_2\text{O}$  and the ketone ( $\text{AcOH}$  etc.). A. H. C.

**Manufacture of urea.** COMP. DE PROD. CHIM. ET ELECTROMÉTALL. ALAIS, FROGES ET CAMARGUE (B.P. 488,404, 30.11.37. Fr., 9.12.36).—Urea is manufactured by passing a suspension in an inert liquid, e.g., a mineral oil, of  $\text{NH}_2\cdot\text{CO}_2\text{NH}_4$  prepared in a closed vessel from residuary gaseous  $\text{CO}_2$  and liquid  $\text{NH}_3$ , both derived from a preceding operation, into a urea-forming autoclave at  $150\text{--}250^\circ$ , together with fresh  $\text{CO}_2$  and  $\text{NH}_3$ . N. H. H.

**Manufacture of colloidal urea-aldehyde condensation products.** KALLE & Co. A.-G. (B.P. 488,686, 28.9.37. Ger., 1.10.36).— $\text{H}_2\text{O}$ -sol. colloids which are particularly valuable as additions to soaps are prepared by condensing a mixture of  $\text{H}_2\text{O}$ -sol. intermediate condensation product of urea or its derivatives with  $\text{CH}_2\text{O}$  or  $(\text{CHO})_2$  in presence of a  $\text{H}_2\text{O}$ -sol. ether (I) of a highly polymeric carbohydrate (cellulose) in aq. solution while stirring. Sp. claim is made where 1.5—2.5 (2.0) mols. of urea are used per mol. of  $\text{CH}_2\text{O}$ , and especially when (I) is a  $\text{H}_2\text{O}$ -sol., preferably highly viscous, methylhydroxyethylcellulose. R. G.

**Manufacture of substituted carboxylic acid amides and their polymerisation products.** E. I. DU PONT DE NEMOURS & Co. (B.P. 489,311, 25.1.37. U.S., 24.1.36).—The interaction of an ester of an  $\alpha$ -methylene monocarboxylic acid (I), e.g.,  $\text{CH}_2\cdot\text{CMe}\cdot\text{CO}_2\text{Me}$  (II), with an alkali-metal derivative of a carboxylamide, e.g.,  $\text{NH}_2\cdot\text{CO}\cdot\text{NHNa}$ , preferably in presence of a catalyst ( $\text{COMe}_2$ ,  $\text{Bu}^\circ\text{OH}$ ) and, if desired, a solvent (excess of the catalyst, or a hydrocarbon), at, e.g.,  $-40^\circ$  to  $200^\circ$  under pressure gives substituted carboxylamides in which at least one H attached to N is replaced by the acyl group of (I). The products are polymerised by known methods, either alone or with other polymerisable substances, to give products useful as moulding, coating, sizing, etc. compositions. E.g., (II) (100) and  $\text{HCO}\cdot\text{NH}_2$  (45) in liquid  $\text{NH}_3$  (623) with Na (23 pts.) for 16 hr. at room temp. give on diluting with  $\text{H}_2\text{O}$ , extracting the product with  $\text{Et}_2\text{O}$ , and distilling monomethacrylylformamide. Methacrylyl-urea and -thiourea

are formed similarly. The polymerisation of these products, both alone and with, e.g., (II), is described. N. H. H.

**Manufacture of nitriles.** R. GREENHALGH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 488,036, 29.12.36).— $\text{COCl}_2$  is heated with monobasic aliphatic saturated or olefinic amides ( $\neq \text{C}_3$ ) either alone or in a solvent (e.g., xylene) at  $100\text{--}150^\circ$ . The prep. of nitriles of stearic, oleic, and mixed coconut oil fatty acids is described. A. H. C.

**Preparation of polymerised nitriles.** ARMOUR & Co. (B.P. 488,808, 10.4.37. U.S., 27.7.36).—Aliphatic acid nitriles  $>\text{C}_4$  are polymerised by treatment with catalysts such as  $\text{AlCl}_3$ ,  $\text{ZnCl}_2$ ,  $\text{FeCl}_3$ ,  $\text{SnCl}_4$ ,  $\text{S}_2\text{Cl}_2$ ,  $\text{PCl}_3$ ,  $\text{P}_2\text{S}_5$ , S, P, at  $>100^\circ$ . In the examples, mixtures of palmito- and stearo-nitriles, lard fatty acid nitriles, and cracked nitriles from these products are treated with  $\text{AlCl}_3$  to give thick, heavy oils suitable for use as dielectrics. S. C.

**Condensation of organic metal derivatives with acylated compounds.** N. V. CHEM. FABR. SERVO, and M. D. ROZENBROEK (B.P. 489,026, 14.10.36).—The interaction of esters of aliphatic carboxylic acids (I) of  $\geq \text{C}_4$  or of OH-fatty acids (II) acylated on the OH with carboxylic acids or their esters or alkali salts with aliphatic, aromatic, carbocyclic, or heterocyclic mono- or poly-hydric primary, *sec.*, or *tert.* alkoxides or phenoxides and/or org. metal compounds in which the H attached to C is replaced by metal, e.g., metal derivatives of  $\text{Et}_2$  malonate, gives the corresponding ether and the metal salt of (I) or (II). E.g., the Ac derivative of octan- $\beta$ -ol (200) with  $\text{PrCO}_2\text{Na}$  (150 pts.) at  $125^\circ$  for 2 hr. gives a product, Ac val. 245, which probably contains an ether. The prep. of ethers from ricinoleic acid and malonic acid, terpenyl alcohol and oleyl alcohol, the Et, Bu, glyceryl, and Ph ethers of ricinoleic acid, etc. is described. N. H. H.

**Manufacture of fluorene derivatives.** G. SHELDRICK, M. WYLER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 489,612, 27.1.37).—Interaction of fluorene,  $\text{NPhEt}\cdot\text{COCl}$  (I), and  $\text{AlCl}_3$  gives fluorene-2-carboxyethylamide, m.p.  $163^\circ$ , and -2:7-bis(carboxyethyl)amide (best in presence of NaCl), m.p.  $174\text{--}175^\circ$ . Fluorene-2-carboxylic, m.p.  $278^\circ$ , and -2:7-dicarboxylic acid are produced on hydrolysis with 65%  $\text{H}_2\text{SO}_4$  or  $\text{KOH}\text{--EtOH}$ . The reaction is applicable to homologues of (I). H. A. P.

**Manufacture of naphthalene derivatives.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 489,236, 22.1.37).—A  $\text{C}_{10}\text{H}_8$  derivative useful as a (reddish-blue) dye for acetate rayon or as an intermediate for dyes is prepared by adding (until the  $p_{\text{H}}$  is  $\sim 9$ ) a non-oxidising basic substance, e.g., aq. solutions of alkali hydroxides and carbonates or  $\text{NH}_4$  or quaternary  $\text{NH}_4$  bases, to solutions obtained by the action of S sesquioxide or  $\text{H}_2\text{S}$  on 1:5- $\text{C}_{10}\text{H}_6(\text{NO}_2)_2$  in conc. or fuming  $\text{H}_2\text{SO}_4$  or  $\text{ClSO}_3\text{H}$  and diluting with  $\text{H}_2\text{O}$ . N. H. H.

**Manufacture of nitrogen compounds.** W. J. TENNANT. From HENKEL & Co., G.M.B.H. (B.P. 489,190, 1.3.37).—*sec.*- and *tert.*-Amines and quaternary compounds are obtained by treating nuclear alkyl-

( $\neq C_4$ )-substituted hydroaromatic amines with alkylating agents, e.g., alkyl halides, sulphates, alkylene oxides, and aldehydes, preferably at elevated temp. The following are described: *compounds* from the interaction of (4-*sec.*-octylcyclohexyl)methylamine and glycide (at 160—170°), and  $(CH_2)_2O$  (at 130—140°); 2-methyl-4-*sec.*-alkyl-( $C_{6-12}$ )-cyclohexylamines, b.p. 135—155°/2 mm., and  $CH_2Cl \cdot CH_2 \cdot SO_3Na$  (in dil. EtOH at 160°);  $CH_2Cl \cdot CH_2 \cdot OH$  and  $C_5H_5N$  (at 100—110°) followed by sulphation with  $ClSO_3H$ , and  $CH_2Cl \cdot CO_2Na$ ; N-(4-dodecylcyclohexyl)-N-benzyl-N-dimethylammonium methosulphate, (2-methyl-*sec.*-4-dodecylcyclohexyl)methylammonium methosulphate, (2-methyl-4-*sec.*-dodecylcyclohexyl)dimethylamine, b.p. 155—160°/2 mm., and the corresponding *amine oxide*. The products are used as foaming, wetting, and capillary-active agents and disinfectants. S. C.

**Manufacture of 4-nitro-2-amino-1-[hydr]oxynaphthalenesulphonic acids.** SOC. CHEM. IND. IN BASLE (B.P. 487,718, 11.5.37. Switz., 14.5.36).—The corresponding 2:4-dinitro-1-naphthol(-6-, -7-, or -8-)sulphonic acid is reduced by O-free S compounds of the alkaline-earth metals. E.g., 1:2:4:8-OH· $C_{10}H_4(NO_2)_2 \cdot SO_3Na$  is reduced by  $Na_2S$  and  $MgCl_2$  in  $H_2O$  at 40—50°. H. A. P.

**Manufacture of aminoalkyl esters of carbocyclic acids and of aliphatic amino-alcohols therefrom.** T. SABALITSCHKA and E. BÖHM (B.P. 487,824, 4.1.38. Ger., 22.6.37).—The esterification of carbocyclic acids with alkylene diols in presence of gaseous HBr (or HCl) and treatment of the resulting halogenoalkyl esters with amines gives aminoalkyl esters of the carbocyclic acids, which are saponified to the corresponding amino-alcohols; e.g.,  $p-OH \cdot C_6H_4 \cdot CO_2H$  (40) with trimethylene glycol (50 pts.) and gaseous HCl by warming for 8 hr. gives *n*- $\gamma$ -chloropropyl *p*-hydroxybenzoate, m.p. 72°, which on warming with  $NHEt_2$  yields  $\gamma$ -diethylamino-*n*-propyl *p*-hydroxybenzoate, m.p. 133°, the latter being converted by KOH into  $\gamma$ -diethylamino-*n*-propyl alcohol, b.p. 90°/23 mm. Similarly,  $\beta$ -chloroethyl, b.p. 180°/20 mm., and  $\beta$ -diethylaminoethyl anisate and  $\beta$ -diethylaminoethyl alcohol, b.p. 70°/35 mm., and  $\beta$ -chloro- and  $\beta$ -diethylamino-isopropyl, b.p. 178°/35 mm., *p*-hydroxybenzoate are prepared. N. H. H.

**Manufacture of derivatives of  $\alpha$ -[hydr]oxy-[2-]naphthoic acid.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 487,770, 22.9.36).—Halides of 1:2-OH· $C_{10}H_6 \cdot CO_2H$  are condensed with colourless or slightly coloured polynuclear amines which have the property of forming substantive azo dyes. The products are used as intermediates for the prep. of non-diffusible azo dyes for colouring (gelatin) photographic layers. Examples are the *bis*-1-hydroxy-2-naphthoyl derivatives of benzidine, *m*-tolidine, 3:3'-dichlorobenzidine, 2:7-diaminocarbazole, benzidine-3':3'-dicarboxylic acid, 4:4'-bis-*p*-aminobenzamidostilbene-2:2'-disulphonic acid, and 4:4'-bis-*p*-aminobenzamidodiphenylurea-2:2'-disulphonic acid ("Dialzo light yellow 2G"). H. A. P.

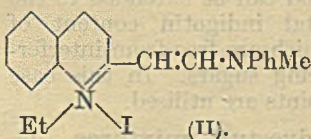
**Recovery of phenols.** BAKELITE, LTD. (B.P. 488,574, 9.1.37. U.S., 30.6. and 24.7.36).—Phenols, e.g., mixtures of PhOH, cresols, and xlenols, are 4 H (B.)

extracted from aq. liquors by treatment with tar acids of higher b.p. than the phenols (b.p.  $\neq 230^\circ$ ). After separation of the layers, which can be facilitated by adjusting the  $p_H$  to  $< 3$  (1.5—1.8), the phenols are recovered by distillation. Tars or tar oils are subjected to at least two washes with NaOH, the first with  $<$  enough to combine with all the phenols and the last with an excess; the last wash is used as the first in subsequent stages. The Na phenolate is distilled and the distillate passed in contact with neutral oil (saturated with  $C_5H_5N$ ) to absorb phenols carried over. N. H. H.

**[Preparation of] chlorinated aralkyl ketones.** [Lubricants.] ARMOUR & Co. (B.P. 487,642, 7.10.37. U.S., 9.3.37).—Aralkyl ketones, e.g., Ph, tolyl, or xylyl heptadecyl ketone, are chlorinated (in presence of a trace of I) to give, according to the temp. and amount of  $Cl_2$ , mono-, di-, or higher chlorinated derivatives, which are lubricants of increased oiliness compared with the parent substances. N. H. H.

**Manufacture of anthraquinone derivatives.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 487,877, 30.12.36).—The interaction of  $CH_2Ph \cdot CN$  (I) with a 2-cyanoanthraquinone-3-carboxylic acid, its amide, or ester, or of CuCN and (I) with a 2-halogenoanthraquinone-3-carboxylic acid, its amide, or ester gives products (dye intermediates) distinguished by a cornflower-blue vat. E.g., Me 2-chloroanthraquinone-3-carboxylate, CuCN, and excess of (I) at the b.p. for 2 hr. give a yellow Cl-free product, with intermediate formation of Me 2-cyanoanthraquinone-3-carboxylate, m.p. 247°. Similar products are obtained from 2-chloroanthraquinone-3-carboxylic acid (m.p. 343—344°) and its amide. H. A. P.

**Manufacture of heterocyclic nitrogen compounds containing aldehyde groups.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 486,780, 9.12.36).—Cyclic  $NH_4$  salts containing reactive Me or their  $\psi$ -bases are condensed with formamides  $NR \cdot CH \cdot NR''$  in which R and R' are aryl and R'' is alkyl, aryl, or aralkyl, or  $NR'R''$  is part of a saturated ring; the polymethine dyes thus obtained are split by alkalis with the formation of aldehydes. E.g., 2-methylquinoline ethiodide and diphenylmethylformamide,  $NPh \cdot CH \cdot NPhMe$  (I), give the yellow dye (II), which with aq. NaOH ( $d$  1.2) at 100° gives an aldehyde, m.p. 101—102°, probably 1-ethyl-2-aldehydo-methylenequinoline,



and  $NPhMe$ . Corresponding aldehydes are obtained from (I) and 1:2:3:3-tetramethylindolenium bromide (III), m.p. 117—118°, b.p. 170—172°/2 mm. (IV), 1-methylbenzthiazole methosulphate, m.p. 111—112°, and 2-methyl- $\beta$ -naphththiazole methosulphate, m.p. 189—190°. Interaction of (III) with phenyl-2-methylindolinyformamide gives a bright yellow basic dye, split as before by alkalis to 2- $\omega$ -aldehydomethylene-1:3:3-trimethylindoline [(IV) above] and 2-methylindoline; similar products [5-*OME*-derivative of (IV)], m.p. 107—108° are obtained with 5-methoxy-1:2:3:3-tetramethylindolenium bromide. Similarly,

1:2:3:3-tetramethyl- $\beta$ -naphthindoleninium bromide and phenyltetrahydroquinolylformamide give a yellow basic dye of good light-fastness, converted by alkaline hydrolysis into an aldehyde, m.p. 152°, and tetrahydroquinoline. H. A. P.

Apparatus for reactions between liquids and gases or vapours.—See I. Hydrocarbons for motor fuels. Alkenes from hydrocarbon mixtures. Polybasic acid derivatives of hydroformed naphthas. High-boiling polymerides. Products for imparting colour etc. to lubricating oils.—See II. Anthraquinone etc. compounds. Phthalocyanines.—See IV. Catalysts for org. synthesis.—See VII. Condensation products. Polymeric amides.—See XIII. Preservation of rubber. Vulcanisation accelerators.—See XIV. Bu<sup>o</sup>OH·COMe<sub>2</sub>·EtOH fermentations.  $\beta\gamma$ -Butylene glycol.—See XVIII.

#### IV.—DYESTUFFS.

Electrometric determination of free sodium hydrosulphite or free indigo in reduced indigo preparations. N. STRAFFORD and W. V. STUBBINGS (J.S.C.I., 1938, 57, 242—245).—Both determinations depend on the preferential oxidation of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> in presence of alkali salts of leucoindigo (I) by Fehling's solution (II). A special apparatus is used from which air is displaced by purified N<sub>2</sub>. The course of the titration is followed potentiometrically. A full potentiometric titration exhibits two sharply defined end-points, the first due to a considerable rise in oxidation-reduction potential as soon as all the Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> has been oxidised, the second to a further considerable rise in potential as soon as all the (I) has been oxidised. Only the first end-point is utilised in the determinations described. For the Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> determination, (II) is standardised in the same apparatus against a Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> solution previously standardised by an established method, e.g., by 0.1N-I. For the free indigo determination, a measured quantity (excess) of standard Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> solution is added and, after "vatting," the excess is determined as above with (II). In this case the Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> solution is standardised by the same technique against standard indigo powder. The method can be extended to the determination of the total indigotin content of reduced indigo products which are free from interfering substances, e.g., reducing sugars. In this case both potentiometric end-points are utilised.

Colour intensity of dyestuff mixtures. G. SZAMEK (Tech. Kurir, 1938, 9, 55—57).—The vals. are obtained by adding the vals. ( $F_A$ ,  $F_B$ , etc.) for the components ( $A$ ,  $B$ , etc.) in proportion to their concn. ( $C_A$ ,  $C_B$ , etc.).  $F_{A+B} = C_A F_A + C_B F_B + \dots$ . To compare the colour intensity of two dyes, to  $a$  g. of  $A$   $p$  g. of "chromoxide-green" (I) must be mixed to obtain the colour intensity of  $b$  g. of  $B$  mixed with  $q$  g. of (I);  $F_A = F_B(bq/ap)$ . E. P.

Diazosulphonates in textile printing.—See VI. Fluorescence analysis.—See XI.

See also A., II, 359, Coupling methone with tetrazonium compounds. 364,  $\alpha$ - and  $\beta$ -Naphth-

oic acids. 370, Alizarates. 378, Dyes from thiohydantoin. 380, Flavinduline derivatives.

#### PATENTS.

Production of monoazo dyes. SOC. CHEM. IND. IN BASLE (B.P. 487,621, 31.3.37. Switz., 31.3.36. Cf. B.P. 424,354; B., 1935, 717).—Diazo compounds derived from amines  $p$ -NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·N(CH<sub>2</sub>Ph)·SO<sub>2</sub>Ph (I) in which each of the C<sub>6</sub> nuclei may be substituted, e.g., by halogen, alkyl, or alkoxy, are coupled with 2:8:6-NHBz·C<sub>10</sub>H<sub>5</sub>(OH)·SO<sub>3</sub>H (II) in which the Bz may be substituted by halogen, alkyl, or alkoxy. Alternatively (I) may be coupled with 2:8:6-NH<sub>2</sub>·C<sub>10</sub>H<sub>5</sub>(OH)·SO<sub>3</sub>H and the azo dye treated with BzCl or its substituted derivatives. The example is the dye (I)  $\rightarrow$  (II) coupled in presence of excess of NaOAc. The dyes afford brilliant red shades on animal fibres; those on wool possess good fastness to perspiration and milling, whilst natural silk is dyed from a neutral bath with complete exhaustion. K. H. S.

Production of azo dyes. COMP. NAT. DE MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES ETABL. KUHLMANN (B.P. 489,458, 3.8.37. Fr., 4.8.36).—Aminoxanthenes free from H<sub>2</sub>O-solubilising groups are diazotised and coupled in substance or on the fibre with arylamides of *o*-hydroxycarboxylic acids or acylacetic acids. Among examples, 2-acetamido-2'-carboxydiphenyl ether, m.p. 178°, is ring-closed (H<sub>2</sub>SO<sub>4</sub>) to 4-aminoxanthone, m.p. 201—202°, or 2-methyl-, m.p. 82°, is oxidised (KMnO<sub>4</sub>) to 4'-chloro-2'-acetamido-2-carboxy-diphenyl ether, m.p. 201°, and cyclised to 2-chloro-4-aminoxanthone, m.p. 233—234°, also obtained by reducing 2-nitro-, m.p. 154°, to 4-chloro-2-amino-6-carboxydiphenyl ether followed by ring-closure. Similarly 5-amino-2:4-diphenoxybenzoic acid, m.p. 123°, affords 2-amino-3-phenoxyxanthone, m.p. 210°. Orange, red, and scarlet shades of great fastness to boiling are produced by diazotising the above xanthenes and also 2-aminoxanthone and coupling on the fibre with arylamides of 2:3-OH·C<sub>10</sub>H<sub>6</sub>·CO<sub>2</sub>H; yellow shades are produced with the bis-*o*-tolidide of CH<sub>2</sub>Ac·CO<sub>2</sub>Et. K. H. S.

Manufacture of azo dyes [ice colours]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 489,035, 16.12.26).—Aminotriphenidioxazines (cf. B.P. 411,132 and 416,887; B., 1935, 794, 1051) containing NH<sub>2</sub> meta to the azine-N are diazotised and coupled on the fibre with arylamides of 2:3-OH·C<sub>10</sub>H<sub>6</sub>·CO<sub>2</sub>H (I) or CH<sub>2</sub>Ac·CO<sub>2</sub>Et, SO<sub>3</sub>H or CO<sub>2</sub>H being absent from the mols. Among examples, the following are tetrazotised and coupled: 2:6-diamino-3:7-diethoxy-, with diacetoacetyl-*o*-tolidide (bluish-black), 4:8-diamino-, with the anilide of (I) (bluish-black) and 9:10-dichloro-2:7-diamino-triphenidioxazine with the  $\alpha$ -naphthalide of (I) (navy-blue). K. H. S.

Manufacture of disazo dyes. I. G. FARBENIND. A.-G. (B.P. 489,562, 29.1.37. Ger. 31.3.36).—Diazotised aminoazo compounds or tetrazotised aromatic diamines, free from NO<sub>2</sub> or H<sub>2</sub>O-solubilising groups, are coupled with phenols having an alkyl group of  $\neq$  C<sub>3</sub> para to the OH. Among examples are the dyes *m*-aminoazotoluene  $\rightarrow$  *p*-isohexylphenol (I) (brownish-orange), Ph·N·N·C<sub>6</sub>H<sub>4</sub>·NH<sub>2</sub>  $\rightarrow$  *p*-isoamylphenol (brownish-orange), benzidine  $\rightarrow$  (I) (reddish-

yellow), and  $\text{CH}_2(\text{C}_6\text{H}_4\text{-NH}_2\text{-}p)_2 \rightarrow$  (I) (greenish-yellow). The dyes are used for colouring fats, oils, lacquers, and artificial masses and also for double-toned printing; they are more sol. than similar dyes made from PhOH or cresol, are free from odour, and of good fastness to light.

K. H. S.

**Production of anthraquinone dyes.** J. R. GEIGY A.-G. (B.P. 487,830, 9.3.38. Switz., 11.3.37).—Quinizarin (I), leucoquinizarin (II), or other anthraquinone derivatives having replaceable 1:4-substituents are condensed with aminodiphenyl ethers and the products sulphonated to give blue-green dyes of good fastness to washing, milling, potting, light, and alkalis. *E.g.*, a mixture of (I) and (II) is heated with 4- $\text{NH}_2\text{-C}_6\text{H}_4\text{-OPh}$  and  $\text{H}_3\text{BO}_3$  at  $150^\circ$ , and the product is sulphonated (conc.  $\text{H}_2\text{SO}_4$  at  $30^\circ$ ); a similar product is obtained from 2:2'- $\text{NH}_2\text{-C}_6\text{H}_4\text{-O-C}_6\text{H}_4\text{Me}$  with (I) + (II) or with 1:4-dichloroanthraquinone at  $180\text{--}190^\circ$ , followed by sulphonation. H. A. P.

**Manufacture of compounds [dyes] of the anthraquinone series.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 489,697, 30.12.36).—The prep. is claimed of 1-hydroxyalkylamino-4-di- or -poly-hydroxyalkylaminoanthraquinones in which the group at 1 has  $\leq \text{C}_2$  and the di- or poly-hydroxyalkyl group has  $\leq \text{C}_3$  by interaction of an appropriate 4-halogeno-, 4-nitro-, or 4-methoxy-1-hydroxyalkylaminoanthraquinone with a di- or poly-hydroxyamine, or of a 1:4-dihydroxy-, 1:4-diamino-, or 1-amino-4-hydroxy-anthraquinone and a reducing agent (or the corresponding leuco-compound alone, or admixed with the unreduced compound) with an appropriate mixture of a mono- and di- or poly-hydroxyamine. *E.g.*, quinizarin (24), leucoquinizarin (6),  $\text{NH}_2\text{-}[\text{CH}_2]_2\text{-OH}$  (I) (9.2), and  $\beta\gamma$ -dihydroxy-*n*-propylamine (II) (13.6 pts.) in  $\text{Bu}^\beta\text{OH}$  at  $60^\circ$ —the b.p. gives (after air-oxidation) mainly 1- $\beta$ -hydroxyethylamino-4- $\beta\gamma$ -dihydroxypropylamino- (III) with some 1:4-bis- $\beta\gamma$ -dihydroxypropylamino- (both  $\text{H}_2\text{O}$ -sol.) and 1:4-di- $\beta$ -hydroxyethylamino-anthraquinone; a similar product is obtained from leuco-1:4-diaminoanthraquinone, (I), and (II). Bromination of 1- $\beta$ -hydroxyethylaminoanthraquinone (Br in HCl at  $40^\circ$ ) and interaction of the product with (II) gives (III). A product, mainly 1- $\beta$ -hydroxyethylamino-4- $\beta\gamma$ -dihydroxypropylamino-5:8-dihydroxyanthraquinone, is obtained from leuco-1:4:5:8-tetrahydroxyanthraquinone, (I), and (II). The products are blue to bluish-green  $\text{H}_2\text{O}$ -sol. dyes for cellulose esters and ethers.

H. A. P.

**Manufacture of compounds [dyes] of the anthraquinone and anthrapyrimidine series.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 487,878, 30.12.36).—Amino-anthraquinones or -anthrapyrimidines free from  $\text{CO}_2\text{H}$  or esterifiable OH are treated with a P halide followed by an aq. alkali to give a  $\text{H}_2\text{O}$ -sol. salt of a phosphamic acid (of the P<sup>III</sup> or P<sup>V</sup> series). The products dye wool and artificial silk in fast shades. *E.g.*,  $\text{POCl}_3$  is added at  $<0^\circ$  to 1:4-diamino-2-methoxyanthraquinone (I) in excess of anhyd.  $\text{C}_5\text{H}_5\text{N}$  and, when the product is completely sol. in aq.  $\text{Na}_2\text{CO}_3$ , it is added to this, the  $\text{C}_5\text{H}_5\text{N}$  is removed by distillation in steam, and the Na salt (of a mixture of mono- and di-phosphamic

acids) is pptd. with NaCl. It is stable in  $\text{H}_2\text{O}$  or alkalis but regenerates (I) with hot acids and is a bluish-red dye for wool, viscose, and cellulose acetate. Similar products are obtained from 1:4-diamino-, 1-amino-4-*p*-aminoanilino-, 1-amino-4-*p*-anisidino-, 1:4:5:8-tetra-amino-anthraquinone, and 4-amino-1:9-anthrapyrimidine (and  $\text{POCl}_3$ ), and from  $\text{PCl}_5$  and 1-aminoanthraquinone. H. A. P.

**Manufacture of [anthraquinone] vat dyes.** F. IRVING, C. SHAW, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 487,798, 24.12.36).—Grey vat dyes are obtained by condensing a 4:4'-bismono-chloro- or -bromo-benzamido-1:1'-dianthrimide with 1-amino-4-, -5-, or -8-benzamidoanthraquinone (in an inert solvent, *e.g.*, boiling  $\text{C}_{10}\text{H}_8$ , in presence of anhyd.  $\text{Na}_2\text{CO}_3$  and CuCl). The products from 1-amino-4-benzamidoanthraquinone and 4'':4'''-dibromo- and 3'':3'''-dibromo-4:4'-dibenzamido-1:1'-dianthrimide (prepared from 4:4'-diamino-1:1'-dianthrimide and the appropriate bromobenzoyl chloride in  $\text{PhNO}_2$ ) are described. H. A. P.

**Vat dyes of the dipyrazoleanthronyl series.** I. G. FARBENIND. A.-G. (B.P. 487,723, 7.6.37. Ger., 9.6.36).—Vat dyes are made by interaction of an alkali salt of 2:2'-dipyrazoleanthronyl (I) with the cyclohexyl ester of an arylsulphonic acid or such an ester alkylated in the cyclohexyl nucleus, isolating the product, converting it into alkali salt, and interaction with the alkyl ester of an arylsulphonic acid. In the example, the K salt of (I) (530 of 97%) is mixed with  $\text{K}_2\text{CO}_3$  (69), *o*- $\text{C}_6\text{H}_4\text{Cl}_2$  (II) (5000), and  $\text{PhSO}_3\text{-C}_6\text{H}_{11}$  (620) and heated at  $120\text{--}125^\circ$  for 8–10 hr. The intermediate thus formed is isolated, milled with KOH (640), mixed with (II), and treated with *p*- $\text{C}_6\text{H}_4\text{Me-SO}_3\text{Et}$  (530 pts.) and the mixture heated at  $120\text{--}125^\circ$ . Cotton is dyed bright red from a blue vat and the dyes are distinguished from those not containing the cyclohexyl nucleus by better solubility of the leuco-compounds, better fastness properties, and yellower shades. K. H. S.

**Manufacture of vat dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 487,380, 15.12.36, 23.2., 11.3., 29.5., and 20.7.37).—Carboxyl halides of aromatic or heterocyclic azoxy- or azo compounds are condensed with vatable amines, *o*-diamines, *o*-hydroxyamines, or *o*-aminothiols, both components being free from  $\text{H}_2\text{O}$ -solubilising groups. The dyes produced can apparently be vatted without reduction of the azo or azoxy-group. Examples are the dyes from azobenzene-4:4'-dicarboxylic acid and 1-amino- (I) (green-yellow), 1-amino-4- (II) (orange-red) and -5-benzamido- (III), 2-amino-1-thiol-, 1-amino-2-hydroxy-, and 3-bromo-2-amino-1-methylamino-anthraquinone (all yellow), 4- or 5-aminoanthrapyrimidine, 2'-chloro-4-amino-*p*-phenylanthraquinone-1:2-iminazole, and (II) + (III) (equimols.); azoxybenzene-4:4'-dicarboxylic acid and (III) (?); azobenzene-4-carboxylic and -3:3-dicarboxylic acid, 3:3'-dimethoxyazobenzene-4:4'- (IV) and 4-methoxyazobenzene-3':5'-dicarboxylic acid (V), 4:4'-azodiphenyl-4'':4'''-dicarboxylic acid (VI), 4:4'-azoxydiphenyl-4'':4'''-dicarboxylic acid (VII), 7:7'-azodiphenylene oxide-2:2'-dicarboxylic acid (VIII), 1:1'-azonaphthalene-5:5'-dicarboxylic acid (IX),

4':4'''-azo-1-phenylbenzthiazole-5 : 5''-dicarboxylic acid (X), 4:4'-azocinnamic acid (XI), 4:4'-azophenoxyacetic acid (XII), 4:4'-azophenylacetic acid, 4:4':4''-benzeneazobenzeneazobenzeneazobenzene-1:4''-dicarboxylic acid (XIII), 4:4'-bisbenzeneazobenzophenone-4'' : 4''-dicarboxylic acid (XIV), and 4:4':4''-trisbenzeneazotriphenylmethane-4'' : 4'' : 4''-tricarboxylic acid (XV) and (III); 3:3'-azocinnamic acid and aminodibenzanthrene; (VI), (VIII), 4:4'-bisbenzeneazodiphenyl-4'' : 4''-dicarboxylic acid (XVI), azobenzene-4 : 3 : 5'-tricarboxylic acid (XVII), and azo- (XVIII) and azoxy-benzene-3 : 5 : 3' : 5'-tetracarboxylic acid (XIX) and (I); (VIII) and (II), and (VI) and 6-chloro-1-aminoanthraquinone. The azo and azoxy-compounds (IV), (VI) (dichloride, m.p. 250°), (VII) (dichloride, m.p. 192°), (VIII), (IX) (dichloride, m.p. 215°), (X) (dichloride, m.p. > 300°), (XI) (dichloride, m.p. 221°), (XIII) (dichloride, m.p. 125°), (XVII) (tetrachloride, m.p. 133°), and (XIX) are prepared by alkaline reduction [glucose; Na<sub>2</sub>AsO<sub>3</sub> in the case of (XIX)] of the corresponding NO<sub>2</sub>-compounds; (V) (dichloride, m.p. 135°) is obtained by condensing diazotised 5:1:3-NH<sub>2</sub>-C<sub>6</sub>H<sub>3</sub>(CO<sub>2</sub>H)<sub>2</sub> with PhOH and methylating, and (XIII) (dichloride, m.p. > 300°), (XIV) (dichloride, m.p. 242°), (XV) (trichloride, m.p. 215°), (XVI) (dichloride, m.p. 235°), and (XVII) (trichloride, m.p. 135°) are prepared by condensation of an appropriate NH<sub>2</sub>-compound with *p*-NO<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>-CO<sub>2</sub>Me and hydrolysis. 7-Nitro-2-carboxydiphenylene oxide is obtained by nitrating diphenylene oxide (HNO<sub>3</sub>-AcOH), condensing with AcCl (AlCl<sub>3</sub>), and oxidising (hypochlorite). H. A. P.

**Manufacture of vat dyes.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 487,071, 18.1.37).—*Pz*-Arylamino-*Pz*-hydroxypyrazino-*o*-aroylbenzoic acids and their arylamides are cyclised by dehydrating catalysts (strong acids, BF<sub>3</sub>) to the corresponding anthraquinone derivatives. *E.g.*, 2-*p*-toluidino-3-hydroxy-6-*o*-carboxybenzoylquinoxaline, prepared by interaction of 3' : 4'-diamino-2-benzoylbenzoic acid with H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, and condensation of the resulting dihydroxyquinoxaline with ZnCl<sub>2</sub> and *p*-C<sub>6</sub>H<sub>4</sub>Me·NH<sub>2</sub>, is converted by 96% H<sub>2</sub>SO<sub>4</sub> at 80° into a mixture of a yellow-red and a blue-red vat dye, presumably 5 : 6-*phthaloyl*- and 6 : 7-*phthaloyl*-2-*p*-toluidino-3-hydroxyquinoxaline. Similar products are obtained from the corresponding 2-*p*-chloro-anilino- and 2-*m*-toluidino-derivatives and from 3' : 6'-dichloro-2-*p*-toluidino-3-hydroxy-6-*o*-carboxybenzoylquinoxaline. H. A. P.

**Manufacture of dye powders.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 488,193, 29.12.36).—The use is claimed as dispersing agents for H<sub>2</sub>O-insol. dyes of the products of interaction of CH<sub>2</sub>O (< 1 mol.), phenols (2 mols.), and primary or *sec.* amines (< 1 mol.), *e.g.*, the product from CH<sub>2</sub>O (2 mols.), PhOH (1 mol.), and NHMe·[CH<sub>2</sub>]<sub>2</sub>·SO<sub>3</sub>Na (1 mol.) (cf. B.P. 470,335; B., 1937, 1177) is used to form very fine dispersions, giving ink-like liquids with H<sub>2</sub>O, of 1-methylamino-4-β-hydroxyethylamino-anthraquinone and the vat dye produced by fusion of 3-anthraquinonylamino-benzanthrone with alkali; other dyes dispersed by similar products are Cu

phthalocyanine, a sulphonated green triphenylmethane dye from CO(C<sub>6</sub>H<sub>4</sub>·NMe<sub>2</sub>-*p*)<sub>2</sub> and 4 : 3'-C<sub>6</sub>H<sub>4</sub>Me·NH·C<sub>6</sub>H<sub>4</sub>·OEt, and the sulphuric ester of leuco-4-2' : 5'-dichlorobenzamido-1 : 9-anthrapyrimidine. H. A. P.

**Production of dye pigment pastes.** I. G. FARBENIND. A.-G. (B.P. 487,601, 22.12.36. Ger., 6.1.36).—A dry, powdered pigment is made into an aq. paste with a quaternary NH<sub>4</sub> or PH<sub>4</sub> or a ternary SH<sub>3</sub> compound having an aliphatic or cycloaliphatic residue of > C<sub>5</sub>, *e.g.*, *n*-NMe<sub>3</sub>Cl·C<sub>12</sub>H<sub>25</sub>. H. A. P.

**Manufacture of cyanine dyes and their application to photographic emulsions.** B. GASPARD (B.P. 488,093, 27.11.36).—Carbocyanine dyes containing two heterocyclic nuclei linked by < 3 CH, *e.g.*, benzthiocarbocyanine, 1-naphthothiocarbocyanine, and benzselenocarbocyanine 1-etho-*p*-toluenesulphonates, are sulphonated with oleum. The sulphonated dyes are more sol. and less sensitive to pptn. with, *e.g.*, *s*-C<sub>6</sub>H<sub>3</sub>(OH)<sub>3</sub> or acid dyes and are particularly suitable for sensitising dyed emulsions and for dyeing filters and antihalation layers. S. C.

**Manufacture of phthalocyanines.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 488,201, 1.1.37).—Metallic (Cu, Ni, Co, Fe) phthalocyanines having very yellowish green shades are obtained by heating *o*-arylene dicyanides containing oxazole, thiazole, or glyoxaline nuclei with metals or metallic compounds, preferably in presence of a solid or liquid diluent. The corresponding metal-free phthalocyanines are obtained by heating the appropriate nitrile with an alkali metal (Na) in presence of an alcohol. Alternatively the *o*-dinitrile may be produced during the reaction by using the corresponding halogeno-compounds and heating with CuCN. The products may be dispersed to form pigments by appropriate agents and they afford sol. compounds suitable for dyeing textiles and for making metallic lakes on sulphonation with oleum. 4' : 4'' : 4''' : 4''''-Tetra-(5-methylbenzthiazolyl)phthalocyanine (Cu and Ni derivatives), 4' : 4'' : 4''' : 4''''-tetra-(1-benzoxazolyl)- and -(2-benzimidazolyl)-copper phthalocyanines are described. Asymmetric phthalocyanines are obtained by replacing part of the substituted *o*-dinitrile with *o*-C<sub>6</sub>H<sub>4</sub>(CN)<sub>2</sub>. The compounds may be brominated and sulphonated. 1-3' : 4'-Dicyanophenyl-5-methylbenzthiazole, m.p. 260—261°, is prepared by heating Na or K 1-4'-cyanophenyl-5-methylbenzthiazole-3'-sulphonate with NaCN and K<sub>2</sub>Fe(CN)<sub>6</sub> in vac. at 340—380°. 1-3' : 4'-Dibromophenylbenzoxazole, m.p. 149°, and 2-3' : 4'-dibromophenylbenzimidazole are obtained by interaction of 3 : 4-C<sub>6</sub>H<sub>3</sub>Br<sub>2</sub>·COCl with *o*-NH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>-OH and *o*-C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub>, respectively, in quinoline. S. C.

**Dry powdered dye materials.**—See I. C<sub>10</sub>H<sub>8</sub> derivative. Heterocyclic N compounds.—See III. Dyes for acetate silk.—See VI.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Effect of salts in water for silk reeling on colour and lustre of raw silk.** I. Salts in water for heating and reeling of cocoons. II. Effect

of salts on colour and lustre. T. NAKAHARA and S. NISIMURA (J. Agric. Chem. Soc. Japan, 1938, 14, 843—848, 849—853).—I, II. When raw silk is reeled with very dil. solutions (0.001—0.0001%) of salts of Fe, Cu, Al, Ca, Mg, Na, and K there is no appreciable effect on the colour of the silk, but with 0.01% solutions the white content of raw silk is decreased markedly by Fe and Cu, moderately by Al, and slightly by Ca. It is suggested that the effect of the salt on the colour is due to adsorption of the cation. The degree of gloss of the raw silk  $\propto$  amount of fat or loss of sericin on reeling, but there is no relationship between effect of cations or anions and lustre.

J. N. A.

**Action of cyanamide on wool.** J. B. SPEAKMAN, B. NILSSEN, and C. S. WHEWELL (Nature, 1938, 141, 688—689).—The curves showing the reduction in the resistance to extension of purified Cotswold wool staple fibres in buffer solutions of different  $p_H$ , with and without previous  $CN \cdot NH_2$  (I) treatment, are reproduced. For fibres treated with (I) for 47 hr. at 22.2°, resistance to extension is independent of  $p_H$  between  $p_H$  5 and 10, owing to the conversion of cysteine side-chains into corresponding isothioureia derivatives. The results support the salt-linking hypothesis (cf. A., 1938, III, 436).

L. S. T.

**Enzymic digestion of wool.** J. I. ROUTH and H. B. LEWIS (J. Biol. Chem., 1938, 124, 725—732).—A  $H_2O$ -sol. fraction containing N and S, extracted from wool which had been ground in a ball mill for 75 and 125 hr., was not attacked by trypsin, although the residue and the original powdered wool were readily attacked. Keratins produced by the reducing action of Na thioglycollate solutions on wool were still more extensively digested by pepsin and trypsin than were the proteins of powdered wool (cf. A., 1934, 1238).

T. F. D.

**Bibliography on the chemistry of wool.** A. K. SMITH and M. HARRIS (Amer. Dyestuff Rep., 1938, 27, 363—392 p).—A further 250 references (cf. B., 1938, 767) to de-amination, theories of dyeing, treatment with halogens, felting, fine structure, and fibre swelling are given.

A. J. H.

[Comparison of the] **warmth-retaining properties of various wools.** E. MIDGLEY (Wool Rec. & Text. World, 1938, 51, 1228—1231).—The warmth-retaining powers of six wools (Cape merino, New Zealand crossbreds, Puntas Arenas, Shropshire, and cashmere) in the form of knitted fabrics, the manufacture of which was controlled at all stages from the original wool in top form, were compared (special calorimeter described) by measuring the rate of heat flow through the fabrics while these covered a  $H_2O$ -filled Cu cylinder cooling from 38° to 35°. The results show the heat-retaining powers to be independent of the fibre structure, but dependent on the prevailing climate where the wool is produced. Thus cashmere wool obtained from Tibet (cold) is superior to Cape (hot) wool.

A. J. H.

**Progress in manufacture of synthetic wool from casein [in Italy].** G. GÉNIN (Lait, 1938, 18, 481—484).—The S content is 0.7 as against 3.7% in natural wool. No loss of wt. is met on boiling with

$H_2O$  for 3 hr. and no greater loss is shown on boiling with an alkaline soap solution than is given by merino wool. A mixed artificial wool and regenerated cellulose fibre which has 7% N has been prepared.

W. L. D.

**Microphotometer for analysing X-ray diffraction patterns of raw cotton fibre.** E. E. BERKLEY and O. C. WOODYARD (Ind. Eng. Chem. [Anal.], 1938, 10, 451—455).—Apparatus and technique are described. Preliminary results for cotton fibres are reported, and the application of the procedure to the study of the influence of various factors on the structure of the fibre and its tensile strength is discussed.

E. S. H.

**Utilisation of species of Euphorbia of Italian African colonies.** G. SOLLAZZO (Boll. Chim. farm., 1938, 77, 429—432, 435—436).—Data for the cellulose and lignin contents of the wood and the resin (60%) and sol. gum content (24%) of the milky excretion are given and the commercial utilisation of the latter is discussed.

F. O. H.

**Iodometric determination of oxycellulose.** P. V. MORIGANOV and E. I. VLASIUK (J. Appl. Chem. Russ., 1938, 11, 711—718).—Oxidation of cotton fabrics during bleaching is best determined iodometrically. Additional information is given by determination of CHO and  $CO_2H$  groups in a 1% NaOH extract of the fabric.

R. T.

**Cellulose formate. X. Effect of quantity of sulphuric acid [employed as catalyst] on properties of the product.** Y. UYEDA and S. NAKAMURA (J. Cellulose Inst. Tokyo, 1938, 14, 276—280; cf. B., 1938, 769).—The yield,  $HCO_2H$  content, sp.  $\eta$ , strength, and elongation of cellulose formate film in relation to the quality of the  $HCO_2H$  and the quantity of  $H_2SO_4$  used in its prep. are given in the form of curves and tables.

W. A. R.

**Preparation of cellulose acetate with superior optical properties.** M. S. SVERDLIN (J. Appl. Chem. Russ., 1938, 11, 660—667).—The linters is soaked in AcOH for 20 hr., and then acetylated in 1:9.5  $Ac_2O$ -AcOH. The product should be washed with Fe-free  $H_2O$ . The cellulose acetate so obtained gives solutions in  $COMe_2$  which are highly transparent and practically colourless.

R. T.

**Methods of measurement [for acetate-silk fibres] from the [Dutch] Textile Research Laboratory.** V. E. GONSALVES (Chem. Weekblad, 1938, 35, 606—610).—The following methods employed in the A.K.U. Res. Lab. at Arnheim are described. The Viviani apparatus records continuously differences in the diameter of acetate-silk threads by means of the electrical resistance of a capillary filled with Hg, through which the thread passes. By suitably setting the Wheatstone bridge the apparatus will read directly in deniers. The Schultz apparatus directly records the elasticity curve of a thread on rectangular co-ordinates. The thread is stretched by a wt. operating on a continuously increasing plane and carrying a stylus which records the elongation on a rotating drum. The same apparatus is used for the determination of the breaking strain. The Heim apparatus records extension-load diagrams on short

(~3 cm.) elementary threads and consists of a specially constructed balance fitted with a photographic recording device. The wt. ( $10^{-6}$  g.) of elementary threads is determined with an accuracy of 1% by means of a vibroscopic apparatus measuring the  $\nu$  of vibration of a short thread under a known load.

S. C.

**Stabilisation and stability tests of cellulose nitrates.** E. BERL, G. RUEFF, and C. CARPENTER (Ind. Eng. Chem. [Anal.], 1938, 10, 219—224).—Stabilisation with (a) distilled  $H_2O$  at  $100^\circ$ , (b) 0.2N- $NaHCO_3$  at  $100^\circ$ , (c) 0.1N-HCl at  $100^\circ$ , (d) 0.1N-HCl + 0.02N- $NaHCO_3$  at  $100^\circ$ , (e) 0.1N-HCl + 2% of a wetting agent at  $100^\circ$ , (f) AcOH + NaOAc at  $105^\circ$ , (g) 0.1N- $H_2SO_4$  + MeOH at  $65^\circ$ , (h) 0.1N- $H_2SO_4$  + MeOH at  $101^\circ$ , (i) 0.1N-HCl + MeOH at  $65^\circ$ , (j) 0.1N-HCl + MeOH at  $101^\circ$ , and (k) 0.1N- $H_3PO_4$  + MeOH at  $65^\circ$  reduced the  $SO_3$  content of cellulose nitrate (I) from 0.31—0.68% to 0—0.60%, and raised the ignition point from  $125$ — $156^\circ$  to  $140$ — $190^\circ$ . All the treatments except (e) and (j) lowered the  $\eta$ ; with (e) it remained unchanged and with (j) it was increased. The N content was lowered by treatments (a)—(e) and (h), and increased with (f), (g), (i), (j), and (k). A stability test for (I) is described, in which use is made of a feather manometer. 5—20 mg. of (I) are heated at  $157^\circ$  and the pressures developed are indicated by the deflexion of a needle. The decomp. products remain in contact with (I) during the test. Decomp. curves are given (i) to show the effects of traces of moisture, of washing unstabilised (I) with tap or distilled  $H_2O$ , and of pulping, (ii) for unstabilised (I), washed with tap  $H_2O$ , and EtOH-stabilised (I), (iii) to show the catalytic action of  $NO_2$  in the test, and (iv) to show the effect of removing the decomp. gases. Unstabilised (I) shows an enormous decomp. at the beginning of the test, but this soon slows down. The stabilised material shows a max. of changing pressure after the 32nd hr. (I) that is found to be stable by this test is also found to be stable by the Bergmann-Junk test.

W. J. W.

**Viscosity of solutions of cotton in copper oxide-ammonia.** R. SMIT and J. C. PFEIFFER (Chem. Weekblad, 1938, 35, 590—595).—Provided the solvent has the same composition and  $\eta$  is determined in similar viscosimeters under identical conditions a relationship can be traced between  $\eta$  of 1% cellulose solutions and a loss in strength due to attack by NaOCl in the bleaching process. The lowest tolerable  $\eta$  is 50 centipoises for new bleached cotton and 35 centipoises for mercerised cotton. The bleaching process with NaOCl can be controlled in the laboratory by  $\eta$  measurements and reference to a curve showing loss of strength against  $\eta$ . New unbleached cotton and mercerised cotton must be boiled before being used for  $\eta$  measurements.

S. C.

**Recent developments in the use of viscose rayon in the textile industry.** A. E. DELPH (J. Soc. Dyers and Col., 1938, 54, 366—371).

**Milkiness of viscose rayon.** J. SEIBERLICH (Rayon Text. Month., 1938, 19, 477).—Milky viscose yarns are obtained when under-ripe viscose is too

rapidly coagulated. The X-ray diagrams of milky and normal viscoses differ, on account of structural differences in the regenerated cellulose. Milkiness can be removed from rayon by a swelling treatment, followed by washing and drying.

W. A. R.

Could rayon be tested to express its mechanical characteristics as sold? H. L. BARTHELEMY (Rayon Text. Month., 1938, 19, 425—426).—55% R.H. at  $21.1^\circ$  is recommended as the standard condition for testing rayon yarns.

W. A. R.

**Paper pulp fibres from tropical woods.** W. MÜHLSTEPH (Papier-Fabr., 1938, 36, 341—345, 352—356).—The papermaking properties of a no. of tropical woods are investigated. The woods were cooked with NaOH and the resultant pulps, evaluated by means of beating tests and fibre measurements, are classified roughly into the following four groups: (I) catappa (good strength), the fibres, which are flat (ribbon-like), having a weak structure and being easily destroyed by beating; (II) poplar and eucalyptus (good strength), the  $d$  of the wood being  $>$  that of (I) because the fibres are more rounded, and the fibres, being very elastic, make sheets in the moist state which are therefore very susceptible to pressure; (III) beech and mangrove, which make loose and bulky sheets, the formation and strength of which are considerably improved on beating; the fibres, which are resistant to beating, are cylindrical and hard, and have a high  $d$ ; and (IV) coniferous woods, the sheet structure of which resembles that of (III) and the  $d$  that of (II). The dependence of sheet property on the shape of the fibre is indicated.

D. A. C.

**Paper [pulp] from fruit tree prunings and forest slash.** T. L. CROSSLEY (Pulp and Paper Mag. Canada, 1938, 39, 568—570).—Unbarked and unchipped prunings can be economically converted into satisfactory pulp by the sulphite, sulphate, or soda process.

H. A. H.

**Chemical investigations of wood substances. X. Soda-pulps [from Japanese timbers] and their properties.** K. NISHIDA, R. MIYAMA, Y. YONEZAWA, and H. IMAZATO (J. Cellulose Inst. Tokyo, 1938, 14, 246—253; cf. B., 1938, 633).—The yields of soda-pulp from the 5 Japanese timbers already described (cf. *loc. cit.*) have been determined after treatment under various conditions with dil. NaOH solutions. They had  $\alpha$ -cellulose 82.4—94.2 and ash 0.58—1.71%; the Cl and Cu nos. of the products are also tabulated.

W. A. R.

**Wood pulp. VII. Hydrolysis of lauan wood.** E. HU and T. ITO (J. Cellulose Inst. Tokyo, 1938, 14, 240—241; cf. B., 1938, 770).—Lauan woods when subjected to the sulphite process under identical conditions fall into 3 classes which (a) hydrolyse with difficulty, (b, c) contain 8% and 3% of lignin after treatment, respectively. Analytical data on pulps from 8 samples are presented.

W. A. R.

**Nitric acid process for preparing wood pulp. V. Action of dilute nitric acid on purified cellulose.** I. SHIMODA. VI. Action of dilute nitric acid on sulphite pulp and wood powder. I. SHIMODA and T. DAIMON (J. Cellulose Inst. Tokyo,



1938, 14, 242—243, 244—245; cf. B., 1938, 1023).—V. Cellulose when boiled for 2 hr. with 0.1—10%  $\text{HNO}_3$  is hydrolysed, but oxidised only to a small extent. The high yield of furfuraldehyde from wood pulp prepared by the  $\text{HNO}_3$  process therefore arises from unremoved pentosans, and not from oxycellulose (cf. B., 1936, 267).

VI. Sulphite pulp cannot be purified by treatment with dil.  $\text{HNO}_3$  followed by boiling with alkali. There is no simple relation between Cu no., degree of polymerisation, and pentosan and  $\alpha$ -cellulose contents; an increase in Cu no. does not always imply a diminution in  $\alpha$ -cellulose. W. A. R.

**Influence of nitric acid on wood.** J. SEIBERLICH (Paper Trade J., 1938, 107, TAPPI Sect., 23—24).—Recent literature is briefly reviewed. H. A. H.

**Breaking down of fibrous plant material with polyhydric alcohols.** H. ERBRING and H. GEINITZ (Kolloid-Z., 1938, 84, 215—222).—Straw and similar material can be broken down by treatment with glycol or glycerol for 20 min. at  $160^\circ/2$  atm. The process is accelerated by addition of 0.2% of  $\text{HCO}_2\text{H}$  or  $\text{AcOH}$ . Lignin is dissolved and the swollen residue yields cellulose fibres on rubbing and washing with  $\text{H}_2\text{O}$ . A similar result is obtained in 25 min. by using 50% aq. glycol or glycerol, and a solution of the residual cellulose in  $\text{NH}_3\text{-CuSO}_4$  has a higher  $\eta$  than when the undiluted solvents are used. Yields and ash content of the products from different materials are given. F. L. U.

**Grindstones for brown mechanical pulp.** G. NOTRES (Papier-Fabr., 1938, 36, 370—372).—The excessive wear in artificial stones which occurs in producing brown mechanical pulp is ascribed to the formation, by the action of the steam on the wood, of  $\text{HCO}_2\text{H}$ , which decomposes the binding agents in the stone. The corrosive action practically does not occur where hard  $\text{H}_2\text{O}$  is used for spraying the stone face, and if soft  $\text{H}_2\text{O}$  only is available it can be avoided by the use of  $\text{NaOH}$ . The sensitivity of the stone to  $\text{HCO}_2\text{H}$  can be determined by shaking a powdered sample with the acid and determining the excess of  $\text{HCO}_2\text{H}$  by back-titration. Natural stones or artificial stones with ceramic (fired) binders are unaffected by  $\text{HCO}_2\text{H}$ . D. A. C.

**NAF circulation apparatus for sulphite- and sulphate-pulp digesters.** T. SAMSON (Paper Trade J., 1938, 107, TAPPI Sect., 41—48).—This Swedish system of circulating the liquor during cooking, particularly by the sulphite process, is compared with other methods. It is believed that efficient circulation is most important at the end of the heating period. H. A. H.

**Relation between chip length and uniformity of delignification in sulphite pulping [of wood].** H. MORGAN and H. P. DIXSON, jun. (Paper Trade J., 1938, 107, TAPPI Sect., 72—74).—Variations in the length and thickness of individual wood chips tend to produce non-uniform pulp. This lack of uniformity can be overcome to some extent by prolonging the time of the cooking schedule, but it is much more effective to employ high initial pressure conditions

rather than merely to allow the pressure to rise as cooking proceeds. H. A. H.

**Comparison of sulphite pulps from fast-growth loblolly, shortleaf, longleaf, and slash pines.** G. H. CHIDESTER, J. N. MCGOVERN, and G. C. MCNAUGHTON (Paper Trade J., 1938, 107, TAPPI Sect., 32—35).—With these woods springwood growth gives higher pulp quality, and segregation of growth types is advocated when possible. H. A. H.

**Recausticising and lime recovery [in sulphate-pulp mill] at Bogalusa.** J. P. RUBUSH (Paper Trade J., 1938, 107, TAPPI Sect., 35—36).—Features of the continuous recovery equipment at this mill are briefly described. H. A. H.

**Use of time-cycle chart in [wood-pulp bleaching] plant design.** W. L. SAVELL (Paper Trade J., 1938, 107, TAPPI Sect., 60—62).—The val. of such charts in producing satisfactory flow sheets is demonstrated. H. A. H.

**Factors influencing drainage through Fourdrinier [paper-machine] wires.** H. G. SPECHT and C. M. CONNOR (Paper Trade J., 1938, 107, TAPPI Sect., 48—50).—The effect of  $p_H$  [as brought about by both  $\text{Al}_2(\text{SO}_4)_3$  and  $\text{H}_2\text{SO}_4$ ] on drainage rate, employing different stock and other conditions, is discussed. H. A. H.

**High-speed newsprint production.** A. J. EDWARD (Pulp and Paper Mag. Canada, 1938, 39, 559—562, 567).—The significance of a no. of machine variables is discussed. H. A. H.

**Lithographic paper.** R. F. REED (Paper Trade J., 1938, 107, TAPPI Sect., 25—28).—Chemical woodpulp paper intended for multicolour offset lithography should receive a min. of beating and refining, and a fairly large directional difference in formation is desirable. In avoiding register troubles during printing, temp. control is as important as R.H. control, and the paper should contain 0.5—1.0% more moisture than that required for equilibrium at any given R.H. H. A. H.

**Ink-feathering test for writing papers.** A. NOLL (Papier-Fabr., 1938, 36, 349—352).—A method which is said to fulfil all conditions necessary for the standardisation of this test is described. A specially constructed tracer pen (illustrated), enabling the ink to be deposited under conditions of const. line thickness and pressure, is used. The ink consists of a mixture of 10 g. of Trypan-blue (Merck), 10 g. of glycerin ( $d$  1.27), and 1 g. of  $\text{PhOH}$  crystals in 1 l. of  $\text{H}_2\text{O}$ . D. A. C.

**Biological methods in paper diagnosis.** H. N. LEE (Paper Trade J., 1938, 107, TAPPI Sect., 53—59).—Microscopical methods of identifying fibres, starch, rosin, glue, inks, etc. are described. H. A. H.

**Influence of moisture on the d.-c. conductivity of impregnated paper.** D. A. MCLEAN and G. T. KOHMAN (J. Franklin Inst., 1938, 226, 203—220).—For moisture contents of (a) <1%, (b) 1—3%, and (c) >3%, a decrease in apparent conductivity with time was found for (a) and (c) and an increase for (b). This behaviour, and an increase in current with voltage

in all cases, are best explained by Murphy and Lowry's theory (A., 1930, 685) of dielectric adsorption. High conductivity may produce thermal effects.

K. W. P.

Filter-cloths.—See I. Determining cellulose in lignite.—See II. Dyeing cellulose esters.—See VI. Micro-mechanical testing of textiles.—See X. Fluorescence analysis.—See XI. Pptn. of blanc fixe.—See XIII. Butter-box liners.—See XIX. Air for viscose factories. Corrosion control at rayon plant. Textile waste disposal.—See XXIII.

See also A., I, 451, Sorption of vapours by cellulose and its derivatives. 476, Determining birefringence of fibres.

#### PATENTS.

Manufacture of textiles [pile fabrics]. A. W. DROBILE and G. S. HIERS, Assrs. to COLLINS & AIRMAN CORP. (U.S.P. 2,065,853, 29.12.36. Appl., 21.3.34).—Yarn for the backing material of a pile fabric is treated with a binder, e.g., rubber, cellulose derivative, synthetic resin, or bitumen. After weaving, the fabric is rendered tacky by heat, solvents, etc. so that the pile tufts are secured, tackiness being finally removed by application of suitable reagents. The binder may also be made in thread form and incorporated with the yarn. E.g., the yarn is treated with rubber (as latex or solution and containing vulcanising agents) and, after weaving, is heat- or solvent-treated; the rubber can then be vulcanised, e.g., by  $S_2Cl_2$ . Another suitable reagent is a  $H_2O$ -sol. cellulose H phthalate derivative which is softened by  $H_2O$  and subsequently rendered insol. by AcOH.

R. J. W. R.

Treatment of textiles and oils therefor. LISTER & Co., LTD., and W. GARNER (B.P. 487,949, 30.10.36).—Esters of monohydric alcohols, preferably of  $\leq C_6$  (e.g., lauryl alcohol or methylcyclohexanol), or di- or tri-glycols, e.g.,  $(OH \cdot C_2H_4)_2O$ , with polymerised and/or oxidised unsaturated fatty acids of  $\leq C_8$ , esters of di- or tri-glycols with saturated or unsaturated fatty acids, or polymerised and/or oxidised (e.g., by heat or air-blowing) esters of monohydric alcohols and unsaturated fatty acids are used for oiling textile materials containing animal fibres. Diluents may also be present, the  $\eta$  of the lubricant being 200—650 sec. Redwood at  $60^\circ$ . The oils are easily removed on scouring and are resistant to oxidation.

R. J. W. R.

Felting and fulling of textile materials. I. G. FARBENIND. A.-G. (B.P. 476,172, 2.6.36. Ger., 1.6.35).—The materials are placed in a perforated container which is immersed in the treatment liquor and subjected to a rapid oscillatory movement having a frequency of a few hundred to thousands per min. and an amplitude up to a few mm. Apparatus is claimed.

R. J. W. R.

Centrifugal spinning of fibrous material. S. E. BERTHELSEN (B.P. 477,259, 5.6.37).—Fibres, e.g., jute, cotton, wool, are fed to a rotating, bell-shaped, centrifugal drum by bringing the end of a sliver or rove in contact with the wall of the inner drum, whereby they are separated and settle around this

wall. They are then withdrawn along the axis of rotation of the drum in the form of spun yarn. Apparatus is claimed.

F. R. E.

Production of cellulose. H. A. KNOPF (B.P. 488,362, 10.2.38. Addn. to B.P. 468,669; B., 1938, 148).—The quantity of  $Ca(OH)_2$  recommended in the chief patent may be considerably reduced if the  $Ca(OH)_2$  treatment is followed by thorough washing of the digester material, with simultaneous mechanical separation of the fibres in a hollander. This process is particularly advantageous for treating silicate-rich varieties of straw, grasses, bamboo, etc. H. J. E.

Obtaining and treating cellulose. CELLULOSE RES. CORP. (B.P. 478,943—4, 23.4.36. U.S., [A] 24.4.35, [B] 4.9.35).—(A) In the production of pulp by both acid and alkaline methods, the wood chips are impregnated with the liquor used for fibre liberation under conditions which will remove all air from within the chips, but without at the same time producing delignification. This may be done by boiling in the digester under slight pressure with intermittent short gas reliefs, after which the steam is condensed by slightly raising the pressure. The liquor is thus drawn into the interior of the chips to give uniform impregnation. Digestion is then proceeded with. If NaOH is used as the cooking liquor the chips may first be impregnated by boiling at 1 atm. with  $H_2O$  or a dil. aq. electrolyte (e.g., 10—2M-NaCl). Deposition of the positive ions of the electrolyte in the capillary spaces of the chips is stated to aid liquor penetration during the cook. (B) Pulp thus produced is immediately subjected to controlled chlorination with subsequent  $OCl'$  bleaching and NaOH purification steps; it is thus maintained in a swollen condition from the commencement of pulping, in which condition it can be rapidly acetylated. Thus the pulp after the NaOH purification is washed, conc., impregnated with glacial AcOH and  $H_2SO_4$  for  $\geq 30$  min., and treated for 2 hr. with  $\geq 2.2$  pts. of  $Ac_2O$  at  $\geq 45^\circ$ .

D. A. C.

Activation of cellulose for conversion into cellulose derivatives. G. A. RICHTER and J. W. MCKINNEY, Assrs. to BROWN Co. (U.S.P. 2,064,384, 15.12.36. Appl., 11.10.33).—Cellulose which has been refined in an alkaline mercerising solution is treated with aq. glycerin (10—15%) and dehydrated before acetylating.

F. R. E.

Decomposition of cellulosic materials with the aid of hydrogen fluoride to yield products that are of low mol. wt. up to the point of solubility in water. J. BOHUNEK, H. HOCK, and G. MAYRHOFER (B.P. 476,191, 25.7.36. Austr., 13.8.35 and 6.3.36).—Dry cellulosic material is placed in a vessel which is then exhausted to remove air therefrom and also to a large extent from the cells of the cellulosic material. A small quantity (10 wt.-% of the cellulose) of conc. HF is introduced in absence of diluting gas, and the reaction with gaseous HF is allowed to proceed *in vacuo* and under alternate compression and dilution of the gas, brought about by cooling and heating. When interaction is complete, the mass is comminuted and the HF drawn off. If desired, products prepared with small quantities of HF which are substantially insol. in  $H_2O$  are converted into

H<sub>2</sub>O-sol. products by after-treatment with dil. mineral acid at elevated temp. F. R. E.

**Production of cellulose esters.** "AFAG" FINANZIERUNGS A.-G. (B.P. 488,707, 12.10.36. Fr., 10.10.35).—Cellulose esters lower than the tri-ester are prepared by first converting cellulose (which may have been pretreated with a swelling agent) into an intermediate mixed ester (I) with a controlled content of SO<sub>4</sub> by adjusting the proportions of esterifying agent, sulphating agent, and also the solvent power of the diluent preventing dissolution of the ester; the SO<sub>4</sub> radicals are removed by means of agents of B.P. 411,260 and 468,880 (B., 1934, 714; 1938, 150) which do not contain unesterified CO<sub>2</sub>H and/or OH. Sp. claim is made to the production of cellulose acetate containing <61% OAc by using Ac<sub>2</sub>O and H<sub>2</sub>SO<sub>4</sub> 30 : 1 for the esterification and at least one liquid hydrocarbon as diluent. R. G.

**Manufacture of cellulose esters of lower aliphatic acids.** A. J. WATTERS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 489,110, 19.1.37).—Cellulose esters of aliphatic acids (>C<sub>4</sub>) which have been prepared by acylating in presence of H<sub>2</sub>SO<sub>4</sub> are stabilised by freeing from H<sub>2</sub>SO<sub>4</sub> either before or after pptn. (but after hydrolysing excess of acylating agent) in substantial absence of H<sub>2</sub>O by heating (e.g., in C<sub>6</sub>H<sub>6</sub>) with a minor proportion of an aliphatic acyl halide or easily hydrolysed halogenated lower fatty acid (<2 mols./mol. of H<sub>2</sub>SO<sub>4</sub>). The stabilisation of esters of AcOH and EtCO<sub>2</sub>H with AcCl, CH<sub>2</sub>Cl·CO<sub>2</sub>H, and CH<sub>2</sub>Cl·COCl is described. A. H. C.

**Manufacture of [fibrous] highly acetylated cellulose.** L. S. E. ELLIS. From P. KOETSCHET (B.P. 478,716, 23.7.36).—After pretreatment, if desired, in absence of mineral acid, e.g., with AcOH, cellulose is esterified with Ac<sub>2</sub>O, AcOH, an anti-solvent (Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>), and a catalyst consisting of a mixture of HClO<sub>4</sub> with <5% of H<sub>2</sub>SO<sub>4</sub> and no metallic salt. F. R. E.

**Fibrous esterification of cellulose employing homogenisers.** L. M. MINSK, W. O. KENYON, and H. LE B. GRAY, Assrs. to EASTMAN KODAK CO. (U.S.P. 2,066,571, 5.1.37. Appl., 19.5.33).—Cellulose is esterified with a lower fatty acid anhydride [Ac<sub>2</sub>O, (EtCO)<sub>2</sub>O] and a catalyst, in presence of a saturated hydrocarbon of b.p. 150—210° (petroleum distillate, cyclohexane) as non-solvent, and a lower fatty acid (AcOH) to ensure compatibility of the ingredients of the esterification bath without dissolution of the resulting ester. The prep. of cellulose acetate-propionate is claimed. F. R. E.

**Manufacture of cellulose derivative compositions.** (A, B) DISTILLERS CO., LTD., H. A. AUDEN, and H. P. STAUDINGER, and (A) H. M. HUTCHINSON (B.P. 477,036 and 477,327, 18.5.36).—(A) Acetals (<C<sub>7</sub>) obtained by condensing an aldehyde, e.g., CH<sub>2</sub>O, with a monohydric alcohol, higher aliphatic, hydroaromatic, e.g., cyclohexanol, or aromatic, e.g., CH<sub>2</sub>Ph-OH, either unsubstituted or having no O-containing substituents, and particularly halogen-substituted monohydric alcohols, are incorporated with org. esters of cellulose as solvents, softeners, or plasticisers. (B) An org. ester of cellulose is dissolved

in a solvent, together with an ester of b.p. <185° formed from crotonic acid (I) or halogenated (I) and a monohydric alcohol (<C<sub>5</sub>), e.g., C<sub>5</sub>H<sub>11</sub>·OH, cyclohexanol, CH<sub>2</sub>Ph-OH, or a dihydric alcohol, e.g., simple or polyglycols or their monoalkyl derivatives, as plasticiser or softener. F. R. E.

**Saponification of cellulose esters.** SOC. RHODI-ACETA (B.P. 476,989, 10.5.37. Fr., 29.5.36).—Fabrics composed of cellulose esters interwoven, if desired, with other fibres (animal, vegetable, or regenerated cellulose) are treated with aq. Na<sub>2</sub>CO<sub>3</sub> and H<sub>3</sub>BO<sub>3</sub> (3—7 wt.-% of the cellulose) at <40°, and hydrolysis of the esters is continued at 80° to the desired extent. F. R. E.

**Preparation of cellulose suspensions and of cellulose xanthate solution.** BROWN CO. (B.P. 475,925, 10.12.36. U.S., 10.1.36).—Cellulose fibre pulpboard [thickness 0.03—0.06 in., compactness (as defined) 50—120] is cut into chips (area >1 sq. in.) which are softened by soaking in aq. NaOH (15—18% solution). After addition of H<sub>2</sub>O, the chips are disintegrated and the fibre suspension free from aggregates is treated with CS<sub>2</sub> to form cellulose xanthate solution containing 6—10% each of cellulose and NaOH. F. R. E.

**Xanthation of cellulose.** L. MELLERSH-JACKSON. From BROWN CO. (B.P. 488,761, 10.8.37).—Viscose syrup is prepared by mixing liquid CS<sub>2</sub> with a suspension of cellulose (I) in aq. NaOH, preferably in such proportions as to contain 6—10% of both (I) and NaOH, in presence of an org. liquid (II) miscible with both CS<sub>2</sub> and aq. NaOH and containing ·CHO, ·CO, or ·OH; e.g., COMe<sub>2</sub> (5—20 wt.-% in CS<sub>2</sub>) may be used. (II) may be removed from the xanthated product by distillation, if desired under reduced pressure. R. G.

**Production of viscose.** L. MELLERSH-JACKSON. From BROWN CO. (B.P. 488,768, 7.9.37).—A fluent, aq. pulp suspension of substantially individualised cellulose fibre units containing about 2—4% of fibre, such as is obtained in chemical wood pulp or pulp-refining mills, is thickened, e.g., by centrifuging, to a much higher fibre content (20—40%) while maintaining the fibre thoroughly wet; the thickened wet fibre is rediluted with aq. NaOH to yield a suspension containing about 6—15% of both cellulose and NaOH. Liquid CS<sub>2</sub> is then added and reacts readily with the fibres to yield viscose. A solvent maybe added as in B.P. 488,761 (preceding abstract). R. G.

**Preparation of cellulosic materials [having improved affinity for direct dyes].** R. PICARD and R. FAYS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,064,355—6, 15.12.36. Appl., 17.7.35).—Viscose is mixed with 0.25—5% of (A) Na<sub>3</sub>SbS<sub>4</sub>, (B) Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>, before spinning. F. R. E.

**Preparation of cellulose ethers.** DOW CHEM. CO. (B.P. 475,466, 1.3.37. U.S., 6.3.36).—The reaction mixture obtained by treating alkali-cellulose with an etherifying agent at elevated temp. and pressure is discharged into H<sub>2</sub>O boiling at superatm. pressure, whereby the cellulose ether (I) is pptd.

Pressure is then released to remove volatile materials, and the (I) separated from the aq. solution.

F. R. E.

**Manufacture of cellulose derivatives [ethers].** H. DREYFUS (B.P. 478,259, 16.7.36 and 3.2.37).—Cellulosic materials, *e.g.*, cotton or regenerated cellulose, or cellulose derivatives, *e.g.*, acetate, which contain free OH groups or yield them under the conditions of the reaction, and are preferably in yarn or foil form, are treated with the usual etherifying agents in presence of *tert.* alicyclic org. bases, *e.g.*, dimethylaminocyclohexane (I), in a substantially anhyd. medium. Thus, *e.g.*, regenerated cellulose is autoclaved in a mixture of  $\text{CH}_2\text{Cl}\cdot\text{CH}_2\cdot\text{OH}$  and (I) for 4–10 hr. at 20–30°.

F. R. E.

**Production of alkali-cellulose or other cellulose derivatives, *e.g.*, cellulose ethers.** E. I. DU PONT DE NEMOURS & Co., and F. C. HAHN (B.P. 478,255, 14.7.36).—Alkali-cellulose of predetermined cellulose, alkali, and  $\text{H}_2\text{O}$  contents is produced by emulsifying a solution of alkali in  $\text{H}_2\text{O}$  in the required proportions with an inert liquid, *e.g.*,  $\text{C}_6\text{H}_6$ , and adding to the cellulose. The product is then treated with an etherifying agent in presence of a wetting agent.

F. R. E.

**Ripening of alkali-cellulose.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 488,793, 13.1.37).—Alkali-cellulose obtained in the shredding process in the manufacture of viscose is fed continuously into a tube rotating about its longitudinal axis which is slightly inclined to the horizontal, < half the cross-section of the tube being filled, and the ripening temp., *e.g.*, about 32°, being maintained by a jacket containing a suitable tempering medium. The ripened product is then cooled to  $\approx$  room temp., the difference in temp. between the product and the inner wall of the cooling jacket being kept at  $\approx$  6°.

R. G.

**Manufacture of a cotton fibre-like material from straw.** S. TANAKA (U.S.P. 2,065,877, 29.12.36. Appl., 10.9.35. Jap., 3.12.34).—Straw is digested with NaOH, washed, and filtered off. It is then heated for about 2 hr. at 40–50° with "Nibé" (extract of *Hydrangea paniculata* siet) and myrrh in alkaline solution, and treated with rongalite, when the pulp is gelatinised. The pulp is neutralised, bleached, and treated with  $(\text{NH}_4)_2\text{CO}_3$  to swell and open up the fibres. The resulting product may be used for medical purposes or guncotton.

D. A. C.

**Manufacture of artificial filaments, yarns, and the like.** H. DREYFUS (B.P. 477,529, 1.7.36).—A solution of a film-forming base, *e.g.*, an org. derivative of cellulose, is extruded through a shaping device having stream-lined contours into a coagulating medium moving in the same direction and with substantially the same velocity as the formed filaments through a vessel, which is provided with one or more sheets of gauze extending across the vessel and substantially normal to the direction of flow of the medium, to prevent turbulent flow. Apparatus is claimed.

F. R. E.

**Manufacture of artificial silk.** H. L. BARTHELEMY, Assr. to TUBIZE CHÂTILLON CORP. (U.S.P. 2,066,385, 5.1.37. Appl., 13.8.35).—A mixture of

white mineral oil with a  $\text{H}_2\text{O}$ -insol. alcohol, *e.g.*,  $\text{C}_{18}\text{H}_{35}\cdot\text{OH}$ ,  $\text{C}_{16}\text{H}_{33}\cdot\text{OH}$ ,  $\text{C}_{27}\text{H}_{55}\cdot\text{OH}$ , olefine or diolefine alcohols, or sterols, is incorporated in the spinning solution in such proportion that the resulting filaments exhibit reduced lustre and enhanced suppleness.

F. R. E.

(A) Artificial materials. (B) Manufacture of yarns and filaments. C. DREYFUS (U.S.P. 2,066,039–40, 5.1.37. Appl., [A] 17.6.32, [B] 2.5.34).—A solution of an org. derivative of cellulose containing a dispersion of finely-divided (A) starch, (B) chlorinated starch, is formed by wet- or dry-spinning processes into filaments, which are then treated with hot aq. liquor to reduce their lustre.

F. R. E.

**Manufacture of artificial threads, filaments, and the like.** COURTAULDS, LTD., H. J. HEGAN, and A. D. HEYWOOD (B.P. 488,619, 8.1. and 3.12.37).—The thread, after leaving the hot bath in which it has been stretched, is cooled, by a  $\text{H}_2\text{O}$ -bath at <30°, by rollers at <30°, or by passing through a cold atm., before collection.

H. J. E.

**Treatment of artificial filaments for production of staple fibre.** C. HAMEL A.-G. (B.P. 476,939, 17.6.36. Ger., 9.3.36. Addn. to B.P. 457,328; B., 1937, 128).—A device is claimed for conveying the filament bundles from one bath to the next and causing them to oscillate, whereby the action of the liquids thereon described in the prior patent is intensified.

F. R. E.

**Manufacture of artificial threads, fibres, and like materials from cellulose solutions.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 475,350, 16.4.36).—The freshly-spun filaments are laid continuously on a flat support rotating about a vertical or slightly inclined axis in a form which fills the surface of the support substantially completely, *e.g.*, in zig-zag form, where they are after-treated in various ways and then continuously removed before they can form a second layer.

F. R. E.

**Apparatus for treatment of artificial threads.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 478,513, 17.7.36).—Apparatus is claimed for mounting the spun cakes, without handling, wrapping, or modification of the original prepared form, in a stiffened hollow column preparatory to after-treatment, centrifuging, drying, and winding-off.

F. R. E.

**Manufacture of artificial filaments, yarns, films, and similar materials.** BRIT. CELANESE, LTD. (B.P. 478,937, 4.6.37. U.S., 6.6.36).—The spinning solution consists of cellulose acetate (56–59% of OAc calc. as AcOH) dissolved in a solvent medium (<80% of  $\text{COMe}_2$ ) containing  $\approx$  5 (1–2%) wt.-% of  $\text{H}_2\text{O}$ , together with any desired effect materials (dyes etc.). The dry-spun materials show improved tensile strength and resistance to delustring, and the spinning solution is less corrosive than are the ordinary solutions.

F. R. E.

**Manufacture of artificial filaments and the like.** H. DREYFUS (B.P. 488,500, 7.1.37).—Hollow filaments are prepared by extruding a solution of, *e.g.*, cellulose acetate vertically downwards in a preheated countercurrent of gas with an auxiliary gas stream directed towards a point near the jet.

Conditions of evaporation are such that the issuing gas contains  $\leq 1\%$  of solvent vapour, while the gas stream introduced near the jet is  $\leq 40\%$  of the total evaporative gas stream passing through the cell.

H. J. E.

**Manufacture of threads and films made from cellulose acetate.** L. S. E. ELLIS. From P. EHRENSTEIN (B.P. 475,368, 4.6.36).—Threads or films of cellulose acetate swollen with  $\text{COMe}_2$  as they emerge from the spinneret are stretched in aq.  $\text{COMe}_2$  ( $\leq 30\%$ ) at  $\leq 35^\circ$  ( $50$ – $65^\circ$ ) and wound at a speed of  $\leq 100$  m./min. The product possesses an axially regular cryst. structure visible under X-rays.

F. R. E.

**Manufacture of artificial filaments, foils, and like materials.** R. W. MONCRIEFF and F. B. HILL (B.P. 479,050, 30.7.36).—A modification of the process described in B.P. 443,773 (B., 1936, 539) is claimed in which the materials are run from the outer air into an end chamber containing  $\text{H}_2\text{O}$  at a temp.  $<$  that at which it exerts a softening action on the materials ( $< 35^\circ$ ), and thence through a pressure chamber which contains  $\text{H}_2\text{O}$  at  $> 100^\circ$  and at a pressure substantially the same as that in the end chamber. After stretching in the pressure chamber, the materials finally pass again into the outer air.

F. R. E.

**Treatment of cellulose derivative filaments, yarns, and the like.** R. W. MONCRIEFF and F. B. HILL (B.P. 488,794, 13.1.37. Cf. B.P. 443,707; B., 1936, 539).—The materials (especially cellulose acetate) are passed through  $\text{H}_2\text{O}$  at  $> 30^\circ$  under pressure in an end pressure chamber in substantially non-slipping contact with a positively-driven feed device contained therein, and then through wet steam at  $\leq 100^\circ$  ( $\leq 130^\circ$ ) in a steam chamber in which they are stretched. The difference in pressure between the  $\text{H}_2\text{O}$  and steam is preferably  $> 5$  lb./sq. in.

R. G.

**Manufacture of [decorative] films, foils, coatings, and the like.** BRIT. CELANESE, LTD., J. H. ROONEY, and P. R. HAWTIN (B.P. 476,649, 12.6.36).—A solution of a film-forming base, e.g., cellulose acetate (I), in a volatile solvent is extruded into an evaporative atm. on a suitable surface pretreated with a powder compatible with the base, e.g., (I) plasticised with  $o\text{-C}_6\text{H}_4(\text{CO}_2\text{Et})_2$  or natural or artificial resins, the particles of which act as nuclei for the formation of numerous small bubbles in the product during evaporation of the solvent.

F. R. E.

**Production of bottle caps and the like from cellulose esters.** DISTILLERS Co., LTD., H. A. AUDEN, and H. P. STAUDINGER (B.P. 477,402–3, 25.6.36. Addn. to B.P. 456,973; B., 1937, 128).—A solution of a cellulose ester (acetate) in (A) an aprotic, e.g., ethylene glycol formal, or (B) a chlorohydrin, e.g.,  $\text{CH}_2\text{Cl}\cdot\text{CH}_2\cdot\text{OH}$ , is shaped and coagulated, and the solidified cap removed from the former. (A, B) pigment, filler, dye, or plasticiser may be incorporated in the solution.

F. R. E.

**Manufacture of artificial filaments, yarns, foils, and similar materials.** BRIT. CELANESE, LTD., R. W. MONCRIEFF, and F. B. HILL (B.P. 475,650, 22.5.36).—Materials composed of an org. derivative of

cellulose, which, if desired, have been stretched  $> 200\%$  subsequent to their production, are treated with hot  $\text{H}_2\text{O}$  or wet steam containing a low concn. of swelling agent at  $> 100^\circ$  under pressure but in absence of stretching tension. The product shows enhanced extensibility.

F. R. E.

**Spinning of viscose artificial silk.** N. V. ONDERZOEKINGSINST. RES. (B.P. 478,774, 29.1.37. Ger., 31.1.36. Addn. to B.P. 469,817; B., 1938, 151).—The added org. substance (0.05 wt.-% on viscose) possessing surface-active properties consists of a N, sulphonium, arsonium, or phosphonium base or salt thereof, e.g.,  $\text{NMe}_3\text{RI}$ , where R is toluene- or benzene-azophenyl or  $\text{C}_{10}\text{H}_8\text{Pr}^2$ .

F. R. E.

**Manufacture of artificial threads [containing nitrogen and sulphur].** I. G. FARBENIND. A.-G. (B.P. 476,431, 8.6.36. Ger., 8.6.35 and 4.3.36).—Viscose is spun into a bath containing 0–100% of  $\text{H}_2\text{SO}_4$  or an equiv. acid containing, if desired, salts or other substances commonly used in spinning baths, and the freshly-formed threads still containing xanthate groups are treated with ethyleneimine, a homologue thereof, or an analogously-acting cyclic N base or their salts in the gaseous, liquid, or dissolved state. The products may be dyed with wool dyes.

F. R. E.

**Production of [delustred] rayon.** C. A. HUTTINGER and E. R. TIMLOWSKI, ASSRS. TO ACME RAYON CORP. (U.S.P. 2,064,118, 15.12.36. Appl., 16.10.35).—Cellulose xanthate is dissolved in aq. NaOH containing  $\text{Na}_2\text{SO}_3$  (0.25 wt.-% of the cellulose), and the spun product, after removal of acid and salt, is treated with aq.  $\text{Na}_2\text{SO}_3$  and  $\text{H}_2\text{C}_2\text{O}_4$  (2% solution).

F. R. E.

**Production from whale skins of swollen fibrous masses suitable for preparation of threads, moulded articles, or artificial leather.** W., H., O., and R. FREUDENBERG (B.P. 488,596, 9.7.37. Ger., 18.7.36).—The grain layer of the skin is removed by soaking in aq. NaOH or alkaline sulphide and scraping. The pieces of skin are pressed in a flat-press to remove about 80% of the blubber without damaging the fibres, and the remainder of the blubber may be removed with a solvent (e.g.,  $\text{C}_2\text{H}_4\text{Cl}_2$ ). The skin is then treated with a swelling agent (e.g., CaO, NaOH, HCl, or lactic acid) until it contains 75–95% of non-expressible swelling  $\text{H}_2\text{O}$ , and then comminuted, shredded, and extruded. Alternatively, the swollen material may be crushed to a thin fleece, the fibres neutralised with HCl, washed with  $\text{H}_2\text{O}$ , and the  $\text{H}_2\text{O}$  removed with a  $\text{COMe}_2$ -benzene mixture. The resulting wool-like product is rendered  $\text{H}_2\text{O}$ -repellent by chrome tanning.

H. J. E.

**Decortication of fibrous plants.** A. T. RATLIFF and R. R. GUICE (B.P. 476,569, 5.6.36).—Light and bulky fibrous plants (e.g., maize stalks, pine needles, etc.) are digested under pressure with  $\text{H}_2\text{O}$  in a direct steam-heated digester. They are then explosively disintegrated by rapidly discharging into a second and larger digester, in which fibre liberation is completed by cooking with  $\text{H}_2\text{O}$  or any suitable reagents. Direct steam heating is also used in the second digester.

D. A. C.

**Fractured wood-fibre product.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 2,065,395, 22.12.36. Appl., 27.8.35).—A pulp is claimed which is chemically reactive and has a solution  $\eta$  of  $>2$  c.g.s. (in standard cuprammonium solution). It is produced from screened sawdust by any digestion method, and may be used after suitable bleaching or alkali-refining treatment for esterification. D. A. C.

**Chemical pulping process.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 2,065,396, 22.12.36. Appl., 30.3.36).—Spruce wood, as chips or sawdust, is treated with  $\text{SO}_2$  at  $80^\circ$  and 100% R.H. for  $<1$  hr. The  $\text{SO}_2$  is then expelled, *e.g.*, by hot air, and the wood subjected to an alkaline cook. The product is a pulp of high strength and  $\alpha$ -cellulose content. D. A. C.

**Production and utilisation of fibrous cellulose from plant stalks.** G. A. LOWRY and J. A. GRANT, Assrs. to M. H. ITTNER (U.S.P. 2,064,929, 22.12.36. Appl., 22.6.34).—Flax straw is mechanically scutched to remove about 80% of its non-cellulosic constituents. It is then submerged in  $\text{H}_2\text{O}$  and treated at just  $<100^\circ$ . The  $\text{H}_2\text{O}$  is rapidly removed, and the flax digested in absence of air with 10% aq.  $\text{NaOH}$  at  $160$ – $180^\circ/140$  lb./sq. in. for 3 hr. Digestion takes place in a closed, indirect steam-heated tank, provided with means for liquor circulation. The flax is finally washed, when it may be used for papermaking, or spun on cotton or woollen machinery. D. A. C.

**Apparatus for continuous treatment of wood pulp and the like with chemicals.** J. C. F. C. RICHTER and K. W. ROSÉN, Assrs. to ARTIEB. KAMYR (U.S.P. 2,078,810, 27.4.37. Appl., 20.2.35. Norw., 12.1.34).—A vertical cylindrical tank is provided in the lower part with a cylindro-conical baffle and from the annular space surrounding the baffle a portion of the pulp is continuously withdrawn by the suction of a pump. At this point new material and  $\text{Cl}_2$  or other treating agent are added, the combined stream being reinjected tangentially into the tank at an intermediate level. In the upper part of the tank is a similar inverted baffle, and a tangential stream of dilution  $\text{H}_2\text{O}$  is introduced and diluted finished pulp overflowed. B. M. V.

**Apparatus for bleaching pulp.** M. F. McCOMBS and F. W. DECKER, Assrs. to NIAGARA ALKALI Co. (U.S.P. 2,065,387, 22.12.36. Appl., 5.9.35).—The apparatus consists of an outer, cylindro-conically-roofed vessel with a similarly shaped, inner, concentric vessel. The pulp, thoroughly mixed with  $\text{Cl}_2$ , is fed in against the roof of the inner vessel through a vertical pipe, and travels downward through the vessel and upward through the space between the walls of the two vessels. It is withdrawn at the top of the outer vessel through a pipe into which aq.  $\text{Ca}(\text{OH})_2$  is injected. D. A. C.

**Production of pulp from wood.** A. E. NIELSEN (B.P. 477,543, 2.7.36).—Sulphite waste liquor is conc. by evaporation and its concn. of active reagents restored to pre-cooking conditions by addition of  $\text{CaO}$  and  $\text{SO}_2$ , when the liquor is used for further digestion of wood chips. This cycle is repeated until the concn. of org. matter in the waste liquor is sufficiently high ( $d_{40}^{20}$  1.21) for recovering the dissolved

salts, when a small portion of the liquor is tapped off for recovery and the remainder returned for concn. D. A. C.

**Non-cellulosic material obtained from spent digestion liquor.** MEAD CORP., Asses. of N. N. T. SAMAROS and M. PLUNGUAN (B.P. 476,344, 19.6.36. U.S., 29.6.35).—Conc. black liquor (*e.g.*, from the soda-cooking process) is mixed with  $\text{H}_2\text{O}$  and treated with  $\text{CO}_2$  until the  $p_{\text{H}}$  is reduced to 8.0–8.3. It is then warmed (to about  $60^\circ$ ) to coagulate the lignin, cooled with agitation, and the lignin filtered off and washed. The lignin is freed from org. Na compounds, and particularly sulphonic acid derivatives, by washing with 2–5% aq. solution of a strong acid, and is finally washed with soft  $\text{H}_2\text{O}$ . An ash-free lignin is obtained having m.p.  $185$ – $230^\circ$ , which, however, may be lowered by addition of  $\text{H}_2\text{O}$  and/or furfuraldehyde. The lignin may be used as a binder in moulding compositions. D. A. C.

**Manufacture of paper.** CHAMPION PAPER & FIBRE Co. (B.P. 478,804, 30.7.37. Addn. to B.P. 403,852; B., 1934, 191).—Loading is applied to the under-side of the paper web within the press section of the Fourdrinier machine. Excess of filler is wiped off by passing the web over a scraper bar and the paper is then dried as usual. Sufficient loading is intended thus to be left on the web to fill in surface depressions due to wire mark etc. D. A. C.

**Coating and ornamenting paper and the like.** A. M. OVERTON and H. C. FISHER, Assrs. to RICHARDSON Co. (U.S.P. 2,065,925, 29.12.36. Appl., 9.3.32).—A mixture of rosin, a gum, linseed oil, and kerosene is emulsified with  $\text{N}(\text{C}_2\text{H}_4\cdot\text{OH})_3$ , mixed with  $\text{H}_2\text{O}$ , and coated on paper. If applied at room temp. the coating is smooth and uniform, whilst by heating to  $115$ – $120^\circ$  a mottled or grained coating may be obtained. D. A. C.

**Decorative papers.** DENNISON-VICTOR Co., LTD., Asses. of B. ASNES (B.P. 487,603, 22.12.36. U.S., 8.7.36).—The paper, preferably of open  $\alpha$ -cellulose fibres, is coated with glistening flakes and creped and/or corrugated, the flakes being suspended in the creping solution. B. M. V.

**Copying paper.** S. SOKAL. From KALLE & Co. A.-G. (B.P. 487,751, 29.11.37).—Copying paper is treated on one side with the normal dark dye, while the other side, which faces the back of the copy receiving the impression, is coated with a lighter substance, *e.g.*, chrysoidine, *o*-aminoazotoluene, 4 : 4'-di-*p*-(dimethylaminobenzamido)diphenyl-2 : 2'-disulphonic acid, or *p*- $\text{NMe}_2\cdot\text{C}_6\text{H}_4\cdot\text{COPh}$  (I); these absorb actinic light and so facilitate duplication by the photographic process. Among examples, stearic acid (3000), (I) (500), and pressed tallow (1000 g.) are melted together and applied to the unprepared side of an ordinary C copying paper. Similarly naphthostyryl (4) or *m*-tolyl-1 : 2- $\psi$ -azimino-5-naphthol are melted with stearine (35) and added to paraffin (25 pts.). K. H. S.

**Production of sheet formations of a leather-like character from fibres and polymerisation products of unsaturated organic compounds.** W., H., O., and R. FREUDENBERG (B.P. 488,394,

18.6.37. Ger., 20.6.36).—Fleeces of loose fibres (*e.g.*, skin or leather fibres, vegetable or mineral fibres) are converted into sheet formations by impregnation with a binding agent consisting of polymerisation products of vinyl compounds, acrylic acid compounds, and their homologues and derivatives, and pressing or calendering with heat. The product may be coated with lacquer without previous priming. The binding agent may be applied as an aq. emulsion or suspension, or as a solid.

H. J. E.

**Laminated sheet.** K. RIPPER (U.S.P. 2,066,421, 5.1.37. Appl., 17.9.32. Austr., 15.12.30).—Cellulosic sheets are impregnated with a solution of  $\text{CH}_3\text{O}$  (<2 mols.) and  $\text{CS}(\text{NH}_2)_2$  (1 mol.) or  $\text{CS}(\text{NH}_2)_2$ -urea in an acid medium ( $p_{\text{H}}$  3—5). Drying is effected without heating and the products are hot-pressed. (Cf. U.S.P. 2,029,893—4; B., 1937, 158.) S. M.

**Manufacture of coated sheet material.** A. F. BURGESS. From MARATHON PAPER MILLS Co. (B.P. 482,792, 21.5.37. Cf. U.S.P. 2,054,112—6; B., 1938, 153).—Paper, regenerated cellulose foil, etc. is coated with a thermoplastic rubber-wax composition by passing between a heated top-coating roll and a heated rubber-lined lower roll, with a definite speed differential between the two rolls. The coated side of the web then comes in contact with a chilled roll.

D. A. C.

**Composite wood-pulp boards.** ANKARSVIKS ANSGAGS AKTIEB., and F. I. E. STENFORS (B.P. 477,214, 23.6.36).—Board is claimed which is comprised of alternate layers of pulp sheets with  $d$  in sheet form of 1.0—1.8 and  $d$  0.20—0.30. The outer layers should consist of the hard sheets, the sheets being united where possible at their wire sides, using animal or casein glue. The board is stated to have good sound-proof qualities.

D. A. C.

**Manufacture of impregnated fibre board.** A. R. WYLIE (B.P. 476,327, 2.3.36).—Highly-sized kraft paper is impregnated with asphalt, m.p.  $\leq 60^\circ$ , or a mixture of asphalt with a natural or synthetic resin, under condition which will remove all the  $\text{H}_2\text{O}$  in the paper during impregnation. The paper is then exposed to the atm. for a short period to allow thorough penetration of the impregnant, cooled, and humidified to reduce its brittleness. Alternatively, two or more impregnated sheets may be directly united under pressure, with an adhesive (*e.g.*, starch-rubber latex mixture) in  $\text{H}_2\text{O}$  which contains sufficient  $\text{H}_2\text{O}$  to reduce brittleness. Board is claimed which is highly resistant to shrinkage in a wide range of atm. temp. and humidity.

D. A. C.

**Making leatherboard.** H. W. RICHTER, Assr. to G. O. JENKINS Co. (U.S.P. 2,076,159, 6.4.37. Appl., 29.3.35. Cf. U.S.P. 1,975,556; B., 1935, 819).—Leatherboard is bleached by the action of a 1.75% solution of a sol. oxalate, *e.g.*, the K or Na salt.

D. M. M.

(A) Preparation of compositions for forming flexible sheets, films, or coatings. (B) Coating of web or sheet material. (C) Laminated sheet material. A. F. BURGESS. From MARATHON PAPER MILLS Co. (B.P. 481,426 and Addn. B.P. 481,427—8, 6.6.36).—See U.S.P. 2,054,112—6; B., 1938, 153.

**Manufacture of rayon yarn of varying denier.** CELLULOSE ACETATE SILK Co., LTD., and C. C. TYRER (B.P. 489,714, 2.2.37).

**Drying of thread or the like, particularly artificial silk thread, and apparatus therefor.** A. H. STEVENS. From INDUSTRIAL RAYON CORP. (B.P. 489,781, 9.2.37).

[Press-finish] treatment of hosiery. J. H. WESTON (B.P. 477,710, 31.8.36).

[Mechanism for] production of variegated textile yarns. J. SYKES & SONS, LTD., and E. SYKES (B.P. 489,546, 26.1.37).

[Felt-corrugated paper] packing materials. MEDWAY CORRUGATED PAPER Co., LTD., and F. G. PITTMAN (B.P. 487,709, 24.3.37).

**Binding of dust and fly in spinning mills.** A.-G. J. J. RIETER & Co. (B.P. 488,134, 11.6.37. Ger., 1.7.36).

**Papermakers' dryer felts.** T. HINDLE, S. LORD, and K. W. BUCHANAN (B.P. 488,225, 20.2.37).

**Removal of water and the like from a travelling web of paper, such as a web of waxed paper.** BLACK-CLAWSON Co. (B.P. 487,414, 23.12.36. U.S., 11.7.36).

**Extrusion presses for foils etc.**—See I. Coloration of artificial [cellulosic] materials. After-treating artificial fibres. Decolorising cellulose derivative film etc. Amphoteric colloids.—See VI. Fibres from glass etc. Laminated materials. Abrasive-coated discs.—See VIII. Building materials. Plasterboard.—See IX. Insulating material.—See XI. Plastic masses. Thermoplastic threads etc. Casein products [for paper]. Highly dispersed pigments.  $\text{CaCO}_2$  pigment [for paper]. Printer's blanket.—See XIII. Resilient product.—See XIV. Stiffened leather. Gelatin wrapping. Adhesives.—See XV. Purification of liquids [*e.g.*, white- $\text{H}_2\text{O}$ ].—See XXIII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Colour reaction for detection of wool fibres treated with hydrogen peroxide.** J. PINTE (Rev. Gén. Mat. Col., 1938, 42, 281—282).—The treated fibres are detected by treatment with aq. KI in presence of starch. Residual O in the fibres may be determined by use of KI +  $\text{MnCl}_2$  and titrating the liberated I with aq.  $\text{Na}_2\text{S}_2\text{O}_3$ . Details of these tests, which are sp. for  $\text{H}_2\text{O}_2$ -treated wool, are given.

R. J. W. R.

**Review of past developments in textile processing.** R. HALLER (Textilber., 1938, 19, 186—190).—Selected discoveries in dyeing and printing, mainly connected with the discharging and reserving of Turkey-red, indigo, and Indanthrene dyes, are discussed.

A. J. H.

**Linen dyeing and finishing.** J. MONTGOMERY (J. Soc. Dyers and Col., 1938, 54, 351—360).—A lecture. Methods of dyeing yarn and piece goods are discussed with particular reference to the applic-

ation of azoic and vat colours to fabrics. Processes, *e.g.*, beetling and calendering, employed in finishing various types of linen fabrics, and the production of crease-resistant materials, are reviewed.

R. J. W. R.

**New methods of dyeing cellulose esters and acetate.** J. WAKELIN (*Rayon Text. Month.*, 1938, 19, 483—485).—A review of patented methods for dyeing acetylcellulose (I), including vat dyeing in an acid solution, acceleration of the absorption of leuco-compounds by partly drying the impregnated (I) in absence of air, the use of org. solvents, and the use of certain aminoazo compounds which are readily absorbed by (I) and are decomposed and coupled with naphthols by immersion in an acid bath. W. A. R.

**Velocity of diffusion of substantive dyes, in connexion with their equalising properties for viscose silk.** D. C. KANTER and V. A. DEREVITZKAJA (*Prom. Org. Chim.*, 1938, 5, 485—489).—The dyeing properties of a series of substantive dyes are related to the velocity with which they diffuse through Cellophane at 75°.

R. T.

**Diazosulphonates in textile printing.** R. L. DESAI, T. N. MEHTA, and V. B. THOSAR (*J. Soc. Dyers and Col.*, 1938, 54, 371—381).—Na *anti*-diazosulphonates have been prepared from various chlorinated, nitrated, and methoxylated arylamines and incorporated with Naphthol AS and Na<sub>2</sub>CrO<sub>4</sub> in alkaline printing pastes. The stability of the pastes to light and the development, by light and by treatment with steam (both neutral and with traces of Cl<sub>2</sub>), of prints made with them on cotton have been examined; the production of photographic prints by a contact method, using fabric padded with the *anti*-diazosulphonate and Naphthol AS, was also investigated. A study has been made of the activation of the *anti*-salts by light by exposing aq. solutions (of various concns.) and determining the amounts of the *syn*-isomerides (which alone couple with the Naphthol, the *anti*-form being inactive in colour formation) produced. This was carried out by measuring the I absorption val. and rate of coupling and colour development with β-C<sub>10</sub>H<sub>7</sub>-OH. The decrease of the *p<sub>H</sub>* vals. of the solutions, due to hydrolytic dissociation of the *syn*-isomeride formed on exposure, has also been determined. The effect of the nature and position of the substituent groups in the diazo salts on the rate of conversion of the *anti*- into the *syn*-isomeride and on colour development is discussed. Compounds containing NHBz groups show the most activity and nitrated derivatives the least. Alkoxy-groups have a greater activating effect than alkyl groups, whilst of the derivatives prepared from chloro- and nitro-toluidines, those substituted *meta* to the NH<sub>2</sub>-group show greater susceptibility to photo-activation than the *o*- or *p*-substituted compounds.

R. J. W. R.

**[Production of] unshrinkable wool [in top form].** ANON. (*Wool Rec. & Text. World*, 1938, 51, 1233—1235, 1237).—Application of the Drisol process (B., 1937, 890, 1041) to wool tops for the production of unshrinkable yarn is described. Simple immersion of the tops in a 2% solution of SO<sub>2</sub>Cl<sub>2</sub> in white spirit (I) at room temp. is satisfactory (previous

winding of the wool into skein form is unnecessary) since (I) penetrates the top uniformly within a few sec. The resulting wool spins easily to produce stronger yarns.

A. J. H.

**Chemistry of wool. Determining oxycellulose.**—See V. Fluorescence analysis.—See XI. Detergents.—See XII. Inorg. "resins."—See XIII. Colouring rubber.—See XIV. Textile waste disposal.—See XXIII.

See also A., II, 370, Substantivity of derivatives of 1- and 2-aminoanthraquinone. [Dyeing with] alizarates. III, 755, Textile lighting.

#### PATENTS.

**Bleaching wild silk.** W. B. HILL, B. P. RIDGE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 478,277, 17.7.36).—The material is treated at a raised temp. in an alkaline bath (*p<sub>H</sub>* preferably 9—10.5) containing H<sub>2</sub>O<sub>2</sub> (2—4-vol.) and an alkali or NH<sub>4</sub> persulphate (½—1%). An additional bleach before and/or after this treatment may be given. *E.g.*, Tussah silk, pretreated at 90° for 1 hr. in a soap-Na<sub>2</sub>CO<sub>3</sub> bath, is immersed in a bath at 45° containing H<sub>2</sub>O<sub>2</sub> (3-vol.), 1% K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, 1% Na<sub>3</sub>PO<sub>4</sub>, and 1% Na<sub>2</sub>SiO<sub>3</sub>. After 6 hr. the silk is washed and further bleached in a ½% solution of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> in H<sub>2</sub>O for ½ hr. at 45°.

R. J. W. R.

**Decolorisation of cellulose derivative films, celluloid, and like material.** P. COLEMAN (B.P. 478,856, 7.1.37).—The material is treated with an aq. solution of an org. softening or swelling agent (30—80% AcOH, or HCO<sub>2</sub>H) at room temp. and, after removal of the solution for re-use, the treated material is washed with H<sub>2</sub>O, bleached with aq. Cl<sub>2</sub>, again washed, and dried.

F. R. E.

**Production of fast dyeings on mixed fabrics of wool and viscose artificial silk.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 487,409, 21.12.36).—The fabrics are dyed by treating with H<sub>2</sub>O-sol. salts of diamides obtained by condensing an arylenediamine-carboxylic or -sulphonic acid (1 mol.) with a polynuclear *o*-hydroxyarylarboxylic acid capable of coupling (2 mols.) and converting them into dyes on the fibre by treatment with diazo compounds, especially those free from CO<sub>2</sub>H or SO<sub>3</sub>H. Among examples (18), a 50% wool-50% viscose fabric is treated at 90° for 1 hr. with 5% of the Na salt of the diamide prepared from 1:3:5-(NH<sub>2</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>·SO<sub>3</sub>H and 2:3-OH·C<sub>10</sub>H<sub>6</sub>·CO<sub>2</sub>H (I) in presence of 20% of Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O and developed (red-orange) with the diazo derivative of *m*-NO<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>·NH<sub>2</sub>. The diamides prepared from (I) with benzidine-2:2'- and -3:3'-disulphonic acids, 4:4'-diaminostilbene-2:2'-disulphonic acid, and 1:5:2-(NH<sub>2</sub>)<sub>2</sub>C<sub>10</sub>H<sub>5</sub>·SO<sub>3</sub>H are used similarly.

K. H. S.

**Production of fast dyeings.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 489,429, 24.12.36).—Fast dyeings on material consisting of or containing cellulose or regenerated cellulose are obtained by treating the material at > room temp. with a solution (containing, if desired, wetting agents and dyeing assistants) of H<sub>2</sub>O-sol. diazo compounds, *e.g.*, the salts, double compounds, *anti*-diazotates, diazoamino-



compounds, etc., which have affinity for the fibres and contain at least one substituent consisting of  $\leq 3$  heterocyclic or aromatic rings of 5—6 members joined together so that the mol. contains a continuous chain of conjugated double linkings, or at least one  $\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}\cdot$ , two or more  $\cdot\text{CO}\cdot\text{NH}\cdot$  or modified  $\cdot\text{CO}\cdot\text{NH}\cdot$  containing the grouping  $\cdot\text{C}\cdot\text{N}\cdot\text{NH}\cdot$ , or a group  $\text{R}\cdot\text{N}\cdot\text{N}\cdot\text{R}'$ ,  $\text{R}\cdot\text{CH}\cdot\text{CH}\cdot\text{R}'$ , or  $\text{R}\cdot\text{N}\cdot\text{CH}\cdot\text{R}'$  (R and R' = aryl) and in addition at least one aza-N in a 6-membered ring or a quaternary N or a polyglycol ether radical, and finally treating the material with a coupling component. Suitable diazo components are: 3-amino-7:8-benzacridine, diamino-diquinolinalphthalene, 2-(*p*-aminophenyl)-4:5-benzoquinoline, 4:4'-diaminodiazaterphenyl, m.p. 187—189° (prepared from  $\omega$ -chloro-4-aminoacetophenone and  $\text{NH}_3$ ), 9:10-(4'-aminophenyltriazolo)-4-azaphenanthrene, 5:5'-diamino-8:8'-diquinolylurea, m.p. 237—239°, and 2-(*p*-aminoanilino)-4:6-di-(7)-quinolinylamino-1:3:5-triazine. N. H. H.

**Dyeing of cellulose esters or ethers.** H. BLACKSHAW, H. A. THOMAS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 489,613, 27.1.37).—Coloured films, filaments, etc. of cellulose esters or ethers, e.g., acetate or  $\text{CH}_2\text{Ph}$  ether, are prepared by incorporating a solution in an org. solvent, e.g.,  $\text{COMe}_2$ , of dye components capable of combining to form azoic or ice colours with a spinning solution of the cellulose derivative, and, after the formation of the fibres etc., developing colour by treatment with  $\text{HNO}_2$ . E.g., 2-hydroxy-3-naphthoic 2:5-dimethoxyanilide in  $\text{COMe}_2$  (3600) with 2:1:4- $\text{NH}_2\cdot\text{C}_6\text{H}_3\text{Me}\cdot\text{NO}_2$  (9.4) and cellulose acetate (500 pts.) are extruded from a nozzle and the filaments treated with 95.6%  $\text{H}_2\text{SO}_4$  (20),  $\text{NaNO}_2$  (10), and  $\text{H}_2\text{O}$  (1000 pts.) at 50°. N. H. H.

**Coloration of artificial [cellulosic] materials.** P. F. C. SOWTER, R. BETTERIDGE, and BRIT. CELANESE, LTD. (B.P. 489,727, 4.2.37).—Artificial filaments, films, etc. containing cellulose ethers or esters (the acetate) are coloured in the wet-spinning process by extruding the solution of the org. derivative of cellulose (I) into a coagulating liquid containing a high concn. (25—65%) of a solvent or swelling agent for (I), e.g.,  $\text{Et}_2$  tartrate, dioxan, monoacetin, diacetone alcohol, and in addition a dyestuff ( $\text{H}_2\text{O}$ -insol. dyes which have affinity for cellulose ethers and esters), e.g., "Celliton," "Duranol," etc. dyes, basic dyes, "Solacet" and "Cellit" dyes, or a leuco-compound of a vat dye or a sulphuric ester thereof, or a compound which can be converted into a dye by treatment of the extruded material, e.g., a diazotisable compound. N. H. H.

**Dyeing fibres of cellulose or regenerated cellulose alone or in admixture with other fibres.** I. G. FARBENIND. A.-G. (B.P. 488,099, 30.12.36. Ger., 18.1.36).—Fabrics of cellulose or regenerated cellulose, alone or mixed with other fibres, especially wool, are treated with org. salts of high mol. wt. containing a polyether residue and a guanidine radical, such cations having affinity for cellulose (cf. B.P. 436,592; B., 1936, 143), and then dyed by the metachrome process. In the examples, a mixed fabric of wool and Cu artificial silk is treated for 1—2 hr. at room temp. with a 1% solution of the quaternary  $\text{NH}_4$

salt obtained by interaction of the chlorohydrin of a polyglycerol containing about 7 mols. of glycerol with  $\text{NMe}_2\cdot\text{C}_{18}\text{H}_{37}$  and then dyed for 1.5 hr. at the boil with 3% Metachrome Bordeaux B and 1.5%  $\text{Na}_2\text{Cr}_2\text{O}_7 + 1.5\%$   $(\text{NH}_4)_2\text{SO}_4$ ; a level dyeing is obtained from a bath free from ppt. Similarly a mixed fabric of wool and viscose is pretreated with a cold 0.5% solution of oleyldiguanide, obtained by interaction of oleylamine hydrochloride and dicyanodiamide at 150°, and dyed in a level shade with Metachrome Brown B. K. H. S.

**Manufacture of dyed fibres having a high lustre.** E. ELÖD (B.P. 475,308, 1.12.36. Ger., 2.12.35).—The moistened fibres (e.g., of coconut or sisal), which have been freed from their embedding material, pretreated with alkalis, and dyed (preferably black), are polished by mechanical beating (at 30—45°) in presence of lustring agents, e.g., talc. Wetting and emulsifying agents and small amounts of dyes also may be present. Coconut fibres treated by the process of B.P. 308,720 (B., 1930, 813) are especially amenable to this treatment. R. J. W. R.

**Colouring of textiles.** SOC. CHEM. IND. IN BASLE (B.P. 487,724, 17.6.37. Switz., 25.6.36).—Animal or recovered cellulose fibrous materials are printed or padded with an alkali salt or an alkaline solution of a coupling component (e.g., 2:3-hydroxynaphthoic anilide), a diazotisable amine of relatively high m.p. and low volatility (e.g., *o*-aminoazotoluene), and an alkali nitrite, dried, and passed first through an acid to diazotise the amine, and then through an acid-binding agent to produce the coupling. (Cf. B.P. 449,267 and 452,177; B., 1936, 832, 1089.) [Stat. ref.] H. A. P.

**Treatment of filaments, threads, fabrics, films, and like materials [of regenerated cellulose].** BRIT. CELANESE, LTD., and R. W. MONCRIEFF (B.P. 479,341, 3.6.36).—The affinity of such products (especially those prepared by saponification of a cellulose ester and of high tenacity) for cotton dyes is increased by swelling the undyed material with a  $\text{H}_2\text{O}$ -sol. org. base, e.g.,  $(\text{CH}_2\cdot\text{NH}_2)_2$  (I),  $\text{CH}_2\text{Ph}\cdot\text{NH}_2$ , guanidine, or a quaternary base, preferably having a dissociation const. of  $\leq 10^{-4}$ . E.g., completely saponified cellulose acetate yarn is treated in a 60% aq. solution of (I) at 20—60° for 10—15 min., washed, and then dyed with 10% Chlorazol Black BH. No combined N is introduced into the cellulose. R. J. W. R.

**[Products for] dyeing and printing acetate artificial silk.** I. G. FARBENIND. A.-G. (B.P. 487,725, 28.6.37. Ger., 25.7.36).—The use is claimed of dyes of the type:  $\text{R}\cdot\text{N}_2\cdot\text{C}_6\text{H}_4\cdot\text{NR}'\cdot\text{C}_6\text{H}_4\cdot\text{CO}_2\text{H}$  in which the  $\text{NHPh}_2$  nucleus may be substituted by groups other than  $\text{NO}_2$  and in which R is free from  $\text{H}_2\text{O}$ -solubilising groups. Examples are the dyes:  $p\text{-NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{NO}_2 \rightarrow 2\text{-NHPh}\cdot\text{C}_6\text{H}_4\cdot\text{CO}_2\text{H}$  (orange-brown), and  $\rightarrow 2:2'\text{-OEt}\cdot\text{C}_6\text{H}_4\cdot\text{NH}\cdot\text{C}_6\text{H}_4\cdot\text{CO}_2\text{H}$  (I) (red-brown),  $2:4:1\text{-C}_6\text{H}_3\text{Cl}_2\cdot\text{NH}_2 \rightarrow 2\text{-}$  and  $4\text{-NHPh}\cdot\text{C}_6\text{H}_4\cdot\text{CO}_2\text{H}$  (yellow), and  $4:3:1\text{-NH}_2\cdot\text{C}_6\text{H}_3\text{Cl}\cdot\text{SO}_2\text{Me}$  (II)  $\rightarrow$  (I) (yellow-red). (II) is prepared by interaction of  $\text{NH}_4\text{SCN}$  and Br with  $o\text{-C}_6\text{H}_4\text{Cl}\cdot\text{NH}_2$ , hydrolysis ( $\text{NaOH}$ ), methylation ( $\text{Me}_2\text{SO}_4$ ), and oxidation ( $\text{H}_2\text{O}_2$ ). H. A. P.

**Production of resist effects illuminated with vat dyes.** D. P. MILBURN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 489,235, 21.1.37).—Fabrics composed wholly or partly of cellulosic fibres are printed with vat dye pastes containing org. carbimides, R-NCO, where R = a normal or branched-chain saturated or unsaturated aliphatic radical of  $\leq C_8$ , dried, and steamed. On cross-dyeing the printed areas resist the dye, probably on account of a temporary H<sub>2</sub>O-repellency. Among examples, cotton or viscose is printed with pastes containing flavanthrene (paste A) or dimethoxydibenzanthrone (paste B) together with C<sub>17</sub>H<sub>35</sub>NCO, dried, steamed at 100° for 5–20 min., and dyed with the sulphuric ester of leucodimethoxydibenzanthrone or leuco-6 : 6'-diethoxy-2 : 2'-bisthionaphtheneindigotin; paste A affords a yellow pattern on a green ground and paste B a green pattern on an orange ground. Further, cotton fabric is grounded with 2 : 3-OH-C<sub>10</sub>H<sub>8</sub>·CO-NHPh, printed with paste A, and developed with tetrazotised dianisidine; an orange pattern on a blue ground is produced. K. H. S.

**Production of mordant dyeings on cotton hot-tomed with oil.** I. G. FARBENIND. A.-G. (B.P. 488,054, 18.6.37. Ger., 18.6.36).—Cotton is treated, prior to mordanting with Al or Fe salts, with emulsions of neutral oils (e.g., olive oil) prepared with neutral emulsifying agents, e.g., reaction products of (CH<sub>2</sub>)<sub>2</sub>O or polyglycol ethers with org. compounds containing OH, CO<sub>2</sub>H, NH<sub>2</sub>, or SH groups. Clear, full shades are obtained, and removal of unfixed oil is unnecessary. Protective colloids (glue, polyvinyl alcohol, etc.) may be added to improve the stability of the emulsions. E.g., cotton is impregnated with castor oil emulsified in H<sub>2</sub>O with the reaction product of (CH<sub>2</sub>)<sub>2</sub>O and castor oil, mordanted with basic Al sulphate, and dyed with Alizarin V I extra 20%. R. J. W. R.

**Shrinking of cloth.** BINNY & Co. (MADRAS), LTD., and J. V. S. MILNE (B.P. 476,440, 10.6.36. Brit. India, 29.7.35).—Woven fabric which has in its wet-processing been stretched in length and contracted in width is brought to a condition so that it does not shrink in washing by passage over an arrangement of rollers and rubber belts. Apparatus is claimed. A. J. H.

**Rendering of wool materials unshrinkable.** A. J. HALL, W. N. HICKING, and S. J. PENTECOST (B.P. 474,846, 3.7.36).—The SO<sub>2</sub>Cl<sub>2</sub> used in an earlier process (B.P. 464,503; B., 1937, 1041) is replaced by CrO<sub>2</sub>Cl<sub>2</sub>. A. J. H.

**Gaseous treatment in vacuo of wool and like fibres or materials made therefrom.** WOOL INDUSTRIES RES. ASSOC., H. PHILLIPS, and E. G. H. CARTER (B.P. 475,742, 26.5.36).—In producing shrinkage-resistance by treatment with gaseous Cl<sub>2</sub> etc. (cf. B.P. 417,719; B., 1935, 19), the gas is maintained in circulation in the evacuated vessel to ensure uniform treatment of the material. Apparatus is claimed. R. J. W. R.

**Treatment of wool and fabrics containing wool.** J. W. BROWN (B.P. 475,422, 18.5.36. Austral., 28.12.35).—Scoured wool is treated, preferably after dyeing, for 8–15 min. at room temp. in an acidified

(especially with H<sub>3</sub>BO<sub>3</sub>) aq. solution containing urea, CH<sub>2</sub>O, and glycerol. Condensation in the fibre is then completed by a drying treatment, after which the wool is lightly scoured and dried. Shrinkage and felting tendencies are reduced and the material is rendered more resistant to degradation during wear and to attack by moths, moulds, etc.; the cystine content is unaltered. A suitable padding solution contains aq. CH<sub>2</sub>O (38–40%) 19, H<sub>3</sub>BO<sub>3</sub> 4, glycerol 3, and urea 10 pts. in 95 pts. of H<sub>2</sub>O. R. J. W. R.

**Flameproofing [cellulosic] materials.** W. L. MORGAN, and BRIT. SIDAC, LTD. (B.P. 487,702, 18.2.37).—H<sub>2</sub>O-sol. salts of sulphonated aryl, alkaryl, or aralkyl derivatives of H<sub>3</sub>PO<sub>4</sub>, H<sub>4</sub>P<sub>2</sub>O<sub>7</sub>, HPO<sub>3</sub>, H<sub>2</sub>PO<sub>3</sub>, H<sub>3</sub>PO<sub>3</sub>, or H<sub>3</sub>PO<sub>2</sub> are used in conjunction with NH<sub>4</sub> salts of HBr, HI, H<sub>2</sub>SO<sub>4</sub>, or H<sub>3</sub>PO<sub>4</sub> to flameproof articles, especially sheets and films made of regenerated cellulose. In an example, a sheet of regenerated cellulose is soaked in a solution of 5% of NH<sub>4</sub>Br and 15% of Na<sub>2</sub>C<sub>10</sub>H<sub>5</sub>(SO<sub>3</sub>Na)<sub>2</sub> phosphate and dried; the material does not propagate a flame nor does the ash glow for any appreciable length of time. K. H. S.

**Fireproofing of solid combustible substances.** W. W. TRIGGS. From BAYERISCHE STICKSTOFFWERKE A.-G. (B.P. 476,043, 27.4.36).—Wood, fabrics, etc. are fireproofed by application of a condensation product of dicyanodiamide or derivative [e.g., guanidine, melamine, or dicyanodiamidine] and an aldehyde, e.g., CH<sub>2</sub>O. The condensation product may be prepared in presence of an acid, e.g., H<sub>3</sub>PO<sub>4</sub>, and other fireproofing agents, e.g., HBr, H<sub>3</sub>BO<sub>3</sub>, Na<sub>2</sub>SiO<sub>3</sub>; insecticides may be added to the treatment liquids. R. J. W. R.

**Rendering cloth non-inflammable.** R. ALMOND and A. M. YOUNG (B.P. 487,969, 27.2.37).—A fireproofing composition which does not appreciably discolour the fabric is prepared by adding a suitable quantity of a mixture of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> (5–10), NH<sub>4</sub>Cl (1–4), CH<sub>2</sub>O (1–2), MgCO<sub>3</sub> (1–2), ZnSO<sub>4</sub> (1–2), and Na phosphate (1–2 pts.) to H<sub>2</sub>O (preferably boiling). R. J. W. R.

**Waterproofing of textile materials.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 489,495, 23.1., 18.5., and 10.8.37).—Textile materials are rendered H<sub>2</sub>O-repellent by impregnation with a solution or emulsion of one of the quaternary NH<sub>4</sub> compounds from the chloromethyl derivatives of phenols containing one or more alkyl substituents of  $\leq C_4$  and *tert.* amines (cf. B.P. 478,571; B., 1938, 354) and heating the impregnated material, if desired, after it has been dried. Improved results are obtained when the above compounds are used in combination with mixed polymerides from unsaturated carboxylic acids or derivatives thereof and olefinic compounds and containing an aliphatic radical with  $\leq 12$  C in one chain. (Cf. B.P. 464,860 and 472,613; B., 1937, 1197, 1331.) N. H. H.

**Waterproofing treatment of textile fabrics and the like.** E. B. HIGGINS (B.P. 490,215, 6.2., 7. and 21.12.37).—Textile materials are made waterproof and fast to soap-boiling by impregnating them with a 1–3% solution of albumin (I) and/or globulin, preferably at the isoelectric point, coagulating the (I)

or globulin by steaming, preferably in a "cottage" steamer at  $>100^\circ$  (e.g.,  $120^\circ$ ), and then boiling with a solution of soap containing  $\leq 50\%$  of higher saturated fatty acids at  $p_H$  7—7.5. The material is then washed, soured, and finished. Inferior results are obtained by coagulating the (I) in any other way, e.g., by denaturation. The soap-boil may be omitted by emulsifying fatty acids ( $> C_{12}$ ) in the (I) solution, which may also contain emulsified waxes (e.g., paraffin wax). Emulsification is conveniently carried out in an emulsifying machine and must be done at  $<60^\circ$ . The waxes are not removed by subsequent dry-cleaning processes. Delicate fabrics like rayon may be proofed by steaming at a lower temp. ( $<100^\circ$ ) for a longer time, and the process may be applied to fabrics which have already been proofed but are not fast to soap-boiling.

S. C.

**Rendering textiles water-repellent.** M. FLORES and W. ESSERS (FÄRBEREI-GES. FLORES & Co., VORM. STOLTE-MISSY) (B.P. 487,645, 16.10.37. Ger., 16.10.36. Addn. to B.P. 474,403; B., 1938, 49).—Cellulosic and woollen fibrous materials are water-proofed by impregnation with an aq. mixture of a quaternary  $NH_4$  compound derived from an  $\alpha$ -halogeno-ether or -thioether of an alcohol having  $\leq C_{10}$  and a  $H_2O$ -sol. salt of  $HCNO$ , drying, and heating (e.g., for 5 min. at  $140^\circ$ ). The proof is resistant to washing and dry-cleaning.

H. A. P.

**Rendering materials mothproof.** J. R. GEIGY, A.-G. (B.P. 487,804, 5.3.37. Ger., 14.3.36).—The application is claimed of solutions of sulphonium salts  $SR'R''R'''X$  in which  $R'$ ,  $R''$ , and  $R'''$  are the same or different aromatic residues which may be substituted and/or linked together *oo'* to  $S$  by  $S$ ,  $O$ ,  $CO$ , or  $NR$  ( $R$  being an aliphatic or araliphatic residue), or, alternatively, one of the groups  $R'$ ,  $R''$ ,  $R'''$  may be a lower alkyl radical;  $X$  is the radical of an inorg. acid. Examples are:  $SPh_3Cl$ , 5-phenylthianthrenium chloride, 2 : 5 : 7-trimethylthianthrenium Me sulphate (2 : 7-dimethylthianthrene methosulphate), and thioxanthone methosulphate.

H. A. P.

**Treatment of textile yarns or threads with rubber.** J. H. FENNER & Co., LTD., J. H. FENNER, S. B. HAINSWORTH, and J. H. ANDERSON (B.P. 477,393, 22.5.36).—The material, which may have been pretreated with a coagulant, is passed through a flexible diaphragm fitted into the base of a vessel containing stabilised latex. After passage through the bath, it is removed via the upper surface, which is open to the atm., and dried before being brought in contact with the guide or winding device. Apparatus is claimed.

R. J. W. R.

**Treatment of fabric containing rubber thread.** T. L. SHEPHERD (B.P. 479,026, 26.6.36).—The fabrics, especially those in which a reagent, e.g., glue or gelatin, is incorporated with the rubber to reduce extensibility, are vulcanised under tension (e.g., in boiling  $H_2O$ ) so that the finished materials are flat, elastic, and free from puckers. The setting agent may be partly or completely removed during the operation.

R. J. W. R.

**[Rubber]-coated fabric [artificial leather].** A. M. ALVARADO and H. J. BARRETT, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,065,881, 4 I (B).

29.12.36. Appl., 5.1.34. Renewed 18.6.36).—Gilsonite (I) (bitumen)-drying oil (e.g., tung oil or oil-modified alkyd resin) varnishes containing C black are used for the top coats of rubber-coated fabrics. 160—560 pts. of drying oil vehicle are used with 100 pts. of (I); the proportion of C black present is  $\leq 12\%$  of the (I)-drying oil mixture and  $\geq$  the wt. of oil less  $5/4$  of the wt. of (I). Other pigments may be present. The coating can be applied directly to the rubber-coated fabric or over an intermediate coat of oil varnish, and heated to dry the coat and effect vulcanisation. The finishes are resistant to chalking and retain their lustre and colour.

R. J. W. R.

**Manufacture of material for use as leather substitute.** A. J. HANLEY, Assr. to RESPRO, INC. (U.S.P. 2,065,892, 29.12.36. Appl., 29.7.33).—A film of rubber compound containing fibres, e.g., waste cotton, is applied to one side of a stretched, woven fabric while suction is applied to the other side sufficient to draw the fibres and compound into the interstices. The material is then dried, pressed under tension, and vulcanised.

F. R. E.

**Treatment of fibrous materials, including leather.** N. V. CHEM. FABR. SERVO, and M. D. ROZENBROEK (B.P. 475,478, 15.4.36. Cf. B.P. 473,760; B., 1938, 642).—Solutions of soaps which are prepared from elaidinised fatty acids (e.g., elaidic acid) and are rendered substantially neutral to phenolphthalein by presence of free fatty acid (effected, e.g., by addition of mineral acid) are used for wetting, dispersing, etc. purposes, being more effective than the normal alkaline solutions. Salts of sulphonated polycyclic aromatic hydrocarbons, e.g.,  $C_{10}H_6Pr^b \cdot SO_3Na$ , may be present.

R. J. W. R.

**Adhesive fabrics.** E. I. DU PONT DE NEMOURS & Co. (B.P. 479,111, 27.4.36. U.S., 27.4.35).—Fabrics such as surgical plasters are prepared by coating one side of a fabric base with a plasticised cellulose nitrate (I) composition and the other with a suitable rubber adhesive; the fabric is first rendered waterproof, e.g., by application of carbohydrate amine polymeride (e.g., an aq. solution of an acetate of deacetylated chitin) or cellulose derivative-wax compositions. The (I) coating films must be free from vegetable oils and stearates of high alcohols or ether-alcohols since these have a deleterious effect on the rubber coating, but are suitably plasticised, e.g., with 12—19% of various esters of  $o$ - $C_6H_4(CO_2H)_2$ , esters of glycerol or polyglycerol, and adipic, pimelic, suberic, azelaic, and sebamic acids, or  $(C_6H_4Me)_3PO_4$ , and may contain pigments (11—16%).

R. J. W. R.

**[Sizing] treatment of textile materials and compositions therefor.** BRIT. CELANESE, LTD. (B.P. 476,581, 10.6.36. U.S., 18.6.35).—Fabrics etc. of org. derivatives of cellulose are treated with a liquid containing an alkali silicate (I) and the product formed by interaction of a resin with  $NH_3$ . The liquid medium is preferably  $H_2O$  containing also a solvent for the resin, and it is desirable first to prepare the  $NH_4$  resinate, (I) being added just before use. Fabric is passed through the treating solution, dried, and heated between rollers at 95—115°. A smooth, soft, and non-slip handle is produced. After-treatment of the

sized fabrics with substances which form insol. resinates, *e.g.*,  $\text{BaCl}_2$  (10% aq. solution), produces a delustred effect.  
R. J. W. R.

**Sizing and dressing of textiles.** J. T. SHEVLIN. From PFEIFFER & LANGEN KOMM.-GES. (B.P. 476,778, 16.10.36).—Pectin extracts suitable for use, either alone or with other substances, as sizing and dressing agents are prepared from beet. Readily  $\text{H}_2\text{O}$ -sol. materials, *e.g.*, sugar, are first removed from the beet by lixiviation at 30–80° (>55°) with  $\text{H}_2\text{O}$ , and the pectin is extracted from the lixiviated material, *e.g.*, by autoclaving.  
R. J. W. R.

**[Setting] treatment of textile materials.** J. BRANDWOOD (B.P. 476,571, 9.6. and 27.10.36).—Highly twisted or doubled yarns, fabrics, etc. are treated (to counteract effects due to tension used in previous processing) in a partial vac. with steam at > the b.p. of  $\text{H}_2\text{O}$  at the low pressure used, until a determined fall in the vac. is reached. *E.g.*, the goods are placed in a chamber which is evacuated to 28 in. Hg, and steam at 148° is slowly admitted until the pressure in the chamber rises by 13–18 in. Hg. Apparatus is claimed.  
R. J. W. R.

**Impregnating, stiffening, or sticking fibrous materials.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 477,830, 2.6.36).—The materials are treated with aq. emulsions of polymerised vinyl compounds (*e.g.*, vinyl or acrylic esters, vinyl ethers or ketones, etc.) containing also a hydrocarbon of high mol. wt. The resulting finishes are not affected by  $\text{H}_2\text{O}$ , and loss of shape and spotting are thus prevented. *E.g.*, a 40% aq. emulsion (100) of polymeric  $\text{CH}_2\text{:CH}\cdot\text{CO}_2\text{Et}$  (prepared by emulsion-polymerisation in  $\text{H}_2\text{O}$ ) is stirred with a 15% aq. emulsion of paraffin wax (10 pts.) to give a suitable treatment medium. Mixed vinyl polymerides may also be used.  
R. J. W. R.

**Impregnated [stiffening] material.** CELLULOID CORP. (B.P. 479,257, 30.4.37. U.S., 5.5.36).—Porous material, *e.g.*, fabric or felt, is impregnated with a solution of a plastic material (*e.g.*, natural or synthetic resin or cellulose derivative) in a volatile solvent, *e.g.*,  $\text{COMe}_2$ ,  $\text{MeOAc}$ , and the solvent then evaporated by treatment with an inert vapour, *e.g.*, steam. The mixed vapours are then condensed and the solvent is recovered by distillation or separation. The operation is carried out in absence of air, explosion hazards being thus eliminated. Apparatus is claimed.  
R. J. W. R.

**Stiffened [fabric] materials.** CELLULOID CORP. (B.P. 479,403, 30.4.37. U.S., 5.5.36).—Stiffening materials are prepared by impregnating a base material (fabric, felt, etc.) with a solution of a cellulose derivative (I), *e.g.*, cellulose acetate or nitrate, and then treating with a (superheated) vapour of a pptg. agent, *e.g.*, live steam. (I) is preferably dissolved in a low-boiling,  $\text{H}_2\text{O}$ -sol. liquid, *e.g.*,  $\text{COMe}_2$ , and may be mixed with plasticisers and fire-retarding agents.  
R. J. W. R.

**Production of stiff fabrics stable to washing.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 476,312, 4.6. and 31.7.36).—Fabrics are coated or impregnated with a solution or emulsion of a polymeride of a vinyl alkyl ketone, especially

$\text{CH}_2\text{:CH}\cdot\text{COMe}$  (I), and then heat-treated; yarns and threads can be similarly treated and subsequently made up into fabrics which are permeable to air and  $\text{H}_2\text{O}$ . Mixed polymerides of these ketones and other polymerisable substances, *e.g.*, styrene or Et acrylate, may also be used. The stiffened fabrics are useful as such or as interlayers in laminated fabrics. *E.g.*, cotton fabric impregnated with a 20% solution of polymeric (I) in  $\text{COMe}_2$  or  $\text{CH}_2\text{Cl}_2$  and dried at 50° may either be stiffened by ironing at 120°, or used as an interlayer in a 3-ply fabric (bonding being effected in this case by hot-pressing). Dyes, pigments, and softeners may be added to the treatment liquors.  
R. J. W. R.

**Manufacture of [semi-stiff] textile fabric.** H. L. SHOUB, Assr. to STARCHLESS CURTAIN PERFECTOR INSTITUTE (U.S.P. 2,066,079, 29.12.36. Appl., 18.2.35).—Semi-stiff finishes resistant to laundering are produced by application of a small amount of a colourless vinyl resin (*e.g.*, polymerides of  $\text{C}_2\text{H}_4$ ,  $\text{CH}_2\text{:CH}\cdot\text{OAc}$ , or  $\text{CH}_2\text{:CHCl}$ ) and ironing; the resin is applied by spraying the fabric with a 5–20% solution or dipping in a 3–10% solution in, *e.g.*,  $\text{COMe}_2$  or  $\text{CH}_2\text{Cl}_2$ .  
R. J. W. R.

**Stiffeners, adhesives, and process for stiffening textile articles.** C. F. BOEHRINGER & SOEHNE, G.M.B.H. (B.P. 475,538, 15.10.36. Ger., 17.3.36).—Paper-like felted materials containing a substance, *e.g.*, cellulose acetate, which becomes adhesive on application of a solvent and also fibres, *e.g.*, cellulose pulp, which remain unchanged, are used as interlayers in porous, stiffened, laminated fabrics. Porosity is attained by the presence of the inert fibre and may be assisted by application to the finished felt of a discontinuous protective layer of, *e.g.*, gum arabic or cellulose tribenzoate, which will inhibit the action of the solvent on portions of the felt and can afterwards be removed by washing. The felt can also be improved by incorporating with it, *e.g.*, during manufacture, light, open fabrics. *E.g.*, a suitable felt is prepared from a beaten mixture of cellulose di- or tri-acetate (100) and cellulose pulp (150 pts.).  
R. J. W. R.

**Production of crimped artificial textile fibres and like materials.** L. UBBELOHDE (B.P. 477,428, 13.10.36. Ger., 7.12.35, 5.2., 8.9., and 19.9.36).—Permanent crimped effects on viscose etc. are obtained by subjecting the filaments, before coagulation is complete (if desired, after pptn. of the xanthate and before regeneration of the cellulose), to a crimping operation during which the filaments are stretched beyond the elastic limit; orientation of the micelles is thus adapted to the form assumed by the fibres. Coagulation is then completed, after which the fibres may be cut into staple lengths. Max. permanence of the crimp is dependent on the nature and condition of the filament at the time of crimping, and there is thus an optimum distance from the spinning jet at which the crimping device can be placed. In crimping bundles of filaments with one crimping device, the maturity, temp., and concn. of the respective baths are graduated so that each thread bundle has attained its optimum condition on reaching the crimping device. Substances which reduce the

maturity or speed of reaction, *e.g.*, urea,  $\text{CH}_2\text{O}$  or pre-condensates of urea,  $\text{PhOH}$ , or vinyl resins, may be added to the viscose. The introduction of hardenable resins increases the abrasion-resistance of the threads. Where crimp in  $>$  one plane is desired, the bundles of fibres are given a false twist by means of a suitable rolling treatment before crimping. Crimping is effected by a mechanical treatment. The finished crimped fibres may be after-treated with solutions of resins which can eventually be hardened. Finished fibres etc. may also be crimped by treating the softened materials so that they are stretched and the micelles oriented. Apparatus is claimed.

R. J. W. R.

**Production of crimped artificial filaments, fibres, and the like.** D. FINLAYSON and R. G. PERCY (B.P. 479,347, 30.7.36).—Filaments of thermoplastic materials are placed crosswise over each other, softened by application of hot  $\text{H}_2\text{O}$  or steam, and then pressed and dried. Suitable apparatus is figured.

R. J. W. R.

**Soaking of natural or artificial silk.** WARWICK CHEM. CO. (B.P. 477,066, 11.12.36. U.S., 14.2.36).—The materials, especially natural silk which has not been degummed, are lubricated by soaking in a bath containing a dispersed "soaking oil" and a reagent to discharge this on to the fibre. The discharging reagent may be a sol. salt of an org. compound containing  $\text{OH}$  and/or  $\text{CO}_2\text{H}$  groups, *e.g.*, alkali lactates, whilst the lubricant is preferably a neutralised sulphonated oil (*e.g.*, olive, neatsfoot, sperm, etc.). Silk is treated at  $21$ – $38^\circ$  and rayon at  $51$ – $60^\circ$ , the bath being alkaline ( $p_{\text{H}}$  approx. 10) so that when absorption is complete the  $p_{\text{H}}$  is  $< 7$ . A suitable bath for natural silk skeins (100) consists of  $\text{H}_2\text{O}$  (600), sulphonated olive oil (16),  $\text{K}_2\text{CO}_3$  (2.5), and Na lactate (2–4 pts.).

R. J. W. R.

**Rendering natural silk and artificial silk matt.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 477,049, 25.6.36).—Delustred finishes are obtained by treating the materials at room temp. or slightly above in stable aq. dispersions of pigments (*e.g.*, lithopone,  $\text{ZnO}$ , or  $\text{TiO}_2$ ) free from  $\text{H}_2\text{O}$ -insol. solvents, and drying. The dispersions are prepared by the use of polynuclear aromatic sulphonic acids or their salts, *e.g.*, condensation products of naphthalenesulphonic acids and  $\text{CH}_2\text{O}$ ,  $\text{MeCHO}$ ,  $\text{COMe}_2$ , or  $\text{S}_2\text{Cl}_2$ , and may contain also  $\text{H}_2\text{O}$ -sol. polyvinyl alkyl ethers (*e.g.*, polymeric  $\text{CH}_2\text{:CH}\cdot\text{OMe}$ ), softening agents, and protective colloids.

R. J. W. R.

**Matting of textiles.** A. G. BLOXAM. From Soc. CHEM. IND. IN BASLE (B.P. 478,998, 6.7.37).—Aq. colloidal suspensions of  $\text{H}_2\text{O}$ -insol. condensation products of  $\text{CH}_2\text{O}$  and urea etc. are used for delustring textiles (cf. B.P. 469,688; B., 1938, 366). They are prepared by pretreating the condensate with  $\text{H}_2\text{O}$ , dissolving the product in an acid, and diluting the resulting solution with  $\text{H}_2\text{O}$ ; pretreatment with  $\text{H}_2\text{O}$  facilitates dissolution of the condensate in the acid. *E.g.*, the hardened product (1 kg.) obtained by condensing a neutral solution containing equal pts. of urea and  $\text{CH}_2\text{O}$  is mixed with  $\text{H}_2\text{O}$  (1). After keeping overnight,  $\text{H}_2\text{O}$  (1) and  $\text{HCO}_2\text{H}$  (4) are added and the clear solution is poured into  $\text{H}_2\text{O}$  (600 l.) at  $35^\circ$  and

containing  $\text{NaCl}$  (300 g.). Viscose (20 kg.) is delustred by treatment in this bath for 30–40 min.

R. J. W. R.

**(A) Subduing the lustre of and finishing, (B) treating [softening], (A, B) shaped textile goods, more particularly silk and artificial silk stockings.** BRIT. SCHUSTER BATES MACHINE CO., LTD. From F. SCHUSTER (B.P. 478,677 and 478,686, [A] 24.7.36, [B] 30.7.36).—The stockings are drawn over upright metal shapes attached to an endless chain which continuously traverses a closed path so that they are sprayed with (A) delustring or (B) softening emulsions, led between squeezing rollers, and thence through a hot-air drying chamber.

A. J. H.

**Treatment of cellulose fabrics, yarns, or the like.** L. G. LAWRIE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 475,537, 22.5.36).—The materials are treated with an aq. caustic alkali solution and afterwards with an  $\alpha\beta$ -alkylene oxide, *e.g.*,  $(\text{CH}_2)_2\text{O}$  (cf. B.P. 439,880; B., 1936, 272); if desired, they may then be further treated with aq. caustic alkali solution. The alkali solutions for the pre- and/or after-treatments may contain alkali-sol. cellulose ethers, *e.g.*, methyl-, ethyl-, or glycol-cellulose (I). *E.g.*, bleached calico (100) is immersed in a solution of  $\text{NaOH}$  (12.5) and (I) (5) in  $\text{H}_2\text{O}$  (100), squeezed, and treated for 18 hr. in  $\text{CCl}_4$  (2000) containing  $(\text{CH}_2)_2\text{O}$  (40 pts.), afterwards being acidified, washed, and dried.

R. J. W. R.

**Wetting and penetrating agents for strong alkali lyes.** CHEM. WORKS FORMERLY SANDOZ (B.P. 488,620, 8.1.36. Switz., 9.1.36).—The wetting and penetrating power of conc. alkali lyes (of  $d < 1.08$ ), *e.g.*, those used in mercerising, is increased by incorporating naphthenic acids the alkali salts of which are not salted out by the lye. The max. wetting etc. action is obtained when (a) the solution of the latter in the lye is saturated or (b) the solution is  $<$  saturated and contains in addition an auxiliary wetting agent, *e.g.*, saturated or unsaturated ether alcohols and/or aminoalcohols and/or  $\text{NH}_4$  bases with one or more mono- or poly-hydroxyalkyl groups, viz., ethers of glycol, glycerol, etc. Insol. alkali naphthenates may be used in combination with suitable dispersing agents, *e.g.*, sulphonic acids of mono-nuclear alkylated and/or hydroxylated aromatic hydrocarbons. 25 examples are given. N. H. H.

**Increasing the wetting power of strong alkali lyes.** M. SCHWARZ, F. and E. ZSCHIMMER, and R. and W. SCHWARZ (ZSCHIMMER & SCHWARZ CHEM. FABR. DÖLAU) (B.P. 487,927, 28.9.37. Ger., 3.10.36).—Mixtures of alcohols (preferably those of high b.p. or glycol or glycerol ethers containing free  $\text{OH}$ ) with hydroxyalkylbenzyl alcohols or xylylene glycols are added to the alkaline liquors used in textile processing. The products are almost odourless and do not cause difficulties in disposal of waste liquor. A suitable mixture consists of  $\text{C}_6\text{H}_4(\text{CH}_2\text{-OH})_2$  (85) and  $\text{OH}\cdot[\text{CH}_2]_2\text{-OBu}$  (15 pts.).

R. J. W. R.

**Treatment [saponification] of materials comprising organic esters of cellulose.** BRIT. CELANESE, LTD., H. DREYFUS, R. W. MONCRIEFF, and F. B. HILL (B.P. 478,077, 8.4.36. Cf. B.P. 453,302; B., 1936, 1149).—Partial or complete saponification of

the material (filaments, fabrics, etc.) is effected by treatment with an aralkylamine or quaternary  $\text{NH}_4$  base containing an aralkyl radical, *e.g.*,  $\text{CH}_2\text{Ph}\cdot\text{NH}_2$  (I) (2—5% aq. solution),  $\text{NH}(\text{CH}_2\text{Ph})_2$  (II),  $\text{CH}_2\text{Ph}\cdot\text{NMe}_3\cdot\text{OH}$  (III), in absence of mineral saponifying agents. The treatment may be carried out at  $\leq 1$  atm., and the bath may contain substances to reduce swelling, *e.g.*,  $\text{H}_2\text{O}$ -sol. salts of org. acids,  $\text{NaCl}$ , sugars, etc. Examples show the complete saponification of cellulose acetate yarn by treatment at 60—90° in an aq. solution of (I) (2—4%) or (III) (1—2%). (II) and  $\text{CH}_2\text{Ph}\cdot\text{CH}_2\cdot\text{NH}_2$  are used at higher concn., the latter being dissolved in aq.  $\text{EtOH}$  and used at lower temp. (25—50°). R. J. W. R.

**Continuous deacidification and after-treatment of artificial fibres in endless form.** ZELWOLLE-ARBEITSGEMEINSCHAFT G.M.B.H. (B.P. 475,711, 19.1.37. Ger., 24.1.36).—The band of fibres, either immediately after spinning or after intermediate processing, is treated with liquids or gaseous materials (at  $\sim 100^\circ$ ) while being passed over guide rollers arranged so that tensioning is continually altered. The fibres can also be squeezed at intervals during treatment, and agitated in an oscillatory manner. Apparatus is claimed. R. J. W. R.

**Carroting processes.** W. C. MERCIER (B.P. 477,795—6, [A, B] 13.11.36).—(A) Felting properties are imparted to fur etc. by treatment with a solution containing  $\text{Hg}(\text{NO}_3)_2$ ,  $\text{HNO}_3$  and a per-salt (0.1—0.8m. per l.), *e.g.*,  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  (I),  $\text{NaClO}_4$ , or  $\text{NaBO}_3$ . Oxidising agents, *e.g.*,  $\text{H}_2\text{O}_2$ , may also be present, especially when light shades are required. *E.g.*,  $\text{HNO}_3$  ( $d$  1.38, 100) is added to  $\text{Hg}$  (35 pts.), and  $\text{H}_2\text{O}$  added to bring the  $d$  of the solution to 1.13. Equal quantities of aq. (I) ( $d$  1.15) and  $\text{H}_2\text{O}_2$  (100-vol.) are then added to give a suitable solution. (B) Carroting may be accomplished by treatment with an aq. solution containing  $\text{HClO}_3$ , a peroxygen compound, *e.g.*,  $\text{H}_2\text{O}_2$  (20—60-vol.) or a per-salt (0.1—2.5m. per l.), and a strong acid (0.4—0.8m. per l.), *e.g.*,  $\text{H}_2\text{SO}_4$ , to liberate  $\text{O}_2$ . Such solutions are non-toxic and do not discolour the fibres. In an example, a carroting solution is prepared by adding  $\text{HNO}_3$  ( $d$  1.38, 1) to aq.  $\text{Ba}(\text{ClO}_3)_2$  ( $d$  1.16, 5 pts.); after removal of the  $\text{Ba}(\text{NO}_3)_2$ , an equal quantity of aq. (I) ( $d$  1.15) is added. R. J. W. R.

**Modifying the properties of natural and artificial amphoteric colloids.** ACETA GES.M.B.H. (B.P. 488,783, 13.11.36. Ger., 13.11.35).—The chemical properties of shaped materials containing natural or artificial amphoteric colloids (wool and the products of B.P. 459,711; B., 1937, 469) are modified, *e.g.*, the affinity for dyes is increased, by treatment with a univalent alkylating agent, *e.g.*, alkylene oxide, alkyl halides, toluenesulphonic acids, and/or a substance containing  $\leq 2$  functional groups capable of reacting with the colloid, at least one of which has an acylating action. Such groups are acid anhydride, acid halide, and carbimide groups, whilst the other functional groups are, *e.g.*, isocarbamide, isothiocarbamide, iminoether, aldehyde, cyanamide, and alkylene oxide groups and halogeno- or sulphonic acid ester residues. Examples describe the treatment of wool with 25 wt.-% of  $p\text{-CH}_2\text{Cl}\cdot\text{C}_6\text{H}_4\cdot\text{NCS}$  or  $p\text{-}$

$\text{C}_6\text{H}_4\text{Me}\cdot\text{O}\cdot[\text{CH}_2]_2\cdot\text{NCO}$  for 12 hr. at  $70^\circ/\leq 1$  atm. and of artificial silk dyed with Alizarin Cyanine Green G with a 2% solution of the carbamyl chloride from  $(\text{CH}_2\text{Cl}\cdot\text{CH}_2)_2\text{NH}\cdot\text{HCl}$  and  $\text{COCl}_2$  in  $\text{C}_2\text{HCl}_3$  at  $60^\circ$  for 10 hr. N. H. H.

**[Anti-static] textile dressings.** BRIT. CELAN-SE, LTD. (B.P. 477,639, 8.10.36. U.S., 15.10.35. Addn. to B.P. 450,420; B., 1936, 926).—Anti-static dressings, which can be used as lubricants or applied to previously lubricated yarn and are especially useful in connexion with cellulose ester or ether rayons, are prepared from a polyhydric alcohol (I) and an alkylol-amine soap of a higher fatty acid. The amount of the latter used is 2—8 wt.-% of (I), and the mixture preferably contains sufficient  $\text{H}_2\text{O}$  to bring it into equilibrium with the atm. Lubricants and wetting-out agents may also be added. *E.g.*, a suitable prep. contains  $\text{N}(\text{C}_2\text{H}_4\cdot\text{OH})_3$  oleate (6.5),  $\text{O}(\text{C}_2\text{H}_4\cdot\text{OH})_2$  (100), and  $\text{H}_2\text{O}$  (30 pts.); 2—3% of the mixture is applied to cellulose acetate yarn. R. J. W. R.

**[Feeding yarns during] yarn dyeing, dressing, and loom beaming.** N. A. BATCHELDER (B.P. 478,687, 31.7.36).

**Polymerised alkylene oxides. Surface-active agents. N compounds.**—See III. Azo dyes.—See IV. Bleaching pulp.—See V. Insect- and fungi-toxic fibrous materials.—See IX. Wetting etc. agents.—See XII. Dyeing hardened casein. Horn-like condensation products. Embossing fabrics. Highly dispersed pigments. Cellulose derivative emulsions.—See XIII. Coating [fabric] materials with rubber.—See XIV.

## VII.—ACIDS; ALKALIS; SALTS: NON-METALLIC ELEMENTS.

**Progress in the nitration process of sulphuric acid manufacture.** H. PETERSEN (Metall u. Erz, 1938, 35, 375—378).—A lecture. The necessity for a high ratio of surface to space is emphasised: optimum conditions are much more nearly attained in the tower than in the chamber system, with a large saving of plant space. The stages in the development of the tower process are reviewed; it can compete successfully with the contact process, especially for the treatment of metallurgical gases, if the production of conc. acid is not necessary. I. C. R.

**Portable instrument for analysis of hydrocyanic acid gas-air mixtures.** R. J. WILMOT and T. N. GAUTIER (J. Agric. Res., 1938, 56, 283—290).—The gas sample is circulated over a hot Pt filament, the resulting change in resistance of which is a measure of the  $[\text{HCN}]$ . A. G. P.

**Determination of magnesia and lime in cooking salt.** C. TANNE (Chem.-Ztg., 1938, 62, 572—573).—The % of  $\text{MgO}$  and  $\text{CaO}$  found in a vac.-evaporated sample of  $\text{NaCl}$  decreased with increase in the amount taken for the determination. This result was not influenced by the dilution or amount of pptg. agent. Mixtures of increasing quantities of this salt with a const. wt. of  $\text{MgSO}_4$  also gave decreasing wts. of  $\text{MgO}$  when analysed, but pure  $\text{NaCl}$  did not have this effect. It is suggested that double salts

may be formed with NaCl and Ca and Mg salts present as impurities. S. M.

Determination of small quantities of magnesia in limestone and [sugar-]defecation slime. G. STORCK (Z. Wirts. Zuckerind., 1938, 88, 378—382).—The material is boiled with 20%  $H_2SO_4$  to dissolve MgO and to destroy org. matter. After cooling and dilution with EtOH, the filtered solution is treated with  $NH_4$  citrate to retain  $Fe_2O_3$  and  $Al_2O_3$  in solution, MgO being then pptd. as  $MgNH_4PO_4$  and determined as  $Mg_2P_2O_7$ . I. A. P.

[Determination of water-soluble] nitrogen [in cyanamide]. A. L. PRINCE (J. Assoc. Off. Agric. Chem., 1938, 21, 274—277).—The official method proved more trustworthy than two proposed modifications when tested by collaborative analysis. E. C. S.

Potassium salts as chemical raw materials. J. W. TURRENTINE (Ind. Eng. Chem., 1938, 30, 889—890).—About 3% of all sales of K compounds in the United States are for chemical purposes. These include manufacture of KOH and thence  $K_2CO_3$  principally for potash soaps and glass manufacture.  $KClO_3$ ,  $KClO_4$ , and  $KNO_3$  are imported in appreciable amounts together with felspar for the ceramic industry. It may be possible to replace the latter by a synthetic source of  $K_2O$ . C. I.

Potash in the Permian salt basin [of the United States]. H. I. SMITH (Ind. Eng. Chem., 1938, 30, 854—860).—Geological and historical. Whilst polyhalite occurs over a wide area, sylvite co-exists over a limited one, now well prospected and in commercial exploitation. C. I.

Exploitation of potash salts in relation to the [German] four-year plan. SCHELIGA and JAISLE (Bergbau, 1938, 51, 83—85).—A discussion. R. B. C.

Production of potassium chloride in New Mexico. T. M. CRAMER (Ind. Eng. Chem., 1938, 30, 865—867).—The material mined is essentially KCl + NaCl. It is dissolved in mother-liquor heated to 110°, cooled in vac. evaporators, and the cryst. KCl washed and dried in Oliver filters. The waste NaCl is stored in a natural reservoir. C. I.

New Mexico sylvinite: occurrence and mining. R. M. MAGRAW (Ind. Eng. Chem., 1938, 30, 861—864).—Sylvinitic deposits about 12—14 ft. thick at a depth of about 1000 ft. are worked by methods similar to those employed in coal mining. C. I.

Potassium chloride from the brine of Searles Lake. R. W. MUMFORD (Ind. Eng. Chem., 1938, 30, 872—878).—This brine fills the voids of a cryst. deposit. It forms the raw material for the manufacture of borax,  $Na_2CO_3$ , and  $Na_2SO_4$  in addition to KCl. In the separation of KCl by the Trona process triple-effect evaporators with countercurrent flow are used with continuous separation of Na salts. The conc. liquor is cooled to 38° in 3-stage vac. coolers with addition of make-up  $H_2O$ . The solid KCl is centrifuged and then containing 5% of free  $H_2O$  is dried in oil-fired rotary dryers. The mother-liquor is cooled to 24° with liquid  $NH_3$  expanded in helical

coils, the distillate refluxed, and crude borax separated. C. I.

Chemistry of the Trona process from the standpoint of the phase rule. W. A. GALE (Ind. Eng. Chem., 1938, 30, 867—871).—The  $H_2O$  of Searles Lake forms a 9-component system. On concn. a  $PO_4$  compound separates before the end-point (at which no further change of composition of the liquid phase occurs) is reached. There should therefore be 6 solid phases in equilibrium with the brine. These have been identified as NaCl,  $Na_2B_4O_7 \cdot 10H_2O$ ,  $Na_2CO_3 \cdot NaHCO_3 \cdot 2H_2O$ ,  $K_2Na(SO_4)_2$ ,  $9Na_2SO_4 \cdot 2Na_2CO_3 \cdot KCl$ , and  $2Na_2SO_4 \cdot NaCl \cdot NaF$ . In practice the additional constituents  $AsO_4$ , S, Br, and Li gradually accumulate by concn. It is therefore too complicated for complete investigation, but partial studies show why separation of K and Na by evaporation at 20° is impossible. Evaporation at 100°, however, enables NaCl to be removed continuously, followed by  $Na_2CO_3 \cdot H_2O$ . On cooling, KCl is crystallised, borax remaining in supersaturation and being separated later. C. I.

Production of potassium sulphate by the potassium-magnesium [sulphate] method. N. S. KURNAKOV and E. I. LUKJANOVA (Bull. Acad. Sci. U.R.S.S., 1938, Sér. Chim., 39—46).—Production of  $K_2SO_4$  from  $K_2SO_4 \cdot MgSO_4 \cdot 6H_2O$ , obtained by agitation of raw kainite with  $H_2O$ , is recommended. E. S. H.

Potash in the fertiliser industry. F. S. LODGE (Ind. Eng. Chem., 1938, 30, 878—882).—Historical and economic survey of American conditions. C. I.

Concentration of phosphorites. N. S. ULJANOV (Bull. Acad. Sci. U.R.S.S., 1938, Sér. Chim., 69—74).—Concentrates suitable as raw material for the production of fertilisers and containing 30—31% of  $P_2O_5$  are obtained by calcination and flotation. E. S. H.

Thermal concentration of Austrian phosphorites. R. MEISSNER (Berg u. Hüttenmänn. Monatsh., 1938, 86, 45—50, 57—67).—For the manufacture of fertiliser from phosphorite (analysis given)  $Na_2CO_3$  and  $CaCO_3$  are added, the mixture is sintered at 1200°, and steam passed over it. Various patented processes are summarised.  $Na_2SO_4$  may, but NaCl may not, be substituted for  $Na_2CO_3$ . The reactions involved are discussed. R. B. C.

Decomposition of phosphates by nitric acid. S. I. VOLFKOVITSCH, A. I. LOGINOV, and A. M. POLJAK (Bull. Acad. Sci. U.R.S.S., 1938, Sér. Chim., 101—109).—A review. R. T.

Geologic factors in the interpretation of fluorspar reserves in the Illinois-Kentucky field. L. W. CURRIER (U.S. Geol. Survey, 1937, Bull. 886B, 5—14).—The estimated average yearly consumption of fluorspar in the United States for some years to come is 175,000 tons. At this rate estimated reserves of the field will last 30—35 years. L. S. T.

Evaluation of commercial arsenious oxide by titration with iodine. P. LUNDMAN (Ind. Eng. Chem. [Anal.], 1938, 10, 387—388).—Direct titration with I by the usual method of commercial grades, 95—97 and 98—99%, of  $As_2O_3$  gives results approx.

0.5% high. Good results are obtained when the I solution is standardised against pure  $As_2O_3$  to which the common contaminants,  $As_2O_5$ ,  $Sb_2O_3$ , and Se, have been added. Lower-grade samples, 90—91%  $As_2O_3$ , containing appreciable amounts of  $SeO_2$ ,  $Sb_2O_3$ , and  $SO_3 + S$ , must be distilled. L. S. T.

**Electro-fusion of ammonia-synthesis catalyst.** V. G. TELEGIN and N. V. SIDOROV (J. Appl. Chem. Russ., 1938, 11, 588—596).—The catalyst is prepared by fusing magnetite,  $Al_2O_3$ , and  $KNO_3$  in an electric furnace. R. T.

**Chromium-vanadium catalyst with a high velocity coefficient.** I. E. ADADUROV and E. G. SEDASCHEVA (J. Appl. Chem. Russ., 1938, 11, 597—603).—80 c.c. of 0.1N- $Cr(NO_3)_3$  and 0.5 g. of  $SbCl_3$  in 25 c.c. of HCl are added to a solution of 80.6 g. of Na silicate in 180 c.c. of 0.1N- $NH_4VO_3$ . The ppt. is collected, washed, and dried at  $110^\circ$ , to yield a catalyst of  $SO_2$  oxidation, of unsurpassed activity. R. T.

**Titanium. XI. Hydrolysis of tetrachloride.** A. V. PAMFILOV, V. E. KISELEVA, and G. V. MILINSKAJA. **XII. Decomposition of titanomagnetite concentrate with sulphuric acid.** A. V. PAMFILOV, E. G. IVANTSHEVA, V. V. ALJAEVA, and I. M. SOBOLEVA (J. Appl. Chem. Russ., 1938, 11, 621—630, 631—637; cf. A., 1938, I, 319).—XI. Aq.  $TiCl_4$  is added to dil. aq.  $Na_2SO_4$  or  $(NH_4)_2SO_4$  at 60— $100^\circ$ , when hydrated  $TiO_2$  is obtained in theoretical yield, in a form which settles readily.

XII. 91% decomp. of the concentrate is obtained by heating at 140— $170^\circ$  with 1.5 vols. of 77%  $H_2SO_4$  for 40 min., when 0.5 vol. of  $H_2O$  is added and the mass is allowed to cool. R. T.

**Determination of carbon monoxide and oxygen in industrial gases.** H. H. MÜLLER-NEUGLÜCK (Wärme, 1938, 61, 280—285).—Factors affecting the accuracy of the  $I_2O_5$ -oleum method for determining CO in gases have been investigated. The oxidation of CO is influenced by the acid concn. and the fineness and exposed surface of the  $I_2O_5$ .  $H_2$  and  $CH_4$  are unattacked by the reagent;  $C_2H_6$  is affected only if >2% is present in gas containing 20% of CO;  $C_3H_8$  is strongly and  $C_4H_{10}$  almost completely oxidised. Conc. aq. alkaline hydroxyquinol (I) is not a satisfactory absorbent for  $O_2$  in the Orsat apparatus; only in dil. solution is it as effective as aq. alkaline pyrogallol (II).  $O_2$ -Multi-Rapid (III), a proprietary compound, is less effective than (II) containing pyrogallol and alkali in the ratio 1:1.5. Since (II) decomposes with the formation of CO in  $O_2$ -rich gases it can only be applied in the analysis of gases containing <25% of  $O_2$ . (I) and (III) do not break down in this way, and hence are superior to (II). Steel shavings saturated with (III) have much superior  $O_2$ -absorptive properties than have shavings saturated with (I) or (II). R. B. C.

**Rate of sublimation of Dryice in relation to its density.** L. RIEDEL (Z. Ver. deut. Ing., 1938, 82, 524).—Tabulated data for compressed solid  $CO_2$  (Dryice) ( $d$  1.24—1.55 g./c.c.) show that the rate of sublimation is independent of the  $d$  within the limits of experimental error. R. B. C.

**Industrial preparation of hydrogen.** L. JACQUÉ (Rev. l'Ind. Min., 1938, 257—268).—An outline of the methods for the manufacture of  $H_2$  and water-gas. P. G. McC.

**Apparatus for purification of inert gases and hydrogen.** W. WEIZEL (Z. tech. Physik, 1938, 19, 146—148).—Apparatus for the separation of the inert gases and  $H_2$  from gaseous mixtures by the passage of the electric discharge through the gas between Mg electrodes is described. The apparatus makes it possible to separate A, He, and Ne from a mixture with 75% of air, to obtain spectroscopically pure  $H_2$  from mixtures such as coal gas, and spectroscopically pure inert gases. The time for which it is necessary to pass the discharge is short. CO is removed from coal gas in 1 min., and  $N_2$  in 10 min. A. J. M.

**Obtaining elementary sulphur from the furnace gases in pyritic smelting.** T. D. AVERBUCH (Sotz. Rekonstr. i Nauka, 1935, No. 4, 168).—The gases contain up to 40% of S +  $SO_2$ . The S could be recovered by changing the existing side-charging of the furnace to top-charging, so as to obtain perfect tightness of the furnace and uniformity of operation. CH. ABS. (e)

**Oxidation of phosphorus vapour and hydrogen phosphide with oxygen in presence of nitric acid.** I. S. ROSENKRANTZ [with M. O. DEMBO] (Bull. Acad. Sci. U.R.S.S., 1938, Sér. Chim., 75—99).—P vapour and  $PH_3$  may be removed from (CO +  $H_2$ ) fuel gas by oxidation with  $HNO_3$ . The N oxides formed are absorbed by alkali. Expenditure of  $HNO_3$  is not great, as oxidation continues through the agency of  $O_2$ , NO acting as a catalyst.  $H_3PO_4$ ,  $H_2PO_3$ , and  $H_3PO_2$  accumulate in the  $HNO_3$  and may be converted into  $H_3PO_4$  by heating the mixture at 90— $92^\circ$ . The liquid may be converted into phosphate or mixed phosphate-nitrate fertilisers. E. S. H.

**Reaction agglomerating furnace.**—See I. Pyrites from coal.  $NH_3$  from gas. S from fuel gases.  $NH_3$  as fuel.—See II.  $Na_2S_2O_4$  in reduced indigo preps.—See IV. K in glass industry.—See VIII.  $FeSO_4$  from pickling acid waste. Behaviour of Cr-Ni in  $H_3PO_4$  solutions.—See X. Electrolysis of alkali chlorides.—See XI.  $TiO_2$ .—See XIII. Fertilisers. Determining  $H_2O$ -sol.  $PO_4'''$ , K, Mg, and Mn in fertilisers, and citrate-insol.  $PO_4'''$  in mixtures.—See XVI. Determining  $O_2$  in air. Detecting HCN.—See XXIII.

See also A., I, 466, Prep. of neutral Li phosphate. 467, Prep. of very pure  $O_2$ , and of neutral  $ZnCO_3$ . 468, Prep. of rare earths, and of  $SOBr_2$ . 469, Mo-blue.

#### PATENTS.

**Denitration of waste sulphuric acid mixtures or dilute nitric acid.** W. BUSCHING (B.P. 476,136, 10.11.36).—Part of the  $H_2SO_4$  vapour, generated by boiling  $H_2SO_4$  in a closed still provided with a stirrer, is introduced into the waste acids in a denitrating column kept at  $170^\circ$ , whence the dil.  $H_2SO_4$  is conducted to another column and conc. by another part of the vapours. The conc. acid is passed to the still



through a pipe of such diameter that the acid leaving the lower end has the same concn. as that in the still. The  $\text{HNO}_3$  vapours and  $\text{NO}$  are drawn off from the denitrating tower by a fan and led to a cooler, where they are condensed.

W. J. W.

**Manufacture of sodium hydroxide.** H. LAWARRÉE (B.P. 477,230, 9.12.36. Fr., 18.12.35 and 9.7.36).—A Na salt ( $\text{NaCl}$ ) is caused to react with a metal oxalate (I) ( $\text{CuC}_2\text{O}_4$ ) in presence of  $\text{H}_2\text{O}$  and a volatile alkali ( $\text{NH}_3$ ). The pptd.  $\text{Na}_2\text{C}_2\text{O}_4$  in aq. solution or suspension is causticised with  $\text{CaO}$  or  $\text{Ca}(\text{OH})_2$ , yielding  $\text{NaOH}$  solution and pptg.  $\text{CaC}_2\text{O}_4$ . The latter is removed from the mother-liquor and caused to react with the mother-liquor remaining from the first step after part of the  $\text{NH}_3$  in this has been removed by distillation and sufficient acid has been added to dissolve sol. constituents of the resulting ppt., the original (I) being thus obtained. Alternatively, acid may be added to the ppt. obtained by removal of volatile alkali and the resulting mixture treated with the  $\text{CaC}_2\text{O}_4$ . The final mother-liquor may be distilled with  $\text{CaO}$  to recover residual volatile alkali.

W. J. W.

[Manufacture of] anhydrous sodium sulphite. R. W. JAMES. FROM A. R. MAAS CHEM. CO. (B.P. 485,215, 23.1.37).—Aq.  $\text{Na}_2\text{CO}_3$  (*d* 1.17) is treated with  $\text{SO}_2$  in an absorbing tower until it becomes 0.4N in acid, and the liquor is filtered from  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , etc., boiled to remove  $\text{CO}_2$ , and neutralised with aq.  $\text{NaOH}$  to ppt.  $\text{Fe}(\text{OH})_3$  etc., which are filtered off. The solution is then evaporated under reduced pressure (at  $55^\circ$ ) to incipient crystallisation, when it is transferred to a salt-catch and blown with hot air until it is nearly dry; the product is centrifuged, rinsed with a little  $\text{H}_2\text{O}$ , and dried in a rotary dryer.

L. C. M.

**Production of crystallised anhydrous sodium metasilicate.** C. L. BAKER, ASSR. TO PHILADELPHIA QUARTZ CO. (U.S.P. 2,067,227, 12.1.37. Appl., 25.11.33).—An aq. solution of  $\text{Na}_2\text{SiO}_3$  (of  $\leq 50\%$  concn.) is conc. at  $72^\circ$  to  $\leq 57\%$  concn. and then agitated and heated to  $>72^\circ$  ( $120^\circ$ ), when the anhyd. salt crystallises.  $\text{NaOH}$  or  $\text{Na}_2\text{CO}_3$  is added to absorb excess of  $\text{H}_2\text{O}$ , the mixture yielding, on further heating, an anhyd. product.

F. M. L.

**Manufacture of alkali subsilicates.** PENNSYLVANIA SALT MANUFG. CO. (B.P. 477,518 [A] and 477,578 [B], 31.3.36. U.S., 10.4.35).—(A) Finely-powdered  $\text{SiO}_2$ , powdered  $\text{NaOH}$  ( $\text{Na}_2\text{O} : \text{SiO}_2 \leq 1 : 1$  mol.), and preferably  $\text{H}_2\text{O}$  ( $<7\%$  of the total wt.) are heated in a stirred mixer. The mass becomes viscous ( $175^\circ$ ), then plastic ( $200$ — $225^\circ$ ), and finally, after evolution of steam, is converted into a granular monohydrate which may be used as such, or is heated, *e.g.*, in a rotary dryer at  $450^\circ$ , to form an anhyd., free-flowing,  $\text{H}_2\text{O}$ -sol. product. The use of  $\text{NaOH}$  in the approx. ratios 1.25, 1.5, or 2.0 mols. of  $\text{Na}_2\text{O} : 1$  of  $\text{SiO}_2$  to produce substantially  $\text{Na}_2\text{SiO}_3$ ,  $\text{Na}_6\text{Si}_2\text{O}_7$ , or  $\text{Na}_4\text{SiO}_4$ , respectively, is claimed. For use as detergents  $\text{Na}_3\text{PO}_4$  or  $\text{Na}_2\text{CO}_3$ , *e.g.*, may be incorporated. (B) The ratio  $\text{Na}_2\text{O} : \text{SiO}_2$  may be adjusted, *e.g.*, to 1 : 1 by mixing the required amount of water-glass with the anhyd. product and drying at  $\sim 200^\circ$ .

I. C. R.

**Manufacture of alkali thiocyanates.** E. L. HELWIG, ASSR. TO ROHM & HAAS CO. (U.S.P. 2,067,606, 12.1.37. Appl., 9.4.36).—Waste products from coal-gas manufacture, *e.g.*, spent alkaline scrubber liquor containing  $\text{Na}_2\text{S}_2\text{O}_3$ , is boiled with finely-divided S,  $\text{Zn}(\text{CN})_2$ , and  $\text{Na}_2\text{CO}_3$ ; the  $\text{ZnCO}_3$  is filtered off and the  $\text{NaCNS}$  crystallised from the filtrate. F. M. L.

**Manufacture of double or treble (calcium) superphosphate.** W. H. MACINTIRE (U.S.P. 2,067,538, 12.1.37. Appl., 5.1.34).—High-grade, finely-divided limestone (100) and  $\sim 85\%$   $\text{H}_3\text{PO}_4$  (230 pts.) are agitated for  $\sim 2\frac{1}{4}$  min., the mixture is than set aside for 8—10 min., and again agitated until a granulated product is formed.

F. M. L.

**Manufacture of stable calcium thiosulphate.** SCHERING-KAHLBAUM A.-G. (B.P. 480,414, 27.8.36. Addn. to B.P. 467,139; B., 1937, 1337).—A stable 10% aq. solution of  $\text{CaS}_2\text{O}_3$  is pptd. with sufficient  $\text{H}_2\text{O}$ -miscible org. solvent ( $\text{EtOH}$ ,  $\text{MeOH}$ , dioxan) to give a liquid consisting of 50% of  $\text{H}_2\text{O}$ .

W. J. W.

**Manufacture of calcium carbide.** C. H. HARRISON (B.P. 477,100, 13.8.36).— $\text{CaS}$  obtained by heating  $\text{CaSO}_4$  with C at  $800$ — $1000^\circ$  is heated with carbonaceous material (coke, brown coal) at  $1600$ — $1750^\circ$ , the reaction being made continuous. Impurities ( $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ ) in the  $\text{CaSO}_4$  do not interfere with the subsequent conversion of the  $\text{CaS}$ .

W. J. W.

**Manufacture of calcium carbide.** A.-G. F. STICKSTOFFDÜNGER (B.P. 479,763, 10.8.36. Ger., 9.8.35).—The interaction of  $\text{CaO}$  and C is carried out in two stages in a form of electric furnace (claimed) having two hearths separated by a dividing wall with an opening near the bottom. A mixture of  $\text{CaO}$  and C is introduced into the smelting hearth in such proportions that a molten product containing 60—70% of  $\text{CaC}_2$  is obtained, and this is caused to flow into the second hearth containing a high [C], so that the final product contains 85—95% of  $\text{CaC}_2$ .

W. J. W.

**Removal of salts from [aqueous] solutions.** PERMUTIT A.-G. (B.P. 478,134, 10.7.36. Ger., 27.7.35).—The solutions, *e.g.*, boiler feed- $\text{H}_2\text{O}$  or sugar solutions, are passed over a carbonaceous H-exchange material, and the resulting acid liquor is passed through a bed of granular insol. oxide or hydroxide gel, *e.g.*,  $\text{Fe}_2\text{O}_3$ , which forms an insol. basic compound with the acid. Alternatively, the acid solution may be mixed with a suspension of the oxide or hydroxide (or these may be produced *in situ* by passing the liquid over metals, alloys, or voltaic couples; with Fe, oxidation is necessary) and then filtered. The gel may be regenerated by treatment with alkali.

I. C. R.

**Obtaining beryllium fluoride free from oxide.** SERI HOLDING SOC. ANON., ASSEES. OF SOC. ANON. PROCESSI PRIVATIVE INDUSTRIALI (B.P. 485,711, 19.7.37. It., 15.7.36).—A mixture of  $\text{Be}(\text{OH})_2$  (M) with  $\text{NH}_4\text{HF}_2$  (2M) is heated to  $450$ — $500^\circ$  during 3— $3\frac{1}{2}$  hr. in a vessel (preferably of the metal with which the Be is subsequently to be alloyed); a stream of  $\text{CO}_2$  carries away the  $\text{H}_2\text{O}$  and  $\text{NH}_4\text{F}$ , the latter being condensed in a cold chamber and treated with HF to regenerate  $\text{NH}_4\text{HF}_2$ .

L. C. M.

**Production of a magnesium oxychloride suitable for chemical reactions.** I. G. FARBENIND. A.-G. (B.P. 480,698, 25.8.36. Ger., 4.1.36).—Basic Mg oxychloride is obtained as a powder by heating an intimate mixture of MgO (*e.g.*, 20 pts. by wt.) and ground hydrates of MgCl<sub>2</sub> in the desired proportion (*e.g.*, MgCl<sub>2</sub>·6H<sub>2</sub>O 50 pts.) in an internally heated rotary furnace, in countercurrent to the flame gases, so that the product is discharged at 130° (140° for lower hydrates). The product may be moistened with H<sub>2</sub>O or aq. MgCl<sub>2</sub> and moulded, or the materials may be moulded before heating. I. C. R.

**Extraction of alumina from bauxite.** T. R. HAGLUND (B.P. 478,489, 29.6.37. Swed., 23.10.36).—Bauxite (>5-mm.) is introduced into the top of a pressure tower, and alkaline liquor, *e.g.*, NaOH-NaAlO<sub>2</sub> solution, from which Al(OH)<sub>3</sub> has been recovered, at the bottom, preferably tangentially and/or with stirring, while liquor containing sludge in suspension is removed (continuously) from the top, the temp. being 160–180° and the pressure > the v.p. of the solution. Liquor is also removed from the top (or middle) section and reintroduced at the lower section. The tower is arranged thus: diameter of the top > the middle > the lower section, and the flow of liquid up the tower is such that coarse particles are held in suspension in the lower section but only fine sludge particles in the top section. Sludge removal is interrupted for a time after introducing a fresh charge of bauxite. Two or more towers may be arranged in series, fresh liquid being introduced into that tower in which reaction has proceeded furthest. The hot liquor containing the sludge passes to another tower where the reaction is continued, and then, after removal of sludge, *e.g.*, by centrifugal action, Al(OH)<sub>3</sub> is pptd. by stirring. I. C. R.

**Manufacture of copper oxychloride.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 480,697, 25.8.36).—3Cu(OH)<sub>2</sub>·CuCl<sub>2</sub> (I) is prepared in a granular, filterable form by oxidation of CuCl (*e.g.*, 90 g./l.) in, *e.g.*, aq. NaCl (150–200 g./l.) by O<sub>2</sub> or (enriched) air in presence of sulphates (2–5 g./l., calc. as S) of other elements (K, Zn), at 60–80° and preferably in presence of (I) from a previous operation. I. C. R.

**Production of lead arsenate.** "MONTECATINI," SOC. GEN. PER L'IND. MIN. ED AGRIC. (B.P. 477,431, 1.2.37. It., 1.2. and 17.7.36).—An aq. suspension of PbSO<sub>4</sub> (*e.g.*, chamber white-Pb) is warmed and agitated with excess of aq. Na<sub>3</sub>AsO<sub>4</sub> (obtained, *e.g.*, during Pb refining) to form Pb<sub>3</sub>(AsO<sub>4</sub>)<sub>2</sub>, which is converted by addition of acid (H<sub>2</sub>SO<sub>4</sub>) into substantially pure PbHAsO<sub>4</sub> (30% of As<sub>2</sub>O<sub>5</sub>). After filtration, excess of As is pptd. as Ca<sub>3</sub>(AsO<sub>4</sub>)<sub>2</sub> by CaO. Na<sub>2</sub>SO<sub>4</sub> is recovered from the mother-liquor. I. C. R.

**Manufacture of catalysts for organic syntheses.** E. I. DU PONT DE NEMOURS & Co. (B.P. 478,085, 26.10.36. U.S., 26.10.35).—BF<sub>3</sub> catalysts, *e.g.*, for the production of aliphatic acids by interaction of CO with alcohols or olefines, are prepared by the interaction of liquid HF with a boric acid or a borate, preferably at 0–10°. Acids, *e.g.*, conc. H<sub>2</sub>SO<sub>4</sub>, may be present. I. C. R.

**Catalysts and methods of preparing and using them.** AMER. PLATINUM WORKS (B.P. 477,026, 16.3.36. U.S., 20.3.35).—A contact mass for SO<sub>3</sub> synthesis is prepared by wetting a rigid porous carrier, such as SiO<sub>2</sub> gel, meerschäum, etc., with a solution of Pt or 9:1 Pt:Rh, preferably containing alcohols to reduce its  $\gamma$ , drying, and heating at >600° so as to produce an active deposit directly associated with the pore surface of the carrier and amounting to 0.1–0.5 wt.-%, then bringing the material in contact with a solution or suspension of a promoter substance, and re-drying so as to leave a deposit of the latter on the active surface but not chemically combined or mechanically mixed with it. The promoter may consist of oxides of one or more metals of groups IV–VII, or of sulphates of one or more metals of groups I–III, or of mixtures of these oxides and sulphates. Fe<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>, MgSO<sub>4</sub>, and MgSO<sub>4</sub> + As<sub>2</sub>O<sub>5</sub> are specifically claimed as promoters. [Stat. ref.] A. R. PE.

**Fluorescent substance.** DEGEA A.-G. (AUVERGES.) (B.P. 485,232, 22.3.37. Ger., 21.3.36).—Yellow-fluorescent material is prepared by heating a mixture of ZnO and SiO<sub>2</sub> (ZnO:SiO<sub>2</sub> = 1:1.5–5), containing an activating agent (*e.g.*, 1.3 wt.-% of Mn silicate), at 1300–1600° for 30 min. L. C. M.

**Inorganic luminescent substance.** M. SERVIGNE (B.P. 485,373, 18.11.36).—The use of mixtures of phosphorogen, *e.g.*, Li<sub>2</sub>O, or Ca + Na tungstates, with diluent, *e.g.*, CaMoO<sub>4</sub> and PbMoO<sub>4</sub>, and a stabiliser, *e.g.*, Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>, is claimed. L. C. M.

**Purification of hydrogen.** H. E. MOORE, Assr. to CAPITAL CITY PRODUCTS Co. (U.S.P. 2,074,311, 16.3.37. Appl., 7.9.34).—Commercial H<sub>2</sub> from the steam-Fe or other process and containing 1% of CO is freed from H<sub>2</sub>S and CO<sub>2</sub> and then passed over a catalyst consisting of Cu 5–15, ThO<sub>2</sub> 2, and Ni 93–83%, whereby the CO is converted into CH<sub>4</sub> and H<sub>2</sub>O. D. M. M.

(A, B) **Production of hydrogen peroxide (A) from solutions of per-compounds.** H. SCHMIDT (B.P. 476,262–3, 12.6.36. Ger., [A] 22.6.35, [B] 26.9.35).—(A) Distillation from solutions of per-compounds is discontinued before the residues reach a concn. beyond which further amounts of H<sub>2</sub>O<sub>2</sub> cannot be distilled without decomp., the remaining solutions being treated to yield the compounds containing O<sub>2</sub> as cryst. per-salts. Separation of part of these per-salts may be effected during distillation by lowering the acidity of the solutions. (B) In a closed-cycle process, electrolytically obtained solutions of compounds which split off active O are purified by subjecting them, prior to distillation, to supersaturation or cooling so as to separate per-compounds. These are ultimately replaced by adding to the solutions equiv. amounts of compounds which will yield the per-compounds again on electrolysis. W. J. W.

**Cartridges and like moulded bodies for generation of pure oxygen for respiratory purposes.** I. G. FARBENIND. A.-G. (B.P. 476,254, 5.6.36. Ger., 27.6.35).—The moulded O<sub>2</sub>-generating mass (chlorates or perchlorates) is surrounded with a material, such

as slag wool, impregnated with a catalyst, such as  $MnO_2$ , with or without  $CuO$ , to convert the  $CO$  evolved on heating into  $CO_2$ . The material may also be impregnated with an alkali or alkaline-earth metal to absorb the  $CO_2$  thus formed, as well as that originally in the gas mixture, and generate  $O_2$  during such absorption.

W. J. W.

**Recovery of sulphur dioxide from gases.** METALLGES. A.-G. (B.P. 485,955, 6.5.37. Ger., 23.6.36. Addn. to B.P. 435,116; B., 1936, 146).—In the process of the prior patent,  $Na_2SO_4 \cdot 10H_2O$  (I) is recovered continuously from the effluent liquor by withdrawing a portion of it, cooling it to  $\sim 2^\circ$ , collecting the (I), and returning the mother-liquor to the absorber.

L. C. M.

**Recovery of sulphur dioxide from gases.** R. F. BACON, Assec. of R. FANELLI (B.P. 478,544, 24.12.36. U.S., 24.12.35).— $SO_2$  is absorbed in a solution of  $(NH_4)_2SO_3$  containing an appreciable amount of a free acid weaker than  $SO_2$  and, preferably, not easily volatile, e.g., lactic acid (I), or in a solution of a salt of a weak acid and the same or another weak acid, e.g.,  $NH_4$  lactate and (I). Loss of  $NH_3$  during the recovery of  $SO_2$  by boiling is prevented, and the recovery is much more rapid.

I. C. R.

**Production of concentrated sulphur dioxide.** METALLGES. A.-G. (B.P. 480,519, 19.5.36. Ger., 5.7.35).—Gases containing  $SO_2$  are treated with a mixture of  $H_2O$  and an org. base (I), e.g., an arylamine such as a xylydine, with addition of an auxiliary substance (II), such as hydroxides, oxides, carbonates, or neutral or acid sulphates of alkali metals, which decompose the sulphate of (I) and form inorg. sulphates. The amount of (II) must be such as to give the mixture an acidity  $\succ$  that of a solution of the same concn. of the bisulphite of the particular (I) used, so that autoxidation of  $SO_2$  to  $SO_3$  is avoided. The mixture is ultimately heated to expel  $SO_2$ .

W. J. W.

**Blast-roasting  $CaO$ . Retorts for distilling  $Ca(OAc)_2$ . Cooling mixtures.**—See I. Treating gas-scrubbing liquor.—See II. Urea.—See III. Making cement and  $CaO$ .—See IX. Treating nickeliferous Fe ores. Treating Pb-bearing ore.—See X. Electric furnace for making Na silicate. Hyposulphites. Electrolytic cell for  $Cl_2$ .—See XI. Washing media.—See XII.  $CaCO_3$  pigment. Coloured Ti pigments.—See XIII. Fertilisers.—See XVI. Blasting explosives.—See XXII. Emulsifiable  $SO_2$  solutions.—See XXIII.

### VIII.—GLASS; CERAMICS.

**Tunnel-kiln firing. II. Choosing a tunnel kiln for pottery firing.** N. WILSON (Trans. Ceram. Soc., 1938, 37, 255—278).—The various fuels available for tunnel-kiln firing are compared and factors requiring consideration in choosing a tunnel kiln discussed. Preference is given to direct firing for most pottery articles in a medium- or large-sized tunnel kiln, to a muffle with open placing for glost firing in most factories, and to the electric kiln for enamel-kiln firing.

A. L. R.

**Potash in the glass industry.** A. N. FINN (Ind. Eng. Chem., 1938, 30, 891—892).— $K_2O$  glass is less subject to weathering than  $Na_2O$  glass.  $K_2O$  is also essential for a no. of coloured glasses and is preferred for optical glass though it is not absolutely necessary for this purpose. Specifications of K compounds for glass making require very low Fe and Cl' contents; in some cases  $SO_4$ ' is also objectionable.

C. I.

**Influence of sodium nitrate and arsenious oxide on reactions occurring during melting of glass.** M. A. BESBORODOV, N. D. ZAVJALOV, T. A. LADE, and G. M. MINKIN (J. Soc. Glass Tech., 1938, 22, 218—234T).—The thermal decomp. were studied by measuring the loss in wt. at const. temp.  $As_2O_3$  volatilises readily below  $300^\circ$ , but when heated with  $NaNO_3$  at  $600^\circ$  or with  $Na_2CO_3$  at  $500^\circ$  it forms sol. compounds. A batch giving the glass  $SiO_2$  75,  $CaO$  10,  $Na_2O$  15% melted much more readily at  $800^\circ$  when  $NaNO_3$  was a batch constituent and also in presence of cullet. Free  $NaOH$  was formed in the  $Na_2CO_3 + CaCO_3 + SiO_2$  systems at  $400$ — $800^\circ$ .

J. A. S.

**Application of colloidal graphite to glass-bottle moulds.** J. S. MARSHALL and W. G. MACKAY (J. Soc. Glass Tech., 1938, 22, 214—217T).—A new mould was provided with a good "skin" (which did not need oiling) by thoroughly degreasing, heating to  $100^\circ$ , painting with Aquadag (paste 1 pt., distilled  $H_2O$  2 pts.), and polishing as soon as the layer of graphite had dried. A second application was made.

J. A. S.

**Exact and rapid polishing of glass, quartz, and agate on a hard polisher.** I. B. GREBENSCHTSCHIKOV and S. V. NESMELOV (J. Opt. Mech. Ind. U.S.S.R., 1934, 5, No. 6, 18).—The polisher is of glass, Ni, or cast Fe.  $Cr_2O_3$  used as a polishing powder is obtained by igniting  $K_2Cr_2O_7$  with S, washing, and re-igniting at temp. suitable to the purpose required. Org. substances forming emulsions with  $H_2O$  are used for lubrication.

CH. ABS. (p)

**Spectral-transmissive properties and use of coloured eye-protective glasses.** W. W. COBLENTZ and R. STAIR (U.S. Bur. Stand., 1938, Circ. C421, 28 pp.).—The characteristics of many commercial coloured glasses are recorded. The problems of the elimination of glare, distortion of colours, confusion of traffic signals, the danger of coloured glasses for night-driving, and the need for standards of shade, are discussed in detail.

J. A. S.

**Influence of tensions on variation of the refractive index of a glass.** (MME.) N. WINTERKLEIN (Compt. rend., 1938, 206, 1627—1629).—Experiments are described in which unstrained specimens of a flint and a borosilicate glass were heated slowly to different temp. ( $\theta$ ), cooled quickly in the atm., and their  $n$  was measured. As  $\theta$  rises,  $n$  shows an initial rapid decrease ( $\Delta n_1$ ) due to the  $\alpha \rightarrow \beta$  transformation, followed above the softening point by a much smaller increase ( $\Delta n_2$ ) attributed to mechanical tensions in the specimen. A specimen originally in a strained state gives only a smaller initial decrease, representing the difference  $\Delta n_1 - \Delta n_2$ .

A. J. E. W.

**Viscosity measurements of the  $\text{Na}_2\text{O}$ - $\text{SiO}_2$  glasses by E. Preston.** G. HEIDTKAMP and K. ENDELL (J. Soc. Glass Tech., 1938, 22, 235—236T).—Exception is taken to certain conclusions drawn by Preston (B., 1938, 782). J. A. S.

**Viscosity of the soda-silica glasses at high temperatures.** E. PRESTON (J. Soc. Glass Tech., 1938, 22, 237—242T).—Misinterpretations by Heidtkamp and Endell (see preceding abstract) are corr. and the author's belief in the presence of compounds in molten silicates is further supported. J. A. S.

**Molar volumes of glasses.** O. KNAPP (Keram. Rund., 1938, 46, 231—234).—Biltz (B., 1933, 915) has claimed that a glass is composed of the most siliceous cryst. silicates known in the particular oxide systems, together with the remaining  $\text{SiO}_2$  in the free state, and that proof of this hypothesis exists in the agreement between the calc. and measured mol. vols. of the glass. The author shows that the hypothesis is founded on an erroneous basis. A case is cited where the disilicate is the highest one known, and yet equally consistent results are obtained if the glass is regarded as containing di-, tri-, or tetra-silicate. J. A. S.

**Influence of iron oxide on stability of enamels.** F. H. SCHULZ (Keram. Rund., 1938, 46, 257—258).—The acid-resistance (to boiling 4%  $\text{AcOH}$ ) of a "commercial enamel" ( $\text{SiO}_2$  54.0,  $\text{Fe}_2\text{O}_3$  6.4,  $\text{CaO}$  1.6,  $\text{Na}_2\text{O}$  13.2%) and an "acid-resisting" enamel ( $\text{SiO}_2$  54.7,  $\text{Fe}_2\text{O}_3$  0.3,  $\text{CaO}$  11.05,  $\text{Na}_2\text{O}$  15.15%) were greatly decreased by addition to the mill of 10% of  $\text{Fe}_2\text{O}_3$ . The min. amount of  $\text{Fe}_2\text{O}_3$  should therefore be left as a mill addition for colour matching. J. A. S.

**Recent opacifiers for white covering enamels.** H. KOHL (Z. Ver. deut. Ing., 1938, 82, 546—548).—Developments in the use of antimonates,  $\text{ZrO}_2$ , and  $\text{CeO}_2$  for opacifying finishing coats of vitreous enamel, and in gas opacifiers, are reviewed.  $\text{CeO}_2$  obtained from monazite sand and gas opacifiers are replacing  $\text{SnO}_2$  in Germany. R. B. C.

**Opacification [of enamels] with the oxides of zirconium and cerium.** W. SCHULZ (Keram. Rund., 1938, 46, 277—279).—The theory and practice of the use of these opacifiers are discussed. J. A. S.

**Ceramic raw materials for dry-plastic working.** C. SCHUSTERIUS (Naturwiss., 1938, 26, 394—398).—The use of steatite as a raw material for "pressed" ceramic work is described. The difficulties encountered in using mica for this purpose are mentioned, and the advantages of sericite are described. A. J. M.

**Magnesia and special [ceramic] bodies.** F. DETTMER (Keram. Rund., 1938, 46, 253—256).—The formulæ, nature, and properties of various bodies composed of  $\text{MgO}$  minerals and of  $\text{TiO}_2$  are briefly described. J. A. S.

**Metallising in the pottery industry.** G. W. JARMAN, jun. (Bull. Amer. Ceram. Soc., 1938, 17, 328—329).—The applications of methods of metallising by spraying are briefly described. J. A. S.

**Steelworks' refractories.** D. PETIT (Rev. Mét., 1938, 35, 250—262, 296—308).—The properties and

practical applications of  $\text{SiO}_2$ ,  $\text{MgO}$ ,  $\text{Cr}_2\text{O}_3$ - $\text{MgO}$ , and some special refractories are described in detail.

J. A. S.

**Factors influencing durability of chrome-magnesite bricks in basic open-hearth steel-making furnaces.** W. HUGILL and A. T. GREEN (Trans. Ceram. Soc., 1938, 37, 279—295).—From analyses and microscopical examination of bricks from the roof, side-walls, and end-blocks of a basic open-hearth furnace the mechanism of the disintegration of chrome-magnesite bricks in the open-hearth furnace is suggested as follows:  $\text{Fe}$  oxide is absorbed by the hot face of the bricks, in which zone the chromite grains grow by taking up  $\text{Fe}_3\text{O}_4$  into solid solution. The matrix, relatively rich in  $\text{MgO}$ , is largely converted into the less refractory  $\text{MgO}$ ,  $\text{Fe}_2\text{O}_3$  and metasilicates. Strains induced by the growth of the chromite grains cause (1) disintegration of the working face of the brick, (2) the mobile matrix to move to and solidify in a zone at a lower temp. Following the disintegration of the hot face the temp. of the second zone increases and liquefaction occurs, resulting in a more serious breakdown of the surface. A. L. R.

**Effect of fluxes on cupola linings.** C. K. DONOHO and J. T. MACKENZIE (Bull. Amer. Ceram. Soc., 1938, 17, 317—321).—The mechanism of slag attack is discussed with reference to the testing of cupola refractories. The correct fluidity of a slag is of primary importance. The high  $\eta$  of a slag may increase the attack of cupola linings by increasing the  $\text{Fe}_2\text{O}_3$  content of the slag (inefficient protection of the metal surface), deflecting the air blast directly against the lining, and preventing the mixing of the slag and so the dilution of the corrosive constituents.  $\text{MgO}$ ,  $\text{CaF}_2$ , and  $\text{Na}_2\text{CO}_3$  are flux constituents which increase the life of the refractory. J. A. S.

**Behaviour of refractory materials under torsion at different temperatures.** III. (A) Modifications to apparatus. (B) Ultimate strength of kaolin and kaolin-quartz mixtures. A. L. ROBERTS and J. W. COBB (Trans. Ceram. Soc., 1938, 37, 296—311; cf. B., 1936, 835).—Modifications made to extend the scope of the original apparatus (B., 1933, 228) to the measurement of ultimate shear strength at high temp., and to permit the use of a simple type of test-piece, are described. At 20—800° the shear strength ( $S$ ) of previously-burned kaolin was found to change little, and the rigidity modulus ( $R$ ) not at all; with kaolin-quartz test-pieces both  $S$  and  $R$  increased considerably and to approx. the same extent. At 800—1100° the development of plasticity in both materials was marked by a pronounced increase in the  $S/R$  ratio, and it is concluded that the increased resistance to spalling which accompanies the development of plasticity in a refractory material at high temp. (e.g., 800—1100°) is due, not only to the reduction in  $R$ , but also to the absence of a corresponding change in the effective  $S$ . A. L. R.

**Radiation of some silico-alumina refractories.** A. BARITEL (Chaleur et Ind., 1938, 19, 237—244, 299—311).—The black-body radiation from five commercial refractory materials containing  $\text{SiO}_2$  97—6

and  $\text{Al}_2\text{O}_3$  1—91% at 950—1200° was investigated. Details are given of the apparatus and technique employed, and data are tabulated. R. B. C.

**Conception of d and ceramics.**—See I. K salts as raw materials.—See VII. Steel for enamelling. Adhesion of enamel to Fe. Refractories in Fe and steel industry.—See X.

See also A., I, 446, Structure of  $\text{SiO}_2$  glass and of the K silicate glasses. 477, Determining vol. resistivity of insulating glasses.

#### PATENTS.

**Melting [of glass].** S. A. FORSTER, Assr. to HARTFORD-EMPIRE Co. (U.S.P. 2,078,794—5, 27.4.37. Appl., [A] 2.7.32, [B] 6.9.32. Renewed [B] 13.10.36).—(A, B) The material progresses by gravity down a sloping hearth from a heap of new material at the hottest point; after leaving the heap the material flows in thin layers in the same direction as the heating gases until it arrives at a working pool, the roof inclining downwards more steeply than the hearth. (B) The thin layer of freshly molten material is merged gradually with the bath without abrupt change in direction. B. M. V.

**Manufacture of borosilicate glasses highly resistant to alkaline liquids.** JENAER GLASWERK SCHOTT & GEN. (B.P. 477,698, 9.7.36. Ger., 22.7.35).—Glasses improved by addition of  $\text{BeO}$  contain  $\text{SiO}_2$  etc.  $\geq 90$ ,  $\text{Al}_2\text{O}_3$   $\geq 10$ ,  $\text{K}_2\text{O}$  ( $\text{Na}_2\text{O}$  etc.)  $\geq 10$ ,  $\text{BeO}$  0.1—15,  $\text{R}_2\text{O}_3$  + RO (other than  $\text{BeO}$ ) 3—70,  $\text{R}_2\text{O}_3$   $\geq 20\%$ . *E.g.*,  $\text{SiO}_2$  72,  $\text{Na}_2\text{O}$  5,  $\text{BaO}$  2.5,  $\text{BeO}$  3,  $\text{B}_2\text{O}_3$  15,  $\text{Al}_2\text{O}_3$  2.5%. J. A. S.

**Heat-absorbing glasses.** JENAER GLASWERK SCHOTT & GEN. (B.P. 475,336, 3.7.37. Ger., 13.7.36. Addn. to B.P. 387,778; B., 1933, 307).— $\text{Sb}_2\text{O}_3$  or  $\text{PbO}$  is introduced into the glasses previously claimed (cf. *loc. cit.*), which are melted under oxidising conditions. If heat absorption and transparency to visible light are both desired, the  $\text{B}_2\text{O}_3$  content should be  $\geq 10\%$ . J. A. S.

**Refractory glasses.** GEN. ELECTRIC Co., LTD., and J. H. PARTRIDGE (B.P. 476,400, 26.8.36. Addn. to B.P. 426,129; B., 1935, 496).—Glasses suitable for high-pressure Hg-vapour lamps and having a transmission extending further into the ultra-violet region and a transmission to both the ultra-violet and visible light which does not fall off so rapidly during the life of the lamp, contain  $\text{B}_2\text{O}_3$   $< 7$ , Zn 5—20, Mg  $\geq 2$ , and/or  $\text{P}_2\text{O}_5$   $\geq 2\%$ . J. A. S.

**Glass insulators for electrical purposes.** CORNING GLASS WORKS, Assecs. of J. T. LITTLETON (B.P. 476,401, 20.1.37. U.S., 31.1.36).—The head of a glass "suspension-type" insulator is tempered and the skirt left untempered so that breakage of the skirt does not shatter the whole insulator. Chilling may be effected by jets of air or molten salts (*e.g.*, the eutectic of  $\text{KNO}_3$  +  $\text{NaNO}_3$ ). J. A. S.

**Optical glass composition.** M. R. SCOTT, Assr. to BAUSCH & LOMB OPTICAL Co. (U.S.P. 2,064,361, 15.12.36. Appl., 28.3.35).—A coloured glass,  $n$  1.50—1.55 and dispersion ( $v$ ) 51.0—58.5, contains compounds of Pb, Mn, and Ce. *E.g.*, 5.5% of  $\text{PbO}$ ,

$> 1$  (2)% of Mn compound ( $\text{MnO}_2$ ), and  $< 2$  (1%) of  $\text{CeO}_2$  may be present. J. A. S.

**Manufacture of photo-luminescent glass.** I. G. FARBENIND. A.-G. (B.P. 476,945, 18.6.36. Ger., 18.6.35. Addn. to B.P. 460,210; B., 1937, 346).—The glass, containing phosphates to the extent that the  $\text{P}_2\text{O}_5$  content is  $< 20\%$  of the acid constituents +  $\text{SnO}_2$  or the oxide of another excitable metal, is fused in an oxidising or neutral atm. This glass may be flashed with another luminescent or non-luminescent glass. J. A. S.

**Tempering of glass.** R. MONNIER, Assr. to Soc. ASSUREX LE "ROI DES VERRES DE SECURITE" MAGNIEN, MONNIER & Co., and L. A. E. PETIT (U.S.P. 2,078,541, 27.4.37. Appl., 9.1.35. Fr., 16.1.34).—Apparatus is claimed. B. M. V.

**Manufacture of tempered articles of lime-soda glass.** CORNING GLASS WORKS, Assecs. of W. W. SHAVER and H. K. MARTIN (B.P. 477,460, 30.6.36. U.S., 1.7.35).—The glass is chilled (in a bath of inorg. salts, *e.g.*,  $\text{NaNO}_3$  +  $\text{KNO}_3$  at 405°) to a degree of temper just insufficient to cause its fracture when cold to be explosive (max. tension, 2—3 kg./sq. mm.). J. A. S.

**Manufacture of tempered glass articles.** CORNING GLASS WORKS, Assecs. of W. W. SHAVER (B.P. 477,585, 30.6.36. U.S., 31.7.35).—The chilling bath consists of molten inorg. salts, *e.g.*,  $\text{KNO}_3$  +  $\text{NaNO}_3$ , having a low v.p. at the temp. to which the glass is heated and of m.p.  $< 310^\circ$ . The nitrites may also be used, and a substance, *e.g.*,  $\text{WO}_3$ ,  $\text{B}_2\text{O}_3$ ,  $(\text{NH}_4)_2\text{SO}_4$ , added to neutralise any alkalinity which may develop. J. A. S.

**Chilling baths for tempering glass.** CORNING GLASS WORKS, Assecs. of J. T. LITTLETON, W. W. SHAVER, and H. R. LILLIE (B.P. 477,593, 30.6.36. U.S., 24.9.35).—The baths of molten inorg. salts described in B.P. 477,585 (cf. preceding abstract) may be operated at lower temp. (*e.g.*, lower by 100°) by adding to them 5% of finely-ground  $\text{CaSO}_4$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{MgO}$ , kaolin, or feldspar. J. A. S.

**Manufacture of compound glass and other laminated materials.** A. ROSENTHAL (B.P. 477,964, 10.7. and 9.11.36).—The interlayers of plastic material (*e.g.*, cellulose acetate, polyvinyl compound, etc.) are treated with a wetting liquid comprising at least one  $\text{H}_2\text{O}$ -insol. org. compound (I), which is a strong solvent for the plastic material employed, and at least one  $\text{H}_2\text{O}$ -sol. compound (II), the proportions of (I) and (II) being so selected that the mixture retains the high solvent action on the plastic but is substantially  $\text{H}_2\text{O}$ -insol. The manufacture conforms with standard practice, but sealing of the edges is unnecessary since penetration of  $\text{H}_2\text{O}$  into the laminations during processing does not occur. Ornamental effects may be obtained by dissolving org. dyes in the wetting liquid, and, if desired, the glass (or other material) may be coated with a  $\text{H}_2\text{O}$ -sol. adhesive prior to pressing. Many examples of (I) and (II) are quoted including, for (I), glyceryl diacetate, condensation products of aromatic sulphonamides and  $\text{CH}_2\text{O}$ , methyleugenol, etc., and, for (II),

neutral org. liquids (*e.g.*,  $\text{HCO}_2\text{Me}$ ,  $\text{COMe}_2$ ,  $\text{MeOH}$ ), alkalis, acids, and salts. J. W. CR.

**Hardened vitreous articles.** W. J. TENNANT. From AMER. OPTICAL CO. (B.P. 478,759, 3.11.36).—A composite glass article (sheet, lens, etc.) consists of an outer or exposed glass which is relatively thick ( $\frac{3}{16}$  in.) and "toughened," laminated by a layer of non-brittle (org.) material with an inner or unexposed glass which, being relatively thin ( $\frac{1}{16}$  in.) and un-toughened or strain-free, is not liable to detach dangerous splinters when subjected to impact. The thicknesses mentioned are suitable for a motor-car windscreen. J. A. S.

**Decoration of glass.** G. B. WATKINS, Assr. to LIBBEY-OWENS-FORD GLASS CO. (U.S.P. 2,066,497, 5.1.37. Appl., 2.6.33).—A design is printed on, or otherwise applied to, a synthetic (alkyd) resin-pigmented film which is supported on glass and the whole is heated. S. M.

**Coating glass sheets with polymerised vinyl compounds.** D. C. POLDEN and E. W. ALDRIDGE (B.P. 478,163, 31.3.37).—A viscous solution (*e.g.*, <50% in an aliphatic acetate) of a vinyl compound resin (preferably the acetate) is heated to reduce the  $\eta$ , applied (roller-coated) to a cooled glass sheet, and the solvent evaporated. Preferably the solution is deaerated prior to application and the coated glass before drying is placed in a closed chamber containing vapour of a resin solvent (*e.g.*,  $\text{COMe}_2$ ) where evaporation is prevented while the film smooths out and loses the air bubbles. The coated glass may be employed in the manufacture of splinterless glass. J. W. CR.

**Production of fibres from a viscous substance such as glass.** A. E. EDWARDS. From N. V. MAATS. TOT BEHEER EN EXPLOIT. VAN OCTROOIEEN (B.P. 475,406, 18.1.37).—Fibres having a curved or sinuous form (and therefore capable of being "felted") are made by carrying the fibre, while still in the plastic condition, by means of a gas stream through a set of suitably disposed baffle plates. Apparatus is claimed. J. A. S.

**Drawing of fibres [glass wool] from a viscous substance.** N. V. MAATS. TOT BEHEER EN EXPLOIT. VAN OCTROOIEEN (B.P. 475,384, 18.1.37. U.S., 17.1.36).—A blast of gas is directed on to one side of, and in approx. the same direction as, a stream of the semi-fluid material, which is held against the blast by an induced air current on the other side: The attenuated stream of pasty material may, if desired, be directed along a curved surface to produce a curled fibre. Apparatus is claimed. F. R. E.

**Manufacture of fibres from glass and other materials.** N. V. MAATS. TOT BEHEER EN EXPLOIT. VAN OCTROOIEEN (B.P. 487,648, 21.10.37. U.S., 23.10.36).—Fluid glass is formed into thin streams by gravity flow through jets, and the streams are introduced into gas blasts flowing in substantially the same direction but turbulently, *e.g.*, into steam flowing at the velocity of sound. B. M. V.

**Preparing an enamel frit for production of white clouded iron enamel.** I. KREIDL (B.P. 476,194, 11.8.36. Austr., 25.2.36).—A frit suitable for "gas-clouding" is made from a batch containing

F (wholly or in part from cryolite) 8—12,  $\text{B}_2\text{O}_3$  >10,  $\text{Al}_2\text{O}_3$  (from constituents other than cryolite) as low a % as possible, with 10% of  $\text{B}_2\text{O}_3$  or >7% with <6% of  $\text{B}_2\text{O}_3$ . *E.g.*, borax 22, cryolite 18, soda 4,  $\text{KNO}_3$  2,  $\text{SiO}_2$  54%. J. A. S.

**Tunnel kiln for firing ceramic ware.** GEN. MOTORS CORP. (B.P. 488,262, 1.1.37. U.S., 1.4.36).—In the firing zone the goods are passed through upper and lower tunnels; in the latter burners play direct on the goods, producing surface combustion, and the gases escape to the upper tunnel to effect preheating. [Stat. ref.] B. M. V.

**Activation of clays or like siliceous crude earths.** OESTERR. DYNAMIT NOBEL A.-G. (B.P. 478,911, 8.2.37. Austr., 22.2.36).—The process of activation by treatment with  $\text{H}_2\text{SO}_4$  and alkali chloride is supplemented (simultaneously or later) by suitable treatment with HCl (or an alkali chloride) so that any  $\text{CaSO}_4$  present or formed in the material is removed. J. A. S.

**Ceramic material [for electrical insulation].** STEATIT-MAGNESIA A.-G. (B.P. 478,794, 7.6.37. Ger., 8.6.36).—A soapstone material having a low dielectric loss, an increased surface resistance, and a reduced firing shrinkage, contains (2—10% of) MgO spinel ( $\text{MgO}, \text{Al}_2\text{O}_3$ , preferably formed by fusion in the arc) in addition to the other advantageous constituents. A typical mixing is soapstone 85, spinel 4, clay 6, BaO 4,  $\text{CaCO}_3$  1%.  $\text{ZrO}_2$  may also be included. J. A. S.

**Ceramic masses for electrical insulation.** C. SCHUSTERIUS (B.P. 487,961, 1.2.37. Ger., 30.1. and 9.10.36).—A sintered mixture of  $\text{TiO}_2$  (and, if desired,  $\text{ThO}_2$  or  $\text{ZrO}_2$ ), with 3—10% of CdO and/or Bi oxide to lower the sintering temp. to approx. 1200°, is claimed. B. M. V.

**Production of [magnesium silicate] ceramic materials.** STEATIT-MAGNESIA A.-G. (B.P. 476,393, 18.6.36. Ger., 20.6.35).—A steatite body of increased mechanical strength is prepared by using in the ceramic mix at least two soapstones (talcs or steatites) having different crystal sizes. J. A. S.

**Refractory heat insulation.** F. CHRISTENSON and H. N. CLARK (U.S.P. 2,078,753, 27.4.37. Appl., 22.8.36).—A facing of castable monolithic material is backed by a blanket of fibrous material supported between metal screens, the castable material bonding into one of the last. B. M. V.

**Refractory products.** NON-METALLIC MINERALS, INC. (B.P. 475,508, 19.5.36. U.S., 16.9.35. Addn. to B.P. 464,047; B., 1937, 1207).—The tendency of merwinite refractories to interact with CaO present to form a layer which cracks or disintegrates on cooling is eliminated by incorporating in the mix a small amount (*e.g.*, >1%) of a material containing B, P, or Cr. J. A. S.

**Production of siliceous refractory articles.** A. F. BURGESS. From NAT. ALUMINATE CORP. (B.P. 476,623, 22.4.37).—A  $\text{SiO}_2$  mix containing compounds of Ba and Fe is fired under reducing conditions which are later replaced by oxidising conditions either during a continuance of the firing or during the slow cooling. *E.g.*, ganister 100,  $3\text{BaO}, \text{Al}_2\text{O}_3$  2 pts.,  $\text{Fe}_2\text{O}_3$  0.7 pt.

The product has a high tridymite content and improved strength and resistance to spalling.

J. A. S.

**Abrasive article.** D. E. WEBSTER, Assr. to NORTON Co. (U.S.P. 2,078,354, 27.4.37, Appl., 25.4.35).—A thin wheel (for cutting off) is bonded with vulcanised rubber and has the grains closer together on the side faces than on the periphery, supplemental and preferably harder grains being pressed in during manufacture.

B. M. V.

**Abrasive-coated discs.** W. J. TENNANT. From CARBORUNDUM Co. (B.P. 488,303, 25.2.37).—A tough, ductile backing disc (of hydrolysed cellulose or reinforced plastic material) is coated with cloth, and that with abrasive grains, by means of an adhesive containing synthetic resin, preferably phenolic. [Stat. ref.]

B. M. V.

**Testing the abrasion-resisting properties of materials.** C. P. and O. F. A. SANDBERG and C. W. HUMFREY (B.P. 487,428, 29.1.37).—In apparatus in which one member or both members is/are made of the material under test and is/are subjected together to combined rolling and sliding contact under pressure, the members take the form of a roller and shallow dish, the former pressing on the rim of the latter; the axes are at 90° and besides the common rotation the roller is oscillated on its axis.

B. M. V.

**Production of coloured or decorative glass [having a "shot" effect].** F. D. PARKINSON (B.P. 478,301, 13.7.36).

**Method and apparatus for forming [moulded] ceramic bodies.** GEN. MOTORS CORP. (B.P. 477,287, 22.6.36. U.S., 27.6.35).

**Filter-presses [for clay etc.].**—See I. Electric furnace for glass. Sealing W or Mo through quartz glass etc.—See XI. Plastic masses [for safety glass]. Polyvinyl resins and compounds. Finishing material [abrasive].—See XIII.

## IX.—BUILDING MATERIALS.

**Puzzuolanas and cements.** II. Hydrated monocalcium silicate. V. CIRILLI (Annali Chim. Appl., 1938, 28, 239—244; cf. B., 1938, 911).—Passage of aq. CaO over SiO<sub>2</sub> gel gives CaSiO<sub>3</sub>·H<sub>2</sub>O (I) which is not readily dehydrated, loss of H<sub>2</sub>O commencing at 130° and being complete at approx. 700°. Thermal decomp. of (I) yields Ca<sub>2</sub>SiO<sub>4</sub> and SiO<sub>2</sub> which, at higher temp., produces wollastonite.

F. O. H.

**Correlation of methods for measuring heat of hydration of cement.** R. W. CARLSON and L. R. FORBRICH (Ind. Eng. Chem. [Anal.], 1938, 10, 382—386).—Three types of calorimeter, viz., the adiabatic, heat of dissolution, and vane-and-conduction types, are described and their limitations and advantages pointed out. Practically identical results can be obtained when possible sources of error are taken into account. The immediate heat of hydration, carbonation of heat-of-dissolution specimens, the H<sub>2</sub>O : cement ratio, and variations in sp. heat of concrete with temp. are important factors to be considered if agreement in the results is to be reached.

L. S. T.

(A) Temperature and humidity effects in unpainted and painted brick structures. (B) Painting cement-asbestos compositions. ANON. (Sci. Sect. Nat. Paint, Var. Assoc., Inc., July, 1938, Circ. [A] 562, [B] 563, [A] 152—153, [B] 154—157).—(A) Graph and charts show temp. and R.H. variations during the summer of 1937 in two penthouses. One of them was painted white, had max. temp. 35° and const. R.H.; the other was unpainted, had probable max. temp. 43°, and the R.H. varied from 55 to 70%.

(B) Two boxes made of corrugated asbestos-cement were similarly exposed; one painted white was 3—5° cooler than the unpainted box. The painted material absorbs negligible amounts of H<sub>2</sub>O, whereas the unpainted composition absorbs 4 oz./sq. ft. of surface. Exposure tests with two coats of a green paint on a series of primers show that the material can be satisfactorily painted.

S. M.

**Trend of progress in the building industry.** R. FITZMAURICE (Chem. and Ind., 1938, 845—847).

**Trend of progress in road design.** J. O. WILLIS (Chem. and Ind., 1938, 847—849).

**Bituminous road materials in Canada.** C. MACK (Chem. and Ind., 1938, 836—837).—The special requirements for road construction in Canada are outlined and a brief account is given of the nature of the research work undertaken to find suitable materials.

T. W. P.

**Mechanical testing of bituminous road mixtures.** J. P. PFEIFFER (J.S.C.I., 1938, 57, 213—225).—Data are given on compression and tensile strength tests on bitumen-mineral aggregate road mixtures, from which it is concluded that the tests are unsuitable. A new method of testing resistance to shear, based on the principles of soil mechanics, is described.

T. W. P.

**New method of treating bituminous roof coatings.** H. A. GARDNER (Sci. Sect. Nat. Paint, Var. Assoc., Inc., July, 1938, Circ. 561, 146—161).—Dusting with Al powder is recommended, to improve durability and lower the summer temp. of the roof. For large areas the Al paste is extended with mineral spirits and sprayed. Coloured pigments may also be used.

S. M.

**Waterproofing and consolidating the walls of mine-shafts.** H. WALDECK (Glückauf, 1938, 74, 385—392, 409—417).—The merits and costs of cementation and chemical processes and the Shellperm process are compared. The Shellperm process consists in injecting a bituminous emulsion into rock pretreated with a reagent which causes the bitumen to be pptd. in the rock pores. In the chemical method a siliceous solution is forced into the rock, followed by a second solution causing a gel to be formed. Experiments have shown that all types of rock can be chemically consolidated. Practical examples of the three methods of treatment are described in detail.

R. B. C.

**Wood as a homogeneous material.** I. Improving wood for structural purposes. P. BRENNER. **Gluing of wood with synthetic resin.** O. KRAEMER (Aircraft Eng., 1938, 10, 129—134, 183—186).—I. The homogeneity and resistance to H<sub>2</sub>O are

improved by lamination and binding with Tegofilm or Kaurit under pressure. The compressive strength is thus increased up to 125% in the direction of the grain and up to 500% perpendicular thereto. The bending strength, modulus of elasticity, and rigidity are improved, but the tensile strength is increased only in a direction perpendicular to the grain.

II. Data on the binding strengths of birch plywood glued with casein, casein-blood-albumin, blood-albumin, Tego film, Kaurit, and Bakelite are given.

R. B. C.

Rapid determination of moisture in willow bark. I. V. GERASIMOV (Utschen. Zap. Univ. Kazan, 1938, 97, 121—126).— $\text{CaC}_2$  is mixed with the bark and the vol. of  $\text{C}_2\text{H}_2$  produced is measured. A determination of  $\text{CaC}_2$  in the specimen of carbide used is essential.

J. J. B.

Grinding machinery for cement etc.—See I.  $\text{H}_2\text{C}_2\text{O}_4$  from sawdust.—See III. Wood substances.—See V. Joint strength of high-grade glues.—See XV. Cane molasses as road-making material.—See XVII.

See also A., I, 459, Equilibrium diagrams of binary [silicate] systems. Solid solutions of Ca ferrite in Ca aluminate.

#### PATENTS.

Treatment of raw materials for manufacture of cement or lime. M. VOGEL-JØRGENSEN (B.P. 476,435, 8.6.36).—The raw materials are heated at 70—350° and then subjected, in a state of fine subdivision, to flotation, for the purpose of cleaning or concn. The preliminary heat-treatment improves the efficiency of the flotation process and also allows less  $\text{H}_2\text{O}$  to be used in the slurry.

T. W. P.

Manufacture of hydraulic gypsum cement material. RUMFORD CHEM. WORKS (B.P. 477,743, 30.3.37. U.S., 14.5.36).—Wholly or partly hydrated or dehydrated gypsum is mixed with a phosphoric acid, e.g.,  $\text{H}_3\text{PO}_4$ , and an alkali phosphate, e.g.,  $\text{NaH}_2\text{PO}_4$ , and calcined at 980—1260°. The mixture may include  $\text{SiO}_2$  or silicates, up to 3%; the phosphate additions are preferably < 2%. The calcined product gives a product with high strength and sand-carrying capacity and normal setting time.

T. W. P.

Hydraulic binders. ETABL. POLIET ET CHAUSON (B.P. 474,917, 11.5.36. Fr., 14.2.36).—Portland cement is mixed with 10—50% of a mixture having a basis of sulphoaluminate, obtained by sintering a mixture of bauxite or clay with limestone and gypsum. Alternatively, the Portland cement is made with an excess of  $\text{CaSO}_4$ . In either case the production of hydrated sulphoaluminates during setting and hardening causes an expansion in vol. which counteracts the initial shrinkage during drying.

T. W. P.

Waterproof concrete. H. L. LEVIN, Assr. to PATENT & LICENSING CORP. (U.S.P. 2,067,772, 12.1.37. Appl., 22.8.32).—A composition containing asphalt 50—65, rosin soap 3—5, and  $\text{H}_2\text{O}$  30—47% is prepared by running molten pressure-tar asphalt, m.p. 60—93°, into a mill containing the soap solution and grinding the mixture. This dispersion can be mixed

with concrete to render it waterproof, only coalescing when all  $\text{H}_2\text{O}$  is removed.

F. M. L.

Manufacture of light-weight calcium sulphate plasters. V. LEFEBURE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 479,452, 5.8.36).—Anhydrite plasters containing chips or sawdust of Balsa wood (2—20 wt.-%) are claimed. The small amount of filler necessary does not materially reduce the fire-resistance of the plaster.

T. W. P.

Production of building and like materials. W. SCHÖBITZ (B.P. 475,081, 7.12.36).—The material is prepared by mixing washed and pulped paper material,  $\text{CH}_2\text{O}$  solution, water-glass, and Portland cement.

T. W. P.

Composite building material. E. C. LOETSCH (U.S.P. 2,067,012, 5.1.37. Appl., 9.10.33).—Various mixtures of synthetic resins and  $\text{CaO}$  with resinous and relatively non-resinous woods are treated (method specified) to produce sheets which are hard and dense, have a lustrous surface, and can be worked or cut. About 10% of S may be added to the mix to increase plasticity.

T. W. P.

Composite building plates and the like. G. SCHUSTER and F. HOFFMANN (B.P. 488,127, 14.5.37. Ger., 15.5.36).—The centre (or back) is composed of sound-insulating, compacted or bound fibrous material and both faces (or one face) are/is of asbestos-cement or cork-cement which is caused to adhere by an intermediate layer of rubber deposited *in situ* from natural or synthetic dispersion (latex).

B. M. V.

Manufacture of bituminous pavements of high structural strength. C. M. BASKIN, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 2,074,010, 16.3.37. Appl., 27.6.33).—A strong paving mixture which will not crack is prepared by mixing 6% of an asphalt of low penetration at 25° and low furoil  $\eta$  at 149°, 6% of fine mineral dust of < 200-mesh, 48% of mineral aggregate ( $\frac{1}{4}$  in. max. particles), and 40% of aggregate of 1—2 $\frac{1}{2}$  in. size.

D. M. M.

Preservation of wood. G. B. SHIPLEY (U.S.P. 2,066,583, 5.1.37. Appl., 29.4.33).—The tarry oil recovered from the condensing system of a by-product coke oven separately from the heavy tar deposited in the hydraulic main is used undistilled as a wood-preserving impregnant. Such oil should have abs.  $\eta$  2.47 centipoises at 85° and a distillation residue of > 33.5% at > 180°.

T. W. P.

Preserving treatment for wood. H. M. A. VAUTHERIN (B.P. 477,742, 22.3.37. Fr., 28.3.36).—Wood is treated in an autoclave with live steam and the temp. then maintained at 115—170° for a period long enough to render the tissue unalterable and unattackable. Preservatives, e.g., creosote, may be introduced with the steam.

T. W. P.

Wood-preservative oil. A. HOLMES, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 2,078,570, 27.4.37. Appl. 6.6.33).—The oil consists of a solution of creosote in a petroleum oil, acid-sludge distillate oil, preferably containing > 5% (10—15%) of S.

D. M. M.

(A) Toxic agent and its application to fibrous products. (B) Insect- and fungi-toxic fibrous materials. G. H. ELLIS, Assr. to INSULITE Co.



(U.S.P. 2,067,046—7, 5.1.37. Appl., [A] 31.8.34, [B] 29.6.35).—(A) Methods of making a toxic agent, comprising treatment of a mixture of a rosin, a coal-tar product of the class containing hydrocarbons of the type  $C_{14}H_{10}$ , creosote and its homologues,  $C_5H_5N$ -like substances (<0.5%), and a caustic are claimed. The suspension of the agent in  $H_2O$  is added to the dil. suspension of the fibres in  $H_2O$ . (B) Various types of fibrous products containing the toxic agent are claimed. T. W. P.

**Artificial lumber.** J. V. NEVIN (U.S.P. 2,078,269, 27.4.37. Appl., 3.4.34).—70—95% of fibrous woody material is bound by 30—5% of an infusible, final condensation product of urea, *m*-cresylic acid, and  $CH_2O$  and the natural binders present in the wood. B. M. V.

**Manufacture of plasterboard.** H. O. KAUFFMANN and D. D. CRANDELL, Assrs. to BUFFALO ELECTRO-CHEM. CO., INC. (U.S.P. 2,064,800, 15.12.36. Appl., 27.1.33).—In making porous plasterboard which is reinforced by wood-pulp fibres, uniform pore distribution and pore size are obtained by  $O_2$  evolution from  $H_2O_2$  which is dispersed in the pulp during its disintegration. The  $H_2O_2$  is stabilised by maintaining the pulp suspension at about  $p_H$  4.7, and sol. salts of Mn, Fe, Co, etc. are also added as latent catalysts. The pulp is then mixed with plaster and the mixture, after making alkaline ( $p_H$  7.5—11.0) with NaOH, is formed into board. The NaOH ppts. the hydrated oxide of the catalyst, the particles of which act as nuclei for the evolution of  $O_2$ . D. A. C.

(A) Manufacture of [bituminous wood-fibre] composition. (B) Manufacture of plastic material. T. ROBINSON, Assr. to LANCASTER PROCESSES, INC. (U.S.P. 2,072,686—7, 2.3.37. Appl., [A] 16.12.35, [B] 29.1.36).—Compositions which are plastic on prep. and become hard on setting, suitable as roofing material etc., are made by mixing a fibrous material,  $H_2O$ , and asphalt, with CaO or rosin if desired, in a closed mixer in which the fibrous material is disintegrated and sufficient pressure maintained to prevent the evaporation of the  $H_2O$  even if the temp. reaches 100°. Suitable mixtures are: (A) wood chips 175,  $H_2O$  100, CaO 3.5, and oxidised asphalt, m.p. 220—235°, 175 pts.; (B) shredded waste paper 220,  $H_2O$  110, rosin 15, CaO 5, and oxidised asphalt, m.p. 100°, 220 pts. D. M. M.

**Sand-facing of bricks, building blocks, and the like.** LONDON BRICK CO., LTD., G. H. C. RACTLIFFE, and C. W. D. ROWE (B.P. 488,278, 3.11.36).

**Blast-roasting cement. Drying slurry.**—See I. Emulsions.—See II. Fibre board. Wood-pulp boards.—See V. Fireproofing.—See VI. Shaped elastic masses.—See XIII. Wood glue. Adhesive for wood etc. Synthetic resin adhesive.—See XV.

## X.—METALS; METALLURGY, INCLUDING ELECTROMETALLURGY.

**Austrian mining industry.** E. FERJANCIC (Bergbau, 1938, 51, 131—137, 147—154).—A review.

Analyses of Fe ores, magnesite, and bituminous and brown coals are given. R. B. C.

[Need for] more intensive field studies for laboratory investigations of ore deposits. R. H. SALES (Econ. Geol., 1938, 33, 239—250).—An address. L. S. T.

**Mixing and its importance in metallurgical industry.** E. BELANI (Montan. Runds., 1938, 30, No. 12, 1—4).—The principles of machines designed for effecting the intimate mixing of powdered materials, e.g., ores from which sponge Fe is made, are discussed. R. B. C.

**Desulphurisation [of iron ore] in a Martin furnace.** J. BULINA (Chem. Listy, 1938, 32, 243—246; cf. B., 1930, 1068).—When the slag contains >40% of CaO, with ~10% of  $SiO_2$  and 12—22% of FeO, desulphurisation is complete, but with <40% of CaO it is insignificant. R. T.

**The blast furnace.** ESTOUR (Chaleur et Ind., 1938, 19, 425—432).—Reactions taking place between Fe oxide and reducing agents and gases in the furnace, the composition of the gases and their distribution in the furnace, slag composition, and the composition of solid and molten materials in the shaft are discussed. R. B. C.

**The blast furnace and its operation.** J. G. WEST (Blast Furn. Steel Plant, 1938, 26, 77—78, 98, 183—185, 208, 290—293, 314, 386—387, 431, 496—499, 614—615, 627—629).—The construction and design of the blast furnace and its auxiliary equipment are comprehensively reviewed. R. B. C.

**Blast-furnace economy by pretreating the charge.** K. GUTHMANN (Stahl u. Eisen, 1938, 58, 857—865).—It is shown that low-grade German Fe ores can be smelted economically provided they are previously roasted or sintered. M. A.

**Efficiency of thermal operations and equilibrium operations—applications to the blast furnace.** L. GRENET (Chaleur et Ind., 1938, 19, 422—424).—Reactions taking place in the blast furnace, e.g., the production of the ferrous phase by reduction of Fe oxide by C, and the desulphurisation of the metallic phase, are discussed. R. B. C.

**Melting experiments with a coreless standard-frequency furnace.** G. MARS (Stahl u. Eisen, 1938, 58, 833—840, 865—868).—A specially constructed, coreless furnace worked at the standard frequency of the supply network (details of construction are given) was found to be superior to a high-frequency furnace for melting heavy pieces and large charges, but inferior for small charges. The current losses are approx. the same as in a high-frequency furnace. M. A.

**Refractory problems in the iron and steel industry.** F. SINGER (Iron Steel Ind., 1938, 11, 316—320, 479—482).—A review. R. B. C.

**Poumay cupola control in a Danish [iron] foundry.** ANON. (Found. Tr. J., 1938, 58, 404).—Data obtained for three cupolas melting Fe before and after conversion to the Poumay system show that an increased thermal efficiency is obtained in the converted cupolas as a result of better combustion of the coke. Oxidation is also negligible. R. B. C.

**Radiant-tube annealing covers.** C. H. CARPENTER (Iron Steel Eng., 1938, 15, No. 5, 36—43).—Recent types of radiant-tube annealing furnaces and temp.-control devices are described. R. B. C.

**Desulphurisation of pig iron with sodium carbonate.** N. THEISEN (Stahl u. Eisen, 1938, 58, 773—779).—0.5% of  $\text{Na}_2\text{CO}_3$  will desulphurise pig Fe containing 0.1% of S. Although a higher proportion of  $\text{Na}_2\text{CO}_3$  will give a greater degree of desulphurisation, excess of  $\text{Na}_2\text{CO}_3$  is not recommended as the amount of S removed is not  $\propto$  the amount of  $\text{Na}_2\text{CO}_3$  added. Desulphurisation may be effected as the metal runs from the blast furnace, or in the mixer; the latter method is advantageous since the composition of the Fe is more uniform, the temp. less variable, and a smaller quantity of  $\text{Na}_2\text{CO}_3$  is required. Desulphurisation is accompanied by a drop in the Si content of the metal, but Mn, P, and C are only slightly affected. The operation is favoured by low temp., since  $\text{Na}_2\text{CO}_3$  decomposes and Na vaporises at high temp. The requirements of the steelworks and the fall in temp. due to the  $\text{Na}_2\text{CO}_3$  treatment must, however, be borne in mind. If the reaction between the  $\text{Na}_2\text{CO}_3$  and the pig Fe is too prolonged S may return to the metal:  $\text{SiO}_2 + \text{Na}_2\text{S} + \text{FeO} \rightarrow \text{Na}_2\text{SiO}_3 + \text{FeS}$ . A reaction time of 6—10 min. is recommended. Increasing Mn content (up to 0.5%) results in greater desulphurisation, but a higher Mn content has no beneficial effect. With dolomite or magnesite linings the desulphurisation is  $>$  when a grog lining is employed. These linings, however, develop cracks very readily. Methods of recovering the  $\text{Na}_2\text{CO}_3$  from the slag are given, and the possibility of using the  $\text{Na}_2\text{CO}_3$ -rich slag in the glass industry is discussed. M. A.

**Tempering of iron containing ferrous oxide.** W. BRONIEWSKI and S. MAZGIS (Compt. rend., 1938, 207, 342—344).—The mechanical and electrical properties of Armeo Fe have been studied in relation to the temp. of tempering (500—1450°). In general, tempering increases the hardness, tensile strength, and electrical resistance, and decreases the work required for breakage, the elongation on breakage, and dissolution e.m.f. Seasoning at room temp. enhances the effect of tempering in most cases. A. J. E. W.

**Extraction of oxide inclusions in high-carbon iron—electrolytic iodine method. I.** F. W. SCOTT (Met. and Alloys, 1938, 9, 171—174).—The theories that have been proposed to explain the graphitisation of pig Fe are briefly discussed and methods of analysing for oxide inclusions in ferrous alloys are outlined. M. A.

**Extraction of oxide inclusions in high-carbon iron—electrolytic iodine method. II.** F. W. SCOTT (Met. and Alloys, 1938, 9, 201—206; cf. preceding abstract).—In order to study the effect of large variations in analysis of Fe alloys by this method of extraction, the normal concn. of the various metalloids was exceeded. A series of Fe—C alloys, containing Mn, Si, P, and S, was prepared and their oxide contents were determined. The results indicate that the metalloids do not give high vals. or wide variations, and that different rates of cooling do not

cause any difficulty. It is concluded that the method can be applied to the study of oxides in foundry Fe with reasonable assurance of obtaining reproducible, comparative, and accurate results. Owing to recent small changes and modifications in the practical details of the extraction process, a brief review of the latter is appended. P. G. McC.

**Ingot moulds.** N. H. BACON (Iron Steel Ind., 1938, 11, 326—331).—Data obtained by Messrs. Steel, Peech, and Tozer on factors determining the life of moulds are discussed. The ideal ratio of wall thickness to mould wt. is 0.06, and the best mould thickness is that which results in 50% of the mould failures being due to cracking, and 50% to crazing, of the working face. A statistical study of 1000 moulds showed that when the Si content of the mould was  $\sim$  2% it was advantageous to increase the Mn content, but as the Si was reduced this was no longer true. With  $<$  1.80% Si addition of 1% of Mn or more increased the life, whereas below 1.60% Si 0.80—0.89% of Mn gave the better results. Other factors affecting mould life are the design, the stripping time and method of stripping, and the mixture of hæmatite and scrap used to make the mould. R. B. C.

**Moulding sand, with special reference to blind scabs.** S. CARTER and A. W. WALKER (Found. Tr. J., 1938, 58, 423—428).—Data on the tendency of certain moulding sands to produce scabs on Fe castings are given. The causes of this defect and methods for its elimination are discussed. R. B. C.

**Randupson process of cement moulding [of iron].** F. W. ROWE (Found. Tr. J., 1938, 58, 520—526).—The advantages and disadvantages of using  $\text{SiO}_2$  sands bonded with Portland cement are discussed. Castings made by this method are illustrated. R. B. C.

**White cast iron.** P. L. WARD (Found. Tr. J., 1938, 58, 441—443).—Cupola and moulding practice for the production of white-Fe castings, and the effects of various elements in the Fe are discussed. R. B. C.

**Modern manufacture of machine-tool [iron] castings.** J. BLAKISTON (Found. Tr. J., 1938, 59, 3—7).—The properties desirable in cast Fe used for machine tools, methods of melting employed, and running and moulding practice are discussed. R. B. C.

**Titanium in cast iron.** E. R. STARKWEATHER (Trans. Amer. Found. Assoc., 1937, 45, 816—830).—The use of Ti for deoxidising and grain-refining, and its effect on the physical properties of Fe, are reviewed. R. B. C.

**Diesel-engine metals.** R. W. MAUGHAN (Metallurgia, 1938, 18, 123—124).—The quality-improving properties of Ni on Fe castings for engine parts is briefly discussed. Reference is also made to bearing metals and some compositions are given. P. G. McC.

**Heat-treatment of copper-silicon ferrous castings.** ANON. (Metallurgia, 1938, 18, 133—134).—These alloys require a double treatment, viz., (1) heating at  $\sim$  925° for some time, followed by rapid cooling to  $\sim$  590°, and (2) reheating to 760°, followed

by controlled cooling. Details of the furnaces installed at the Ford Motor Co.'s works, Dagenham, for this purpose are outlined. P. G. McC.

**Casting defects [in iron].** G. H. PIPER (Found. Tr. J., 1938, 58, 336).—Conditions tending to the production of gas-, sand-, and slag-holes, and methods of differentiating between the different types of defect, are discussed. When the casting is treated with Cu chloride in aq. ethylene glycol Cu is deposited on the metal; the sand and slag inclusions are unaffected. R. B. C.

**Use of cast-iron borings.** A. PALMUCCI (Found. Tr. J., 1938, 58, 437—438).—Comparative costs of melting the borings in an electric furnace and in a cupola are given. R. B. C.

**Oxidation [of iron] and adhesion [of enamel].** J. WHITE (Found. Tr. J., 1938, 59, 14—16).—A review. R. B. C.

**Determination of aluminium in cast iron.** E. TAYLOR-AUSTIN (Analyst, 1938, 63, 566—592).—The methods of determining Al by pptn. as  $\text{Al}(\text{OH})_3$  and  $\text{AlPO}_4$ , and with 8-hydroxyquinoline (I), are compared critically. The procedure recommended for pptn. with (I) is to dissolve the sample of cast Fe in 10%  $\text{H}_2\text{SO}_4$  and ppt. the Al, P, and some of the Fe by judicious addition of  $\text{NaHCO}_3$ . The ppt. is redissolved in HCl, and Cu, Mo, etc. are removed by pptn. with  $\text{H}_2\text{S}$  in presence of tartaric acid. Fe is then removed by double pptn. with  $\text{H}_2\text{S}$  from a solution slightly alkaline with  $\text{NH}_3$ , the solution being completely saturated with  $\text{H}_2\text{S}$ . The Al solution is evaporated, HCl added, and then Br until the solution is slightly yellow. After filtration, neutral-red is added and the solution made just alkaline with  $\text{NH}_3$  and then just acid with AcOH. A solution of (I) is then added, followed by aq.  $\text{NH}_4\text{OAc}$ . The coagulated ppt. is dried at 105—110°. Ti is not pptd. under the conditions described. In presence of Cr the  $\text{NaHCO}_3$  ppt. is dissolved in hot 10%  $\text{H}_2\text{SO}_4$ , and  $\text{AgNO}_3$  and  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  are added to the boiling solution. Fe and Al are then pptd. with  $\text{NH}_3$ , the solution is boiled until the vapours are free of  $\text{NH}_3$ , and the ppt. filtered off and treated as in absence of Cr. The ppt. obtained with (I) can also be dissolved in conc. HCl, indigo-carmin indicator added, and  $\text{KBrO}_3$ -KBr solution added until the solution is yellow. The solution is then shaken with  $\text{CS}_2$  and KI, starch is added, and titration carried out with  $\text{Na}_2\text{S}_2\text{O}_3$  until no blue colour remains in the  $\text{CS}_2$ . J. W. S.

**Strength [of iron, sulphur, and tin] at the transformation point.** F. ROLL (Z. Metallk., 1938, 30, 244—245).—The tensile strength-temp. curves of Fe, S, and Sn show marked irregularities at temp. in the neighbourhood of the transformation points, e.g., at 750—950°, 90—100°, and around 162°, respectively. A. R. P.

**Water-impact endurance tests on pure iron.** M. VATER (Z. Ver. deut. Ing., 1938, 82, 672—674).—Endurance tests were carried out on Fe with loads in the neighbourhood of the  $\text{H}_2\text{O}$ -impact endurance strength. The data obtained confirm the view that destruction of the material is caused by gradual breakdown under the combined effects of alternating

mechanical stress and corrosion. Stressing of this type is compared with other wearing processes.

R. B. C.

**Corrosion research.** F. MÜLLER (Z. Ver. deut. Ing., 1938, 82, 841—845).—Recent work is reviewed.

R. B. C.

**Principles of corrosion and its prevention.** F. N. SPELLER (J. New England Water Works Assoc., 1938, 52, 228—232).—The electrochemical corrosive action of soil and  $\text{H}_2\text{O}$  on Fe pipes is discussed and it is shown that control of cathodic reaction is safer than anodic polarisation. Protection of metals by alloying, surface coatings, and soil treatment are discussed. O. M.

**Underground corrosion of pipe.** K. H. LOGAN (Chem. Met. Eng., 1938, 45, 422—425).—A review.

A. R. PE.

**Cathodic protection. Combating pipe corrosion with electricity.** A. V. SMITH (J. New England Water Works Assoc., 1938, 52, 233—243).—Electrolytic corrosion from stray tramway currents etc., corrosion by galvanic currents set up by soil, and the control of the latter type of corrosion by cathodic protection are discussed. O. M.

**Atmospheric corrosion of iron.** W. H. J. VERNON (Korros. u. Metallschutz, 1938, 14, 213—220).—The increase in wt. of abraded specimens of Fe containing approx. 0.1% C has been followed for periods of 70—160 days. Supersaturated and unsaturated pure and  $\text{SO}_2$ -containing air were employed, the specimens being tested in closed glass vessels containing 10 or 30 l. The effects of addition of  $\text{CO}_2$  and of inoculating the specimens with particles of  $(\text{NH}_4)_2\text{SO}_4$ , C, or quartz were examined in the different atm.  $\text{SO}_2$  causes increased corrosion, whilst  $\text{CO}_2$  reduces the attack in pure or impure air. Particles of  $(\text{NH}_4)_2\text{SO}_4$  cause greatly increased corrosion, especially in supersaturated air, but C and quartz have little effect. C. E. H.

**Phosphoric acid as protective agent against corrosion [of iron].** A. FOULON (Montan. Runds., 1938, 30, No. 14, Stahlbau-Techn., 4—5).—A review.

R. B. C.

**Corrosion-resistant alloys.** H. C. HICKEN (J. Proc. Austral. Chem. Inst., 1938, 5, 256—264).—Properties and uses of austenitic cast Fe and monel metal for corrosion-resisting purposes are described.

L. N.

**Recent developments in the American iron and steel industry.** C. E. WILLIAMS (Iron and Steel Inst., Oct., 1938, Advance copy, 22 pp.).

**Development of the open-hearth steelmaking processes in recent years in the United States.** L. F. REINARTZ (Iron and Steel Inst., Oct., 1938, Advance copy, 47 pp.).

**Basic Bessemer [steel] process.** H. MALCOR (Chaleur et Ind., 1938, 19, 409—412).—Thermal aspects of the process, e.g., the heat generated by combustion of the various elements in the liquid Fe and temp. regulation at the completion of blowing, are discussed. R. B. C.

**Fuel-air ratio control for open-hearth [steel] furnaces.** T. A. PEEBLES (Blast Furn. Steel Plant,

1938, 26, 404—405).—The control system employed by the Steel Co. of Canada is described. Either fuel oil or coke-oven gas can be employed. R. B. C.

**Scaling losses in rolling-mill furnaces.** IV. F. WENZEL (Stahl u. Eisen, 1938, 58, 481—491; cf. B., 1937, 560).—Scaling losses increase very rapidly at temp.  $>1100^\circ$ . Relatively small losses occur in a strongly reducing atm. even at  $1400^\circ$ , but in a feebly reducing atm. they are much greater, particularly at high temp. Up to  $1250^\circ$  the scaling losses in a feebly oxidising atm. are  $>$  in a strongly oxidising atm. At higher temp. the influence of temp. preponderates and pronounced scaling occurs even in strongly oxidising atm. Within the limits studied, C (0.35—0.6%) appears to have no effect on the tendency to scaling. Scaling losses increase rapidly at first with increasing duration of heating, and then, with a more prolonged heating, the curve flattens out. Heating ingots in such a way as to minimise the temp. gradient from the outside to the centre results in increased scaling losses. M. A.

**Gases for controlled atmospheres [for use in heat-treating steels]—requirements for purification.** E. E. SLOWTER and B. W. GONSER (Met. and Alloys, 1938, 9, 163—168).—The sources, applications, and effects of various furnace atm. are considered, and methods of removing or avoiding harmful constituents such as  $H_2O$  and  $CO_2$  are given. Complete removal of  $CO_2$  is possible, but the removal of the final traces is difficult and costly. A study was made of the effect of small traces of  $CO_2$ , and the possibility of counteracting these effects by addition of  $CH_4$ . By lowering the  $CO_2$  content to 0.2—0.5% decarburisation of high-C steels can effectively be prevented by addition of 0.5—1.0% of  $CH_4$ , but such an atm. is carburising to low-C steels. M. A.

**Surface sensitivity of steels towards certain fuel gases.** F. NEHL (Stahl u. Eisen, 1938, 58, 779—784).—Specimens of steels in the form of bend-test pieces were heated in various fuel gases at  $800$ — $1200^\circ$  and then bent through an angle of  $90^\circ$  to ascertain the extent to which surface-cracking occurred. Under such conditions surface-cracking is due to the selective attack of small quantities of  $O_2$  on the grain boundaries at temp.  $>800^\circ$ .  $O_2$  formed by the dissociation of  $H_2O$  vapour is particularly injurious. Grain-boundary attack by  $O_2$  is noticeable only if the Cu content of the steel is  $>0.08\%$ , and is the greater the higher is the Cu content. Addition of small quantities of Ni prevents cracking of high-Cu steels. The selective action of  $O_2$  on the grain boundaries is decreased by increasing the  $[O_2]$  as well as by adding S to the gases. M. A.

**Improving steel-foundry practice.** J. DESCHAMPS (Found. Tr. J., 1938, 58, 309—312).—Factors controlling the production of satisfactory plain C-steel castings are discussed. R. B. C.

**Survey of recent work on mild steel.** H. H. STANLEY (Iron Steel Ind., 1938, 11, 273—278, 483—486).—A review. R. B. C.

**Tool steel past and present.** IV. **Induction-furnace melting.** H. C. BIGGE (Met. Progr., 1938, 33, 489—493). R. B. C.

**Fabrication of corrosion-resistant alloys.** E. J. RAYMOND (J. Proc. Austral. Chem. Inst., 1938, 5, 265—267).—The welding of, and methods of avoiding weld decay in, 18-8-type alloys are described. L. N.

**Use and fabrication of austenitic steels in manufacture of organic acids.** J. L. MARSHALL (J. Proc. Austral. Chem. Inst., 1938, 5, 268—278).—The properties of the 18-8-type steels and experience in their use for the manufacture of citric and tartaric acids are described. The alloy modification containing 2—4% Mo was found satisfactory for use with boiling citric acid solutions. Contact with C, e.g., as used for decolorising purposes, resulted in failure of austenitic steels. Earlier troubles due to weld decay have been overcome by using the Mo variation of the alloy. For corrosion tests, heating the specimen for 7 hr. at  $85^\circ$  in an aq. solution containing 5% of HF and 40% of  $HNO_3$  was quicker than the usual  $CuSO_4$  test. To obtain max. corrosion-resistance on welded surfaces these are well polished and then made passive with 40%  $HNO_3$ . L. N.

**Temperature measurement and regulation in [steel-]hardening furnaces.** G. KEINATH (Arch. Tech. Mess., 1938, No. 84, 71—72r).—A review. R. B. C.

**Effect of cooling rate on quantity of proeutectoid ferrite [in steel].** J. A. VERÖ (Roy. Hung. Palatin-Joseph Univ. Tech. Econ. Sci., Publ. Dept. Min. Met., 1937, 9, 192—209).—Experiments in which the amount of structural constituents in unalloyed steels, heated at  $50^\circ$  above the Ac3 point for 1 hr. and cooled at varying rates, was measured by Rosiwal's method showed that the slow cooling usual in practice does not produce conditions of equilibrium. In industrial steels there is much less ferrite than would be expected from an examination of the equilibrium diagram. The dependence of the quantity of ferrite on the C content is represented by a straight line only in a state of equilibrium; in other cases it is indicated by curves differing from this straight line to an increasing extent with greater cooling rates. Steels consisting of nearly equal proportions of ferrite and pearlite are most affected by variations in cooling rate, whilst steels consisting chiefly of one constituent are influenced to a much smaller extent. A simple relationship exists between the quantity of constituents present and the type of structure produced. R. B. C.

**Effect of low velocity of cooling on transition temperatures of carbon steel.** F. WEVER (Naturwiss., 1938, 26, 393—394).—The effect of variation of the rate of cooling on the transition temp. of various steels has been determined. The temp. of the Ar1 transformation is considerably lowered as the velocity of cooling is increased. A. J. M.

**Cooling of permanent-magnet alloys in a constant magnetic field.** D. A. OLIVER and J. W. SHEDDEN (Nature, 1938, 142, 209).—When alloys of the Ni-Al-Fe Mishima type are cooled from  $1200^\circ$  in a unidirectional magnetic field the coercivity is not markedly affected, but the remanence and the val. of  $BH_{max}$  are increased. Typical results are re-

produced graphically for Alnico. An interpretation of the results is discussed. L. S. T.

**Hot-galvanising of steel wire.** A. KELLER and K. A. BOHACEK (Stahl u. Eisen, 1938, 58, 402—405).—Hot-galvanised wire is classified according to whether it is stripped of some of the Zn acquired in the bath, or whether it retains its thick galvanised coating; in the former case the coating varies between 30 and 150 g./sq. m., and in the latter between 225 and 300 g./sq. m. In the former case either the Pb-Zn or the alloy-galvanising process is adopted, the latter giving the better finish. Reference is made to the Crapo and galvannealing processes and to recent developments in Germany. A short account is given also of a wire-galvanising plant. The economics of the processes are examined. C. M. A.

**Present position regarding hot-galvanising of steel.** R. HAARMANN and W. RÄDEKER (Stahl u. Eisen, 1938, 58, 397—401).—The literature is reviewed. C. M. A.

**Regeneration of [metal] pickling acid waste.** F. HEINRICH (Stahl u. Eisen, 1938, 58, 617—623).—The  $[\text{FeSO}_4]$  of the regenerated waste should be so low as to have no detrimental effects on the goods pickled, and the crystallising plant should be economically efficient. The solubility of  $\text{FeSO}_4$  in  $\text{H}_2\text{O}$  containing  $\text{H}_2\text{SO}_4$  rises with the temp. up to  $60^\circ$  and falls with  $[\text{H}_2\text{SO}_4]$ . The crystal pptn. of  $\text{FeSO}_4$  is stimulated by concn. of the brine and maintenance of a min. temp. and max. acid content. The different methods of regeneration are compared and the effect of the  $\text{FeSO}_4$  content on the rate of pickling is reviewed. The selection of continuous or intermittent regeneration and the method of increasing concn. must be determined by works conditions. Means of avoiding dilution of the pickling waste through steam and methods of effecting heat economy are discussed, and some crystallising plants are described. Reference is made to the effect of scum in a continuous process, to the economic efficiency of the regeneration of pickling acid wastes, and to the possible uses of the recovered  $\text{FeSO}_4$ . C. M. A.

**Utilisation and regeneration of iron sulphate from pickling acid waste.** F. SIERP (Stahl u. Eisen, 1938, 58, 491—497).—A review. C. M. A.

**Pickling steel [for enamelling].** B. T. SWEELY (Steel, 1937, 101, No. 20, 56, 74—75).—Pickling of steel in "aq. FeS" followed by a Ni flash promoted the adherence of enamel. Pickling in  $\text{H}_2\text{SO}_4 + \text{HNO}_3$  mixture followed by a Ni flash was not advantageous. Good results were obtained by passing  $\text{H}_2\text{S}$  into the  $\text{H}_2\text{SO}_4$  pickling bath. R. B. C.

**Surface preparation.** Various methods of treatment prior to painting and enamelling steel-work. E. ARMSTRONG (Auto. Eng., 1938, 28, 187—188, 273—275).—Various types of primers, paints, and enamels were applied to samples of degreased and sand-blasted mild steel either directly, or after Parkerising or Bonderising (phosphate treatment), and the samples were exposed to a salt spray. Tabulated data showing the relative protective efficiencies of the various treatments and finishes employed illustrate the advantages of phosphate treatment. R. B. C.

**Rate of corrosion of steel in the course of prolonged exposure to a rural atmosphere.** K. DAEVES and K. F. MEWES (Stahl u. Eisen, 1938, 58, 841—842).—Measurement of the change in dimensions which had taken place in four bars of steel (C 0.25—0.40, Cu 0.2—0.3, and P 0.110—0.160%) after 60 years' exposure in a rural atm. showed that the average rate of corrosion was 20—40 g./sq. m./year. The rate of corrosion decreased each year, and in the final year was only 2—3 g./sq. m. This diminishing rate of corrosion explains the good state of preservation of Fe parts exposed to rural atm. M. A.

**Behaviour of chrome-nickel [steel] alloys in phosphoric acid solutions.** W. C. WALLIN and C. S. GROVE, jun. (J. Elisha Mitchell Sci. Soc., 1935, 51, 214—215).—Tests for 15 steels in 0.1—5.0N- $\text{H}_3\text{PO}_4$  are described. The max. rate of corrosion was attained in 100—200 hr. The resistance of an alloy at one concn. was not a reliable guide to its behaviour at another. The rate of corrosion was not correlated with the amount of Cr or Ni in the alloy. The best resistance for all  $\text{H}_3\text{PO}_4$  concns. was obtained with Cr 10, Ni 20%; Cr 18, Ni 8%; and Cr 29, Ni 9%. CH. ABS. (e)

**Corrosion of steel by cathodic polarisation.** A. HILDEBRAND (Mitt. Kohle u. Eisenforsch. G.m.b.H., 1937, 1, 199—212).—From investigations on the loss in wt. of 0.12% C steel in dil. aq. NaCl and  $\text{Na}_2\text{SO}_4$  it has been established that under prolonged cathodic polarisation corrosion decreases as c.d. increases. Complete protection is not obtained at c.d. calc. by Faraday's law from the loss in wt. in absence of an applied current, but only at higher c.d.; this is explained by the action of local couples. The same findings apply in general to the use of a discontinuous polarising current, but the current required in practice to give complete protection can be adjusted more closely to the theoretical val. by suitable adjustment of its characteristics. The most favourable conditions for protection can be determined only by corrosion tests under the particular type of polarisation. R. B. C.

**Use of the Lovibond Tintometer in colorimetric analysis as applied to steel.** B. BAGSHAWE (J.S.C.I., 1938, 57, 260—265).—The Lovibond Tintometer has been applied to the colorimetric determination of small amounts of Cu, Ti, and Cr in steels and curves are produced expressing metal concn. in terms of the Lovibond colour scale. Cu is determined by measurement of the blue amine colour after isolation by the thiosulphate method, Ti by the  $\text{H}_2\text{O}_2$  reaction following a cupferron pptn., and Cr by the diphenylcarbazide reaction after removing Fe etc. with  $\text{Na}_2\text{O}_2$  and NaOH.

**Volatilising chromium as chromyl chloride. Rapid method applicable to determination of manganese in stainless steel.** F. W. SMITH (Ind. Eng. Chem. [Anal.], 1938, 10, 360—364).—In the determination of Mn in stainless and other high-Cr steels, sufficient Cr can be rapidly volatilised as  $\text{CrO}_2\text{Cl}_2$  by treatment with NaCl in  $\text{HClO}_4$  solution to eliminate interference in the persulphate- $\text{AsO}_3^{3-}$  method for Mn. A sample (1 g.) is dissolved (5—10

min.) in 5 ml. of 6*N*-HCl and 20 ml. of 70—72% HClO<sub>4</sub>, 2—4 g. of NaCl are added gradually, and the CrO<sub>2</sub>Cl<sub>2</sub> is volatilised. Excess of Cl' is removed, and the analysis completed by the usual method. Data comparing the method with the ZnO separation are recorded. The extent of the removal of Cr by various methods of adding NaCl has also been investigated. With alternate additions of NaCl to a HClO<sub>4</sub> solution and rinsing of the containing vessel, 99% of the Cr from steels containing up to 25% Cr can be volatilised. When the presence of Na salts is undesirable an equally effective, but longer, method involves the use of small vols. of HCl. A preliminary survey of the effect of the NaCl-HClO<sub>4</sub> treatment on the determination of other elements shows a loss of As and Sn, little, if any, of Fe, and none of Se, Al, P, V, Mo, Ti, Co, Ni, Cu, Nb, Zr, W, U, Zn, Be, B, or S, but interferences occur in the determination of Se, Ti (by cupferron), Al, and W by certain methods.

L. S. T.

**Determination of boron in steel by special organic reagents.** H. A. KAR (Met. and Alloys, 1938, 9, 175—177).—Methods of determining small amounts of B in steel are outlined and the difficulties associated with each method enumerated. Two new methods giving accurate results are described. (A) The steel sample is dissolved in dil. H<sub>2</sub>SO<sub>4</sub>, the solution oxidised with H<sub>2</sub>O<sub>2</sub>, cooled, and filtered, and the filtrate electrolysed (Hg cathode) to separate Fe, Mn, Cr, etc. The solution is filtered, acidified with conc. H<sub>2</sub>SO<sub>4</sub>, and evaporated to fumes, then quinalizarin solution is added, the solution diluted with conc. H<sub>2</sub>SO<sub>4</sub>, and the blue colour compared with that of a standard. (B) The steel sample is dissolved in dil. HCl. The solution is oxidised (KClO<sub>3</sub>) and boiled, then transferred to a Pt dish, and oxidised (H<sub>2</sub>O<sub>2</sub>). The solution is boiled, cooled, diluted to 200 c.c., and half is filtered into a Pt dish. The filtrate is acidified with HCl, 0.5 g. of H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> and 0.5 g. of curcumin solution are added for each estimated 0.01% B present, and the solution evaporated to dryness. The salts are dissolved in H<sub>2</sub>O, the solution is filtered, and the filtrate discarded. The B is washed from the filter-paper with EtOH, the solution diluted to 50 or 100 c.c. with EtOH, and the colour compared with that of a standard.

M. A.

**Hydrogen-reduction method for determination of oxygen in steel.** J. G. THOMPSON and V. C. F. HOLM (J. Res. Nat. Bur. Stand., 1938, 21, 79—86).—An examination has been made of the modified Ledebur method devised by Brower *et al.* in which the finely-milled sample is preheated in H<sub>2</sub> at 500—550° in order to remove surface O from the millings, followed by high-frequency induction heating in order to maintain the sample at 1200° for the determination of the remainder of the O. The method yields accurate results for the total O content of plain C steels, but low yields are experienced with steels containing large amounts of Al<sub>2</sub>O<sub>3</sub> or FeO.

C. R. H.

**Determinations of oxygen in alloy steels.** J. G. THOMPSON and V. C. F. HOLM (J. Res. Nat. Bur. Stand., 1938, 21, 87—93).—Comparison has been made between the modified reduction method (cf.

preceding abstract) and the vac. fusion method of Vacher and Jordan (cf. B., 1931, 976). Agreement is generally good, but where one method gives a higher O val. than the other, the higher val. is probably more nearly correct. The former method is less susceptible to Mn interference than the latter.

C. R. H.

**Influence of alloying elements on crystallisation of copper. I. Small additions and the effect of atomic structure.** L. NORTHCOTT (J. Inst. Metals, 1938, 62, Advance copy, 9—28).—The effect on the size of the columnar crystals of cast Cu of additions of 0.1—2% of numerous elements has been determined, using special moulds to ensure unidirectional solidification. The results show that the effect on crystal size is a characteristic property of each individual element and is quite independent of differences in lattice type or dimensions, but seems to be due to the adsorption of foreign atoms on the surface of the growing crystals. The "crystal-growth restriction factor" (*R*) appears to be related to the valency and at. structure of the added element, since elements of high valency are usually more effective than those of low in refining the grain size, although the arrangement of the valency electrons is also important. This empirical relationship can be expressed in a formula of the type  $R = N^1/S'C$ , where *N* is the no. of effective electrons, *S'* the screening factor to allow for variation in degree of binding, and *C* a const. which depends on the second quantum no. Experimental results for 35 elements are in agreement with this theoretical deduction.

A. R. P.

**Oxidation-resistance in copper alloys.** L. E. PRICE and G. J. THOMAS (J. Inst. Metals, 1938, 63, Advance copy, 253—260).—From theoretical considerations based on the results of other investigators it is shown that if an alloying constituent is to act as a protection against the high-temp. oxidation of a metal it should have a high affinity for O and its oxide should have a high electrical resistance; the radii of the cations in the protective oxide should also be small and this oxide must not be deficient in cations. These conditions are fulfilled by Be and Al additions to Cu, but the protective power of the oxide films produced under normal conditions is  $\ll$  the theoretical. By selective oxidation of the Be or Al so as to produce a film free from Cu oxides the protective power of these metals is considerably increased, *e.g.*, to a val. 200,000 times as great as that of Cu coated with its own oxide. Thus such a protective film is produced on a 5% Al-Cu alloy by heating it at 80° for 15 min. in H<sub>2</sub> containing a 0.1 mm. partial pressure of H<sub>2</sub>O, which is obtained by bubbling the H<sub>2</sub> through 84 wt.-% H<sub>2</sub>SO<sub>4</sub>; after this treatment the alloy remains practically unaffected on heating for 4 hr. in air, whereas the untreated alloy becomes covered with a thick black oxide film under these conditions.

A. R. P.

**Casting of high-strength brasses.** J. E. NEWSON (Metal Ind. [Lond.], 1938, 52, 625—630).—The collective effect of certain physical factors, *viz.*, sp. heat, latent heat of fusion, thermal conductivity, freezing range, casting temp., and mould material, on the primary crystallisation of high-strength brasses

cast in sand is discussed. Experimental evidence in favour of certain assumptions regarding recrystallisation and grain growth in large castings is given.

P. G. McC.

**Non-ferrous foundry practice. IX. Unsoundness in bronze castings.** J. LAING and R. T. ROLFE (*Metal Ind.* [Lond.], 1938, 53, 51—54; cf. B., 1938, 794).—The nature of various kinds of porosity encountered in bronze castings is discussed and evidence of the prejudicial nature in this direction of reducing atm. (especially CO) present during melting is reviewed. Experimental results are given which endorse the val. of melting in crucibles and using a cover.

P. G. McC.

**Relief of internal stress in [non-ferrous metal] castings.** L. E. BENSON and H. ALLISON (*Metal Ind.* [Lond.], 1938, 53, 78).—A procedure is described for determining the degree of stress relief obtained in castings after different annealing treatments, and experimental particulars are tabulated for gunmetal and a high-tensile bronze, which indicate the min. annealing temp. required for the removal of stress in these alloys.

P. G. McC.

**Monel metal solves problem of economic pickling crates.** N. C. MARPLES (*Sheet Met. Ind.*, 1938, 12, 456—457).—The advantages, *e.g.*, increased service life, of monel metal over wood or bronze for pickling-crate construction are discussed.

R. B. C.

**Metallurgy of zinc and coal distillation.** LATOURTE (*Rev. l'Ind. Min.*, 1938, 247—256).—The low thermal conductivity of the charge and the consequent small size of the furnaces, combined with manual charging and discharging, are inherent disadvantages in the classical method for the manufacture of Zn which are found again in the problems of coal distillation. The manner in which the experience obtained in the latter process may be applied to the case of Zn is indicated and an example of such collaboration is the continuous New Jersey process, details of which are outlined. The help given by this experience to the thermal process in its competition with the electrolytic process is pointed out.

P. G. McC.

**Irvinebank tin smelter.** W. EDLINGER (*Metal u. Erz*, 1938, 35, 396—398).—Smelting and refining procedure, carried out at Irvinebank, Queensland, yielding 99.580% Sn, is described.

E. S. H.

**Determining the corrosion-resistance of tinplate. The hydrogen-evolution test.** V. W. VAURIO, B. S. CLARK, and R. H. LUECK (*Ind. Eng. Chem.* [Anal.], 1938, 10, 368—374).—The test worked out for the corrosion of tinplate is based on the rate of formation of  $H_2$  from a die-formed sample by 1N-HCl. Apparatus, procedure, typical results, and limitations of the method are described. Correlation of the  $H_2$ -evolution vals. with can-service vals. of plain cans packed with various fruits in their syrups indicates that the test gives satisfactory predictions of the expected service life of the cans. Certain alloying constituents of the steel base, *e.g.*, Cu, affect the service vals. with some foods, but do not show a corresponding effect on the  $H_2$ -evolution val.

L. S. T.

**Determination of arsenic, antimony, and tin in lead-, tin-, and copper-base alloys.** J. A. SCHERRER (*J. Res. Nat. Bur. Stand.*, 1938, 21, 95—104).—As, Sb, and Sn are separated from most of the Pb in Pb-base alloys by digestion in HF,  $HNO_3$ , and  $H_2SO_4$ , followed by removal of  $HNO_3$ , and from most of the Cu, Zn, and Pb in brasses and bronzes by digestion in  $HNO_3$  or by pptn. with aq.  $NH_3$ , followed, in either case, by redissolving in  $H_2SO_4$ . As, Sb, and Sn are then successively removed from solution by fractional distillation according to the author's method (cf. A., 1936, 812) and subsequently titrated.

C. R. H.

**Lead for use in chemical plant.** B. JONES (*J.S.C.I.*, 1938, 59, 251—259).—Some of the physical properties of Pb and its alloys suitable for use in chemical plant are considered. The importance of softness as a practical asset is pointed out and the effects of deformation on the crystal structure of commercially pure Pb are illustrated. The behaviour of Pb under conditions of temp. variations is considered and the results of vibration tests are given. The addition of Cu to Pb is discussed and the effects of 0.06% Cu on softness, crystal structure, and vibration-resistance are described. The properties of Te-Pb are briefly reviewed and the results of "thermal creep" tests and vibration tests indicated. The effects of adding Cu to Te-Pb are described. It is shown that the crystal structure of small castings of Cu-Te-Pb is capable of extreme refinement; this refinement is crit. and takes the form of a transformation in structure coinciding with a Cu content of 0.06%. The practical importance of the addition of Cu to Te-Pb is revealed in the crystal structure of welded joints where the formation of coarse structures is prevented.

**Rapid analysis of type metals.** L. LAURENT (*Ann. Chim. Analyt.*, 1938, [iii], 20, 208).—Sb is determined by its insolubility in aq. KI-HCl, Pb as  $PbSO_4$ , and Sn colorimetrically by addition of AuCl to a solution of the alloy in aq. HCl.

E. C. S.

**Thermal expansion and effects of heat-treatments on growth, density, and structure of some heat-resisting alloys.** P. HIDNERT (*J. Res. Nat. Bur. Stand.*, 1938, 20, 809—824).—Coeffs. of linear expansion over the range 20—1000° are given for an alloy of Fe-Cr-Al and for three alloys of Fe-Cr-Al-Co. No polymorphic transition was observed between 20° and 1000°. Heat-treatment up to 1400° caused growth of the order 1—3%, with increase in alloy densities. Excessive grain growth occurred at elevated temp.

M. R.

**Refractory gold tailing.** L. M. ABELL and H. W. GARTRELL (*Chem. Eng. Min. Rev.*, 1938, 30, 378).—A sample from a large dump of weathered siliceous tailing assaying 2 dwt. of Au per ton was examined, but no profitable method of extraction could be suggested.

L. N.

**Analysis of dental gold alloys.** R. GILCHRIST (*J. Res. Nat. Bur. Stand.*, 1938, 20, 745—771).—Details are given of the separation and gravimetric determination of Ag, Ir, Sn, Au, In, Cu, Zn, Ni, Pd, Rh, and Pt. AgCl and Ir are pptd. when the alloy

is decomposed by aqua regia. Sn is first separated as stannic acid at  $p_H$  1.5, converted into sulphate, and reprecipitated by hydrolysis. Au is precipitated by  $\text{NaNO}_2$ , the In, Cu, Zn, and Ni being collectively separated from Rh and Pt, at the alkalinity controlled by thymolphthalein, and subsequently determined, in order, by precipitation. In by aq.  $\text{NH}_3$ , Cu and then Zn by  $\text{H}_2\text{S}$ , and Ni by dimethylglyoxime. Mn, Fe, Co, and Cr are quantitatively precipitated in a solution containing  $\text{NO}_2^-$  at the end-point of thymolphthalein. Pb may be separated as carbonate from Pd and Pt at the end-point of xylene-blue. M. R.

**Determination of gold and silver in cyanide [leach] solutions.** W. E. CALDWELL and L. E. SMITH (Ind. Eng. Chem. [Anal.], 1938, 10, 318—319).—Details of an assay method, using 2-l. samples (approx. 66.67 assay tons), the accuracy of which is 0.01—0.02 mg. for Au and 2 mg. for Ag, are given.  $\text{CN}^-$  is first converted into  $\text{Fe}(\text{CN})_6^{4-}$  by treatment with  $\text{FeSO}_4$  and then the Au and Ag are collected by means of a precipitate of  $\text{Hg}-\text{Hg}_2\text{Cl}_2$  in an acid solution (cf. A., 1938, I, 47). L. S. T.

**Permanent-magnet alloys of copper, cobalt, and nickel.** W. DANNÖHL and H. NEUMANN (Z. Metallk., 1938, 30, 217—231).—The miscibility gap in the Co-Cu system is closed by addition of 19% of Ni; with a 1:1 Cu-Co ratio the min. amount of Ni required to produce a homogeneous solid solution is 61% at 20°, 52% at 600°, 40% at 800°, 28% at 1000°, and 19% at 1150°. The equilibria in the ternary system are shown in a ternary diagram and several pseudobinary diagrams for const. Ni contents. The variations in magnetic properties produced by various heat-treatments are also shown in ternary diagrams. Coercivity vals. of 100—1000 oersted, remanence vals. of 7000—2000 gauss, and max. Brinell hardness vals. of 10<sup>6</sup> gauss  $\times$  oersted may be obtained by appropriate heat-treatment of selected alloys. The 20:20:60 Ni-Co-Cu alloy has the max. coercivity and the 24:41:35 alloy the max. Brinell hardness val. These alloys have the advantage over special Fe-base magnet alloys that they can be worked by drilling or turning. A. R. P.

**Experimental smelting of Podozhgor titanomagnetites in an electric furnace.** A. P. LIUBAN (Metallurg, 1935, 10, No. 7, 72—80).—By magnetic concn. of the ore ( $\text{TiO}_2$  8—14, Fe 25—30%), the Fe was increased to 55%. Pig Fe with V 0.6—0.8, S 0.03, and P 0.015% was obtained by smelting the sintered concentrate. The slag contained 35% of  $\text{TiO}_2$ . CH. ABS. (e)

**Properties of rolled molybdenum sheet.** C. E. RANSLEY and H. P. ROOKSBY (J. Inst. Metals, 1938, 62, Advance copy, 29—38).—Pure sintered Mo bars were rolled sideways at 1200° from 12 mm. to 1.5 mm., cleaned from scale, annealed in  $\text{H}_2$  at 970°, and rolled cold to 0.85 mm. in the same direction; the sheet was then either straight- or cross-rolled to 0.1 mm. with suitable intermediate anneals. Annealing at 1000—1100° has no effect on the X-ray pattern and even after annealing at 1600° any preferred orientation introduced by working persists. Severe straight-rolling develops an orientation such that a (110) plane is perpendicular to the direction of rolling.

Cross-rolling, on the other hand, produces a completely-preferred orientation in which a (100) plane is in the plane of the sheet and two other similar planes lie at 45° to the rolling direction and perpendicular to the surface of the sheet. Bend tests on straight-rolled sheet show that there are no preferentially brittle directions apart from the normal inter-fibre brittleness, but similar tests on cross-rolled sheet show marked brittleness at 45° to the rolling direction since the (100) planes oriented in this direction are the cleavage planes in Mo. Cross-rolled sheet is very susceptible to penetration by surface impurities such as Fe, which increases the brittleness, and by  $\text{H}_2$  when the metal is made the cathode in an electrolytic bath. Sheet which has been annealed at 1600° is brittle in all directions owing to the lack of cohesion between the grains after complete recrystallisation. A. R. P.

**Flotation of scheelite.** J. G. HART (Chem. Eng. Min. Rev., 1938, 30, 379—381).—Concentrates assaying 71.5%  $\text{WO}_3$  from ore containing 1.66%  $\text{WO}_3$  were obtained by flotation. Other methods of concentrating gave lower yield. L. N.

**Sintered alloys. I. Copper-nickel-tungsten alloys sintered with a liquid phase present.** G. H. S. PRICE, C. J. SMITHELLS, and S. V. WILLIAMS (J. Inst. Metals, 1938, 62, Advance copy, 117—132).—Mixtures of Cu, Ni, and W powders were pressed into compacts which were then sintered in  $\text{H}_2$  and the progress of alloying was followed by micro-examination and  $d$  measurements. With a 2:5:93 Cu-Ni-W mixture no measurable shrinkage occurs at 950°, but at higher temp. shrinkage proceeds at a rapidly increasing rate, especially above 1300°; at about 1450° the  $d$  reaches almost the theoretical val. Micrographical examination shows that at 1050° the Ni and Cu have alloyed completely, but the size of the W particles is unchanged; at 1300° porosity is much reduced and the W particles have increased in size, and at 1400° voids have practically disappeared and the W exists as large, rather rounded grains embedded in a continuous Ni-Cu phase. The latter dissolves completely in aqua regia, leaving a residue of pure W single crystals the diameters of which are 50—100 times those of the original W grains. The growth of the W is attributed to continuous dissolution of the smallest grains in the liquid Ni-Cu alloy and crystallisation of the dissolved W on the remaining grains. This is confirmed by the behaviour of alloys containing Cu only; here the molten Cu wets the W, but no grain growth occurs since W is insol. in liquid Cu. With Ni only grain growth of the W is slow unless the temp. is  $>1450^\circ$  so that the Ni melts, and with Ni and Cu in a ratio  $<2:1$  the solubility of the W is so much reduced that growth is again slow. The time required for sintering is greater and the sintering temp. higher the coarser are the W particles. Fully-sintered alloys with max.  $d$  have a tensile strength of up to 40 tons/sq. in. A. R. P.

**Metals and binary alloys.** J. SEIGLE (Rev. l'Ind. Min., 1938, 293—312).—A review of present knowledge from the viewpoint of the at. theory and crystal structure. P. G. McC.



**Crystal nuclei and their importance in metal casting.** G. PHRAGMÉN (Jernkont. Ann., 1938, 122, 108—125).—A review. Theories of supercooling and crystallisation are discussed, with reference to grain size in castings. No satisfactory theory of the occurrence and behaviour of crystal nuclei has yet been advanced. Industrial methods for improving the fineness of grain of castings, *e.g.*, shaking, addition of oxides or slag to the melt, controlled cooling, etc., are discussed. It is often impossible to say whether increased fineness of grain is due to an increased or decreased no. of crystal nuclei. M. H. M. A.

**Corrosion of metals.** V. ČUPR (Chem. Listy, 1938, 32, 268—278).—A lecture. R. T.

**Gases and metals.** C. J. SMITHELLS (J. Soc. Arts, 1938, 86, 936—949, 951—964, 971—983).—Cantor lectures.

**Consequences of gas evolution from metals at ordinary temperature.** G. CHAUDRON, A. PORTEVIN, and L. MOREAU (Compt. rend., 1938, 207, 235—237).—Data are recorded for changes in lattice parameter, electrical resistance, and Brinell hardness of Pd and Ta, accompanying the removal of H<sub>2</sub> by ionic bombardment at <100°. Under these conditions the removal of H<sub>2</sub> does not restore these physical properties to their normal vals., since the lattice distortion persists. Heating restores normal vals. H. J. E.

**Determination of sulphur in alloys.** L. SILVERMAN (Ind. Eng. Chem. [Anal.], 1938, 10, 433).—Published procedure (B., 1935, 634) is applicable, with minor individual modifications, to the determination of S in Cu and its alloys with Sn, Pb, Zn, Fe, and Al; monel metal, Ni, Co, and Ni-Co; Fe-Mn, and Fe-Mo. E. S. H.

**Application of micro-mechanical testing methods to metals and textiles.** P. CHEVENARD (Bull. Soc. Ind. Mulhouse, 1938, 104, 265—293).—A lecture.

**Belgian and foreign regulations for welding high-pressure vessels and steam boilers.** M. GUERIN (Rev. Univ. Min., 1938, 81, 407—427).—The choice of weld metal, the arrangement of joints, welding techniques, and the testing of welds are reviewed. R. B. C.

**Gas-welding of Class I pressure vessels.** G. W. PLINKE (Welding J., N.Y., 1936, 15, No. 4, 20—24).—A report on the technique of gas-welding with a slightly reducing flame, designed to bring the ductility of the weld in sections heavier than  $\frac{1}{2}$  in. up to standard ductility for Class I welds. CH. ABS. (e)

**Production of "Raffinal" (high-purity aluminium) and its use in the chemical industry.** A. VON ZEERLEDER and E. ZURBRÜGG (Aluminium, 1938, 20, 365—378).—The different methods of producing highly purified Al are described. A comprehensive investigation has been made of the effects of adding Fe, Si, Cu, Zn, Mg, or Mn in varying amounts up to 1% to Al of high purity. Figures have been obtained for electrical conductivity and tensile strength, and for rate of corrosion by 1% and 5% NaOH, 10% HCl, 20% H<sub>2</sub>SO<sub>4</sub>, 5%, 25%, and

conc. HNO<sub>3</sub>, and NaCl under oxidising conditions. Specimens were tested in the rolled condition and after different heat-treatments. C. E. H.

**Properties of high-purity aluminium.** C. S. TAYLOR, L. A. WILLEY, D. W. SMITH, and J. D. EDWARDS (Met. and Alloys, 1938, 9, 189—192).—The m.p., thermal expansivity, lattice parameter, *d*, electrical and mechanical properties, and radiation characteristics of 99.996% Al have been determined and the methods used in their determination are given. P. G. McC.

**Examination of a sheet of aluminium exposed for 40 years in an urban atmosphere.** C. PANSERI (J. Inst. Metals, 1938, 63, Advance copy, 181—186).—Al sheet 1.3 mm. thick forming part of the cupola on a church in Rome and erected in 1897 was found to be only very slightly corroded after 40 years' exposure to an urban atm. The metal contained Si 1 and Fe 0.6% and had the structure and physical properties characteristic of the "half-hard" state. Much of the sheet was practically unaffected and in the corroded parts the depth of penetration was only 0.05—0.15 mm.; these parts had a tensile strength of only 5.5% and an elongation of 7.4% < the corresponding vals. of the clean parts. The good behaviour of the sheet is attributed to the satisfactory design and ventilation of the cupola, which prevented accumulation of H<sub>2</sub>O. A. R. P.

**Attack on light metals by corrosive tap-waters.** W. MIALKI (Aluminium, 1938, 20, 315—320).—Specimens of commercial Al, Al-Mg, and Al-Mg-Si alloys and of steel were tested in sheet form in waters containing additions of CO<sub>2</sub>, Cl<sub>2</sub>, or H<sub>2</sub>O<sub>2</sub>, and by the salt-spray test. The effect of protecting the light metals by the Eloxal or M.B.V. processes and by lacquering, and the steel by galvanising, lacquering, enamelling, and phosphate treatment, was investigated. The light metals were, in general, superior to the steel, but were only satisfactory in the different waters when protected by the Eloxal process. The salt-spray test was unsuitable for determining the resistance of Al alloys to tap-waters. C. E. H.

**Protection [from corrosion] of aluminium by anodic oxidation.** A. GLAZUNOV (Chem. Listy, 1938, 32, 317—320).—A description of known methods. R. T.

**X-Ray as an aid in manufacture of aluminium castings.** G. E. STOLL and A. T. RUPPE (Trans. Amer. Found. Assoc., 1937, 45, 801—815).—The radiographic testing technique adopted by an American firm in the production of aeroplane carburettors is described. R. B. C.

**Characteristics of copper-aluminium alloys made from aluminium of very high purity.** M. L. V. GAYLER (Mrs. HAUGHTON) (J. Inst. Metals, 1938, 63, Advance copy, 261—275).—The properties of 4% Cu-Al alloy made from French super-pure Al (I) are compared with those of similar alloys made from Hoop's Al (99.96%) (II) and commercial Al (III). Alloy (I) cannot be obtained in a condition such that it age-hardens uniformly to the max. extent at 20°; hot-working followed by prolonged annealing at 500° produces little age-hardening at

room temp. since even after this treatment there are segregated areas of fine  $\text{CuAl}_2$  particles. The lack of homogeneity in age-hardening is attributed to the difficulty of work-hardening the alloy owing to the Al matrix deforming plastically without appreciable work-hardening, probably due to self-annealing, and to the particles of  $\text{CuAl}_2$  acting like the hard particles in a bearing metal. Alloy (I) does not supercool, but its micro- and macro-structures after casting are similar to those of alloy (III). Alloy (II) age-hardens at  $20^\circ$  after hot-forging at  $450^\circ$ , annealing at  $500^\circ$  for 20 hr., and quenching, but, like alloy (I), it does not harden uniformly; addition of 0.32% of Fe reduces the hardening obtained at room temp., but 0.24% of Si has no effect. Fe reduces the grain size of alloys (I) and (II) by pptn. of an insol. Fe-rich constituent along the boundaries. A. R. P.

**Hardening of an aluminium-copper alloy by ageing.** J. CALVET, P. JACQUET, and A. GUINIER (Compt. rend., 1938, 206, 1972—1974).—An Al-Cu alloy (5.2% Cu) was heated for 24 hr. at  $540^\circ$ , plunged in  $\text{H}_2\text{O}$ , and then maintained for various periods at different temp. between  $25^\circ$  and  $300^\circ$ . The hardness was determined, and X-ray and microscopical analyses were made. At  $250$ — $300^\circ$  hardness is produced after the appearance of a new oriented tetragonal phase. Between  $25^\circ$  and  $100^\circ$  no indication of a constituent pptd. from the solid solution was found. X-Rays indicate that Cu atoms separate in small quantities arranged parallel to the faces of the crystal structure of the solid solution. Between  $150^\circ$  and  $200^\circ$  both effects are superposed and the tetragonal phase always separates first. The conditions under which  $\text{Al}_2\text{Cu}$  separates are discussed. W. R. A.

**Influence of iron and magnesium on age-hardening of copper-aluminium alloys.** D. A. PETROV (J. Inst. Metals, 1938, 62, Advance copy, 81—93).—The presence of very small amounts of Fe in Cu-Al alloys practically prevents room-temp. age-hardening; this is completely prevented in the 4% Cu alloy by 2% of Fe since all the Cu is converted into the insol. ternary compound  $\text{Al}_7\text{Cu}_2\text{Fe}$ . Addition of about 0.03% of Mg to commercial Cu-Al alloys which have lost their hardening capacity owing to the presence of a little Fe completely restores it. A. R. P.

**Manufacture and fabrication of light alloys.** J. A. RABBITT (Japan Nickel Rev., 1938, 6, 367—407).—A detailed review dealing with Al alloys containing Ni. P. G. McC.

**Properties of light alloys containing nickel.** T. MISHIMA (Japan Nickel Rev., 1938, 6, 338—366).—Commercial Ni-Al alloys are classified as (1) those possessing high strength and containing Ni, Cu, and Mg as the chief alloying elements, and (2) those having a low heat expansion and low  $d$  and containing a considerable amount of Si in addition to Ni, Cu, and Mg. The composition, physical properties, structure, mechanical properties at room temp. and above, and the required heat-treatment of several of these alloys, including Y alloy, the R.R. series, and a few die-cast alloys in (1) and Lo-Ex, KS 245, and KS 280 in (2), are tabulated and discussed. P. G. McC.

**Industrial applications of nickel-bearing light alloys.** K. TOMIZUKA (Japan Nickel Rev., 1938, 6, 408—424).—A review of the uses of Ni-Al alloys in the various transport and electrical industries. P. G. McC.

**Light metals which age-harden slowly and their use as rivets.** K. MATTHAES (Z. Metallk., 1938, 30, 238—244).—Duralumin rivets harden rapidly at room temp. after quenching, but by reducing the Cu and increasing the Mg content the rate of ageing may be considerably decreased. The best results in this respect combined with the best mechanical properties are obtained with the alloy containing Cu 2.1, Mg 2.1, Si 0.8, and Mn 0.5%, which starts to age-harden only after 9 hr. after quenching from  $500^\circ$ . In the quenched state the yield point is 11.1 kg./sq. mm., the breaking strain 28.9 kg./sq. mm., the elongation 2%, the Brinell hardness 61, and the shear strength 17.6 kg./sq. mm.; after fully ageing at room temp. (50 days) the corresponding vals. are 23.1, 40.4, 21, 102, and 24. Rivets made from the alloy do not split in the head even after ageing, and their shear strength reaches 28.4 kg./sq. mm. when hammered and aged. A. R. P.

**Problem of age-hardening of duralumin.** D. A. PETROV (J. Inst. Metals, 1938, 62, Advance copy, 63—80).—Si and  $\text{Mg}_2\text{Si}$  are shown to take no part in the age-hardening of duralumin at room temp., but  $\text{Mg}_2\text{Si}$  or an Al-Mg-Si-Cu complex produces hardening at elevated temp. Tests on high-purity Cu-Mg-Al alloys indicate that room-temp. hardening is due, not only to the pptn. of  $\text{CuAl}_2$ , but also to pptn. of  $\text{Al}_5\text{Cu}_2\text{Mg}_2$ , and it is considered that the ternary compound also plays a part in the room-temp. hardening of duralumin. A. R. P.

**Dynamic tensile properties of light-metal alloys at low temperatures.** K. BUNGARDT (Z. Metallk., 1938, 30, 235—237).—Cooling to  $-65^\circ$  increases the fatigue limit of duralumin, 4.68% Mg-Al alloy, and 1.7% Mn-Al alloy considerably, but has little effect on that of 7% Al-Mg or 9% Mg-Al alloys. The notched-bar impact val. is affected by cooling in a similar manner to the fatigue limit. A. R. P.

**Heat-treatable aluminium-silicon casting alloys.** G. G. GAUTHIER (Metal Ind. [Lond.], 1938, 52, 631—636).—Alloys containing Mn and Co (in addition to Mg) are successively considered, an account being given of the investigations made for perfecting their heat-treatment and of the variations in mechanical properties under the influence of such treatment and of varying amounts of Si, Fe, Mg, Mn, Co, and Na. P. G. McC.

**Preparation of aluminium alloys for microscopical examination.** G. MANN (Metallurgia, 1938, 18, 121—122).—The major difficulties encountered in this work are indicated and details of satisfactory methods for overcoming them are given. Etching agents are also recommended for sp. purposes. P. G. McC.

**Determination of zinc in light aluminium alloys containing lead, copper, antimony, tin, iron, manganese, and magnesium.** E. PACHE (Chem.-Ztg., 1938, 62, 585).—Pb, Cu, Sb, and Sn

are removed as sulphides, Fe, Mn, and Mg as hydroxides, and the Zn is determined electrolytically as zincate.

F. L. Ü.

**Alloys of magnesium. VII. Mechanical properties of some wrought aluminium-magnesium and silver-aluminium-magnesium alloys.** J. L. HAUGHTON and A. E. L. TATE (*J. Inst. Metals*, 1938, **62**, Advance copy, 95—116; cf. A., 1937, I, 454).—The alloys examined contained 4—10.5% Al and up to 4% Ag. In alloys which have been forged at 380° addition of Ag has little effect on the mechanical properties in the as-forged, annealed, or aged states, and the max. in mechanical properties previously reported to occur at 9% Al could not be confirmed. In forging a 3.5 in. diameter rod the work penetrates to the centre after 40—50% reduction and no further hardening is obtained until after 90% reduction. If the cast ingot is first forged at 380° and then finished at 200° the tensile strength is considerably improved without marked loss of ductility. The presence of Ag in alloys so worked considerably improves the mechanical properties; thus an alloy with Al 8.2 and Ag 4.2% has a tensile strength of >30 tons/sq. in. and an elongation of 5%, whilst the corresponding vals. for an alloy with Al 6.5 and Ag 1.5% are >27 tons and 11.5%, and for an alloy with Al 4 and Ag 1.5% 24 tons and 13.4%; the last alloy has a proof stress of 21.3 tons/sq. in., which compares favourably with this val. for RR 56 Al-base alloy.

A. R. P.

**Avoidance of accidents in the working (machining) of magnesium alloys.** E. RAUSCHER (*Z. Ver. deut. Ing.*, 1938, **82**, 856—858).—Precautions adopted for preventing the explosion of Mg dust, and methods for extinguishing dust fires, are discussed.

R. B. C.

**Hydronalium and Elektron metal.** E. BARONI (*Österr. Chem.-Ztg.*, 1938, **41**, 283—287).—A review of the mechanical properties of these alloys.

E. S. H.

**Spotted appearance on the Eloxal-treated surface of hydronalium pressure-castings.** E. NITZSCHE (*Aluminium*, 1938, **20**, 385—389).—The castings often consist of a coarsely cryst. core and a very thin layer of fine eutectic. The core shows a spotted appearance after Eloxal treatment if exposed by machining etc. The trouble may be overcome by raising the casting temp. so that the alloy is completely fluid when it enters the mould.

C. E. H.

**Process of flow in [alloy] bar material extruded through one or several dies.** H. WALBERT (*Aluminium*, 1938, **20**, 379—385).—The structure of extruded Al-Cu-Mg bars at different stages of the extrusion process is illustrated. There is considerable variation in cryst. size across the bars, and the zones of coarse and fine crystals are distributed differently according to whether only one bar is extruded or several are extruded simultaneously. The difference in tensile strength between the different zones is small.

C. E. H.

**Causes of grain-boundary corrosion in age-hardened alloys of the Al-Cu-Mg series.** M. BOSSHARD and H. HUG (*Aluminium*, 1938, **20**, 389—394).—The influence of a no. of factors on the liability

of two alloys of the Duralumin type to intergranular corrosion in the salt-spray test has been examined. Liability to this form of attack is diminished by reducing the delay between removal from the annealing furnace and quenching, by quenching in H<sub>2</sub>O at as low a temp. as possible, and by ageing below 100°. Variations in the annealing medium or time have only a slight and indefinite effect. Thin sheets are more subject than thicker sheets.

C. E. H.

**Electrolytic galvanising and tinning of iron and steel.** G. ELSSNER (*Stahl u. Eisen*, 1938, **58**, 405—409).—Electrolytic and hot-galvanising processes are compared, the many advantages of the former being stressed and outlined. Reference is made to the various chemical and electrolytic descaling processes, and the relative advantages of acid and alkaline electrolytes are discussed. The recent introduction of the bright Zn electrolyte, consisting of an alkaline Zn(CN)<sub>2</sub> salt bath containing Mo, has greatly increased the corrosion-resistance and appearance of galvanised goods. In a discussion, reference is made to the Tainton descaling and the Bethlehem Steel Co.'s "bethanising" processes.

C. M. A.

**Zinc electroplating of small objects.** J. L. VERTZMAN (*J. Appl. Chem. Russ.*, 1938, **11**, 604—620).—Satisfactory results were not obtained with acid baths. Uniform, adherent deposits were obtained on objects of highly irregular profile, using the following bath: ZnO 43, NaCN 120, NaOH 30, sulphonated castor oil 5 g. per l. (*p<sub>H</sub>* 12.5—13.5), with a c.d. of 5—10 amp. per sq. dm., at 40°, in a revolving drum. The plated surface is passivated by treatment with 10% aq. CrO<sub>3</sub> (10 sec.) or K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> (25—30 sec.).

R. T.

**Adherent nickel-plating.** E. E. HALLS (*Metalurgia*, 1938, **18**, 139—140).—Efficient preparatory operations are essential if proper adherence of the coatings is to be obtained. Pre-cleaning processes must completely cater for the miscellaneous surface conditions encountered in practice and, for this purpose, individual pretreatments for brass and steel components are given as well as a general process in conveyerising practice. Surface oxidation between cleaning operations is also associated with adhesion troubles.

P. G. McC.

**Nickel-plating of aluminium.** M. BALLAY (*Japan Nickel Rev.*, 1938, **6**, 425—427).—The degreased Al surface is anodically treated in 10% aq. Na<sub>2</sub>CO<sub>3</sub> and then immersed for a time in a hot solution consisting of H<sub>2</sub>O 100, Fe perchlorate solution (*d* 1.45) 2—5, and HCl (*d* 1.18) 1—5 l. The reaction in the latter solution is mainly the deposition of Fe by displacement, the Al being only slightly attacked by the acid. A very adherent Ni deposit can be obtained on an Al surface so prepared.

P. G. McC.

**Determination of nickel and boric acid in nickel-plating solutions.** G. S. SMITH (*Analyst*, 1938, **63**, 593—596).—The solution is diluted tenfold, 25 c.c. of the product are neutralised to Me-red, and Ni is determined by titration with 0.5N-KCN until the Ni(CN)<sub>2</sub> ppt. just disappears. The solution is then again neutralised to Me-red with H<sub>2</sub>SO<sub>4</sub>, 10—25 c.c. of neutral glycerin are added, and H<sub>3</sub>BO<sub>3</sub> is

determined by titration (phenolphthalein) with 0.1N-NaOH.  
J. W. S.

**Electrolytic analysis of nickel bronzes and light aluminium alloys.** S. TORRANCE (*Analyst*, 1938, 63, 488—492).—The electrolytic separation of Ni and Zn is carried out in ammoniacal tartrate solution, using a Pt-gauze electrode and a saturated calomel electrode of the Lindsey and Sand type. In the analysis of standard solutions of Ni, Zn, and Al salts,  $\text{Na}_2\text{SO}_3$  is added and the solution electrolysed at 70° with an auxiliary p.d. of 1.0—1.1 v., when Ni is deposited alone. The liquor is then cooled to room temp. and electrolysed (3 amp.) at 20° to deposit Zn. For Ni bronzes (Cu, Sn, Pb, Zn, and Ni, with small amounts of Fe, Al, and Mn), the alloy is dissolved in HCl and  $\text{KClO}_3$ , excess of  $\text{KClO}_3$  is removed, HCl and  $\text{N}_2\text{H}_4\cdot\text{HCl}$  are added and the solution is electrolysed at 50° (p.d. 0.4 v.) to deposit Cu and then at 20° (p.d. 0.7 v.) to deposit Sn + Pb. Fe and Al are removed by pptn. and Ni and Zn are determined as above. Mn is determined by means of  $\text{NaBiO}_3$ . In the case of light Al alloys (Al, Cu, Zn, Ni, and Si, with Fe, Sn, Pb, Mn, and Mg), these are heated with HCl,  $\text{KClO}_3$ , and  $\text{H}_2\text{SO}_4$ , the residue being  $\text{SiO}_2$ . The solution is treated as above to determine Cu, Sn + Pb, Ni, and Zn electrolytically. Fe and Mn are pptd. as sulphides and determined gravimetrically. The filtrate contains Mg and Al. The Mg is pptd. by 8-hydroxyquinoline, which does not ppt. the Al in caustic tartrate solution. Al is determined by difference. Full details and sample analyses of all applications are given.  
T. H. G.

**Ammonia in the electrodeposition of brass.** L. C. PAN (*Trans. Electrochem. Soc.*, 1938, 74, Preprint 9, 125—143).—Addition of 0.2—1.5 g. of  $\text{NH}_3$  per l. to the complex cyanide bath enables bright yellow deposits of brass to be obtained over wide variations in the cathode c.d. and in the Cu : Zn ratio of the electrolyte. The Cu : Zn ratio of the deposit depends on the operating conditions, but in presence of  $\text{NH}_3$  the composition of the plate may alter without appreciable change in its colour.  $\text{NH}_3$  improves the cathode efficiency.  
J. W. C.

**Current-density variations in the electrolysis of magnesium from carnallite.** A. J. TAITZ (*Legk. Metal*, 1935, 4, No. 9, 14—22).—Variations of c.d. from 0.5 to 2.0 amp./sq. cm. do not affect the current efficiency.  
CH. ABS. (e)

**[Electrodeposition of aluminium from] aluminium bromide [solutions].** C. C. DOWNIE (*Metallurgia*, 1938, 18, 134).—The solution consists of  $\text{AlBr}_3$  (with or without  $\text{AlCl}_3$ ), EtBr,  $\text{C}_6\text{H}_6$ , and xylene, the EtBr being added to prevent the solution from being affected by the atm. Absence of moisture and external cooling by running  $\text{H}_2\text{O}$  are required and the plated metal is clean, smooth, and strongly adherent.  
P. G. McC.

**Determination of aluminium in certain non-ferrous materials by use of ammonium aurintricarboxylate.** J. A. SCHERRER and W. D. MOGERMAN (*J. Res. Nat. Bur. Stand.*, 1938, 21, 105—111).—A dil.  $\text{H}_2\text{SO}_4$  solution of the material is electrolysed in a Hg-cathode cell to remove interfering elements.

Al is determined colorimetrically by adding HCl, AcOH, and  $\text{NH}_4$  aurintricarboxylate, followed by neutralisation with aq.  $\text{NH}_3$  and comparison with standard solutions. The depth of colour is affected by the rate of neutralisation. If the solution is re-acidified and re-neutralised, any trouble due to fading is considerably reduced. From a study of such variations a reliable technique has been developed which is suitable for the determination of 0.02—0.08 mg. of Al. Of those elements which interfere and are not removed by electrolysis, Be is the only one the removal of which is essential. Details for this are given.  $\text{HNO}_3$ ,  $\text{SO}_2$ , HF, and large amounts of  $\text{H}_3\text{PO}_4$  bleach the colour, but, with the exception of the last, they can be removed by gently fuming it with  $\text{H}_2\text{SO}_4$ .  
C. R. H.

**Erratum.**—On p. 794, col. 1, line 30, for BUCHOVER read BUCHNER.

**Reaction agglomerating furnace.**—See I. Ore-roasting furnaces. Steels for petroleum industry. Cathodic protection of metals.—See II. Metallising pottery. Steelworks refractories. Steel-furnace bricks. Effect of fluxes on cupola linings.—See VIII. Electric salt-bath furnace [for steels]. Mo for electrical heating. Welding electrodes.—See XI. Rust-preventive paints.—See XIII. Rubber in plating industry.—See XIV. Corrosion of Fe and Pb in soils.—See XVI. Water-pipe corrosion control.—See XXIII.

See also A., I, 447, Systems Mn-Bi, Co-Sb, Ni-Sb, As-Sb, Structure of Cu and Ni in certain Cu-Ni alloys. Co-Sn alloys compared with Fe-Sn and Ni-Sn alloys. Co-W and Cd-Ir alloys. Phases  $\text{CoTe-CoTe}_2$  and  $\text{NiTe-NiTe}_2$ . Co-Mo alloys. 448, Cu, Au. Zn-Cu alloy. Alloys of Sn with Bi. Liquid layers in Cu-Fe alloys. Cu-Al and Be-Ni alloys. Alloys of Be with Ag, Pd, and Au. Systems Ti-Fe and Mg-Cu-Al. 449, Ternary Fe-Ni-Al alloys. Au-Cu, Au-Ag, Ag-Cu, and Cu-Ni alloys. System Ni-Mo. Austenite-martensite transformation. Solid solubility in alloys. 462, Passivity of Fe to natural waters. 464, Solid solutions of Mg-Cd alloys. 465, Cu and Ag deposition. 468, Isolation of Eu. 469, Mutual displacement of metals from the vapours of their salts; application to corrosion. 478, Phase transformation of ferrous alloys.

#### PATENTS.

**Treatment of iron ores containing nickel.** E. H. BROWN and S. J. BRODERICK, Assrs. to BETHLEHEM MINES CORP. (U.S.P. 2,067,874, 12.1.37. Appl., 29.8.32).—Removal of Ni is effected by reducing the ore in  $\text{H}_2$  at 600—700°, chlorinating at 200°, and dissolving the  $\text{NiCl}_2$  in  $\text{H}_2\text{O}$  at 85°. Loss of Fe during leaching can be minimised by chlorinating at 300°, when  $\text{FeCl}_3$  sublimes and can be recovered.  
F. M. L.

**Magnetic iron alloys.** R. A. CURRY, Assr. to INDIANA STEEL PRODUCTS Co. (U.S.P. 2,066,911 and 2,066,926, 5.1.37. Appl., [A] 29.3.35, [B] 20.4.36).—The alloy consists of Fe with (A) Ni 15—25 (20), Be 6—15 (12), and Co 3—5%, or (B) Ni 15—25 (20), Be 0.25—2 (1), and Co 1—10 (3.5)%.  
A. R. P.

**Chill-cast iron-alloy roll.** P. D. MERICA, J. S. VANICK, and T. H. WICKENDEN, Assrs. to INTERNAT. NICKEL CO., INC. (U.S.P. 2,066,848, 5.1.37. Appl., 26.12.34).—The Fe contains C 2—4 (3), Si 0.25—2 (1), Mn 0.25—2 (0.6), Ni 2—10 (4.5), Cr  $\geq$  4 (1.5)%, and a little Mo as hardener. A. R. P.

**Manufacture of steel.** RÖCHLING'SCHE EISEN- U. STAHLWERKE GES. M.B.H. (B.P. 485,881, 24.8.36. Ger., 12.9.35).—Steel is produced from low-grade ore, high in  $Al_2O_3$  and  $SiO_2$ , by smelting in a shaft furnace with an acid slag, and desulphurising the metal by blowing in a Thomas converter before refining, e.g., by the open-hearth process. L. C. M.

**Furnaces for heat-treatment of metals.** A. L. SADLER. From S. F. KEENER (B.P. 488,062, 31.7.37).—A soaking pit with facilities to remove ash and slag from the gas-outlet downtake is described. B. M. V.

**Reheating furnace for the heating of steel ingots.** S. SOKAL. From OFENBAU U. INDUSTRIE- ANLAGEN G.M.B.H. (B.P. 488,344, 28.10.37).—Elongated furnaces are divided into at least two chambers by transverse walls which are open at the bottom. The fuel burners are through the long sides and the flue-gas outlets/hot-air inlets are through the short walls of the end chambers and are each capable of taking the gases from all the chambers in one line. Preheating furnaces for ingots and "economisers" (regenerators) for air are inserted in the path of the flue gases. B. M. V.

**Heat-treatment of cast drill bits.** W. C. REA, Assr. to DETACHABLE BIT CORP. OF AMERICA (U.S.P. 2,066,853, 5.1.37. Appl., 18.7.33).—A bit for rotative percussion drilling is cast in a temperable Fe alloy and the cutting portion only is heated to 930—980°, quenched, reheated at 900—920°, and again quenched; the whole bit is then tempered at 232°. A. R. P.

**Rolling of [steel] rails.** A. P. SPOONER, Assr. to BETHEHEM STEEL CO. (U.S.P. 2,067,293, 12.1.37. Appl., 16.7.34).—Steel is hot-rolled to form blooms, which are then rapidly cooled to  $<205^\circ$ , reheated, and rolled to shape. F. M. L.

**Manufacture of hardened [steel] rails.** EISEN- WERK-GES. MAXIMILIANSHÜTTE (B.P. 485,474, 23.11.36. Ger., 23.11.35).—Rails rolled from steel containing C 0.3—0.4, Mn 0.7—0.9, and Si 0.7—0.8%, with or without 0.5% of one or more of the metals Cr, Mo, V, and Ti ( $\geq$  2% in all), are hardened by passing from the rolls into flowing  $H_2O$ . L. C. M.

**Electric heat-treatment of steel rails.** WELD- ING SERVICE, INC., Asses. of R. E. FRICKEY (B.P. 485,966, 16.7.37. U.S., 27.7.36).—A method of hardening the wearing surfaces of the ends of rail sections by controlled heating by electromagnetic induction, followed by quenching, is claimed. L. C. M.

**Annealing furnaces and the like.** W. J. MILLAR, and ELECTRIC RESISTANCE FURNACE CO., LTD. (B.P. 488,285, 7.1.37).—A liquid (oil) seal for the shaft of a fan passing through the base of the furnace is described. B. M. V.

**Annealing pots or covers.** F. BARNÉ (B.P. 485,233, 23.3.37).—The boxes are constructed of

thin cast steel or wrought Fe, and are covered by a coarse wire mesh (of heat-resisting Fe alloy containing Cr 25, Ni 20, and C 1%), the interstices of which are filled with refractory clay. L. C. M.

**Method of annealing.** L. WILSON (U.S.P. 2,078,356, 27.4.37. Appl., 16.10.36).—Coils of wire or stacks of sheets are annealed by radiant heat which is applied over a large proportion of the height (reckoning from the bottom) during the early stages of heating and over a progressively reduced height as the finally desired temp. is approached. B. M. V.

**[Cold]-drawing metal.** H. K. IHRIG (U.S.P. 2,067,530, 12.1.37. Appl., 28.12.35).—Steel tubes are coated with a mixture of lampblack and aq.  $Na_2SiO_3$ ; the coating is allowed to dry, and the tubes are cold-drawn, the coating acting as lubricant. F. M. L.

**Manufacture of composite metallic article.** W. W. TRIGGS. From ANTACIRON, INC. (B.P. 485,718, 10.8.37).—A method of casting a coating of Si-Fe on a steel core, e.g., for use as an agitator in chemical plant, is claimed. L. C. M.

**Coating [ferrous] metal.** V. M. DARSEY, Assr. to PATENTS CORP. (U.S.P. 2,067,007, 5.1.37. Appl., 5.3.34).—The metal is treated in a hot solution containing  $ZnH_2P_2O_7$  15,  $FeSO_4$  2,  $CdSO_4$ ,  $CuSO_4$ , or tartar emetic 2, and  $NaNO_3$  7.5 g./l. Zn- and Mg- base alloys can also be given a protective coating in this solution. A. R. P.

**[Ferrous metal] welding rods.** (A) R. FRANKS, (B) F. M. BECKET and R. FRANKS, Assrs. to UNION CARBIDE & CARBON CORP. (U.S.P. 2,067,630—1, 12.1.37. Appl., [A, B] 28.7.34).—(B) The rod contains Fe with Cr 12—30, Ni 5—30, C 0.01—0.3, Si 0.7—2 (0.7—1), and Nb 0.1—3%, the Nb content being  $\leq$  8 times the C content. (A) It is covered with a flux containing 1—50% of metallic Si. F. M. L.

**Apparatus for detecting cracks or flaws in iron or steel parts.** EQUIPMENT & ENG., CO., LTD., and H. B. SWIFT (B.P. 487,427, 29.1.37).—A.c. magnetic methods are employed and the test-piece forms part of the secondary winding of the transformer. B. M. V.

(A) **[Copper] alloys.** (B, C) **Stainless copper-base alloys.** R. A. WILKINS, Assr. to REVERE COPPER & BRASS, INC. (U.S.P. 2,067,306—8, 12.1.37. Appl., [A] 12.4.34, [B, C] 1.11.34. [A] Renewed 1.7.36).—Claim is made for white-metal alloys containing (A) Cu 65—93 and Ni 4—25 (20.2), the Cu + Ni content being  $\sim$  80—97, Cr 0.5—5 (1.3), Mn 0.5—5 (2.5), Fe 2—15 ( $\leq$  twice the Cr content) (6), and V trace—0.1%; (B) Cu 65—93, Ni 4—20, Cr 0.5—5, Zn  $\geq$  19.5, and V trace—0.1%, the amount of Cr varying roughly linearly and inversely with the Cu content within the ranges specified, and the Cu + Ni content being 80—95%; and (C) Cu 66.5—95.3, Ni 3.6—2.5, Cr 0.5—5.5, Mn 0.5—5.5, and V trace—0.1%, the Cu + Ni content being 89—99%, and the Cr varying as in (B). F. M. L.

**Treatment of complex lead-bearing ore materials.** T. A. MITCHELL, Assr. to HUGHES-MITCHELL PROCESSES, INC. (U.S.P. 2,067,778, 12.1.37. Appl., 3.7.35).—Sulphide ore containing  $ZnS$ ,  $PbS$ ,

and  $\text{FeS}_2$  is roasted in air, then treated with  $\text{Cl}_2$  to convert the  $\text{ZnO}$  into  $\text{ZnCl}_2$ , and the product is further chlorinated in presence of a sulphating agent, *e.g.*,  $\text{ZnS}$  or  $\text{S}$ , to form  $\text{PbSO}_4$ . After heating to  $300^\circ$ ,  $\text{ZnCl}_2$  is leached out with  $\text{H}_2\text{O}$ , the residual  $\text{PbSO}_4$  and  $\text{Fe}_2\text{O}_3$  being boiled with brine, the  $\text{Fe}_2\text{O}_3$  filtered off, and the  $\text{PbCl}_2$  in the filtrate converted into  $\text{PbCO}_3$  by addition of  $\text{Na}_2\text{CO}_3$ , and this is ignited to  $\text{PbO}$ .

F. M. L.

**Treatment of molten metal [lead] with gas.** A. E. HALL, Assr. to UNITED STATES SMELTING, REFINING & MINING Co. (U.S.P. 2,067,394, 12.1.37. Appl., 20.12.33).—To effect the removal of Zn from Pb, the molten metal is circulated and  $\text{Cl}_2$  discharged into the downward stream; the  $\text{ZnCl}_2$  formed is removed, before it reaches the surface, from the upward stream.

F. M. L.

**Treatment of lead and lead alloys.** G. O. SMITH, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 2,067,507, 12.1.37. Appl., 28.8.30).—High-m.p. metals, *e.g.*, Cu, Co, Ni, Ag, are alloyed with Pb or Pb-Sb alloys at a low temp. ( $500^\circ$ ) by treating the molten Pb with the chloride or oxide of the metal and removing  $\text{PbCl}_2$  or  $\text{PbO}$  formed during the reduction process.

F. M. L.

**Production of metallic coatings.** P. W. NOBLE and A. R. POWELL (B.P. 485,977, 9.10.37).—Coatings of alloys consisting of  $\leq 2$  of the metals Cu, Ag, Sn, Au, and the Pt group are produced on metal articles by applying (*e.g.*, as a paste or paint with amyl acetate) a finely-powdered mixture of their oxides, hydroxides, or carbonates, which may also contain finely-divided Au or Pt metals, and heating in a reducing atm. at  $\geq 1000^\circ$ . (Cf. B.P. 483,156; B., 1938, 930.)

L. C. M.

**Recovering metals [gold] from ores etc. [pyrites].** C. R. ARNOLD (U.S.P. 2,067,006, 5.1.37. Appl., 6.6.34).—The material is smelted with fluxes and, if necessary,  $\text{FeS}_2$ , to produce a slag and a FeS matte containing the Au. The matte is granulated and dissolved in  $\text{H}_2\text{SO}_4$  to obtain aq.  $\text{FeSO}_4$  and a residue containing all the Au.

A. R. P.

**Cyanidation and flotation of [gold and silver] ores.** J. M. MORRIS, Assr. to BENGUET CONSOLIDATED MINING Co., S.A. (U.S.P. 2,067,014, 5.1.37. Appl., 3.8.35).—The ore is crushed in stages, the slimes are separated by classification and treated by flotation to recover the vals., and the sands cyanided by percolation.

A. R. P.

**Settling of inorganic mineral slimes.** A. M. HERBSMAN, Assr. to INDUSTRIAL PATENTS, LTD (U.S.P. 2,066,778, 5.1.37. Appl., 27.3.34).—The slime is flocculated by addition of 0.5% of a sulphonated glyceride (*e.g.*, a sulphonated vegetable oil) to the suspension.

A. R. P.

**Alloys of rhodium and nickel.** E. M. WISE and R. F. VINES, Assrs. to INTERNAT. NICKEL Co., INC. (U.S.P. 2,066,870, 5.1.37. Appl., 5.12.34).—An alloy, especially suitable for pen nibs, consists of Ni with 25–65 (35 or 65)% Rh, deoxidised with 0.1% Mg.

A. R. P.

**Alloy.** H. D. FITZPATRICK. From CONSOLIDATED CAR-HEATING Co., INC. (B.P. 485,226, 5.3.37).

—Addition of Be 0.1–5 ( $\pm 1.6$ ) to alloys consisting of Cr 10–35 (20–30), Ni 21–50 (35–40) (Cr + Ni 55–80), and Co 20–50 (25–40), with or without Mo  $\geq 8$  (3–7)%, is claimed to increase the fluidity of the molten metal.

L. C. M.

**Tantalum carbide alloy.** C. W. BALKE, Assr. to RAMET CORP. OF AMERICA (U.S.P. 2,067,166, 12.1.37. Appl., 6.3.33).—A non-porous tool is made by alloying 3–15% of Ni with low C content, deoxidised TaC or NbC containing Ta or Nb 94.25, and C 5.75%.

F. M. L.

**Treatment of materials containing tantalum and/or niobium.** W. W. TRIGGS. From Soc. GÉN. MÉTALLURG. DE HOBOKEN (B.P. 485,318 and 485,797, [A] 23.3.37, [B] 19. and 22.3.37).—(A) Oxidised Ta-Nb concentrate ( $\text{Ta}_2\text{O}_5$  29.8 and  $\text{Nb}_2\text{O}_5$  35.4%, with  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ , etc.) 1000 is reduced by heating in an electric furnace in admixture with 30 : 70 Fe-Al alloy 325, CaO 100, and  $\text{CaF}_2$  32 kg. The resulting alloy (465 kg.) contains Ta 15.0 and Nb 38.3%, and the slag (930 kg.)  $\text{Ta}_2\text{O}_5$  21.3 and  $\text{Nb}_2\text{O}_5$  10.0%. (Cf. B.P. 467,483–4; B., 1937, 1361.) (B) The material is reduced by  $\text{CaC}_2$  (*e.g.*, in an electric furnace), with addition of Fe and slag material. If an insufficiency of  $\text{CaC}_2$  be employed, the Nb : Ta ratio in the alloy is  $>$  in the initial material.

L. C. M.

**Coating magnesium.** H. J. LODEESEN, Assr. to PATENTS CORP. (U.S.P. 2,066,842, 5.1.37. Appl., 22.12.34).—The metal consisting chiefly of Mg is immersed in boiling 1% aq.  $\text{H}_2\text{C}_2\text{O}_4$  until a sufficiently thick protective film is obtained.

A. R. P.

**Manufacture of corrosion-resistant, precipitation-hardenable, clad aluminium alloys.** W. H. A. THIEMANN. From DÜRENER METALLWERKE A.-G. (B.P. 485,292, 17.11.36).—Plates consisting of a core of Al alloy containing Cu 2–5 (4.2), Mg 0.3–2 (0.9), Si 0.2–1.5 (0.4), and Mn 0.2–1.5 (0.2)%, faced with a pptn.-hardenable alloy containing the same alloying metals but without Cu, after solution heat-treatment and quenching, are annealed at  $130$ – $230^\circ$  (*e.g.*, for 2–6 hr. at  $200^\circ$  or 15–50 hr. at  $170^\circ$ ) until a particularly high yield point is attained.

L. C. M.

**Plating metal [aluminium alloys].** ALUMINIUMWALZWERK WÜTÖSCHINGEN GES.M.B.H. (B.P. 485,956, 19.5.37. Ger., 27.5.36).—A corrodible core, *e.g.*, of Al alloy containing Cu 3.5–5.5, Mg 0.3–1.4, Mn 0.3–1.2, Si 0.3–1, and Fe  $<$ 0.5%, is coated with a corrosion-resisting alloy containing, *e.g.*, Mg 0.3–2, Si 0.3–1.5, Mn 0–1.5, and Fe  $<$ 0.5%, by first applying a thin (0.02-mm.) layer of the second alloy or one of lower m.p., *e.g.*, Al containing 1–10% of Sn or Cd, to serve as a solder, by spraying or brushing on a suspension of the powdered alloy, and then heating to the plastic temp. and hot-rolling on the coating alloy.

L. C. M.

**Cleansing agent for light-metal melts.** METALLOCHEM. FABR. A.-G., and W. CLAUS (B.P. 485,698, 27.2.37. Ger., 30.3.36).—The use of briquettes (of greater *d* than that of the melt) consisting of, *e.g.*, a mixture of Pb powder 75, cryolite 15,  $\text{NH}_4\text{Cl}$  9, and  $\text{LiCl}$  1% is claimed.

L. C. M.

**Thermal deposition of metals or other materials.** P. ALEXANDER (B.P. 485,965, 16.7.37. Ger., 17.7.36).—In an apparatus for deposition of metal (*e.g.*, Al) or quartz etc. by sublimation at  $\sim 10^{-3}$  mm., the material in the form of wire, strip, or granules is fed automatically upon the heater, which consists of a grid of W or Mo wire, at such a speed that vaporisation is practically instantaneous, and consequently rapid deterioration of the heater by alloying with the material is avoided. L. C. M.

**Heat-treatment of [metal] articles.** F. S. DENEEN, W. C. DUNN, and C. N. MITCHELL (B.P. 485,455, 19.9.36).—An apparatus for surface-hardening the articles by heating by electromagnetic induction and subsequent quenching is claimed.

L. C. M.

(A, B) Surface-hardening of, (C) electric induction heaters for heat-treatment of, (D) electric heat-treatment of, (E) surface-hardening of hollow, (A—E) metal articles. (A—C) F. S. DENEEN, W. C. DUNN, and C. N. MITCHELL, (D, E) F. S. DENEEN and W. C. DUNN (B.P. 485,651, 485,728—9, and 485,752—3, 19.9.36).—Methods and apparatus for the surface-hardening of (ferrous) metals by local heating by electromagnetic induction and subsequent quenching by jets of cooling liquid are claimed.

L. C. M.

(A) Production of electrode for arc-welding light metals and their alloys. (B) Sheathed electrode for arc-welding aluminium and its alloys. (A) VEREIN. ALUMINIUM-WERKE A.-G. (B) W. W. TRIGGS. From (A) (B.P. 485,676 and 485,741, 4.12.36. Ger., [A] 4.12.35).—The use of electrodes coated (by dipping in the molten composition) with layers of fluxing materials, the inner being of hygroscopic and the outer of non-hygroscopic nature, is claimed; *e.g.*, the inner flux may consist of (A) NaCl 22.5—40, KCl 37.5—50, LiCl 10, and  $5\text{NaF}\cdot 3\text{AlCl}_3$  (I) 9.5—18, or (B) NaCl 5, KCl 5, and chiolite 90, and the outer of (A) NaCl 15—45, KCl 30—50, (I) 5—15, and  $\text{Na}_3\text{AlF}_6$  10—20, or (B) NaCl 35, KCl 40,  $\text{Na}_3\text{AlF}_6$  15, and chiolite 10 pts. L. C. M.

**Electroplating apparatus.** ELECTRICAL RES. PRODUCTS, INC. (B.P. 477,263, 30.6.37. U.S., 9.7.36).—Anode and cathode rotate in opposite directions, and electrolyte is sprayed on the cathode.

J. S. G. T.

**Electrodeposition of nickel-cobalt alloys.** BOZEL-MALETRA SOC. INDUSTR. DE PROD. CHIM. (B.P. 485,288, 17.9.36. Fr., 2.10.35).—Ni-Co alloy (30—60% Co) is deposited from baths containing salts of both metals and buffered at  $\eta_{\text{H}}$  2.5—4.5; *e.g.*, a 50 : 50 alloy may be deposited from an electrolyte at 60° containing  $\text{NiSO}_4$  190,  $\text{CoSO}_4$  47, NaCl 2, KBr 2,  $\text{Na}_2\text{SO}_4$  3,  $\text{K}_2\text{SO}_4$  2, and  $\text{H}_3\text{BO}_3$  30 g./l., using a c.d. of 0.5—5 amp./sq. dm. and anodes of the same composition. L. C. M.

**Protecting an article made of a tarnishable metal such as silver.** ÉTABL. MARRER, BONNIN, LEBEL, & GUIEU (B.P. 485,905, 11.3.37. Fr., 11.3.36).—In order to improve the corrosion-resistance of Rh-plated Ag articles, a preliminary, non-porous layer of Au is deposited electrolytically on the Ag. (Cf. B.P. 480,145; B., 1938, 675.) L. C. M.

**Method of and electrolyte for depositing rhodium.** C. W. KEITEL, ASSR. to BAKER & CO., INC. (U.S.P. 2,067,534, 12.1.37. Appl., 19.9.31).—The electrolyte is prepared by suspending 2 g. of  $(\text{NH}_4)_3\text{Rh}(\text{NO}_2)_6$  in 1 l. of 5% aq.  $\text{H}_2\text{SO}_4$  and boiling until dissolution is complete. The bath is operated at 50°, 4 v., and 20—30 amp./sq. dm. F. M. L.

**Manufacture of electrolytes for plating rhodium.** F. ZIMMERMANN, ASSR. to BAKER & CO., INC. (U.S.P. 2,067,747, 12.1.37. Appl., 19.9.31).—25 g. of  $(\text{NH}_4)_3\text{Rh}(\text{NO}_2)_6$  are suspended in 100 c.c. of  $\text{H}_2\text{O}$  and heated under reflux for 10—30 hr. The resulting solution can be used as an electrolyte in acid, alkaline, or neutral baths. F. M. L.

**Electrolytic production of sodium and other light metals.** E. I. DU PONT DE NEMOURS & CO. (B.P. 485,587, 20.11.36. U.S., 20.11.35. Addn. to B.P. 433,633; B., 1935, 957).—A modified cell is used, consisting of a cylindrical, brick-lined, steel casing, with a central, cylindrical, graphite anode and an annular steel cathode of 1.5 in. greater radius. In order to improve the circulation of the electrolyte the cathode is pierced with a series of holes, equiv. to 4—8% of its area. L. C. M.

[Machine for] surface-hardening of metal articles [cogwheels]. W. W. GROVES. From W. E. SYKES (B.P. 485,231, 19.3.37).

Fe enamel.—See VIII. Magnetic materials. Filaments for lamps. Ni or Ni-alloy electrodes. Purifying Hg.—See XI. Coating [metal] materials with rubber.—See XIV.

## XI.—ELECTROTECHNICS.

**Behaviour of refractory metals, particularly molybdenum, at high temperatures.** New molybdenum electrical-resistance furnace. J. MICHEL (Bull. Acad. roy. Belg., 1938, [v], 24, 333—346).—The variation of the physical properties of Mo with rise of temp. and under various heat-treatments is examined, and loss by volatilisation and the action of  $\text{O}_2$ ,  $\text{N}_2$ ,  $\text{H}_2$ , and impurities from refractories in contact are investigated. A furnace embodying a flat Mo strip ( $2 \times 8$  mm.) formed into a helical coil and heated in an atm. of  $\text{H}_2$  is described. The rigidity of Mo at high temp. renders support necessary only at the ends. N. M. B.

**Use of unsupported molybdenum for electrical heating up to 2000°.** O. DONY-HÉNAULT (Bull. Acad. roy. Belg., 1938, [v], 24, 285—286).—The use of Mo heating elements is discussed. N. M. B.

**Internally-heated electric salt-bath furnace.** F. P. PETERS (Met. & Alloys, 1938, 9, 183—188).—The design, operation, and advantages of the Ajax-Hultgren furnace are briefly reviewed (cf. Davis, B., 1937, 578). The results of experience with three kinds of work, viz., hardening of alloy-steel gears, heat-treatment of hardware parts, and hardening of steel engravings, are given and questions relating to pot life, quality of products, and possible difficulties are answered. P. G. McC.

**Reactions at the positive electrode of the lead accumulator.** V. SINN (Compt. rend., 1938, 206,

1801—1803).—The consumption of  $H_2SO_4$  at the positive plate during discharge is in good agreement with the double-sulphatation theory of Gladstone and Hibbert. A. J. E. W.

**Optical study of the negative plates of a Planté accumulator during working.** G. VALENTI (Nuovo Cim., 1938, 15, 100—103).—Observations by means of a Babinet compensator of the light reflected from the negative plates of a Planté accumulator during charging indicate that dark grey  $Pb_2SO_4$  is formed. This is in agreement with the Féry theory; the mechanism of the charge and discharge processes is discussed. O. J. W.

**Electrolysis of alkali chlorides with the aid of mercury cathodes.** O. DONY-HÉNAULT and A. DE JAER (J.S.C.I., 1938, 57, 265—272; cf. B., 1937, 1073, 1229).—Apparatus and technique are described. Using saturated solutions at 45—60°, an anode of C or Pt and Ta, a cathode of special steel containing Cr, Ni, and Mo (which catalyse the decomp. of the amalgam), and providing centrifugal agitation of the Hg, the current yield increases with the c.d. up to a max. of 5000—6000 amp./sq. m. The yield is higher for NaCl than for KCl, but the increase of yield with increasing c.d. is the greater for KCl. The energy of amalgamation can be recovered partly by addition of amalgam, with the aid of an auxiliary cell, and without using a shunt. E. S. H.

**Electrolytic protection of steam-power plant [against corrosion].** H. BENDFELDT (Arch. Warmewirts., 1938, 19, 123—124).—The Hauptvogel method of protection is diagrammatically described. In the case of a condenser the tubes form the cathode and the anode is insulated. A d.c. (6 v., 2—5 amp.) is employed. R. B. C.

**Metrovick welding electrodes.** ANON. (Metro-Vickers Gaz., 1938, 17, 308—309).—Various types are diagrammatically described. R. B. C.

**Gas-filled electronic tubes.** O. W. LIVINGSTON and W. J. WALKER (Gen. Elect. Rev., 1938, 41, 354—360).—Various types of tube are illustrated, and their applications discussed. R. B. C.

**Cold flow of [electrical] insulating materials.** R. BURNS and I. L. HOPKINS (Amer. Soc. Test. Mat., Consistency Symp., 1937, 71—73).—Methods of parallel-plate plastometry have proved too laborious for engineering or production control. An empirical deformation test which has proved an accurate and reliable method of evaluating insulating materials subject to deformation under heat is described. R. B. C.

**Permanent magnet materials and their heat-treatment.** J. B. PEILE (Heat Treat. Forg., 1938, 24, 245—246).—A review. R. B. C.

**Non-destructive testing of materials by the magnetised powder process.** E. A. W. MÜLLER (Siemens Z., 1938, 18, 249—254).—An illustrated review. R. B. C.

**Measurement of magnetic fields with bismuth spirals and plates.** G. BUBLITZ (Arch. Tech. Mess., 1938, No. 83, 61—63r).—The thermo-electric properties of Bi are reviewed. Resistance anomalies

can be reduced or eliminated by suitable preliminary heat-treatment. R. B. C.

**Fluorescence analysis in ultra-violet light as applied to dyes and fibrous materials.** J. GRANT (J. Soc. Dyers and Col., 1938, 54, 361—366).—The production of ultra-violet light and the use of fluorescent indicators and fluorescence microscopy in the analysis and testing of dyes and examination of textiles are reviewed. R. J. W. R.

**Reproducing daylight.**—See I. Carbonising bituminous coal. Treating gasoline. Cathodic protection of metals.—See II. Conductometric analysis of mixed  $H_2SO_4$ - $BuHSO_4$ .—See III. Microphotometer for cotton. Measurements for acetate-silk fibres. D.-c. conductivity of paper.—See V. Analysis of HCN-air mixtures. Purifying inert gases and  $H_2$ .—See VII. Melting in network-frequency furnaces. Corrosion. Determining B in steel. Magnet alloys. Analysis of Ni bronzes and light Al alloys. Mg from carnallite. Protecting Al from corrosion. X-Rays and Al castings. Applications of Ni-bearing light alloys. Determining Al in non-ferrous materials. Electrodepositing brass. Zinc electroplating. Galvanising. Determining Ni and  $H_3BO_3$  in Ni-plating solutions. Ni-plating Al. Electrodepositing Al. Plating Ni-Co alloys.—See X. Pptn. of blanc fixe. Electron microscope and paint research. Varnish kettle.—See XIII. Determining soil- $p_H$ .—See XVI. Sugar-factory control. Photometric control of colours and  $p_H$  vals.—See XVII. Staling of bread. Determining  $p_H$  of lactic casein.—See XIX. Measuring turbidity of sewage etc. Determining Pb in drinking  $H_2O$ .—See XXIII.

See also A., I, 465, Cu and Ag deposition. Anodic oxidation of acid-ester salts. 468, Isolation of Eu. 477, Determining vol. resistivity of insulating glasses. 478, Electron ultramicroscope. Phase transformation of ferrous alloys. II, 347, Prep. of Ca gluconate and other salts of aldonic acids. 376, Reduction of glutarimide and its derivatives. III, 755, Textile lighting.

#### PATENTS.

**Electric furnaces [for making glass, sodium silicate, etc.].** W. W. TRIGGS. From Y. R. CORNELIUS (B.P. 477,027, 19.3.36).—An arrangement and spacing of submerged electrodes in a polyphase furnace, whereby the charge in the fusion zone is maintained at a uniform temp. without local overheating, is claimed. J. S. G. T.

**Electric accumulators.** COMP. GÉN. D'ÉLECTRICITÉ (B.P. 487,715, 29.4.37. Ger., 15.5.36).—Grids (for pasted electrodes) are formed of a metal or alloy lighter than Pb (Al or Mg, or alloy thereof) attackable by the electrolyte but protected by an incorrodible and non-conducting material (micro-porous rubber or ebonite) which is traversed by numerous small perforations (preferably invisible to the naked eye) filled with a conductor other than Sb or graphite. The active Pb paste may also be deposited in the pores by interaction of a Pb salt with  $H_2SO_4$ . B. M. V.



**Production of electrodes for electric accumulators.** R. D. PONTE and E. MÜLLER (B.P. 479,081, 20.5.37).—Metal, *e.g.*, Cd, Ag, Ni, or Fe, cathodically deposited on the grid by means of a nozzle from an electrolyte under pressure, is anodically oxidised after change of electrolyte. J. S. G. T.

**Negative electrode for alkaline accumulators.** ACCUMULATOREN-FABR. A.-G., and E. LANGGUTH (B.P. 480,109, 15.8.36).—Pores in a porous plate of Cu and/or Ni are filled with Cd and/or Fe.

J. S. G. T.

**Electrodes for secondary cells, especially those having alkaline electrolytes.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 487,607, 24.12.36).—The supporting conductor comprises a porous metal body, and the active mass metal hydroxides or basic salts deposited in the pores by cathodic polarisation from a solution of the nitrate of the metal (Cd or Ni) in presence of inorg. salts of alkali or alkaline-earth metals, Mg, and/or Al, to ensure conductivity. B. M. V.

**[Manufacture of aluminium electrodes for] electrolytic device.** P. ROBINSON and J. L. COLLINS, ASSRS. to SPRAGUE SPECIALTIES CO. (U.S.P. 2,067,703, 12.1.37. Appl., 28.3.31).—Al electrodes for use in condensers or rectifiers are cleaned in a bath containing aq. NaOH and borax or  $\text{Na}_3\text{PO}_4$ ; the bath is then acidified with  $\text{H}_3\text{BO}_3$  and the electrode subjected to oxide film formation while still immersed.

F. M. L.

**Electrolytic condensers and electrolytes therefor.** MALLORY PATENTS HOLDING CO., LTD. (B.P. 475,757, 24.12.36. U.S., 7.1.36).—An electrolyte prepared by boiling a mixture of mannitol (20–25 g.), a solvent, *e.g.*, ethylene glycol or glycerin (85 c.c.), an ionogenetic agent, *e.g.*,  $\text{H}_3\text{BO}_3$  (226 g.), and  $\text{H}_2\text{O}$  is claimed. If desired, aq.  $\text{NH}_3$  (32 c.c. of 28% solution) may be added. J. S. G. T.

**Electrolytic manufacture of [water-soluble] hyposulphite.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 480,334, 20.8.36).—Solutions containing  $\text{HSO}_3^-$  are cathodically reduced at  $>15^\circ$  (25–50°) with a c.d.  $>2$  amp. per 100 c.c. of catholyte while the  $p_{\text{H}}$  is maintained at 4.0–6.5 (4.5–6.0) during the electrolysis. J. S. G. T.

**Electrolytic cell [for chlorine].** L. D. VORCE, ASSR. to WESTVACO CHLORINE PRODUCTS CORP. (U.S.P. 2,078,517, 27.4.37. Appl., 16.10.34).—An anode comprising a ring of C rods is surrounded inside and out with pairs (in succession) of diaphragms, wire mesh, perforated sheet-metal cathodes, and impervious walls. Dimensions are given. B. M. V.

**Electrolytic purification [of mercury].** R. E. VIVIAN, ASSR. to GEN. ELECTRIC CO. (U.S.P. 2,067,361, 12.1.37. Appl., 31.12.34).—Hg is purified, particularly from Fe, by making it the anode in a cell containing 10% aq.  $\text{Na}_2\text{SO}_4$  or  $\text{K}_2\text{SO}_4$  and operating at 50 amp./sq. ft. of anode, 4–6 v., and 16–38°.

F. M. L.

**Production of evacuated or partly evacuated vessels for electrical apparatus.** J. T. SHELVIN. From SIEMENS & HALSKE A.-G. (B.P. 487,679, 18.12.36).—In the construction of apparatus having

walls wholly of ceramic material and metal, the following operations are effected without interruption in a vac. furnace: evacuation, soldering of ceramic parts and lead-in conductors, degassing, and sealing.

B. M. V.

**Manufacture of filaments for electric incandescence lamps.** VEREIN. GLÜHLAMPEN U. ELEKTRIZITÄTS A.-G. (B.P. 481,227, 6.2.37. Austr., 3.3.36).—Refractory metal wire, *e.g.*, of W, is wound (helix or coiled coil) on a core, *e.g.*, of Mo wire, which is protected at intervals by, *e.g.*, shellac, so that on subsequent treatment with conc.  $\text{H}_2\text{SO}_4 + \text{HNO}_3$  the Mo core is dissolved out at all unprotected points only.

J. S. G. T.

**Manufacture of [electric] lamp filament.** R. M. ZABEL, ASSR. to HYGRADE SYLVANIA CORP. (U.S.P. 2,067,746, 12.1.37. Appl., 8.11.34).—W wire is coiled around a Mo mandrel and the whole is then coiled around a steel mandrel of larger diameter. The combination is heated at  $\sim 1100^\circ$ , the steel completely dissolved away, the remainder heated to  $1650^\circ$ , and the Mo removed, leaving the doubly-convoluted W filament.

F. M. L.

**Electrodes and other articles made from nickel or nickel alloy.** N. V. PHILIPS' GLOEILAMPEN-FABR. (B.P. 485,220, 15.2.37. Ger., 17.2.36).—The use of Ni, or Ni alloys in which Ni is the main constituent, containing Zr 0.05–3% and free from Mn and Si is claimed for making the electrodes of incandescence lamps or discharge tubes. The alloy is prepared, *e.g.*, by fusing pure Ni (1000) at  $1500^\circ$  in a reducing atm. and adding Zr (15 g.); it is then cast into moulds, and swaged at 750–800° before rolling or drawing. L. C. M.

**Metallic electric conductors sealed through quartz.** GEN. ELECTRIC CO., LTD., N. L. HARRIS, and J. W. RYDE (B.P. 477,462, 30.6.36).—A strip of oxidisable refractory metal, *e.g.*, of W or Mo,  $>20 \mu$ . thick, is coated (by distillation) with a layer of refractory non-oxidisable metal, *e.g.*, Rh,  $<1 \mu$ . thick, before being sealed into a quartz tube. J. S. G. T.

**Vacuum-tight sealing of tungsten or molybdenum conductors through quartz glass or glasses of high silica content.** GEN. ELECTRIC CO., LTD. From PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 485,786, 12.2.37).—The part of the wire which passes through the glass is coated with a finely-powdered mixture of a readily oxidisable material (*e.g.*, C, Mg, Mo, S, or W) 40–55%, with an inert filler (*e.g.*, kaolin or felspar) and aq. Na silicate as binder. L. C. M.

**Thermionic electrodes.** GEN. ELECTRIC CO., LTD. From PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 476,908, 2.11.36. Addn. to B.P. 451,989; B., 1936, 1214).—Paste of high thermionic emissivity, *e.g.*, molten alkaline-earth nitrate, placed in the gaps between a helix of refractory metal and a solid rod of high thermionic emissivity, is formed into a hard coherent mass by heating. J. S. G. T.

**Production of oxide cathodes by spraying.** EGYESÜLT IZZÓLÁMPA ÉS VILLAGOSSÁGI R./T. (B.P. 476,385, 5.6.36. Hung., 5.6.35).—A coating which

dries quickly is applied to the core and this is followed by a wet coating which is dried at  $<200^{\circ}$ . Thus, *e.g.*, a suspension containing  $C_5H_{11}OAc$  (I) 160 c.c.,  $COMe_2$  160, nitrocellulose 6,  $BaCO_3$  100,  $SrCO_3$  70, and  $CaCO_3$  30 g. is sprayed on a Ni core and then covered, by spraying, with a suspension containing (I) 320 c.c., nitrocellulose 6,  $BaCO_3$  100,  $SrCO_3$  70, and  $CaCO_3$  30 g., and finally dried at  $80-100^{\circ}$ . J. S. G. T.

**Production of oxide cathodes for electric-discharge tubes.** EGYESÜLT IZZÓLÁMPA ÉS VILLAMOSSÁGI R./T. (B.P. 477,767, 5.6.36. Hung., 5.6.35).—A cleaned core, *e.g.*, of W, Mo, or Ta wire, is coated by being drawn through a suspension of, or by spraying with, *e.g.*, a suspension containing nitrocellulose 6 g., amyl acetate 320 c.c.,  $BaCO_3$  100 g.,  $SrCO_3$  76 g., and  $CaCO_3$  30 g., and the coating is dried at  $<200^{\circ}$  and then converted into oxide by heating *in vacuo* at about  $1000^{\circ}$ . J. S. G. T.

**Thermionic electrodes [cathodes] for electric-discharge tubes.** S. A. ABBOTT (B.P. 487,699, 5.2. and 8.6.37).—A hot cathode comprises a carrier body of insulating refractory material ( $SiO_2$ ) of which the surface has been locally reduced (by burning in contact with Mg or Al) and the oxides have been removed. An electron emitter comprising a mixture of refractory metal and compounds of rare or alkaline-earth metal [melted  $Ba(OH)_2$  75, powdered Ni 20, Si 5%; or W 90, BaO 5, Si 5% as a first layer and BaO 80,  $ThO_2$  20% as second layer] is applied to the reduced parts and the whole heated to cause bonding, which is aided by the presence of Si in the emitter mixture. B. M. V.

**Electron-accelerating apparatus.** O. KLEMPERER (B.P. 487,778, 19.12.36).—The electrons are restrained in orbital paths in increasing length by a magnetic field, and accelerated by an electric field alternating in synchronism with the orbital periodic time. B. M. V.

**Electric-discharge devices.** GEN. ELECTRIC CO., LTD. FROM PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 476,066, 21.7.36).—Electrophoresis in the discharge is abnormally increased by using a gas filling compound of a mixture of gases or vapours having different ionisation potentials, *e.g.*, Ne and Hg, whereby the positive column of the discharge acquires a positive characteristic, *i.e.*,  $dV/di$  is positive, where  $i$  is the current through the tube and  $V$  the voltage across it. J. S. G. T.

**Electric-discharge lamps.** GEN. ELECTRIC CO., LTD. FROM PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 476,240, 4.6.36).—In a gas-filled electric-discharge lamp containing Ne with 0.25–2% of A, and/or Kr, and/or Xe, luminescent material composed of Zn silicate or tungstate or  $CaWO_4$  is arranged in contact with the discharge. J. S. G. T.

**Electric-discharge tubes.** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 475,772, 12.4.37. Holl., 14.4.36).—A gas-binding substance, *e.g.*, an alkaline-earth metal, is arranged between the oxide cathode and a fluorescent screen containing a S compound. J. S. G. T.

[Cathode for] electric-discharge tube. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 476,090, 16.2.37. Holl., 19.2.36).—A thin intermediate layer (0.01–0.1  $\mu$ . thick) of Ni is applied between the electron-emitting material and its Cu support. J. S. G. T.

**Electric-discharge tubes comprising secondary-emission electrodes.** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 477,262, 21.6.37. Holl., 23.6.36).—The electrode system comprises at least one secondary-emission electrode, *e.g.*, of Cu or Cu alloy, coated by cataphoresis with a layer of one or more alkaline-earth metal oxides, *e.g.*, BaO and/or Mg, of thickness  $\geq 3$  ( $\geq 1$ )  $\mu$ . J. S. G. T.

[High-pressure] mercury-vapour electric-discharge lamps. SIEMENS ELECTRIC LAMPS & SUPPLIES, LTD., and J. N. ALDINGTON (B.P. 476,190, 13.7.36).—The thermionically-activated tips of the lead-in wires of the lamp are completely immersed in the Hg electrodes of the lamp. J. S. G. T.

**High-pressure metal-vapour electric-discharge devices.** GEN. ELECTRIC CO., LTD., V. J. FRANCIS, and J. W. RYDE (B.P. 477,105, 22.9.36. Addn. to B.P. 461,211).—The resistant sheath arranged between the discharge path and the sealed envelope, in accordance with the prior patent, is composed of quartz or of translucent or transparent material having a softening temp.  $>$  that of quartz. Thus, *e.g.*, the sheath may consist of  $ZrO_2$  80,  $ThO_2$  14, and CaO 6%. J. S. G. T.

**Mercury-cathode discharge tubes.** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 477,816, 3.7.37. Ger., 6.7.36).—The "anchoring" body submerged in the Hg cathode for retaining the cathode spot in position is composed of material, *e.g.*, Ni, which readily oxidises at high temp., coated with a very thin covering of non-oxidisable metal, *e.g.*, Pt, Ir, Pd, or Rh. J. S. G. T.

**Production of semi-conducting coatings for dry-plate rectifiers and light-sensitive cells.** BRIT. THOMSON-HOUSTON Co., LTD. (B.P. 476,790, 21.6.37. Ger., 20.6.36).—Evaporation and deposition of the semi-conductor, *e.g.*, a mixture of Se and I, in the carrier electrode is effected in presence of a neutral gas or vapour, *e.g.*, a rare gas or  $N_2$ , under pressure. J. S. G. T.

**Photoelectric cells.** F. B. DEHN. FROM ZEISS IKON A.-G. (B.P. 476,719, 25.3.37).—A covering of partly or wholly transparent material, impermeable to moisture, *e.g.*, resin, wax, paraffin, or cellulose derivative, is applied to an intermediate layer, *e.g.*, of regenerated cellulose, extending over at least the sensitive layer of the cell. J. S. G. T.

**X-Ray contrast media.** SCHERING-KAHLBAUM A.-G. (B.P. 478,108, 14.5.37. Ger., 15.5.36).—Such a medium for use in stomach examinations is composed of  $BaSO_4$  and 5–10% of dextrin, *e.g.*, erythro-dextrin. J. S. G. T.

**Fluorescent screens.** TELEFUNKEN GES. F. DRAHTL. TELEGRAPHIE M.B.H. (B.P. 478,302, 13.7.36. Ger., 11.7.35).—The  $\lambda$  of light emitted by the screen is increased by replacing part (25%) of the Zn of the Mn-activated Zn silicate of the screen by an alkaline-earth metal (Be). J. S. G. T.

**Fluorescent screens [for electric-discharge lamps etc.].** BRIT. THOMSON-HOUSTON CO., LTD. (B.P. 477,192, 21.1.37. U.S., 22.1.36).—A coating of fluorescent material is applied at spaced intervals to a base member, the thickness of coating being  $>$  that required to complement the spectrum of the discharge if the coating were uniformly applied to the whole of the base member. [Stat. ref.]

J. S. G. T.

**Luminescent screen for electric lamp bulbs or tubes filled with rare gas or mercury vapour.** H. G. G. DESMAREST (B.P. 480,225, 1.5.37. Fr., 2.5.36).—One or more extremely thin and transparent layers (0.01—0.1 mm. thick) formed by a colloidal dispersion of rubber are applied to a transparent support and covered with a luminescent metallic salt, e.g., ZnS.

J. S. G. T.

**Luminescent materials for use in electric lamps and the like.** BRIT. THOMSON-HOUSTON CO., LTD. (B.P. 482,071, 9.4.37. U.S., 9.4.36).—A mixture of CdO and SiO<sub>2</sub>, in substantially the mol. proportions required to form CdSi<sub>2</sub>O<sub>5</sub>, with MnO<sub>2</sub> (the Mn content of the mixture being  $\sim$  1.25 wt.-% of the CdO) is treated at 1100° with a flux containing K or KCl (the K content being  $\sim$  2.1 wt.-% of the CdO) to remove, or render non-detrimental, all traces of Ni, Pb, Cu, Co, and Fe.

J. S. G. T.

**Production and use of secondary electron-emissive substances.** H. G. LUBSZYNSKI (B.P. 481,563, 10.9.36).—A surface capable of emitting secondary electrons (of intensity  $I_p$ ), e.g., a Ni surface treated with UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>, is bombarded by electrons of a given velocity and subsequently by electrons of a higher velocity (and intensity  $I_p$ ) so that the ratio  $I_s/I_p$  in the second bombardment is  $>$  for the first. Secondary electrons emitted in the second bombardment are used to provide signal currents.

J. S. G. T.

**Coating of articles by vaporisation of the coating materials.** B. BERGHAUS (B.P. 481,842, 9.6.37. Ger., 17.6.36).—Fused coating material is vaporised at  $<$  1 atm. in a magnetic field while the material and an accelerating electrode are connected to the poles of a source of electric current.

J. S. G. T.

**Electrical purification of gases.** E. C. ST. JACQUES (B.P. 487,547, 21.6.37).—A cyclone separator has the outer cylindrical wall positively and the axial clean gas outlet negatively charged with electricity.

B. M. V.

**Detection of faults in solid, liquid, or gaseous bodies.** S. SOKOLOFF (B.P. 477,139, 13.5.37).—Ultra-acoustic oscillations, generated by piezo-electric means, after passage through the body, excite a piezo-electric crystal plate, producing a distribution of electrostatic charges which is analysed by television processes to provide a graphical picture of the distribution.

J. S. G. T.

**Electrical insulating material.** ALLGEM. ELEKTRICITÄTS-GES. (B.P. 475,684, 31.8.36. Ger., 31.8.35).—Cellulose tri-ester (triacetate) is deposited on the threads of woven silk fabric.

J. S. G. T.

**Electrical insulating materials.** CALLENDER'S CABLE & CONSTRUCTION CO., LTD., and E. TUNNI-4 L (B.)

CLIFF (B.P. 478,222, 29.9.36).—Powdered SiO<sub>2</sub> (e.g., passing 130—150-mesh) is thoroughly dried by prolonged heating (e.g., 10 hr. at 200°) and mixed with an oil (mineral or vegetable type) or oil compound (e.g., oil-rubber or oil-resin solutions) which has previously been dried by prolonged heating at temp. (e.g., 24 hr. at 120°)  $<$  the b.p. of the oil and under a non-oxidising atm. The mixing is carried out under conditions which inhibit adulteration by metal particles, and vac. is applied after the powder has been "wetted." The product is suitable for filling insulating joints etc. of high-tension cables.

J. W. CR.

**Manufacture of electrically insulating [layered-paper] materials.** BRIT. THOMSON-HOUSTON CO., LTD. (B.P. 475,756, 23.12.36. Ger., 28.12.35).—Synthetic resin varnish is applied to layers of paper having perforations, and the whole bonded by heat and pressure.

J. S. G. T.

**Electrical insulating materials.** SIEMENS & HALSKE A.-G. (B.P. 482,172, 9.3.27. Ger., 9.3.36).—Fluid or semi-fluid materials consisting of polyvinyl compounds containing air and gas bubbles is extruded as strings, bands, etc. through nozzles which widen outwardly so that the bubbles of air and gas expand and the material stretches.

J. S. G. T.

**Electric resistances.** S. E. C. I., SOC. ELETTO-TECNICA CHIM. ITAL. (B.P. 488,293, 29.1.37. Italy, 13.2.36).—Enamelled or cemented-type resistances are enclosed in a case with gas in the space, there being no contact between the resistance and the outer case.

B. M. V.

**Electric resistances or condensers.** STEATIT-MAGNESIA A.-G. (B.P. 488,289, 9.1.37. Ger., 9.1.36).—Resistance layers supported on solid insulating material are protected from effects of humidity and temp. (even tropical) by two or more coats of wood or tung oil (or varnish containing such oil), each layer being heated at 120° and the last to 200°.

B. M. V.

**Fusible elements for protecting electric circuits.** BRIT. THOMSON-HOUSTON CO., LTD. (B.P. 487,829, 7.3.38. Ger., 6.3.37).—Metal of high m.p. is coated with one of lower m.p. and the latter is distributed throughout the cross-section of the conductor by the use of stranded ( $>$  0.5 mm. diameter), concentric or other laminar forms  $>$  0.2 mm. thick.

B. M. V.

**Manufacture of magnetic materials suitable for cores of inductors of low dissipation.** GEN. ELECTRIC CO., LTD., C. G. SMITH, and S. V. WILLIAMS (B.P. 486,057, 30.4.37).—Prior to insulating with shellac and forming into cores, powdered Ni-Fe or Si-Fe alloy is treated with dil. HCl or H<sub>2</sub>SO<sub>4</sub>, respectively, so that  $\sim$  5% is dissolved; the residual metal is washed with deoxidised H<sub>2</sub>O and dried in a reducing atm.

L. C. M.

**Magnetic separators.** H. H. THOMPSON, A. E. DAVIES, and M. D. THOMPSON (B.P. 488,420, 2.11.36 and 5.2.37).—A belt running up an incline is studded with magnetic material and passes over spaced magnetic poles; it is also vibrated.

B. M. V.

**Magnetic separator and concentrator.** R. H. STEARNS, ASSR. to STEARNS MAGNETIC MANUFACTURING CO. (U.S.P. 2,078,513, 27.4.37. Appl., 11.12.33).—The apparatus comprises a roll (forming part of the magnetic circuit) rotating between a sharp pole and a blunt pole, the gap to the former being adjustable. In a wet type of apparatus the wound part of the magnetic circuit is above liquid level, the roll is partly submerged, and the magnetic return yoke is submerged. B. M. V.

[Electrodes for] electrolytic condensers. DUBILIER CONDENSER CO. (1925), LTD. (B.P. 476,688, 10.6.36. U.S., 24.6.35).

Treating  $H_2O$ .—See I. Treating tar emulsions. Voltolised oils. Insulating oils.—See II. Polymerised nitriles.—See III.  $CaC_2$ .  $H_2O_2$ .—See VII. Glass insulators. Refractory glasses. Ceramic material.—See VIII. Magnetic Fe alloys. Heat-treating steel rails. Detecting flaws in Fe or steel. Protecting Ag articles. Heat-treatment of metal articles. Arc-welding light metals. Electroplating apparatus. Rh-plating. Na and other light metals by electrolysis.—See X. Shaped elastic masses. Resins from aldol. Coating composition of high dielectric strength.—See XIII.

## XII.—FATS; OILS; WAXES.

Fat of goat's milk. A. CHOLLET and A. CAMUS (Ann. Falsif., 1938, 31, 224—225; cf. B., 1937, 387).—The sol. (S) and insol. (I) volatile acids, and the sap. and Planchon vals. of the milk fat of two individual goats during one year are recorded. The ratio I/S increases almost regularly from the beginning to the end of lactation. E. C. S.

Extracted cacao-waste fats. K. H. BAUER and L. SEBER (Fette u. Seifen, 1938, 45, 342—345).—Full analyses of 5 or 6 samples of commercial crude and refined fats (I vals. 41.3—44.6, unsaponifiable matter 1.3—2.0%) extracted from factory cacao waste (shells, germs, dust, etc.) are given. The high oleic acid content (up to 41.8% of the fatty acids) of the refined fats appears to be due to a selective accumulation of linoleic and saturated acids (as well as of oxidised acids) in the soap stock during refining. In spite of the removal of some unsaponifiable matter during refining, the high content of unsaponifiable matter (average 1.5%) compared with that of cacao-nib butter (max. 0.4%) remains the best method of identification of these offal fats (cf. Grossfeld, B., 1932, 29). The content of unsaponifiable matter may be used to calculate the approx. content of cacao-shell fat. E. L.

Amount and condition of the fat in cacao shells. (A) H. FINCKE (Fette u. Seifen, 1938, 45, 345—346; cf. preceding abstract). (B) K. H. BAUER and L. SEBER (*Ibid.*, 346).—(A) Whilst fat may not have travelled from the nib into the shells during the hot-air roasting of the cacao examined by Bauer and Seber (*loc. cit.*), it apparently did so in the case of the beans (steam-roasted in large-capacity roasters) examined by Fincke (cf. B., 1926, 73); hence different conditions of processing, rather

than bad sorting, may account for discrepancies between the results of different observers.

(B) Further studies on the point are advocated. E. L.

**Composition of ucuúba fat.** F. RAMOS and R. DE CASTRO AYRES DO NASCIMENTO (Rev. Chim. Ind., 1938, 7, 186—188; cf. B., 1938, 957).—The fat was examined by fractional distillation of the Me esters at 1 mm., the mol. wt. and sap. val. for each 5° fraction being recorded. The fat consists of myristin 70, laurin and lower glycerides 5, stearin and higher glycerides 10, olein and unsaturated glycerides 10, and unsaponifiable matter 5%. F. R. G.

Utilisation of animal carcasses with special reference to production of technical fats and animal-meat meals. W. STEINMANN (Fette u. Seifen, 1938, 45, 338—342).—The principal types of plant for the processing of waste carcasses, slaughterhouse waste, etc. are described. E. L.

Improving the quality of marine-animal fats by conjugated hydrogenation. V. M. PUZANOV (J. Appl. Chem. Russ., 1938, 11, 668—669).—The fishy taste and odour of seal, dolphin, and other marine-animal oils are abolished by subjecting the oils to conjugated hydrogenation, with EtOH (Ni catalyst). R. T.

Report of the American Oil Chemists' Society Fat Analysis Committee (Oil & Soap, 1938, 15, 208).—Slight changes in the specification for the beaker and test-tube for use in the Wiley m.p. test, and in the wording of the hot-plate method for the determination of  $H_2O$  in fats, margarine, etc., are recommended. E. L.

Fats. LXIII. Meso- and micro-methods for analysis of fats. II. Thiocyanogen value. H. P. KAUFMANN and L. HARTWEG (Fette u. Seifen, 1938, 45, 346—349; cf. B., 1938, 545).—A meso- (semi-micro-)method for the determination of the SCN val. of fats, which requires only 0.01—0.03 g. of sample and 4 (for non- and semi-drying oils) to 12 hr. (linseed oil) reaction time, is described. E. L.

Analysis of lard. I. D'O C. COSTA NETTO (Rev. Agron., 1937, 25, 244—315).—Analyses are recorded for 30 samples of Portuguese lard from 2-year-old swine, the mean vals. being  $H_2O$  0.075, impurities insol. in  $CHCl_3$  1.31, ash 0.018, Cl in ash 0.005%, I val. of solid acids 1.16,  $d_4^{20}$  0.928,  $n_D^{20}$  1.4547, acid val. 1.937, sap. val. 195.83, Reichert-Meissl val. 0.84, I val. 63.93. The  $CHCl_3$  extract contained unsaponifiable matter 0.143, fatty acids 89.15, glycerol 10.7%. The acids contain saturated acids 38.47, oleic acid 49.18, and linoleic acid 12.45%. Corresponding data are tabulated for earlier analyses of lards and also for other animal fats, cottonseed, and hydrogenated oils. Methods for determining the m.p. and I val. of fats are compared and full analytical details given. F. R. G.

Oven test as an index of keeping quality [of fats etc.]. N. T. JOYNER and J. E. MCINTYRE (Oil & Soap, 1938, 15, 184—186).—The utility of the Schaal oven test (in which the no. of days are noted before rancidity appears in 50 g. of shortening held at  $\sim 63^\circ$  in a covered squat beaker) is discussed.

Samples of any one kind of fat similarly processed nearly always develop organoleptic rancidity at approx. the same peroxide concn., the time taken to reach the crit. concn. being determined by the rate of peroxide formation, which depends on the stability of the fat. Hence, provided the rancidity-peroxide characteristics have been determined for a given fat, the results of the oven test may be predicted from a measurement of the increase in peroxide val. after 2—4 days in the oven. The fat darkens as it approaches the point of rancidity, and then usually becomes paler, the colour change being sufficiently sharp to serve as a check on estimate of rancidity by odour. In the case of baked goods subjected to the oven test, the fat may be recovered for the purpose of a peroxide determination by leaching the gently-warmed, ground test-piece with successive portions of a mixture of AcOH and  $\text{CHCl}_3$ . E. L.

**Viscosimetry of fats.** H. WOLFF and G. ZEIDLER (*Fette u. Seifen*, 1938, 45, 349—350; cf. Kaufmann and Funke, B., 1938, 810).—The relation of  $\eta$  to I val. is complicated by many factors and does not appear to be usable as an expression of the mol. structure of stand oils. If polymerisation were a simple change of monomeric into a definite polymeric form, the I val. of the product during polymerisation would decrease simply according to the law of mixtures, whilst  $\eta$  would alter logarithmically, so that the relation  $\log \eta/I$  val. should be a straight line. In practice the  $\eta$  increases more than this, especially at higher temp. Further other reactions, e.g., isomerisation, as well as polymerisation, contribute to the fall in I val. when linseed oil is heated. E. L.

**Original and hydrolysed nut oil of the "Dum" palm.** I. UBALDINI (*Annali Chim. Appl.*, 1938, 28, 191—199; cf. B., 1933, 113).—Data for the nut oil (m.p.  $24^\circ$ ,  $d_{20}^{20}$  0.902,  $n_{40}^{40}$  1.4534, acid val. 2.1, sap. val. 225, I val. 27.1, Reichert—Meissl val. 5.58, unsaponifiable matter 1.72%) and for the oils extracted from the plant and its hydrolysate (dil.  $\text{H}_2\text{SO}_4$ ) are tabulated. The nut oil contains octoic (1.31), decoic (2.74), lauric (31.58), myristic (14.68), palmitic (13.69), and stearic acid (4.71%), and unsaturated acids (30.45%). F. O. H.

**Ouricury palm-kernel oil.** R. S. MCKINNEY and G. S. JAMIESON (*Oil & Soap*, 1938, 15, 172—174).—Fruits of *Syagrus (Cocos) coronata* from Florida consisted of fibrous pulp (47.5%, containing 3% of red oil) and the "nut" (shell 76.2, kernel 23.8%). Kernels from Brazil contained 2.4% of  $\text{H}_2\text{O}$  and 69.7% of oil, which deposited only a small amount of "stearin" at  $18^\circ$  and had  $d_{25}^{25}$  0.9221,  $n_{25}^{25}$  1.4543, acid val. 11.2, sap. val. 256.9, I val (Hanus) 14.69, SCN val. 12.78, Reichert—Meissl val. 5.93, Polenske val. 18.38, and unsaponifiable matter 0.27%. The fatty acids consisted (as % on oil) of hexoic 1.66, octoic 9.1, decoic 7.64, lauric 42.7, myristic 8.43, palmitic 7.15, stearic 2.15, arachidic 0.096, oleic 12.18, and linoleic acid 2.04. Notes on the technique employed for the Et ester fractionation of the acids, and on the interpretation of the data therefrom, are given. Apparent indications of the presence of lower homologues of oleic acid derived solely from consideration of the I val. and sap. val. of ester fractions

should be checked by other means as the presence of low-mol. decomp. products may invalidate such simple calculations. E. L.

**Emulsification index of olive oils.** (M.L.E.) M. T. FRANÇOIS, G. P. ARCAÿ, and J. ROUZIQUX (*Ann. Falsif.*, 1938, 31, 211—215).—The emulsification index of 33 samples of North African olive oil was almost const. (61—68). Any slight variations were independent of free acidity. Addition of minute amounts of cottonseed, arachis, colza, or sesame oil produced a marked fall in the index. It is therefore possible to detect adulteration of olive oil with any of these oils. A procedure is suggested for the approx. determination of the extent of adulteration. E. C. S.

**Continuous [steam]-deodorisation of edible oils.** D. K. DEAN and E. H. CHAPIN (*Oil & Soap*, 1938, 15, 200—202).—Plant is described (with some typical results) wherein the preheated oil falls through a de-aerating section situated at the top of the high-vac. deodorising tower, and after further heating flows downward through the deodorising section over a series of bubble trays in countercurrent to the steam admitted at the foot of the tower. The properties of Dowtherm vapour (a eutectic mixture of  $\text{Ph}_2$  and  $\text{Ph}_2\text{O}$ ) and its application as a heating medium for the above process are discussed. E. L.

**Collaborate work of the D[utsche] G[esellschaft für] F[ettforschung]. XI. Oilseed analysis.** G. GREITEMANN (*Fette u. Seifen*, 1938, 45, 350—352).—The German standard methods and proposed modifications thereof for the analysis of oilseeds are detailed for purposes of study and criticism. E. L.

**[American Oil Chemists' Society] Crude Mill Operations Committee report** (*Oil & Soap*, 1938, 15, 211).—The recleaning of cottonseed advocated by Bedell improves the quality of lint cut, but does not appear to be profitable unless the mill produces its own power. The blending of expeller with hydraulic-pressed oils (e.g., 10—30: 90—70) did not reduce, but in some cases increased, the refining loss of the latter. A brief note on lint determination is appended. E. L.

**Report of [American Oil Chemists' Society] Seed Analysis Committee** (*Oil & Soap*, 1938, 15, 207—208).—The method (detailed), wherein the (cotton) seed is weighed and dried prior to fuming with HCl, and the lint is then brushed off the seed, which is again dried and weighed, is preferred to a method involving the determination of  $\text{H}_2\text{O}$  in the lint; certain modifications are suggested for further study. E. L.

**Report of the Soya-bean Analysis Committee of the American Oil Chemists' Society.** R. T. MILNER (*Oil & Soap*, 1938, 15, 214).—Comments on the tentative standard methods for  $\text{H}_2\text{O}$  and oil determinations from the U. S. Regional Soya-bean Industrial Products Laboratory are summarised. Drying of whole beans for 3 hr. and ground beans for 2 hr. at  $130^\circ$  in the Freas oven gives results equal to those obtained by distillation with PhMe. E. L.

**Report of the [American Oil Chemists' Society] Moisture Committee, 1937—8** (Oil & Soap, 1938, 15, 211—213).—It is recommended that the Freas forced-draught oven No. 601/233 previously adopted as a tentative standard (cf. B., 1937, 1080) should be replaced by a standard forced-draught oven (make not specified) which conforms to a standard specification now detailed for functional performance. As a result of reports (detailed) by A. D. RICH and by C. P. BRENNER, the Committee recommend that drying intervals of 3 and 4 hr., respectively, for samples of meal and cottonseed be allowed when the forced-draught oven is used.

E. L.

**Report of 1937—8 activities [on soya-bean oil]. AMER. OIL CHEMISTS' REFINING COMTEE.** (Oil & Soap, 1938, 15, 174—181).—The results of collaborate study are detailed and certain modifications are recommended in the A.O.C.S. (pp. 16 D—16 E) crude soya-bean tentative refining method for the testing of expeller and hydraulic pressed oils; a tentative refining test for crude extracted soya-bean oils is detailed, and some preliminary notes on the testing of extracted clarified soya-bean oil are given.

E. L.

**Report of [American Oil Chemists' Society] Committee on Determination of Soap in Refined Oil** (Oil & Soap, 1938, 15, 209—210).—The following procedures are detailed: (1) free fatty acid method (cf. Spielman *et al.*, B., 1936, 1054): the acid val. is determined before and after treating the oil with HCl; (2) Durst ashing method (cf. B., 1936, 107): Na (as NaCl) is determined in the ash from the oil; (3) Durst HCl-extraction method: a hot HCl extract of the oil is evaporated and analysed for Na; (4) a modification of (3) devised by R. C. Stillman; and (5) EtOH-extraction method (cf. Spielman *et al.*, B., 1937, 941): the EtOH extract of the oil is ashed and titrated with acid. As a result of collaborate studies, (1) is rejected as unreliable; (2) is unsatisfactorily troublesome, though good results can be obtained therefrom with care; (3) and (4) (which is less laborious) gave the best results and are recommended for further study together with (5), which, however, tended to give low results.

E. L.

**Conjugated hydrogenation of hempseed oil with ethyl alcohol.** V. M. PUZANOV (J. Appl. Chem. Russ., 1938, 11, 670—673).—Conjugated hydrogenation of the oil with Ni catalyst gives a product containing considerable amounts of linoleic acid (I), which is only to a small extent further hydrogenated to oleic acid. This is ascribed to the presence of linolenic acid (II), since oils containing (I), but not (II), are readily hydrogenated to oleic acid. Substitution of mixed Cu—Ni for Ni catalysts leads to a lower final pressure (smaller decomp. of MeCHO to CO and CH<sub>4</sub>), prevents hydrolysis of glycerides, and gives lower yields of oleic and saturated acids.

R. T.

**Chemical examination of the fixed oil from the seeds of *Nyctanthes arbortristis*, Linn.** S. K. VASISTHA (J. Benares Hindu Univ., 1938, 2, 343—348).—The kernels formed nearly 56% of the seeds of *N. arbortristis* and gave 14% of oil on extraction with light petroleum. This oil had  $d_{20}^{20}$  0.9157,  $n_D^{20}$  1.4675, sap. val. 185.5, I val. (Hanus) 82.24, Ac val. 19.28, acid

val. 15.75, Reichert—Meissl val. 0.1, and unsaponifiable matter 2.4%. Glycerides of linoleic, oleic, lignoceric, stearic, and palmitic acids, with probably some myristic acid, were present. Vitamin-A, parasitosterol, and a new sterol (named *nycosterol*) (C<sub>27</sub>H<sub>44</sub>O<sub>2</sub>), analogous to phytosterol, were found. This crystallised from the oil (0.2% of oil) on long keeping and had m.p. 222°,  $[\alpha]_D^{20}$  +91°. F. M. F.

**Composition of "Akebi" seed oil.** S. KOMORI and S. UENO (Bull. Chem. Soc. Japan, 1938, 13, 505—507).—Hydrolysis of the seed oil,  $n_D^{20}$  1.4652,  $d_{20}^{20}$  0.9326, acid val. 6.6, sap. val. 254.9, I val. 78.6, Reichert—Meissl val. 49.3, of *Akebia lobata*, Decne. ("Mituba Akebi"), yields AcOH and fatty acids (palmitic 23, stearic 2, oleic 53, and linoleic acid 22%). The oil from *A. quinta*, Decne. ("Itutuba Akebi"), is similar.

R. S. C.

**Chemical composition and examination of *Aleurites* [tung] oils.** E. D. G. FRAHM and D. R. KOOLHAAS (Chem. Weekblad, 1938, 35, 643—655).—From a review of the literature the following consts. are recorded for *A. trisperma*, *A. cordata*, and *A. moluccana*, respectively, new measurements being recorded in parentheses:  $d_{15.5}^{15.5}$  0.935—0.937 (0.9344), 0.930—0.940, 0.925—0.931 (0.9273);  $n_D^{25}$  1.4869—1.4971 (1.4980), 1.498—1.508, 1.476—1.478 (1.4759);  $10^4(n_i - n_c)$  (162), —, (97); acid val. 1—42 (4.9), 0.4—0.6, 0.5—8.1 (0.3); sap. val. 191—200 (190.8), 185—197, 175—214 (193.0); I val. (Wijs) 111—166 (133.1), 149—176, 101—160 (159.2); SCN val. 69.9, 78—87, 97.1; diene val. (43.2), —, (2.0), unsaponifiable matter 0.5 (0.5), 0.4—0.6, 0.3—0.9%. The following data are recorded on *A. trisperma* and *A. moluccana*: average seed wt. 4—5.5 (6.4), — (8.1; 9.5); kernel wt. on seed 51—64 (67.6), 30—43 (22.2; 35.4%); % of oil in dry kernel 53.7—62.5 (62.8), 59—69 (59.9—66.5). The difficulty of detecting adulteration is discussed. Analyses of press cakes from representative *Aleurites* are recorded for moisture, ash, N, oil, P<sub>2</sub>O<sub>5</sub>, K<sub>2</sub>O, and CaO. This material is not suitable for food, but it makes a satisfactory manure.

S. C.

**Iodine value of tung oil.** J. D. VON MIKUSCH (Oil & Soap, 1938, 15, 186—188).—Reaction of tung oil with I takes place in two stages, viz., (i) rapid partial saturation corresponding with 2 of the 3 double linkings of elæostearic acid, and (ii) slow addition to the remaining double linking. Whilst completion of stage (i) can be ensured by employing a sufficient excess of the I reagent, the second step can be restrained by reducing the time and lowering the temp. of the reaction. The following method is proposed for the accurate and reproducible determination of the partial I val. of tung oil: 0.12—0.15 g. of oil (or 0.1—0.13 g. of pure elæostearic acid) is mixed with 10 ml. of CHCl<sub>3</sub> in an Erlenmeyer flask (a glass stopper is not needed), held in an ice-H<sub>2</sub>O bath (1 min.), and 25 ml. of Wijs solution are added, the mixture being gently swirled in the bath during the addition and for a further  $2 \pm \frac{1}{2}$  min., after which 10 ml. of 15% KI solution and 40 ml. of H<sub>2</sub>O are added and the solution is titrated as usual. Variations of over 100% in sample wt., and 100% excess in time of reaction, caused a variation of only 2 units and 1 unit, respectively, in the partial I val. so determined. Preliminary tests indicated that

const. I vals. representing complete saturation can be obtained for dehydrated castor oil ("isoline" drying oil) by the ordinary Hanus method, provided a 400% excess of reagent is used. E. L.

**Oiticica oil.** A. MACHADO (Rev. Soc. Brasil. Quím., 1938, 7, 73—81; cf. B., 1938, 402).—The oil from *Licania rigida* seeds has Ac val. 30, acid val. 5.04, sap. val. 187.7, I val. 152.4,  $n_D^{20.5}$  1.5158, and contains  $\alpha$ -couepic acid (75.4%), oleic acid (4.21%), stearic acid (5.2%), palmitic acid (6.1%), OH-acids (2.42%), unsaponifiable matter (0.34%), and glycerol. In the paint and varnish industry the oil is considered superior to tung oil. F. R. G.

**Unsaturation of drying oils.** (A, C) W. H. STITSON. (B) H. K. DEAN (Paint Manuf., 1938, 8, 119—123, 163, 200).—(A) The activity of the ethylenic linkings in linseed, tung, and perilla oils is discussed. Methods of measurement of unsaturation are critically reviewed. (B) Polemical against Stitson (cf. preceding abstract). (C) A reply to criticism. D. R. D.

**New Norwegian whale oil standards.** L. ERLANDSEN (Fette u. Seifen, 1938, 45, 413—419).—The Norwegian tentative standard methods (NS 488—9) for the sampling and analysis of whale oil are reproduced in full in German. E. L.

**Refractive dispersion of oils and fats. I. Dispersion of ghee and vegetable oils.** V. T. ATHAVALE and S. K. K. JATKAR (J. Indian Inst. Sci., 1938, 21A, 15—25).— $n_D$  and  $n_C$  for butter fats from Jersey and Sindhi cows and buffalo, measured on a Pulfrich refractometer, give dispersion consts. which are sufficiently different from those of vegetable oils to account for the colour fringes found with a simple butyrorefractometer. Mixtures of oils having the same consistency as ghee do not have both the  $n_D$  and  $n_D - n_C$  of the unadulterated product. Oils and fats are classified into three groups based on the val. of  $m$  and  $C$  in  $n_D - n_C = m(n_D - 1) - C$  (cf. B., 1918, 629A); ghee has  $m$  0.00015 and  $C$  -0.05996, whilst the usual adulterants have either  $m$  0.000097,  $C$  -0.036069 or  $m$  0.00014,  $C$  -0.056018. F. R. G.

**isoPropyl alcohol as a solvent for free fatty acid titration.** G. W. AGEE (Oil & Soap, 1938, 15, 189—190).—Preliminary tests with crude and extracted cottonseed oils using  $\text{Pr}^\beta\text{OH}$  as solvent yielded concordant results for free fatty acid content, identical with those obtained in the ordinary way with denatured EtOH; dry  $\text{Pr}^\beta\text{OH}$  is fully miscible with the oil, but the presence of >1% of  $\text{H}_2\text{O}$  produces separation, which does not, however, appear to affect the titration. E. L.

**[isoPropyl alcohol as a solvent for free fatty acid titration.]** C. R. BROWN (Oil & Soap, 1938, 15, 208; cf. preceding abstract).— $\text{Pr}^\beta\text{OH}$  has been used satisfactorily: after the titration, anhyd.  $\text{Pr}^\beta\text{OH}$  may be recovered (85—90% yield) by distilling the soap mixture, dehydrating the const.-boiling aq. distillate with solid NaOH, separating the alcoholic layer, and redistilling this. E. L.

**Developments in soap manufacture and fat research.** J. MÜLLER (Fette u. Seifen, 1938, 45, 378—379).—A lecture. E. L.

**Desliming of oils and fats in the soap industry.** C. BAUSCHINGER (Fette u. Seifen, 1938, 45, 385—388).—Experiments on the desliming of oils with  $\text{H}_3\text{PO}_4$  and its acid salts are described and discussed; with the latter tendering of the filter-cloths is not serious. Addition of, e.g., 1.5% of  $\text{SnCl}_2$  as a means to reduce metallic corrosion is mentioned. E. L.

**Boiling of grained soap and [toilet] soap-base by the newer methods.** ANON. (Allgem. Oel- u. Fett-Ztg., 1938, 35, 299—304).—If refined fats are employed, tedious settling of the soap can be avoided by using the simple modern methods (no special plant other than a crutcher being required) of boiling and graining soap from fats or fatty acids, an outline of which is given. E. L.

**Soap-base [chip] dryer.** P. TACKMANN (Fette u. Seifen, 1938, 45, 401—402).—Requirements in the prep. and drying of soap chips (for milling) are discussed, and the Lehmann dryer (G.P. 448,574) embodying horizontal, slatted conveyor belts is illustrated; the amount of drying air required may be reduced by fitting heaters within the dryer to maintain the temp. of the chips. E. L.

**Technical problems with [soap] plodders.** H. ZILSKE (Fette u. Seifen, 1938, 45, 405—407; cf. B., 1938, 935).—Improvements in plate design of modern plodders are discussed with a view of reducing flaking and cracking of the soap; a heated sieve-plate produces the most homogeneous bar. E. L.

**Vapour pressure of commercial soaps.** R. H. FERGUSON and R. D. VOLD (Oil & Soap, 1938, 15, 181—183).—McBain's dewpoint method (cf., A., 1935, 163), modified by substituting a Cr-plated tube for the Ag tube, has been applied to determine the v.p. (at 30.15°) of powdered soaps (one Na soap and one Na-K soap base) containing 1—6% of  $\text{H}_2\text{O}$ . The smooth curve for v.p./ $\text{H}_2\text{O}$  content of the soaps suggests that the composition of the soap phase varies continuously with the  $\text{H}_2\text{O}$  content of the system and affords no evidence of any step-wise absorption of  $\text{H}_2\text{O}$ . Practical tests indicate that soaps exposed to the atm. (i.e., under non-equilibrium conditions) do actually change their  $\text{H}_2\text{O}$  content in accordance with the predictions based on the equilibrium curves for R.H./ $\text{H}_2\text{O}$  content of soap. E. L.

**[Pneumatic] methods in conveyor technique: importance for the detergent industry.** H. HORN (Fette u. Seifen, 1938, 45, 402—405).—A new type of pneumatic conveyor, which effects transportation without disintegration of the particles, is described. It consists of a slightly inclined, trough-shaped pipe divided by a false bottom of porous stone into superimposed channels. Air from the lower chamber is blown through the porous partition and cushions the soap particles etc., which thus flow smoothly along the incline of the upper channel; power consumption is low, and the process has been adapted to the cooling of hot materials under conveyance. E. L.

**Development of chemical tests for detergents in the years 1936—8.** K. BURGDORF (Fette u. Seifen, 1938, 45, 379—382).—The standard methods of the International Commission for the analysis of soaps

are criticised in the light of work done by the Deuts. Ges. f. Fettforschung, and some notes on the German standard methods with proposed modifications (*e.g.*, in the determination of borates and phosphates) are appended. E. L.

Testing the practical [laundering] value of detergents. W. KIND (*Fette u. Seifen*, 1938, 45, 383—385).—Practical washing tests are discussed with special reference to the possible discoloration and tendering of white fabrics; 25—50 washing tests under commercial conditions are required in order to estimate the effect on fabric strength. E. L.

Morpholine in wax polishes. H. F. ROBERTSON and A. L. WILSON (*Soap*, 1938, 14, No. 8, 99—101, 111; cf. *Wilson*, B., 1935, 893).—Highly-dispersed wax-polish emulsions giving high gloss and improved H<sub>2</sub>O-resistance are prepared by the use as emulsifier of morpholine (I) fatty acid soaps, prepared *in situ* by interaction of oleic or other fatty acid in the wax mixture with (I); solutions of alkali-sol. resins (rosin, shellac, manila, etc.) in aq. (I) may be added with advantage. The compounding of typical polishes is described and examples are given. E. L.

Pyrolysis of seeds.—See II. Fish oils in paint. —See XIII. Lubricating greases.—See XVII. Margarine. Butter fat. Vitamin-A in ghee.—See XIX. Hydrogenated castor oil in ointments. —See XX.

See also A., II, 346, Converting stearic into oleic acid. Dehydration of ricinoleic acid. 347, Linoleic acid and its isomerides. 374, Synthesis of  $\alpha$ -tocopherol. Unsaponifiable matter of wheat-germ oil. III, 741—6, Vitamins. 771, Seeds of *Magnolia grandiflora*.

#### PATENTS.

Manufacture of fat-containing powder. E. J. FECHNER, Assr. to PAGE MILK Co. (U.S.P. 2,065,675, 29.12.36. Appl., 1.8.32).—By warming a fat or oil with milk, homogenising, and spray- or roller-drying, a powder is obtained which may be used, *e.g.*, as a means of incorporating fats in bakery mixes. E. B. II.

Expelling oil from various substances. R. T. ANDERSON, Assr. to V. D. ANDERSON Co. (U.S.P. 2,065,848, 29.12.36. Appl., 15.6.34).—The finely-ground oilseeds, nuts, etc. are "cooked" at ~105—130° and dried to a H<sub>2</sub>O content of 2—4% before or after cooling the mass to ~60—85° prior to expressing the oil in a continuous-acting press (expeller). E. L.

[Apparatus for] extraction of cashew nut-shell oils. W. JEFFERIES, and PIERCE, LESLIE & Co., LTD. (B.P. 480,468 and 480,981, [A, B] 9.12.36).—(A) The hot nuts, heat-treated as in B.P. 472,195 (B., 1938, 405), are discharged into a cooling and draining device consisting of a latticed cage constructed of spaced parallel half-hoop strips, within which is a conveyer spiral the blades of which are fitted with radial fins which throw up the nuts into the surrounding cool atm. as the spiral propels the nuts through the cage. A trough is fitted below the cage to collect the oil as it is drained or scraped from the nuts;

a T-section rail secured to the bottom of the cage-strips helps to maintain the spacing and acts as a drip. (B) The dry nuts are surface-wetted by immersion and transported by the perforated buckets of an elevator to a conditioning bin, in which they are kept in bulk for about 24 hr. while slowly descending through the bin to the outlet from which they are taken to the heat-treatment plant for extraction of the shell oil (cf. B.P. 472,195; B., 1938, 405). During the 24 hr. conditioning, the surface H<sub>2</sub>O is absorbed by the shells, bringing them into fit condition for subsequent heat-treatment. E. L.

Polymerisation of drying oils. H. I. WATERMAN, C. VAN VLODRUP, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 480,677, 22.7.36).—Drying oils are rapidly polymerised by heating them in presence of dry SO<sub>2</sub>; by employing a sufficiently large surface of contact or high [SO<sub>2</sub>], *e.g.*, by working under pressure, the rate of bodying is  $\gg$  when H<sub>2</sub>S, N<sub>2</sub>, H<sub>2</sub>, or CO<sub>2</sub> is used under the same conditions (cf. B.P. 343,099; B., 1931, 501). E. L.

Refining of sperm oil. W. HOLWECH (B.P. 479,758, 8.8.36).—Sperm oils are distilled up to 340°/5—10 mm., whereby the wax esters are removed and obtained free from glycerides, which remain in the residue. E. L.

Production of neutral and bleached soaps. C. E. ROST and H. WORTMANN (B.P. 481,481, 12.7.37).—A polybasic OH-acid (*e.g.*, tartaric or citric) is added to the soap as well as the H<sub>3</sub>BO<sub>3</sub> used for the bleaching. E. L.

Liberation of fatty acids in fatty material [shortenings]. E. W. ECKEY and C. C. CLARK, Assrs. to PROCTER & GAMBLE Co. (U.S.P. 2,065,520, 29.12.36. Appl., 26.10.33).—A conc. strong acid (preferably a polybasic acid, *e.g.*, 0.5—1.0 mol. of 50—85% anhyd. H<sub>3</sub>PO<sub>4</sub> or H<sub>2</sub>SO<sub>4</sub> per mol. of fatty acid to be liberated) and an inert filter aid are added to the mixture of fat and soap, and the whole is agitated at, *e.g.*, 80—110° under vac. so as to evaporate excess of H<sub>2</sub>O and to ppt. the dry alkali salt of the mineral acid, which is then removed with the filter aid by filtration; the process is especially applicable to the production of mixtures of glycerides and fatty acids intended as shortenings. E. L.

Processes for obtaining sulphuric acid esters from oils, fats, their fatty acids or mixtures thereof. H. W. K. JENNINGS, From CHEM. FABR. STOCKHAUSEN & Co. (B.P. 475,792, 23.4.36).—The reaction mixture obtained by the sulphonation of oils (*e.g.*, Turkey-red oil) are treated (after removing free acid and/or neutralisation) with sufficient H<sub>2</sub>O and an alkaline-earth or heavy-metal oxide, hydroxide, or salt in order to ppt. the non-sulphonated constituents as insol. salts; the sol. sulphonated esters (as salts) remain in solution and are recovered by salting-out or by means of acids. E. L.

Non-alkaline, non-saponaceous detergent for the body. I. REICHSTEIN (B.P. 479,571, 24.3.37. Switz., 27.3.36).—Tablets made from sulphonates or phosphates of alcohols (>C<sub>10</sub>) [or their Na, K, N(C<sub>2</sub>H<sub>4</sub>·OH)<sub>3</sub> salts etc.] together with a mineral colloid to act as a buffer to acidity (floridin, bentonite,



etc.) and a binder (tragacanth, starch, etc.) are claimed. O. M.

**Detergent for toilet use containing incompletely esterified polyhydric alcohol.** W. P. WILLIAMS. From PROCTER & GAMBLE Co. (B.P. 488,196, 31.12.36).—Detergents in bar or tablet form are prepared by incorporating <95% of a salt of a  $H_2SO_4$  reaction product of an aliphatic compound  $\leq C_8$  {sulphonated or sulphated alcohols, saturated hydrocarbons or olefines, fatty acid esters of  $OH\cdot[CH_2]_2\cdot SO_3H$ , fatty acid amides of methyltaurine (I), ethers of  $OH\cdot CH_2\cdot CH(OH)\cdot CH_2\cdot SO_3H$ } with  $\leq 5\%$  (8–60%) of a polyhydric alcohol partly esterified with a saturated aliphatic acid ( $\leq C_{12}$ ) to reduce the solubility while leaving the detergent properties unimpaired. The use of alkylsulphuric acids derived from coconut oil and fatty acid amides ( $\leq C_8$ ) of (I) with glycol or glycerol partly esterified with stearic acid is claimed. A. H. C.

**Wetting and cleansing agents.** W. P. WILLIAMS. From PROCTER & GAMBLE Co. (B.P. 489,097, 17.12.36).—The lathering and detergent properties of salts of alkylsulphuric acids (I) ( $C_{8-18}$ ) containing unsulphated alcohol (II) are improved by adjusting the (II) content by adding saturated primary ( $C_{8-14}$ ) or *sec.* ( $C_{8-25}$ ) alcohols so that the ratio (II) : (I) is  $\geq 75 : 100$ . The improvement of sulphated coconut oil alcohols ( $C_{8-14}$ ) by increasing or reducing the unsulphated portion [(II) : (I)  $\geq 17 : 100$ ], or by adding other alcohols [ $n-C_{10}$ ,  $n-C_{12}$ , (II) : (I)  $\geq 12 : 100$ ; alcohols derived from oxidised petroleum, (II) : (I)  $\geq 22 : 100$ ], is claimed. A. H. C.

**Washing media.** E. WEYMUTH (B.P. 481,399, 24.6.37. Switz., 25.6.36).—A mixture (preferably 8 : 2) of  $NaBO_3$  (85–70) and borax (15–30 pts.) is used. E. L.

**Voltoilised oils. Lubricants. Castor oil product.**—See II. **Urea-aldehyde products. Polymerised nitriles. Surface-active agents.**—See III. **Oils for textiles.**—See V. **Alkali subsilicates [as detergents].**—See VII. **Drying oil composition. Resins from aldol [in polishes].**—See XIII. **Fish-liver oil.**—See XIX. **Distilling materials containing sterols.**—See XX. **Insecticide sprays etc.**—See XXIII.

### XIII.—PLASTICS; RESINS; PAINTS; COATING COMPOSITIONS.

**Trends in the plastics industry.** V. E. YARSLEY (Chem. and Ind., 1938, 849–851).

**Applications of formaldehyde.** III. In plastics. A. MANTEGAZZA (Mat. Plast., 1938, 5, 121–124; cf. B., 1938, 1132).—A review.

**Plastic masses from resins prepared by condensation of phenols with naphthalenesulphonic acids.** G. S. PETROV and E. V. KANIGINA (Prom. Org. Chim., 1938, 5, 416–417).— $2\cdot C_{10}H_7\cdot SO_3H$  40,  $PhOH$  40,  $MeCHO$  36, and  $EtOH$  10.8 or  $H_2O$  16 g. are condensed. A mixture of the resulting resin 40,  $BaCO_3$  108,  $(CH_2)_6N_4$  32, oleic acid 12, Ca stearate 8, and sawdust 368 g. is dried at  $70^\circ$  and pressed at  $160^\circ$  (300 kg./sq. cm.). The moulded products thus

obtained are of medium mechanical strength, but are exceptionally resistant to the action of  $H_2O$ . R. T.

**Glass-clear artificial materials.** K. BRANDENBURGER (Chem.-Ztg., 1938, 62, 571–572).—The properties and uses of compositions based on nitrocellulose (celluloid), cellulose acetate ("Cellon"), vinyl ("Astralon," "Mipolam"), vinyl chloride ("Vinifol"), acrylic ester ("Plexiglas," "Plexigum"), and polystyrene ("Trolitul") polymerides are briefly described. None of these resists boiling  $H_2O$ ; hence for sterilisers etc. phenol- $CH_2O$  resins are preferred, but these are not quite transparent and darken gradually on exposure to light. Amino-plastics yield translucent products from which lamps can be made. S. M.

**Properties of an ideal plastic.** A. F. RANDOLPH (Amer. Soc. Test. Mat., Plastics Symp., 1938, 1–8).—A discussion. R. B. C.

**Definition of fluidity of a plastic material.** G. GOTUSSO (Mat. Plast., 1938, 16, 125–127).—A discussion of the various factors which determine such fluidity. O. J. W.

**Measurements of flow characteristics of [thermosetting] plastics.** H. L. BENDER, H. F. WAKEFIELD, and H. E. RILEY (Amer. Soc. Test. Mat., Consistency Symp., 1937, 64–70).—A review. R. B. C.

**Flow relations of thermoplastic materials.** C. H. PENNING and L. W. A. MEYER (Amer. Soc. Test. Mat., Plastics Symp., 1938, 23–30).—A review. R. B. C.

**Recent progress in [plastics] consistency measurement.** E. C. BINGHAM (Amer. Soc. Test. Mat., Consistency Symp., 1937, 3–8).—A review. R. B. C.

**Deformation of moulded plastic products.** I. F. KANAVETZ and A. I. LEBEDEV (Prom. Org. Chim., 1938, 5, 412–416).—Deformation is ascribed to stresses caused by more rapid cooling of the surface than of the lower layers, and to greater loss of volatile constituents from the former. It is avoided by slow cooling under conditions preventing volatilisation. R. T.

**Hardness, as applied in the plastics industry.** J. C. PITZER (Amer. Soc. Test. Mat., Plastics Symp., 1938, 31–34).—A review. R. B. C.

**Permanence of plastics.** G. M. KLINE (Amer. Soc. Test. Mat., Plastics Symp., 1938, 35–51).—A review. R. B. C.

**Testing methods for determination and comparison of the strength properties of various organic plastics.** H. M. RICHARDSON (Amer. Soc. Test. Mat., Plastics Symp., 1938, 9–14).—A review. R. B. C.

**Measuring the thermal properties of plastic materials.** W. A. ZINZOW (Amer. Soc. Test. Mat., Plastics Symp., 1938, 15–22).—A review. R. B. C.

**Bleaching of lac.** A. VENKATASUBBAN (J.S.C.I., 1938, 57, 288).—Lac bleached by means of  $HOCl$  and then pptd. from alkaline solution by  $H_2SO_4$  contains  $Cl_2$  and  $H_2SO_4$ ; these can be effectively removed by a wet-grinding process.

**Raising the m.p. of colophony.** P. V. RUFIMSKI (Utschen. Zap. Univ. Kazan, 1938, 97, 115—120).—The m.p. is raised from 61° to 72° when air is bubbled through liquid rosin, or to 102° in presence of CaO. Simultaneously its content of OH-acids increases.  
J. J. B.

**Pyrogenation of Congo copal.** L. HELLINCKX (Comm. du Copal de l'Assoc. des intérêts colon. Belges, 1938, 74 pp.; cf. B., 1936, 380).—Results of pyrogenation of a no. of types of Congo copals indicate that the primary reaction is a decarboxylation of dibasic acids. The CO<sub>2</sub> found experimentally agrees with that calc. from the diminution of acidity. This reaction takes place at about 300° for light-coloured copals but at 350° for dark copals. At raised temp. decarboxylation of monobasic acids, decomp. of OH-acids, and cracking ensue. Best results are obtained if the pyrogenation is carried to such a point that the acidity is reduced to 70% of its original val. More homogeneous heating is obtained in broader kettles (height: base = 1:1) than in the narrow forms usually employed.  
L. A. O'N.

**Methacrylic ester resins.** H. R. DITTMAR, D. E. STRAIN, and R. G. KENNELLY (Trans. Electrochem. Soc., 1938, 74, Preprint 11, 159—169).—The prep. and properties of the monomeric and polymeric forms of the Me, Et, Pr<sup>α</sup>, Bu<sup>α</sup>, and Bu<sup>β</sup> esters are given, together with an account of their applications. As the mol. wt. of the esterified alcohol radical increases, the polymerides become more plastic.  
K. W. P.

**Phenolic resins.** F. SEEBACH (Chem.-Ztg., 1938, 62, 569—571).—A brief review.

**Phenolic varnish resins, with special reference to the heat-hardening type.** H. HÖNEL (J. Oil Col. Chem. Assoc., 1938, 21, 247—269).—Unlike Novolak resins, those resols (cf. B., 1938, 61) which are obtained from phenols having only two unoccupied reactive positions combine chemically at 150—200° with large proportions of fatty oils or inactive neutral resins to form homogeneous products which are sol. in mineral spirits. Visible reaction ceases at about 230°, there is marked increase in  $\eta$ , and a gel may result. Resols obtained from phenols containing a higher alkyl group, e.g., *p*-C<sub>6</sub>H<sub>4</sub>Bu<sup>α</sup>OH, are sol. even in cold oils or on gentle heating. As less CH<sub>2</sub>O is evolved by heating the mixture of oil and resin than from the resin alone, the reaction is perhaps due to CH<sub>2</sub>OH or labile C·O·C groups. Data (tabulated) show that the relative increase in  $\eta$  of stand oil (or the m.p. of an ester gum) is greatest when the resol is introduced in the low-mol. condensation stage. This effect is due to the larger no. of free reaction-favourable positions per unit wt. of resin and is apparently connected with elimination of CH<sub>2</sub>O; accordingly resols prepared from low mol. proportions of CH<sub>2</sub>O have low hardening efficiency, whereas varying the phenol has no effect. The combining power for oils, fatty acids, etc. is not related to resinifying tendency. The influence of substituents and of free reactive positions in the phenol is discussed.  
S. M.

**Evaluation of resins for lacquers. I, II.** G. ZEIDLER and H. WEGHOFER (Farben-Ztg., 1938, 43, 827—829, 877—879, 903—904).—I. It is recommended that films containing the resin under examination be prepared and the % elongation under gradually increasing load ascertained before and after exposure to H<sub>2</sub>O etc. The elastic limit and range of plastic flow become obvious from the curve. Graphs for nitrocellulose films containing two esterified rosins are considered. Onset of plastic flow is unfavourable and is attributed to weak residual valencies of the several components.

II. It is urged that for evaluation of the mechanical properties of varnish films determination of true elasticity is necessary. The Erichsen machine does not measure this and the use of the rumpometer (B., 1927, 563) is recommended; prep. of detached films therefor is described. Load-extension diagrams thus obtained with films prepared from phenol-CH<sub>2</sub>O resins show that elastic and plastic conditions vary with the baking conditions; at 140° the film was wholly elastic, but at 160° plastic behaviour was produced. Films resembling oil varnishes can be made and phenol resins can substitute oil if yellowing is not a disadvantage. Data obtained in an Erichsen machine with baked phenol-resin films containing Fe oxide show that variations in pigment proportions have little effect and a crit. pigment content does not exist. Low flexibility at high baking temp. is attributed to greater saturation of main valencies by condensation reactions; one consequence of incomplete condensation is reduced resistance to solvents.  
S. M.

**Phthalic acid-glycerol artificial resins.** F. KOLKE (Farben-Ztg., 1938, 43, 933—934, 959—961).—The composition and properties of the many alkyd resins produced in Germany are tabulated and a plea is made for their standardisation according to their suitability for air-drying, stoving, and nitrocellulose lacquers.  
S. M.

**Inorganic "resins."** E. ROSENDAHL (Oil, Paint, Drug Rep., 1935, 128, No. 18, 26, 42A).—Use of "Abopon" for sizing, emulsifying, etc. is reviewed.  
CH. ABS. (p)

**Chemical analysis of organic synthetic and natural resins.** G. BANDEL (Angew. Chem., 1938, 51, 570—574).—The suggested system is based on the products of dry distillation and on the elementary analysis and sap. val. Sp. tests for many resins are given.  
K. W. P.

**Physico-chemical research on the resinous state.** E. JENCKEL (Angew. Chem., 1938, 51, 563—566).—A review of such investigations compares the behaviour of thermoplastic resins, e.g., polystyrene, with that of glasses. An account of the influence of fillers and of the structure of resins is given.  
K. W. P.

**Titanium dioxide: its manufacture and uses.** D. H. DAWSON (Off. Digest, 1938, No. 176, 225—234).—Improvements in and extended uses of TiO<sub>2</sub> during the past 10 years are reviewed. Experiments are described demonstrating the difference in properties between two samples of TiO<sub>2</sub> manufactured

in 1928 and 1938, respectively. Improvements include better colour, less yellowing in baking finishes, and reduction of the trouble experienced in the drying of  $\text{TiO}_2$  paints. The use of  $\text{TiO}_2$  in paper, shoe dressings, rayon, and ceramics is discussed briefly.

D. R. D.

**Photo-electric measurement of the influence of precipitation conditions on the properties of blanc fixe, in relation to its use in the paper industry.** F. MÜLLER and H. AURICH (Papier-Fabr., 1938, 36, 357—362, 365—370).—For the paper industry the most necessary quality of blanc fixe is its covering power, which is governed mainly by the size and degree of dispersion of the particles. It is considered that turbidity ( $T$ ) measurement of blanc fixe suspensions is the most useful index of these properties for practical purposes, and variables occurring during the pptn. of  $\text{BaSO}_4$  from  $\text{BaCl}_2$  with  $\text{H}_2\text{SO}_4$  and different sulphates are accordingly investigated. A specially developed apparatus (described) having Se cells is employed. Using the reagents in mol. proportions, increase in concn. causes a sharp rise in  $T$  to a max. which indicates the optimum  $\text{BaSO}_4$  ppt. Further increase in concn. causes a gradual drop in  $T$ . The val. of max.  $T$  is slightly raised by using sulphates with multi-valent cations, and the reagent concn. at max.  $T$  is also higher. With cations of the same valency, max.  $T$  decreases with rise in at. wt. of the cation.  $\text{H}_2\text{SO}_4$  produces a higher max.  $T$  than do sulphates, and has a characteristic curve; in this respect acidic salts behave similarly, which is ascribed to a  $p_{\text{H}}$  effect. Rise in pptn. temp. tends to lower max.  $T$ , which also occurs if the  $\text{BaCl}_2$  is added to the precipitant instead of the converse. The highest max.  $T$  is obtained with a ratio of  $\text{BaCl}_2$  to sulphate of 2 : 1. It is concluded that fine  $\text{BaSO}_4$  can be obtained equally well with  $\text{H}_2\text{SO}_4$  as with sulphates provided the right conditions are chosen, and that the photo-electric method can be satisfactorily applied to evaluating blanc fixe pastes.

D. A. C.

**New-type red lead.** A. BROOKES (Oil and Col. Tr. J., 1938, 93, 1386—1389; cf. B., 1938, 941).—The Vaprolite process of manufacture is described.  $\text{Pb}_3\text{O}_4$  of 97—98% purity is obtained by burning Pb vapour in  $\text{O}_2$ . In comparison with ordinary  $\text{Pb}_3\text{O}_4$ , the packing  $d$  and particle size are very low; consequently, it disperses well in oil and the resultant paints are of high opacity. By modifying the conditions of manufacture,  $\text{PbO}$  can be prepared. Using an inert gas instead of  $\text{O}_2$ , finely-divided Pb is obtained.

D. R. D.

**Artificial drying of pigments.** O. T. KORITNIG (Farbe u. Lack, 1938, 379—380, 389—390).—Factors concerned in the efficient and economical drying of pigments, which may not be heated to  $100^\circ$ , by warm air and under reduced pressure are discussed.

S. M.

**Multi- or single-roll [paint] mills?** (A) F. E. WOOLVERIDGE. (B) L. A. SONSTHAGEN (Paint Manuf., 1938, 8, 177—180, 231—232).—(A) The factors affecting the efficiency of paint mills are discussed. Multi-roll mills are preferred.

(B) Polemical against Woolveridge (see above).

D. R. D.

**Toxicity of the dyestuff in indelible pencils.** A. W. STEWART (Analyst, 1938, 63, 494).—A note on the Queensland Government Analyst's report for 1937 (cf. *ibid.*, 348).

J. L. D.

**Preparation of naphthenate driers.** R. D. ZAMISLOV, D. N. BOGOSLOVSKI, and A. I. BESPALKO (Prom. Org. Chim., 1938, 5, 421—422).—Directions for the prep. of Mn, Co, and Pb naphthenates are given.

R. T.

**Fundamental technology of petroleum solvents.** E. M. TOBY (Off. Digest, 1938, No. 176, 261—274).—A review of their manufacture, refining, and testing.

D. R. D.

**[Paint-manufacturing] plant progress.** ANON. (Paint Manuf., 1938, 8, 187—197).—An illustrated review.

D. R. D.

**Rust-preventive paints containing tall oil.** H. SCHLENKERT (Farbe u. Lack, 1938, 365—366).—Exposure tests indicate that esterified tall oil can be successfully used; ZnO gave better protection as pigment than Fe oxide. With blown and sulphurised oils embrittlement ensued. Drying and durability are improved by incorporating an alkyd resin.

S. M.

**Fish oils in paint technology.** K. BUSER (Farben-Ztg., 1938, 43, 803—805).—Classification, properties, composition, deodorisation, and processing [including condensation with phenols, phenolic resins, maleic acid,  $o\text{-C}_6\text{H}_4(\text{CO})_2\text{O}$ , alcohols, and unsaturated fatty acids, and chlorination] are reviewed.

S. M.

**Permeability of paint films.** H. NIESEN (Farben-Ztg., 1938, 43, 851—852).—A glass electrode is used to measure the e.m.f. of a paint film which is successively immersed in solutions of increasing  $p_{\text{H}}$ . If the film is intact the result is const., but after exposure of the film to  $\text{H}_2\text{O}$  or weathering the e.m.f. increases with the  $p_{\text{H}}$  and the deviation gives a measure of the permeability. Data are given for ten ZnO- $\text{TiO}_2$  paints prepared from an alkyd resin and various oils.

S. M.

**Paint specifications.** R. J. MOORE (Steel, 1938, 102, No. 9, 56, 58—59).—Anticorrosion primers and finishing coats are discussed.

R. B. C.

**Consistency measurements in the paint industry.** D. L. GAMBLE (Amer. Soc. Test. Mat., Consistency Symp., 1937, 15—22).—The methods and instruments employed, e.g., for enamels and flat wall paints, and their limitations are reviewed.

R. B. C.

**Examination of paintings and paint films.** K. WÜRTH (Chem.-Ztg., 1938, 62, 589—592).—A descriptive account is given of the examination of old pictures chemically and with ultra-violet light and X-rays and the detection of causes of failures in common house paints.

S. M.

**The electron microscope and paint research.** D. DAUTZ (Farbe u. Lack, 1938, 402—403).—Because of its great magnification the electron microscope is suggested for investigating pigment shape, packing, etc. Photomicrographs of two samples of ZnO are given.

S. M.

**Determination of mercury in paint.** W. KOPLOWITZ (Rev. Soc. Brasil. Quím., 1938, 7, 85—

88).—Determination of Hg in "anti-fouling" paints by treatment with 25% HCl and 5% KCl, and subsequent determination of Hg<sup>++</sup> by the usual methods, is recommended. F. R. G.

**New-type electric kettle for varnish makers.** G. SUTHERLAND and J. C. HARRIS (Drugs, Oils and Paints, 1936, 51, 156, 158).—A 275-gal. monel kettle is described. CH. ABS. (e)

**Constitution of "lac-drying oil" varnishes.** R. BHATTACHARYA and B. S. GIDVANI (J.S.C.I., 1938, 57, 285—288).—When lac, glycerol (I), and fatty acids are heated together in presence of catalyst, preferential esterification of fatty acids with (I) takes place and the monoglycerides (II) thus produced react with the OH groups of lac forming "ether" linkings. A hypothetical formula for the lac mol. has been postulated which has mol. wt. 1012 and 5 OH groups. It has been shown that 2 of these 5 OH, which are more reactive, take part in the reaction with (II). Under suitable conditions, esterification of the fatty acids with these OH groups of lac can be effected. The CO<sub>2</sub>H group of lac can also be esterified with (II) under suitable conditions.

**Critical resin-oil proportions in oil varnishes.** H. WOLFF and G. J. ZELLNER (Farbe u. Lack, 1938, 405, 413—414).—A series of varnishes prepared with gradually increasing proportions of ester gum had max. elasticity (Erichsen machine) and drying rate with approx. equal wts. of resin and oil; hardness, gloss, and H<sub>2</sub>O-resistance did not, however, show such max. Max. elasticity was also recorded in the case of 111L Albertol with 47.5% of oil, and in the case of a fossil resin with 55% of oil. It is suggested that an optimum proportion is required by each resin. S. M.

**Zinc resinates in phenolic and alkyd varnishes.** E. EDELSTEIN (Amer. Paint J., 1938, 22, No. 39, 10, 12, 14, 44—46; cf. B., 1938, 298).—The durability of long oil, ester gum varnishes containing Zn is > that of those containing Pb, but the reverse is true of short oil varnishes. Incorporation of Zn resinates (I) into alkyd and phenolic resin varnishes is discussed. A 25-gal. (I)-tung oil-linseed oil varnish is an efficient grinding vehicle for Prussian-blue. L. A. O'N.

[Products from] petroleum industry.—See II. [Resin from] *Euphorbia*. Lithographic paper.—See V. Painting brick etc. structures. Gluing wood.—See IX. Surface prep. of steelwork for painting etc.—See X. Oiticica oil.—See XII. Colouring rubber.—See XIV. Resins in perfumery.—See XX. Rubber-like substances in waterworks practice.—See XXIII.

See also A., I, 469, Mo-blue.

#### PATENTS.

**Manufacture of plastic masses [containing polymerised vinyl ethers].** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 475,822, 2.6.36).—One or more vinyl ethers (I) which are sol. in H<sub>2</sub>O, MeOH, or EtOH, or interpolymerisation products of (I) in preponderant amount with other vinyl compounds capable of polymerisation under similar conditions, are incorporated with nitrocellulose

products, *e.g.*, by mixing the solutions and/or emulsions or by kneading, rolling, etc. the components. The films have excellent H<sub>2</sub>O-resistance, elasticity, and ageing properties, and are suitable for photographic films, packing, intermediate layers in non-splintering glass, etc. Examples of (I) are the polyvinyl Me or Et ethers. J. W. CR.

**Manufacture of threads and fibres of organic thermoplastic materials.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 479,202, 5.8.36).—Thin, unilaterally oriented foils, bands, tubes, or threads consisting of or containing org. thermoplastic material (*e.g.*, polymerised vinyl chloride, acrylic compounds, etc. and cellulose derivatives) are split up (*e.g.*, by grating, turning, brushing, etc.) into spinnable threads and films which have good mechanical properties. The products have high resistance to H<sub>2</sub>O, acids, alkalis, and putrefaction. J. W. CR.

**Synthetic [polysulphide] plastics.** W. P. TER HORST, Assr. to WINGFOOT CORP. (U.S.P. 2,067,465, 12.1.37. Appl., 14.10.33).—Odourless and solvent-resistant plastic compositions which are not brittle at temp. >10° are prepared by interaction of the condensation products of an alkaline polysulphide with a *s*-di-(halogenated alkyl) compound of a non-metallic element of group VI (preferably the sulphide, oxide, selenide, or telluride) and with a polyglycerol halohydrin, respectively. Alternatively, the 3 materials may interact simultaneously. The products themselves are not vulcanisable, but can be employed in almost any proportions in vulcanisable rubber compositions. J. W. CR.

**Manufacture of [plastic] polymerisation products.** A. CAREMAEL. From I. G. FARBENIND. A.-G. (B.P. 487,604, 22.12.36).—2-Vinylfuran (I) is polymerised in aq. emulsion (*e.g.*, in aq. Na oleate) to latex-like polymerides which may be pptd. to yield thermoplastic materials forming films of good quality. Polymerisation may be effected in presence of other polymerisable compounds. Polymerides containing a major proportion of (I) are self-hardening in O<sub>2</sub> but this tendency is counteracted by aromatic OH- and NH<sub>2</sub>-compounds. Mixed polymerides with CH<sub>2</sub>:CHPh, CH<sub>2</sub>:CH·CN, and (CH<sub>2</sub>:CH)<sub>2</sub> are described. A. H. C.

**Synthetic resinoid and other analogous mouldable materials.** H. W. ROWELL (B.P. 477,222, 10.8.36).—The individual yarns or threads made from org. (*e.g.*, silk, cotton, rayon, hemp, etc.) or inorg. (*e.g.*, asbestos, glass, etc.) fibres are impregnated with synthetic resin or the like (*e.g.*, by passing through a bath containing a solution of phenolic condensation product), severed into desired lengths ( $\frac{1}{8}$  to 2 in. or more), and moulded under heat and pressure. Long lengths are preferred, but when high flowability is required (*e.g.*, in moulding complex articles) short lengths or mixtures must be used. J. W. CR.

**Manufacture of moulding compositions from phenol-aldehyde synthetic resins.** BAKELITE, LTD. From BAKELITE GES. M.B.H. (B.P. 476,888, 16.6.36).—Hardenable phenol-aldehyde resins [*e.g.*, Novolak mixed with (CH<sub>2</sub>)<sub>6</sub>N<sub>4</sub>, or resin produced directly from PhOH and CH<sub>2</sub>O] are converted into

the resitol (*B*) state (*e.g.*, by stoving) and in the finely-divided form are intensively mixed (*e.g.*, on rolls heated at 80–100°) in presence of H<sub>2</sub>O or other non-solvent, insufficient in amount to form an emulsion with the resin. H<sub>2</sub>O lost by evaporation during mixing may be replaced. Plasticisers, dyes, mould lubricants, etc. may be incorporated, but the products are particularly suitable for moulding (in hot presses or by injection) without fillers.

J. W. Cr.

**Manufacture of shaped elastic masses.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 478,701, 20.7.36).—Hardened urea- or CS(NH<sub>2</sub>)<sub>2</sub>-CH<sub>2</sub>O resins (*e.g.*, made as described in B.P. 446,442; B., 1936, 863) having cellular, foam-like structures are admixed with natural or artificial binding agents having elastic and sticking properties and the mixture, to which dyes, fillers, plasticisers, etc. may be added, are shaped by sheeting, pressing, or moulding. The products, which retain a porous structure (and hence combine good insulation with low *d*), are resistant to compression and cracking and suitable for floor and wall coverings etc. Examples of suitable binders are linoxyn, natural or artificial rubber, hydrogenated rubber, and polymerised *isobutylene*, vinyl ethers, etc.

J. W. Cr.

**Manufacture of plastic masses and shaped articles therefrom.** I. G. FARBENIND. A.-G. (B.P. 488,997, 15.1.37. Ger., 16.1.36).—Highly elastic plastic masses with a high stability towards all solvents are obtained by hydrolysing the polymerisation product from a vinyl ester and a maleic acid, and adding to the saponification product a polyhydric alcohol or OH-acid as a softening agent. In the examples, the saponification product from the polymeride obtained from equimols. of CH<sub>2</sub>:CHCl and maleic anhydride (I) is mixed with 0.3 pt. of glycerol and pressed into plates at 120°; the product from CH<sub>2</sub>:CH·OAc (II) and (I) is mixed with 0.25 pt. of dibutylene glycol and 0.5 pt. of H<sub>2</sub>O; the polymeride from 90 pts. of (II) and 10 pts. of Me maleate is saponified and the product mixed with 30 pts. of OH·CH<sub>2</sub>·CO<sub>2</sub>H, 80 pts. of H<sub>2</sub>O, and 50 pts. of soot and extruded into tubes.

S. C.

**Production of laminated materials.** BRIT. CELANESE, LTD. (B.P. 477,662, 25.1.37. U.S., 24.1.36).—From laminated material in which at least one layer contains thermoplastic material (*e.g.*, consists of or contains yarns of an org. derivative of cellulose) articles are cut by a blade heated at such a temp. (*e.g.*, 350° for COMe<sub>2</sub>-sol. cellulose acetate) that the thermoplastic material present seals the cut edges by fusion and prevents fraying etc. Cutting may be carried out at the same time as the composite sheet is formed by providing the hollow die with a cutting edge. Plasticisers (*e.g.*, arylsulphonamides) may be included with or applied to the thermoplastic layer.

J. W. Cr.

**Manufacture of condensation products [moulding powders].** CHALLENGE ADHESIVES, LTD. (B.P. 476,903, 31.8.36. Ger., 30.8.35).—An arylamine (*e.g.*, NH<sub>2</sub>Ph,  $\alpha$ -C<sub>10</sub>H<sub>7</sub>-NH<sub>2</sub>, etc.) is treated with a carbohydrate (which may be a H<sub>2</sub>O-sol. saccharide, *e.g.*, sucrose, aldose, etc., but may also be a hydrolytically cleavable polysaccharide, *e.g.*, starch and raw materials

containing starch, used in conjunction with a suitable acid for effecting hydrolysis) in presence of H<sub>2</sub>O, the temp. being maintained so low (*e.g.*, well below the b.p. of the solution for at least the first half of the reaction) that there is pptd. directly from the solution a non-resinous, insol., infusible, readily filterable powder; evaporation is thus rendered unnecessary. The products are filtered, washed, and dried.

J. W. Cr.

**Manufacture of [horn-like] condensation products.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 489,330 and 489,343, 21.1.37).—Horn-like materials of high mol. wt., useful as lacquers, plastics, textile assistants, etc., are prepared (A) by the interaction in aq. solution or emulsion at room or slightly elevated temp. of a mono-olefinic aldehyde, ketone, acid, or ester which contains the group CH<sub>2</sub>:CH· and is capable of forming polymerides of high mol. wt., *e.g.*, CH<sub>2</sub>:CH·CHO, alkyl vinyl ketones, CH<sub>2</sub>:CH·CO<sub>2</sub>H, with a polymeric compound containing alcoholic OH, *e.g.*, polyvinyl alcohol (I), sugars, cellulose derivatives, in presence of alkalis (NaOH, *tert.* bases, quaternary N compounds), or (B) by the interaction at room or elevated temp. and in aq. medium of lower saturated aldehydes with polymeric compounds containing reactive H at a C attached to an aldehyde or ketone group and obtained as described in (A). An example is (A) (I) (44), COMe·CH:CH<sub>2</sub> (70), H<sub>2</sub>O (1000), and n-NaOH (5 pts.) at room temp. for 5 hr. give, *e.g.*, on concn., a syrup, or (B) on boiling with aq., 30% CH<sub>2</sub>O (300) and 35% NaOH (10 pts.) an insol. thermoplastic substance.

N. H. H.

**Compositions of comminuted cork and a binder.** CROWN CORK & SEAL CO., INC. (B.P. 476,864, 16.4.36. U.S., 14.5.35).—Comminuted cork (in predominating vol.) is incorporated with a binder comprising an adhesive base (I) and a gelled fatty drying oil prepared by treating the ungelled oil (*e.g.*, tung) with an org. amine [*e.g.*, N(C<sub>2</sub>H<sub>4</sub>·OH)<sub>3</sub>] and heating the product with a polyhydric alcohol and a polybasic acid anhydride (or the initial liquid resinous reaction product of these two materials). A wax may also be added either to the binder or to the mixture. (I) may contain a natural resin (*e.g.*, kauri, elemi, etc.), a synthetic resin (*e.g.*, PhOH- or urea-CH<sub>2</sub>O resins, an alkyd, etc.), glutinous matter (*e.g.*, glue gel), or rubber. Articles may be produced by compressing the mixture in moulds, extruding, or by sheeting. The products are H<sub>2</sub>O-free and -resistant, and consequently show minimised dimensional changes over a wide range of temp. and humidity conditions.

J. W. Cr.

**Manufacture of [plastic] casein products.** C. SCHWARTZ, ASSR. to HALL LABS., INC. (U.S.P. 2,064,387, 15.12.36. Appl., 28.9.34).—An alkali metaphosphate (*e.g.*, "Graham's salt," the sol. NaPO<sub>3</sub>) is added to casein products which contain a solubilising agent (particularly CaO) and also possibly some mineral matter, thus imparting greater control of fluidity and more complete solubility, and minimising foaming. Typical preps. of paper-coating compositions, paints, glues, and casein-H<sub>2</sub>O-oil emulsions are described.

J. W. Cr.

**Hardening of protein materials.** E. I. DU PONT DE NEMOURS & Co., C. DANGELMAJER, and E. C. PERKINS (B.P. 476,869, 11.6.36).—The reaction between a protein and a hardening agent is inhibited or delayed by treating the mixture with a fatty acid amide, particularly  $\text{HCO}\cdot\text{NH}_2$ . If sufficient amide is added, the penetration of the protein mixture is rapid but hardening is prevented until further treatment, e.g., mild heating at  $50\text{--}90^\circ$ , is given. In addition to retarding hardening, the amides exert a solubilising action on milk casein and certain difficultly sol. vegetable proteins. The examples given include the hardening of casein, gelatin, and glue with  $\text{CH}_2\text{O}$ ; the process is also said to be applicable to tanning of leather. J. W. Cr.

**Dyeing of hardened casein.** F. HILL, J. A. RADLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 489,575, 29.1.37).—Hardened casein articles (buttons) which have been polished by treatment with aq. hypochlorite solution are dyed evenly by means of monoazo and anthraquinone dyes containing a hydroxyalkyl radical esterified by an acid sulphuric ester group, e.g.,  $[\text{CH}_2]_2\cdot\text{O}\cdot\text{SO}_3\text{H}$  or its salts, attached to an aromatic nucleus by an O or S linking but devoid of  $\text{SO}_3\text{H}$  or  $\text{CO}_2\text{H}$  attached directly to the nucleus. Examples illustrate the dyeing of casein buttons and slabs with  $p\text{-NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{NO}_2 \rightarrow N\text{-ethyl-}N\text{-}\beta\text{-hydroxyethyl-aniline sulphuric ester}$ ,  $p\text{-aminophenyl-}\beta\text{-hydroxyethyl ether sulphuric ester} \rightarrow p\text{-cresol or 1-methylamino-4-}\beta\text{-hydroxyethylamineanthraquinone}$ . N. H. H.

**Coloured moulded articles.** H. JACKSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 490,005, 1.2.37).—Urea and  $\text{CS}(\text{NH}_2)_2$  moulding powders are coloured green with highly halogenated phthalocyanines (e.g., Cu hexadecachlorophthalocyanine). [Stat. ref. to B.P. 474,740, 470,079, and 461,268; B., 1938, 42; 1937, 1183, 471.] S. C.

**Production of articles containing plastic materials.** CELLULOID CORP. (B.P. 489,467, 13.10.37. U.S., 13.10.36).—Articles showing mottled or variegated effects are produced by moulding under heat and pressure a mixture of chemically different materials (plastics), one of which is preferably a resin from  $\text{CH}_2\cdot\text{CH}\cdot\text{OAc}$ , having different degrees of plasticity under the conditions employed and containing different effect materials (dyes, fillers, etc.). The following components are used in the examples: (a) polyvinyl acetate-MeCHO (15-70) 100 pts.,  $o\text{-C}_6\text{H}_4(\text{CO}_2[\text{CH}_2]_2\cdot\text{OMe})_2$  (I) 3 pts.; (b) cellulose acetate (II) 100 pts., (I) 35 pts., suitably dyed, (a) and (b) being mixed; (a) polystyrene, (b) (II) 100 pts., toluene-sulphonamide- $\text{CH}_2\text{O}$  resin 5 pts., plasticiser 25-75 pts., and dye, (a) and (b) being mixed 1:10; (a) polymerised vinyl chloride-vinyl acetate 100 pts.,  $(\text{C}_6\text{H}_4\text{Me})_3\text{PO}_4$  3 pts., containing nacreous pigment (A,  $\text{HgCl}$ , fish scales), (b) polyvinyl acetate-MeCHO (15-80) 100 pts., coumarone resin 10 pts., Ca stearate 3 pts., cresol- $\text{CH}_2\text{O}$  resin 5 pts., (a) and (b) being mixed 20-40. The necessary dye is preferably dissolved or dispersed in a vehicle which does not attack or dissolve the polyvinyl resin component. S. C.

**Heat-polymerisation [of solvent naphtha].** W. H. CARMODY, Assr. to NEVILLE Co. (U.S.P.

2,067,073, 5.1.37. Appl., 17.2.36).—Light-coloured resins are obtained by heating crude solvent naphtha, consisting mainly of dicyclopentadiene, at  $200\text{--}250^\circ$  ( $235\text{--}250^\circ$ ) in a closed vessel in absence of  $\text{O}_2$  and a polymerisation catalyst. The liquid is heated gradually by circulating it through heating coils from and to the reaction vessel. S. M.

**Production of vinyl resins.** E. W. REID, Assr. to UNION CARBIDE & CARBON CORP. (U.S.P. 2,064,565, 15.12.36. Appl., 25.9.31).—The liquid medium in which polymerisation of  $\text{CH}_2\cdot\text{CHCl}$  proceeds affects the solubility of the product. To form resins which are substantially insol. in PhMe a selected medium is used, e.g., an aliphatic alcohol, hydrocarbon, or ketone, at  $<60^\circ$  in presence of a polymerisation catalyst (e.g.,  $\text{Bz}_2\text{O}_2$ ). S. M.

**Vinyl resins.** H. E. POTTS. From SHAWINIGAN CHEMICALS, LTD. (B.P. 477,885, 7.4.36).—The product obtained by polymerising together at least one vinyl ester of low mol. wt. (e.g., vinyl acetate) and a minor proportion of vinyl ester of high mol. wt. (e.g., vinyl oleate or stearate), or an acrylic ester or a vinyl halide, is hydrolysed and the product condensed with at least one saturated aliphatic aldehyde (e.g., aq.  $\text{CH}_2\text{O}$ , paraldehyde) in presence of  $\text{H}_2\text{O}$ , a catalyst (e.g.,  $\text{H}_2\text{SO}_4$ ), and an org. solvent which may include EtOH. The amounts of  $\text{H}_2\text{O}$  and EtOH (if present) must be sufficient to prevent the resin formed and the aldehyde from discoloration, but must be restricted so that homogeneous reaction conditions obtain when the acetal reaction is 85% complete. To obtain superior  $\text{H}_2\text{O}$ -resistance of the final product, hydrolysis should be carried out under mild conditions. J. W. Cr.

**Manufacture of mixed [resinous] polymerisation products soluble in benzine.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 487,593, 22.10.36).—Copolymerides suitable for use in lacquers and combining desirable characteristics of vinyl and acrylic resins with solubility in aliphatic hydrocarbons and insensitivity to  $\text{H}_2\text{O}$  are produced by polymerising mixtures of a vinyl ester (C chain  $\rightarrow \text{C}_4$ ) with  $\leftarrow$  an equal wt. of a polymerisable olefine ester (of an alcohol of C chain  $\leftarrow \text{C}_6$ ). Examples are  $\text{CH}_2\cdot\text{CH}\cdot\text{OAc}$ -diisobornyl maleate,  $\text{CH}_2\cdot\text{CH}\cdot\text{O}\cdot\text{COEt}$ -terpinyl acrylate, and  $\text{CH}_2\cdot\text{CHCl}$ -heptyl maleate. A. H. C.

**Production of polyvinyl resins.** KODAK, LTD. (B.P. 479,057, 30.7.36. U.S., 3.2.36).—Polyvinyl acetal-ester resins derived from the condensation of hydrolysed polyvinyl esters with substances (e.g., aldehydes or cyclic ketones) containing a reactive CO group are de-esterified, the ester group content (calc. as polyvinyl ester) being reduced to  $\approx 3\%$  and OH content (calc. as polyvinyl alcohol) being raised to about 10-20%. Suitable de-esterification agents are the lower fatty alcohols, and the preferred temp. range is room temp. to  $70^\circ$ . The products have high softening points, form highly flexible, tough, and elastic sheets, and when plasticised form rubber-like masses which are suitable for use in the manufacture of laminated safety glass. J. W. Cr.

**Manufacture of artificial materials from polyvinyl chloride.** I. G. FARBENIND. A.-G. (B.P.

478,965, 27.7.36. Ger., 26.7.35).—Polyvinyl chloride and a softening agent [*e.g.*, the esters of  $H_3PO_4$ ,  $o-C_6H_4(CO_2H)_2$ , and  $BzOH$ ] are worked together (in a kneader or on rolls) under mechanical pressure while the temp. is raised to  $\sim 130^\circ$  or higher, when the mass gelatinises and a homogeneous and non-brittle product is obtained which, if desired, can be further worked at lower temp. The products are  $H_2O$ -resistant, have good insulating properties, and in the form of films and sheets may be used for wrapping and coating purposes.

J. W. CR.

**Manufacture of water-insoluble artificial materials from polyvinyl ethers.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 479,217, 14.10.36).—Polymerisation products of vinyl ethers (*e.g.*, Et vinyl ether etc.) are converted by treatment (*e.g.*, by stirring the aq. solution) with natural tanning agents (*e.g.*, tannin, extracts of oak and pine bark, etc.) into products which are insol. and swell only slightly in  $H_2O$ ; they can be further worked into shaped articles or coatings.

J. W. CR.

**Softening agents for polymerised vinyl compounds.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 478,822, 10.8.36).—Org. esters of monoaryl ethers of polyhydric alcohols (*e.g.*, Ph, tolyl, etc. ethers of glycols and also of glycerin and polyglycerin, the free OH groups of which are preferably esterified or etherified) are incorporated as softening agents with polymerised vinyl compounds (*e.g.*, the polymerisation and interpolymerisation products derived from vinyl chloride, acrylic and methacrylic esters, etc. and the products formed by after-chlorinating these materials). The softeners may be added before polymerisation and may be incorporated together with solvents and swelling agents by rolling or kneading. The products retain the extensibility and other good mechanical properties at comparatively low temp. and can be used as intermediate layers in compound glass, and for films etc.

J. W. CR.

**Production of [polyvinyl acetal] synthetic resins and manufacture of films or sheets therefrom.** KODAK, LTD. (B.P. 477,446, 29.6.36. U.S., 18.6.36).—A partly or completely hydrolysed polyvinyl ester (other than the formate) or polyvinyl alcohol is treated in presence of an acid acetal condensation catalyst (I) (*e.g.*,  $H_2SO_4$ ) with  $CH_2O$  and  $MeCHO$  at  $>30^\circ$  ( $30-50^\circ$ ). The OH content (as polyvinyl alcohol) must be  $>12\%$  and the ester group content (as polyvinyl ester)  $>10\%$ , and the proportion of aldehydes (which can be added simultaneously or successively) is such that the  $CH_2O$  acetal constitutes 15–25% of the mol. proportion of the acetal portion of the resin. If desired, a de-esterifying agent may be included, preferably comprising a neutral agent (*e.g.*,  $H_2O$ ) and an acid agent which may be the same as (I). The product, which is a colourless,  $H_2O$ -resistant resin sol. in org. solvents, may, if desired, be further treated, *e.g.*, the OH groups esterified. Flexible films and sheets prepared from the resin are also claimed.

J. W. CR.

**Manufacture of synthetic substances [from methyl vinyl ketone].** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 478,899, 11.9.36).—Me vinyl ketone is condensed in a slightly acidic or

neutral medium with a  $H_2O$ -sol. aldehyde, *e.g.*,  $CH_2O$ ; the product may be treated with bases to yield either a yellow,  $H_2O$ -insol. resin, which can be hot-pressed with addition of fillers and dyes, or an adhesive. Alternatively, the condensation may be effected in presence of a base, and the properties of the product may be influenced by adding to the reaction mixture a phenol, amine, etc.

S. M.

**Improving artificial resins [prepared from cycloaliphatic ketones].** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 478,306, 13.7.36).—Resins prepared from, *e.g.*, cyclohexanone, alone or in preponderating amounts with phenols, cyclic alcohols, and/or aldehydes, are treated with  $H_2$  in presence of hydrogenation catalysts (*e.g.*, Ni activated with Cr, Pt, Pd, etc.) which, if desired, may be pptd. on carriers. The treatment, which may be effected in a melt of the resin or in solution (*e.g.*, in hydrogenated hydrocarbons), is preferably carried out at  $150-250^\circ/180-200$  atm. The resins are rendered paler and their softening point is raised.

J. W. CR.

**Manufacture of amine resins and application thereof as base-exchange bodies or the like.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 489,173, 20.1.37).—Resinous compounds valuable as absorbing or base-exchange agents are obtained by treating a solution in, *e.g.*,  $H_2O$  or EtOH of an aliphatic basic compound formed from the mol. unit  $NH-[CH_2]_m$ , where  $m=2$  or 3, *e.g.*, polyethylene-imine or -diamine (formed, if desired, *in situ*) with an aliphatic polyhalogeno-hydrocarbon, *e.g.*,  $(CH_2Cl)_2$  or with similar compounds in which the hydrocarbon chain is interrupted by, *e.g.*, N or O, *e.g.*,  $(CH_2Cl-CH_2)_2NH$ , or with aromatic compounds containing halogen in at least two side-chains, *e.g.*, 1:3:4:6- $C_6H_2Me_2(CH_2Cl)_2$ , at about room temp., and cautiously drying the gelatinous product. If desired, mixed resins containing in addition aromatic radicals may be formed by using an aldehyde ( $CH_2O$ ) as resinifying agent.

N. H. H.

**Production of condensation products from formaldehyde and urea.** BAKELITE GES. M.B.H. (B.P. 479,250, 18.3.37. Ger., 28.4. and 25.6.36).—Urea (in absence of  $H_2O$  or other liquid) is uniformly mixed with solid polymeric  $CH_2O$  (*e.g.*, paraformaldehyde), at room temp. if desired and in presence of acid, basic, or neutral catalysts, and the mixture is set aside at  $>35^\circ$  until the odour of  $CH_2O$  has departed. Other compounds, *e.g.*,  $CS(NH_2)_2$ , which form resinous products with  $CH_2O$  may partly replace urea, and dyes, plasticisers, natural or synthetic resins, etc. may also be added. The products may be used as the bases of lacquers or (particularly with filler) moulding compositions.

J. W. CR.

**Manufacture of urea-formaldehyde condensation products.** J. A. HETHERINGTON, and BRIT. XYLONITE Co., LTD. (B.P. 476,608, 13.6.36).—Urea (or a derivative) is heated with  $CH_2O$  (conveniently as aq. solution) in presence of a sulphite (*e.g.*, of an alkali or alkaline-earth metal) which does not give rise to insol. products, 1.3–7.25 mols. of acid (*e.g.*, AcOH) are added per 100 mols. of urea, and the product is dehydrated. Softening agents (*e.g.*,

glycerol) may be added before or during the condensation. The process is rapid and easily controlled; the products, which do not gel before casting, can be hardened in moulds at 70—80° to give clear, easily worked materials. J. W. CR.

**Manufacture of [urea-polyose-aldehyde] synthetic resin compositions.** BRUSH & GEN. MOULDINGS MANUFG. CO., LTD., M. B. ROUSSET, and A. V. KELLER (B.P. 475,686, 8.9.36).—A urea, a H<sub>2</sub>O-sol. polyose, and an aldehyde are caused to interact (*e.g.*, by mingling streams of aq. urea and a solution of sucrose in aq. CH<sub>2</sub>O) in substantially neutral solution, free from added catalysts, and the product is dehydrated at <42°. The dehydrated product may be filled into moulds, heated (*e.g.*, at 75°), and finally pressed at about 145°, or, alternatively, the syrup, prior to dehydration, may be incorporated with fibrous fillers or used to impregnate fibrous sheets, dried at  $\gamma$ ~20°, and hot-pressed at 145°. J. W. CR.

**Manufacture of [resinous] polymeric amides.** W. W. TRIGES. From E. I. DU PONT DE NEMOURS & Co. (B.P. 487,734, 23.8.37).—Polymeric amides, sol. in org. solvents, *e.g.*, alcohols, phenols, org. acids, and useful in coating, plastic, etc. compositions and for making fibres, are prepared by heating together (in absence of O<sub>2</sub>) and if desired in presence of a diluent, diamines and dibasic acids or their equiv. which have a total radical length of  $\leq 7$  and at least one of which contains O or S in the chain of atoms separating the reactive groups. Examples are: CMe<sub>2</sub>(C<sub>6</sub>H<sub>4</sub>·O·CH<sub>2</sub>·CO<sub>2</sub>H-*p*)<sub>2</sub> with decamethylenediamine (I), (CH<sub>2</sub>·NH<sub>2</sub>)<sub>2</sub> or adipic acid + hexamethylenediamine; salicylactic acid with (I); dithioglycollic acid with (I) at about 200—210° give resins. N. H. H.

**Manufacture of resins [from aldol].** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 478,370, 22.7.36).—Aldol (the crude product obtained by condensation of MeCHO under mild conditions is suitable) is heated in presence of *sec.* amines [*e.g.*, NHMe<sub>2</sub>, NH(C<sub>2</sub>H<sub>4</sub>·OH)<sub>2</sub>, etc.], and the products are heated at 160—190°. The resins obtained, which are sol. in EtOH and compatible with nitrocellulose (I), have good electrical properties and when dissolved in (I) can be used in the prep. of hard and durable polishes. J. W. CR.

**Manufacture of resinous reaction products of inner ethers and natural resin acids.** ATLAS POWDER CO. (B.P. 488,907, 12.10.37. U.S., 28.12.36).—Resins of low acid val. ( $\gamma$ 35) are obtained by heating together 1 mol. of hexahydric alcohol (mannitol, sorbitol) or its inner ether and < 3 equivs. of rosin in absence of air at a high temp., preferably 285—300°. S. C.

**Manufacture of films and foils from polymerisation products.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 489,550, 26.1.37).—Films and foils which are stable to light and solvents and do not become deformed or cohere are obtained by incorporating <50% of an oily or resinous condensation product from a dicarboxylic acid derived from an alkyl ether, *e.g.*, diglycollic, dilactic, and di-

hydracrylic acids, with a polyhydric alcohol, *e.g.*, OH-[CH<sub>2</sub>]<sub>2</sub>·CHMe·OH, octylene glycol, dibutylene glycol, dimethoxyethylene glycol, into a polyvinyl ester of medium or low  $\eta$ , which may be a simple polymeride, *e.g.*, of vinylacetate, or an interpolymeride, *e.g.*, with maleic or acrylic esters. S. C.

**Production of synthetic resin.** E. G. PETERSON, Assr. to HERCULES POWDER CO. (U.S.P. 2,067,859, 12.1.37. Appl., 2.9.33).—Maleic anhydride, a compound containing the abietyl radical (*e.g.*, rosin, abietic acid, etc.), and a terpene hydrocarbon (C<sub>10</sub>H<sub>16</sub>) having no conjugated double linkings (*e.g.*, terpinolene or a terpene cut, boiling range 182—190°) are heated together (at 125—250°). The saturation of the resinous products obtained may be reduced by heating with H<sub>2</sub> under pressure in presence of a hydrogenation catalyst. The products are suitable for lacquer and varnish bases. J. W. CR.

**Polymerisation of organic compounds [to bubble-free slabs].** O. RÖHM, Assr. to RÖHM & HAAS CO. (U.S.P. 2,067,580, 12.1.37. Appl., 16.10.34. Ger., 9.10.33).—Bubble-free slabs of polymerised derivatives of acrylic and methacrylic acid are prepared by charging the monomerides into narrow, preferably vertical, cells the walls of which are uniformly heated by circulating fluid the temp. of which is regulated to maintain a polymerising temp. (*e.g.*, 70°) and to conduct away the heat generated during polymerisation. A sack accurately fitting the cell may also be used and may be removed with the slab to protect it against dust, abrasion, etc. J. W. CR.

**Pigmented granular polymerides [from acrylic esters].** W. E. GORDON and W. W. HECKERT, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,067,234, 12.1.37. Appl., 10.3.36).—Pigments are dispersed in a syrup comprising a solution of the polymeride of acrylic or  $\alpha$ -substituted acrylic esters in the monomeride (the  $\eta$  of the latter by itself would be too low) to which soya-lecithin has been added in proportion (generally about 3 times the wt. of the pigments) to stabilise the dispersion during the subsequent polymerisation in a granulating bath. J. W. CR.

**Production of phenolic synthetic resins.** BAKELITE, LTD. (B.P. 478,589, 20.7.36. U.S., 13.3.36).—A phenol is heated (*e.g.*, at 170—180°) with an unsaturated fatty oil (*e.g.*, tung or linseed) in presence of a converting agent comprising an inorg. basic compound (*e.g.*, a basic metal oxide or a salt of a strong base and a weak acid) which is less alkaline than are the alkaline earths and having substantially no saponifying action on the oil. The product may be further treated with aldehydes, the reaction being accelerated by a further addition of the basic converting agent. The products, which are directly thermosetting without the necessity of preparing an intermediate Novolak resin, can be made brittle or flexible by varying the oil content. J. W. CR.

**[Modified phenolic] artificial resin.** E. C. KNEALE and H. H. WOHLGEMUTH (U.S.P. 2,064,614, 15.12.36. Appl., 18.11.31).—An alkaline phenol-CH<sub>2</sub>O partial condensate is heated in presence of



a polyhydric alcohol (glycerol) with a coumarone-indene resin. The last may be replaced by a neutral or acidic natural resin, *e.g.*, rosin, and the product is finally condensed with  $o\text{-C}_6\text{H}_4(\text{CO})_2\text{O}$ , preferably in presence of a fatty acid, *e.g.*, linoleic, and a drying oil, *e.g.*, soya-bean. The products may be used for lacquers, moulding compositions, etc. S. M.

**Treatment of phenol condensation products adapted for manufacture of phenol-aldehyde resins or products thereof.** DORCH, BACKSIN & Co. AKTIEBOLAG (B.P. 478,988, 22.5.37. Swed., 26.5.36).—A phenol-aldehyde, oily,  $\text{H}_2\text{O}$ -free, intermediate condensation product (I) is dissolved in a volatile solvent, *e.g.*, EtOH, and 0.2–10% [calc. on wt. of (I)] of a dihydric aliphatic alcohol, *e.g.*,  $(\text{CH}_2\text{-OH})_2$ ; a small quantity of a volatile acid is added immediately prior to application as a lacquer. Heating is not essential for the hardening, but may be effected at 0–40°. S. M.

**Manufacture of [phenol-aldehyde] resinous condensation products.** BECK, KOLLER & Co. (ENGLAND), LTD. (B.P. 477,954, 6.7.36. U.S., 6.7.35. Addn. to B.P. 459,549; B., 1937, 470).—In addition to, or in place of, the terpene halides of B.P. 459,549 (*loc. cit.*) there are used the halides (formed by addition of halogen or hydrohalide, or by esterification) of saturated or unsaturated OH-compounds of the group of sesqui- and poly-terpenes, or derivatives thereof, or O-containing compounds of terpenes. Raw materials containing high proportions of the above reactants include conifer, pine, cumin, and lemongrass oils etc., gum and wood rosin.

J. W. CR.

**Production of [alkyd] synthetic resin.** A. L. RUMMELSBURG, Assr. to HERCULES POWDER Co. (U.S.P. 2,067,862, 12.1.37. Appl., 1.12.34).—A polybasic acid (glycerol, a glycol, sorbitol, etc.), an org. polybasic acid or anhydride [ $o\text{-C}_6\text{H}_4(\text{CO})_2\text{O}$ , sebacic and adipic acids, etc.], and a hydrogenated ( $\text{H}_2$  saturation of <50%) abietyl alcohol are heated together (at 125–325°) to form a resinous material having a low acid val. and high oil solubility and m.p. which can be used in oil varnishes and as a plasticiser in nitrocellulose lacquers. J. W. CR.

**Manufacture of condensation products [synthetic resins].** (A) A. CARPMAEL, (B) W. W. GROVES. (A, B) From I. G. FARBENIND. A.-G. (B.P. 487,253 and 487,323, [A] 22.12.36, [B] 15.12.36).—(A) Compounds containing twice in a 5-membered ring the group  $\text{NHX-C:N}$  (3:5-diaminopyrazoles or their hydrazino-derivatives) are condensed with aldehydes of low mol. wt. (<1 mol.) or compounds giving such aldehydes under the conditions of reaction. Other resin-forming materials may be present. Colourless resins of good resistance to  $\text{H}_2\text{O}$  and light and of high electrical resistance are produced. *E.g.*, guanazole is condensed with  $\text{CH}_2\text{O}$  in  $\text{H}_2\text{O}$  and the resinous product is hardened at 100°. Other starting materials used are phenylguanazole, dihydrazoamidopyrroldiazole (4-amino-3:5-dihydrazino-1:2:4-triazole), 3:5-diaminopyrazole, and 4:5-diamino-2-phenyl-1:2:3-triazole. (B) Thiodiglycols  $\text{S}_{1-3}(\text{R-OH})_2$ , in which R is a saturated aliphatic hydrocarbon radical, are condensed with di-, poly-,

or hydroxypoly-carboxylic acids, their anhydrides, or esters, to give viscous to elastic products which are in general sol. in org. solvents and compatible with rubber and butadiene polymerides. Condensation products are described (made at 140–170°) of dithioditetramethylene glycol with maleic (I) and succinic anhydrides, of trithiodi-tetramethylene and -ethylene glycol (II) with (I), and of (II) with a hydrolysed polymeride of  $\text{CH}_2\text{:CHCl}$  and  $\text{Me}_2$  maleate; the last-named, when gelatinised with di-*o*-tolylguanidine, forms a rubber resistant to aromatic and aliphatic hydrocarbons. H. A. P.

**[Calcium carbonate] pigment manufacture.** A. M. BROOKS and H. R. RAFTON, Assrs. to RAFFOLD PROCESS CORP. (U.S.P. 2,066,066, 29.12.36. Appl., 10.8.33).—Aq.  $\text{Na}_2\text{CO}_3$  is agitated at >75° (~50°) with slaked CaO containing MgO and the ppt. is filtered off and washed. The product possesses very fine particle size and may be used as a filler or coating for paper. S. M.

**Production of a series of coloured pigments from titanate acid derivatives.** L. PASSERINI (B.P. 477,055, 20.7.36. It., 20.7.35).—Variously coloured pigments are prepared by calcining an intimate mixture of substances giving rise to the formation of <2 isomorphous salts of bivalent metals, at least one of the salts having Ti as the metallic element of the acidic component, the acidic element of the other salt(s) being Al, Fe, and/or Sn. The reactants, which are mixed in such proportions that stoichiometric quantities of acidic and bivalent components are present, may comprise oxides, or compounds or finely-divided metals which form oxides under the conditions of calcining. The products are clearly defined solid solutions. As an example, stoichiometric quantities of  $\text{FeO}$ ,  $\text{SnO}_2$ ,  $\text{MgO}$ , and  $\text{TiO}_2$  are calcined to give the yellow-brown solid solution between stannates and titanates of Fe and Mg. J. W. CR.

**Manufacture of [metal-free phthalocyanine] highly dispersed pigments.** I. G. FARBENIND. A.-G. (B.P. 475,882, 27.5.36. Ger., 1.6.35. Addn. to B.P. 466,042; B., 1937, 1375).—The softness of grain, clarity, and intensity of shade of metal-free phthalocyanines (*e.g.*, made by heating *o*- or 4-chloro-phthalonitrile in presence of  $\text{HCO}\cdot\text{NH}_2$ ) are improved by mixing or milling with  $\text{H}_2\text{O}$ -sol. dispersing agents (*e.g.*, Turkey-red oil) in presence of  $\text{H}_2\text{O}$ , at raised temp. and/or pressure if desired. The products are suitable for colouring lacquers, printing pastes, etc., and particularly for colouring spinning solutions in artificial silk production. J. W. CR.

**Transfer inks for use in production of photo-mechanical printing forms.** CHEM. FORSCHUNGS-GES. M.B.H. (B.P. 478,776, 5.2.37. Ger., 6.2.36).—A compound, *e.g.*, PhOH, which contains reactive OH and exerts mild corrosive action on Zn is added to the ink. The use of fixing agents and heating of the offset plate are rendered unnecessary. S. M.

**Liquid composition for erasing Indian ink from tracing papers and tracing cloths.** W. SIMPKIN (B.P. 487,618, 2.3.37).—The composition

comprises  $\text{CCl}_4$ , "spirit,"  $\text{NH}_3$  (or  $\text{H}_2\text{C}_2\text{O}_4$ ), linseed oil (or turpentine), and  $\text{H}_2\text{O}$ . B. M. V.

**Printer's blanket.** W. C. CALVERT, Assr. to WINGFOOT CORP. (U.S.P. 2,064,780, 15.12.36. Appl., 18.8.33).—A printers' blanket (of rubber or felt) is provided with a flexible coating of a (stabilised) rubber hydrohalide (hydrochloride) to render it oil-proof. D. F. T.

**Vinyl resin coating.** D. M. GRAY, Assr. to HAZEL-ATLAS GLASS CO. (U.S.P. 2,067,316, 12.1.37. Appl., 5.7.34).—To obtain films which dry without forming ridges, 2—12% of a polyhydronaphthalene is added to the vinyl resin lacquer. S. M.

**Manufacture of rubber chloride varnishes.** RAOLIN CORP. (B.P. 489,977, 5.2.37. U.S., 5.2.36).—Rubber chloride varnishes containing a plasticiser of the ester type which contains a halogen atom or an ether grouping are claimed, such plasticisers being, *e.g.*, esters of  $\alpha$ -dichlorohydrin, and the monoalkyl ethers of glycol, particularly those of long-chain fatty acid, including chlorinated ricinoleic acid. (Cf. B.P. 489,954; B., 1938, 1201.) S. C.

**Cellulose derivative emulsions.** E. I. DU PONT DE NEMOURS & Co. (B.P. 477,997, 10.7.36. U.S., 15.8.35).—A stiff colloided solution (I) (*e.g.*, in a substantially  $\text{H}_2\text{O}$ -insol. org. solvent) of a cellulose derivative is kneaded with a viscous aq. medium containing an emulsifying agent (*viz.*, gelatin, glue, methylcellulose, etc., which with  $\text{H}_2\text{O}$  form viscous colloidal solutions), the ratio of (I) to (II) being adjusted to give a paste-like emulsion containing <33% of film-forming ingredients. Additional emulsifying agents, *e.g.*, Gardinol, may be added to lower the interfacial  $\gamma$ . The products may be used as adhesives, bonding agents (*e.g.*, for laminated cloth), and for coating and impregnating purposes. J. W. CR.

**Cellulose derivative coating composition [of high dielectric strength].** R. T. HUCKS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,064,608, 15.12.36. Appl., 7.7.34).—Compositions suitable for electrical equipment contain 10—50 wt.-% of finely-divided  $\text{MnO}_2$ , reduction of which is avoided by selection of the other components. An example claimed consists of  $\text{MnO}_2$  37.5, *o*- $\text{C}_6\text{H}_4(\text{CO}_2\text{Bu})_2$  2.0, castor oil 1.0, nitrocellulose ( $\frac{1}{2}$ -sec.) 9.0, 50% solution of dewaxed shellac in EtOH 20.0, BuOH 5.0, denatured EtOH 6.5, EtOAc 3.0, BuOAc 13.0, and PhMe 3.0%. S. M.

**Production of [pigmented] cellulose derivative coating compositions.** E. I. DU PONT DE NEMOURS & Co., M. V. HITT, D. G. KENNEDY, and H. LE R. PRIDY (B.P. 476,806, 12.6.36).—A cellulose derivative (I), *e.g.*, nitrocellulose, is treated (*e.g.*, tumbled in steel barrels or mixed in kneading machine) with a mixture of volatile solvent and an org. diluent in proportions to wet and swell the (I) to a friable product without causing coalescence; the product is then kneaded with pigment, the temp. being advantageously maintained at  $\sim 80^\circ$ . Plasticisers, solvents, softeners, diluents, etc. may be added after the pigment is dispersed. The process, which is particularly useful for incorporating C-black pigments,

permits the use of heavy-duty kneading equipment with minimised fire and explosion hazards. J. W. CR.

**[Cellulose derivative-wax] composition.** W. H. CHARCH, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,064,292, 15.12.36. Appl., 18.1.34).—The composition comprises a cellulose derivative (*e.g.*, the ethers, or the esters of  $> \text{C}_2$  fatty acids) sol. in  $\text{C}_6\text{H}_6$  of PhMe, or in a mixture of either with >10 vol.-% of an O-containing solvent, a waxy substance (*e.g.*, paraffin wax, m.p.  $60^\circ$ ), an O-free plasticiser (*e.g.*, dixylylethane), and a solvent, *e.g.*, a (halogenated) hydrocarbon, in which the ingredients are sol. Blending agents, which may be resinous (*e.g.*, gum dammar) or non-resinous (*e.g.*, hydrogenated castor oil), may be added. Accelerated drying may be obtained by adding O-containing solvents (*e.g.*, alcohols) in amount >30% of the main solvent. The products dry at temp. < the m.p. of wax to give glossy, transparent, moistureproof films and are suitable for coating regenerated cellulose etc. J. W. CR.

**Manufacture of materials resisting [liquid] fuel.** I. G. FARBENIND. A.-G. (B.P. 478,680, 24.7.36. Ger., 25.7.35).—Articles which are required to come in contact with mixed liquid motor fuels and must therefore be resistant to the solvent action of those fuels are made of, or given a coating of, polyvinyl formate or mixed polymerides prepared from it; the coating is applied to the surface to be protected as a solution of the protecting agent in  $\text{COMe}$ , or  $\text{CH}_2\text{Cl}_2$ . D. M. M.

**Container-sealing composition.** C. J. PARKER, Assr. to CROWN CORK & SEAL CO., INC. (U.S.P. 2,073,648, 16.3.37. Appl., 25.1.33).—A washer for sealing containers is claimed; it consists of rubber 30—35, stearic acid 2, ceresin 15 pts., a filler, and a vulcanising agent. D. M. M.

**Treatment of plasticisable compounds.** BRIT. CELANESE, LTD., and W. H. MOSS (B.P. 476,638, 10.6.36).—A plasticiser is uniformly distributed over the surface of particles of the compound (*e.g.*, cellulose derivatives, polyvinyl and polystyrene compounds, synthetic resins, etc., in a mixer or on a moving belt) moistened with a non-solvent (preferably  $\text{H}_2\text{O}$ , but ethers, hydrocarbons, and alcohols can be used) and the non-solvent removed by evaporation without any mechanical working of the mass, the physical state (*e.g.*, flaky or fibrous condition) thus remaining unchanged. The process is particularly applicable to liquid plasticisers (*e.g.*, triacetin, phthalates, tartates, triaryl and trialkyl phosphates, etc.), but solids dissolved in volatile solvents can be used. J. W. CR.

**Drying oil composition.** H. M. DENT, Assr. to GEN. PLASTICS, INC. (U.S.P. 2,064,786, 15.12.36. Appl., 15.4.35).—An artificial resin-like mass of a hydroxyaryllalkane (I) [*e.g.*, *p*-di(hydroxyphenyl)ethane, and/or -methane] is heated ( $232$ — $288^\circ$ ) with a drying oil, *e.g.*, tung oil, until a homogeneous liquid product is formed from which (I) does not separate on cooling. The products, which are clear without the necessity of using fluxing agents (natural resin or ester gum), yield films which are adherent to metal and chemically resistant. J. W. CR.

Manufacture of article for continuous embossing [of fabrics]. G. C. CHATFIELD and C. EMMEX (U.S.P. 2,067,435, 12.1.37. Appl., 17.12.35).—A coating composition containing PhOH-CH<sub>2</sub>O and o-C<sub>6</sub>H<sub>4</sub>(CO)<sub>2</sub>O resins, mastic, dye, and activating agent, e.g., CuSO<sub>4</sub>, is spread on paper or other carrier, and hot-pressed on the fabric. In the text, other resins are also used. S. M.

Liquid coating composition [for wax surfaces]. W. H. WOODFORD, Assr. to REMINGTON ARMS CO., INC. (U.S.P. 2,067,586, 12.1.37. Appl., 26.7.29).—To overcome slow drying a metallic soap, e.g., Al stearate, is added to a (nitrocellulose) lacquer; alternatively a solution of the soap in, e.g., PhMe may be applied. S. M.

Manufacture of finishing material. L. R. VAN ALLEN (U.S.P. 2,067,297, 12.1.37. Appl., 7.2.34).—Wax-containing by-products obtained in the treatment of shellac are dried and powdered. They may be used for polishing, as abrasives, etc. S. M.

Stretching foils and bands of organic thermoplastic materials [on heated cylinders]. G. W. JOHNSON. From I. G. FARBEIND. A.-G. (B.P. 479,492, 5.8.36).

[Fabric] reinforcement of synthetic resinous materials and objects. N. A. DE BRUYNE, AERO RESEARCH, LTD., and DE HAVILLAND AIRCRAFT CO., LTD. (B.P. 488,373, 8.1. and 12.11.37).

Extrusion presses for foils etc. Friction-element bond.—See I. Polymerisation products.—See III. Dye pigment pastes. Phthalocyanines.—See IV. Non-cellulosic material from spent digestion liquor. Coating paper. Laminated sheet.—See V. Impregnated material.—See VI. Decorating glass. Coating glass sheets. Laminated materials. Abrasive-coated discs.—See VIII. Composite building material. Artificial lumber.—See IX. [Resinous] insulating materials. Layered-paper insulation.—See XI. Polymerised drying oils.—See XII. Rubber-like product. Rubber chloride compositions. Rubber hydrohalide films.—See XIV. Resinous adhesive.—See XV. Brewer's pitch.—See XVIII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

New rubber plant from the Palmiro-Alai. C. S. AFANASIEV (Compt. rend. Acad. Sci. U.R.S.S., 1938, 18, 479—482).—A new species, *Scorzonera kirghisorum*, is described. Dry roots yielded 4.2—9.5% of rubber. Mechanically isolated crude rubber contained pure rubber 66, resins 22.8, protein 0.62, ash 5.02, and H<sub>2</sub>O 26.23%. The  $\eta$  of 1% solutions of the rubber in C<sub>6</sub>H<sub>6</sub> was similar to that of tau-saghitz. A. G. P.

[Rubber] latex research. F. S. ROBERTS (Trans. Inst. Rubber Ind., 1938, 13, 470—479).—The development of manufacturing processes employing latex is historically reviewed. D. F. T.

Stability of *Ficus elastica* [rubber] latex. F. K. DANIEL, H. FREUNDLICH, and K. SOLLNER (India-Rubber J., 1938, 96, 172—175).—The latex 4 M (B.)

as it leaves the tree is very unstable, but after preservation with NH<sub>3</sub> becomes much more stable and is difficult to coagulate. This change is probably caused by saponification of resin esters and the decomp. of sugars; it is not the result of an enzymic process. On creaming, the latex gives a pink upper layer of dispersed rubber particles and a white lower layer of particles of similar size (diameter 1—2  $\mu$ .), but consisting in part of resins. Electrodeposition gives a non-coherent anodic layer. The protective substance in *F. elastica* is different from that in jelutong or in *Abiarana gutta* latex. Coagulation in all 3 cases, however, can be effected with polar-non-polar org. liquids. D. F. T.

Mixed [vulcanisation] accelerators in [rubber] latex compounding. R. L. SIBLEY (Trans. Inst. Rubber Ind., 1938, 13, 453—469).—Examination of the effect of piperidine pentamethylenedithiocarbamate (I) in conjunction with several derivatives of thiolbenzthiazole (II) and 2 aldehyde-aniline products shows that a mixture of (I) with the Na derivative of (II) or with di(benzthiazylthiol)dimethylurea (III) gives well-vulcanised products at a lower cost of acceleration than does (I) alone, (I) being particularly activated by the Na compound. With appropriately adjusted ratios of (I) and (III) wide variation is possible in the rate of vulcanisation and in the physical properties of the product, which also shows excellent ageing. The ageing behaviour can, however, in all cases be enhanced by addition of a recognised antioxidant, especially polymerised 2:2:4-trimethyldihydroquinoline. The CS<sub>2</sub>-reaction product of methylenedi-(*N*-methylcyclohexylamine), which is a liquid insol. in H<sub>2</sub>O, when used as an aq. dispersion with latex, is a powerful accelerator of vulcanisation and is further activated by the Na derivative of (I). The use of the above accelerator combinations is unaffected by NaOH (in small proportions), casein, urea, or the common synthetic wetting agents. D. F. T.

Oxygen-pressure ageing [for rubber]. Improved equipment. L. M. FREEMAN (Ind. Eng. Chem. [Anal.], 1938, 10, 428—431).—Features of the equipment are: small, stainless steel, quick-closing, jacketed pressure vessels, which are easily removable; an electrically heated, const.-temp. system, adjustable to a wide temp. range; a valve which automatically closes the O<sub>2</sub> supply to the pressure vessel; and a safety release. E. S. H.

Colouring of rubber with organic colours. A. BERETTA (Rev. Gén. Mat. Col., 1938, 42, 321—326).—Various classes of org. pigments for rubber are indicated and reference is made to individual advantages and weaknesses and to general methods of use. D. F. T.

Sealing agents for tubes for rubber tyres. H. LUCKMANN (Chem.-Ztg., 1938, 62, 553—554).—The characteristics and disadvantages of the various fluid agents which have been proposed for introduction into inner tubes for sealing punctures and for checking porosity are briefly discussed. Porosity is commonly experienced in countries which need to economise in the consumption of rubber. D. F. T.

**Consistency measurement of rubber and rubber compounds.** J. H. DILLON and L. V. COOPER (Amer. Soc. Test. Mat., Symp. on Consistency, 1937, 52—58).—A review. R. B. C.

**Time effect in the rapid extension of rubber.** V. HAUKE and W. NEUMANN (Naturwiss., 1938, 26, 461).—Owing to possible confusion with papers by other workers bearing on a different aspect of the subject it is proposed to re-title work already reported under the above heading (B., 1938, 948) "Effect of rate of stretching on tension-extension curves for rapid extension." A. J. M.

**Isothermal and adiabatic extension curves of vulcanised rubber.** V. HAUKE and W. NEUMANN (Monatsh., 1938, 72, 22—30).—These curves and the variation of extension with time have been determined for a series of rubbers of defined composition. The temp. variation between 0° and 90° in the isothermal curve has been determined for two rubbers of, respectively, high and low degree of vulcanisation. O. D. S.

**Rubber in the plating industry.** R. H. KITNER (Month. Rev. Amer. Electro-Platers' Soc., 1934, 21, 17—25).—Hard rubber is more resistant to acids and alkalis because of its high (up to 47%) S content. Pigments such as inorg. oxides and silicates raise the temp.-resistance of rubber from 50° to 66°. Rubber linings for steel tanks are discussed. CH. ABS. (e)

**Utilising *Euphorbia*.**—See V. Rubber formation in tau saghyz.—See XVI. Rubber etc. in waterworks practice.—See XXIII.

See also A., I, 476, Ultra-violet microscopy.

#### PATENTS.

**Concentration of [rubber] latex.** RUBBER PRODUCERS RES. ASSOC. From E. RHODES and C. SEKARAN (B.P. 476,073, 22.10.36).—Creaming is effected by addition of a H<sub>2</sub>O-sol. derivative of an aliphatic (C<sub>6</sub>-18) alcohol (e.g., sulphonated lauryl alcohol or the Na salt of the sulphonated alcohols from hydrogenated coconut oil). D. F. T.

**Manufacture of sponge rubber [from latex].** INTERNAT. LATEX PROCESSES, LTD. (B.P. 479,264—5, 25.6.37. U.S., [A] 10.7.36, [B] 13.8.36).—(A) CO<sub>2</sub> is liberated from a carbonate (NH<sub>4</sub>HCO<sub>3</sub> and CH<sub>2</sub>O, or NaHCO<sub>3</sub> and acid) in the latex with substantial prevention of coagulation (by means of a Nekal or Igepon compound etc.). The latex mixture may contain a heat-sensitising agent (e.g., MgCO<sub>3</sub>) and the foamed product is preferably coagulated by heat. (B) Latex is foamed by dissolving in it a H<sub>2</sub>O-sol. gas (CO<sub>2</sub>) under pressure and subsequently reducing the pressure to <1 atm. The latex contains a suitable delayed-action coagulant or gelling agent which operates on keeping the shaped foam or on heating the foam. D. F. T.

**Water-resistant rubber compositions [from latex].** DEWEY & ALMY, LTD. From DEWEY & ALMY CHEM. CO. (B.P. 476,481, 6.3.36).—A composition (for can sealing and adhesive pastes) comprises, in aq. dispersion, an org. hydrophilic colloid (glue, algin, karaya, or casein), a finely-divided inorg. mineral substance (e.g., bentonite,

talc, asbestine, TiO<sub>2</sub>), and rubber, and contains in solution a salt with a complex metal-ammonium or -amine radical [Zn(NH<sub>3</sub>)<sub>4</sub>Cl<sub>2</sub>, or the acetate, benzoate, salicylate, etc.] which, on heating or drying of the mixture, flocculates the dispersed inorg. material and renders the colloidal material resistant to H<sub>2</sub>O.

D. F. T.

**Preparation of unvulcanised rubber in granular or powdered form.** B. D. PORRITT and W. H. WILLOTT (B.P. 475,503, 19.5.36).—Coagulum, (substantially) unvulcanised rubber, plasticised or reclaimed rubber is wetted with an alkaline aq. solution (0.1% NaOH, 1% Na<sub>2</sub>CO<sub>3</sub>, or 1% NH<sub>3</sub>), and thereafter (if desired) dusted (with a H<sub>2</sub>O-insol. stearate) while being subjected to mechanical division. D. F. T.

**Manufacture of rubber-like product.** H. G. KITTREDGE (B.P. 475,598, 17.8.36. U.S., 7.5.36).—A polyhydric alcohol (glycerol) mixed with a substantially fully polymerised product obtained by heating an unsaturated vegetable oil (tung, oiticica, perilla, or cottonseed), possibly together with an acid or acid anhydride (phthalic, succinic, or maleic), or a glyptal resin, and/or with S, is heated (at 210—232°). The product may be vulcanised (with S and an accelerator). D. F. T.

**Manufacture of resilient product [containing rubber].** A. J. BORST, jun. (B.P. 476,163, 2.5.36).—A resilient composition is obtained by mixing short strain-resisting elements, e.g., disintegrated (rubbered) fabric, and resilient vulcanisable material (rubber), sheeting the mixture so that the former become oriented and flattened, and assembling the sheets to the desired thickness. D. F. T.

**Manufacture of rubber compound.** G. S. HIERS, Assr. to COLLINS & AIKMAN CORP. (U.S.P. 2,065,937, 29.12.36. Appl., 20.4.33).—A liquid composition containing a lower % of rubber than ordinary latex, but having a higher  $\eta$ , is obtained by adding to (diluted) latex a gum from *Ceritonia siliqua* or *Conaphallus konjah* (and also borax or H<sub>3</sub>BO<sub>3</sub>). D. F. T.

**Vulcanisation of rubber.** WINGFOOT CORP. (B.P. 488,138, 29.7.37. U.S., 1.9.36).—Rubber is vulcanised in presence of accelerators containing the grouping  $\begin{matrix} \cdot\text{C}\cdot\text{N} \\ \cdot\text{C}\cdot\text{S} \end{matrix} > \text{C}\cdot\text{Se}$ , such as 1-selenylthiazole compounds, 1-selenylarylthiazoles or salts thereof, particularly those of bivalent metals (Zn), or di(arylthiazole) diselenides, particularly *di(benzthiazyl) diselenide*, m.p. 169°. Among the compounds described are: 1-selenylbenzthiazole (I), m.p. 140—143° (decomp.), and its Zn, softens at 170°, Pb, decomp. >275°, Hg<sup>II</sup>, m.p. 178—180° (decomp.), Cd, decomp. >285°, *diphenylguanidine*, m.p. 169—170°, and *piperidinium*, m.p. 149—151°, salts, and 1-2':4'-dinitrophenyl, m.p. 160.5—161.5°, and Bz, m.p. 108°, derivatives, and *di-1-benzthiazyl selenide*, m.p. 102—103°; also a product, m.p. 252—255°, obtained from (I) (2 mols.), CH<sub>2</sub>O (2 mols.), and urea (1 mol.). R. G.

**[Vulcanisation] accelerators.** WINGFOOT CORP. (B.P. 489,202, 29.6.37. U.S., 17.10.36).—Selenazyl 1-selenide compounds are claimed as rubber accelerators. The following are included among many

examples: 1-selenylbenzselenazole (I), m.p. 152—153° (from 1-chlorobenzselenazole and Na<sub>2</sub>Se) [metal (Zn) and amine (cyclohexylamine, m.p. 151—152°) salts], piperidinium benzselenazole 1-selenide, m.p. 143—144°, benzselenazyl 2:4-dinitrophenyl, m.p. 172—174°, and benzthiazyl, m.p. 114°, 1-selenides, di(benzselenazyl) diselenide, m.p. 228—229°, 1-(carbethoxyselenyl)benzselenazole, m.p. 82—84°, and compounds from (I) and CH<sub>2</sub>O alone and together with amines (piperidine, m.p. 126—127°; NH<sub>2</sub>Ph), (CH<sub>2</sub>)<sub>6</sub>N<sub>4</sub> benzyl ester, COCl<sub>2</sub>, quaternary compounds, and compounds containing a variety of reactive halogen atoms. S. C.

**Manufacture of vulcanised rubber.** J. R. GEIGY A.-G. (B.P. 489,205, 30.7.37. Switz., 1.8.36).—Polyhydroxyalkylarylamines, e.g., α- and β-naphthylglucosamines (prepared by hydrogenation of the amine and glucose in presence of a Ni catalyst at 90—100°/108 atm.), are added to the rubber mix before vulcanisation, to improve the ageing properties. S. C.

**Vulcanisation of rubber.** E. I. DU PONT DE NEMOURS & Co. (B.P. 479,451, 5.8.36. U.S., 8.8.35).—Vulcanisation is accelerated by a quaternary NH<sub>4</sub> salt in which, of the four valencies, <3 are satisfied by aliphatic (hydrocarbon) radicals and >2 by an alicyclic (hydrocarbon) radical. Amongst the 15 examples tabulated are NMe<sub>4</sub>Cl, NEt<sub>4</sub>O<sup>+</sup>Ph, and dimethylpiperidinium formate. D. F. T.

**Vulcanisation of rubber.** WINGFOOT CORP. (B.P. 488,910, 9.11.37. U.S., 30.3.37).—Vulcanisation accelerators are prepared by the interaction at about 100° of an aldehyde, e.g., CH<sub>2</sub>O, MeCHO, PrCHO, CHMe·CH·CHO, PhCHO, furfuraldehyde, with the NH<sub>4</sub> or primary, sec., or tert. saturated or unsaturated aliphatic amine salts of org. acids, e.g., fatty acids, carboxyalkylene dithiocarbamates, xanthates, dithio-furoates and -benzoates, and alkoxy-, phenoxy-, and arylenethiazylthio-glycolic acids. E.g., cyclohexylamine benzthiazylthioglycollate (48.7) and C<sub>6</sub>H<sub>13</sub>·CHO (51.3 pts.) at the boil for 3 hr. give, after removing the volatile material, a yellow semi-solid material. N. H. H.

[Accelerators for] vulcanisation of rubber. WINGFOOT CORP. (B.P. 488,691, 9.11.37. U.S., 26.3.37).—Vulcanisation accelerators are prepared by interaction of thiuronium halides, e.g., those formed from CS(NH<sub>2</sub>)<sub>2</sub>, CS(NH<sub>2</sub>Et)<sub>2</sub>, p-ethoxycyclohexylthiourea, etc. and n-C<sub>7</sub>H<sub>15</sub>Br, PrBr, hexahydrobenzyl chloride, CHMeCl·CO<sub>2</sub>H, etc. with a H<sub>2</sub>O-sol. salt of a dithio-acid, e.g., the Na, K, or NH<sub>4</sub> salts of dithiocarbamic acids and xanthic acids, in solvents, e.g., H<sub>2</sub>O, EtOH, CMe<sub>2</sub> (at 0—10°). E.g., S-benzylthiuronium chloride (30.5) in H<sub>2</sub>O (500) is treated gradually with Na pentamethylenedithiocarbamate (27.5) in H<sub>2</sub>O (500 pts.) at 10—15° to give diamino-(benzylthiol)methyl pentamethylenedithiocarbamate, CH<sub>2</sub>Ph·S·C(NH<sub>2</sub>)<sub>2</sub>·S·CS·NC<sub>5</sub>H<sub>10</sub>, m.p. 112—114°. Similarly, diamino-(carboxymethylthiol)-methyl, m.p. 203—204°, -(n-butylthiol)-, m.p. 106—107°, and -(allylthiol)-methyl, m.p. 102—103°, pentamethylenedithiocarbamate, diamino(benzylthiol)methyl dithiobenzoate, m.p. 106—111° (decomp.), dithiopyromucate, m.p. 136—137°, and xanthate, m.p. 93—94°, are obtained. N. H. H.

**Rubber compositions and methods of preserving rubber.** FIRESTONE TYRE & RUBBER CO., LTD. (B.P. 487,980, 24.6.37. U.S., 8.7.36).—Rubber is preserved and vulcanised by incorporating as anti-oxidant a substituted 1:2-dihydroquinoline containing Me at 2, further alkyl (Me) at 2 and 4, and alkyl, aryl, alkoxy, or aryloxy at 6. Sp. claim is made to the use of 2:2:4-trimethyl-1:2-dihydroquinoline substituted at 6 with Me, Bu, Ph, OEt, or OPh.

R. G.

**Preservation of rubber.** MONSANTO CHEM. CO. (B.P. 489,157, 21.2.38. U.S., 25.2.37).—Aryloxydiarylamines, e.g., 4-phenoxydiphenylamine, 2- and 4-phenoxy-, m.p. 94—96°, 4-p-tolyl-, and 4-α-naphthoxy-phenyl-β-naphthylamines (prepared from β-C<sub>10</sub>H<sub>7</sub>·OH and the corresponding amine), are used as antioxidants for rubber. S. C.

**Preservation of rubber.** J. R. INGRAM, Assr. to MONSANTO CHEM. CO. (U.S.P. 2,064,752, 15.12.36. Appl., 12.3.34).—Rubber is treated with a product obtainable by heating 2:2:4-trimethyldihydroquinoline (from COMe<sub>2</sub> and NH<sub>2</sub>Ph) with a strong non-oxidising mineral acid (conc. HCl) at 85—90° and neutralising the product. D. F. T.

**Plasticising rubber.** I. WILLIAMS and C. C. SMITH, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,064,580, 15.12.36. Appl., 11.2.36).—The resistance to flow of unvulcanised rubber is decreased by subjecting it to the action of a thiophenol (I) (C<sub>6</sub>H<sub>4</sub>Me·SH, C<sub>10</sub>H<sub>7</sub>·SH) in absence of more than ~3½% of S and also in absence of sufficient of any basic material to neutralise the (I). D. F. T.

**Improving rubber and synthetic rubber.** I. G. FARBENIND. A.-G. (B.P. 488,701, 16.3.38. Ger., 19.3.37).—The degradation (softening) of rubber during treatment with NPh·NH<sub>2</sub> can be regulated to the desired extent and the product rendered non-toxic by adding substances which bind or destroy NPh·NH<sub>2</sub>, e.g., aldehydes, ketones (PhCHO, COPh<sub>2</sub>, paraldehyde, cyclohexanone), NO-compounds, or (CH<sub>2</sub>)<sub>6</sub>N<sub>4</sub>. N. H. H.

[Preparation of] rubber solution [of low viscosity]. I. WILLIAMS and C. C. SMITH, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,067,299, 12.1.37. Appl., 11.9.35).—The η of a solution of rubber (in a petroleum solvent) is reduced by adding ~0.1—10% of an α-substituted N<sub>2</sub>H<sub>4</sub> compound, e.g., NPh·NH<sub>2</sub> or (NPh·NH<sub>2</sub>)<sub>2</sub>·ZnCl<sub>2</sub> (rubber 100), and ~1—10% of an alcohol, e.g., MeOH or EtOH (solvent 100), and allowing sufficient time for the effect to take place. D. F. T.

**Coating materials with rubber.** G. W. WORRALL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 475,737, 24.4.36).—The base material (a metal or fabric) is coated with a mixture containing latex and Na silicate solution, the dried coating being subsequently treated with acid to render the silicate insol. The coating may then be washed with H<sub>2</sub>O and/or receive subsequent treatment with latex and a fluid wax or wax-like composition. D. F. T.

**Manufacture of rubber chloride and like compositions.** RAOLIN CORP. (B.P. 489,954, 5.2.37. U.S., 5.2.36).—A solution of (milled) rubber in CCl<sub>4</sub>

is boiled under reflux until the  $\eta$  is permanently and substantially lowered;  $\text{Cl}_2$  is then led into the (irradiated) dry, boiling liquid. The heating may afterwards be continued to remove  $\text{HCl}$  and excess of  $\text{Cl}_2$ . A dry, insol. neutralising agent may also be added and the chlorinated rubber recovered from the (filtered) solution by addition of a non-solvent or introduction into boiling  $\text{H}_2\text{O}$ . Its use as a varnish base is indicated. D. F. T.

**Chlorinated rubbers.** RUBBER PRODUCERS RES. ASSOC., G. MARTIN, W. S. DAVEY, and H. C. BAKER (B.P. 476,743, 15.6.36).—Chlorinated rubber (<60%  $\text{Cl}$ ) is obtained from latex by adding reactants ( $\text{HCl}$  and oxidising agents such as chlorates, permanganates, dichromates, hypochlorites, or peroxides) to liberate  $\text{Cl}$  *in situ*. The latex may previously be preserved with  $\text{HCO}_2\text{H}$  or, if  $\text{NH}_3$ -preserved, may be treated with  $\text{CH}_2\text{O}$  and saponin before introduction of the reactants. Products containing >3.8%  $\text{Cl}$  may be "vulcanised" without  $\text{S}$  by heating with addition of metallic oxides ( $\text{ZnO}$ ,  $\text{MgO}$ ). D. F. T.

**Manufacture of chlorinated rubber products.** E. MOORES, A. P. LOWES, IMPERIAL CHEM. INDUSTRIES, LTD., and J. P. BAXTER (B.P. 488,938, 15.1.37. Addn. to B.P. 416,056; B., 1934, 1024).—Porous chlorinated rubber blocks of more uniform pore size are obtained when the dispersing agent used in the process of the main patent is the product (or alkali salt thereof) obtained by condensing a naphthalenesulphonic acid with  $\text{CH}_2\text{O}$ , *e.g.*, the product from  $\text{C}_{10}\text{H}_8$  (1) and 100%  $\text{H}_2\text{SO}_4$  (1) at  $160^\circ$  for 10 hr. diluted with  $\text{H}_2\text{O}$  (0.4), heated with 40%  $\text{CH}_2\text{O}$  (0.3) at  $95$ – $100^\circ$  for 6 hr., and finally treated with 19%  $\text{NaOH}$  (1.2 pts.). N. H. H.

[Manufacture of] vulcanised halogen-containing rubber derivatives. W. J. TENNANT. From MARBON CORP. (B.P. 488,035, 29.12.36).—A rubber hydrohalide (I) is heated with a vulcanising agent (S) in the absence of a basic stabiliser and preferably in presence of an org. accelerator ( $\text{PrCHO}\text{-NH}_2\text{Ph}$ ). Polymerised chloroprene may also be present, and resin assists the curing. A mix containing a relatively small proportion of  $\text{S}$  yields a relatively non-thermo-plastic composition, *e.g.*, by heating a mixture of saturated rubber hydrochloride (100),  $\text{PrCHO}\text{-NH}_2\text{Ph}$  (2), and  $\text{S}$  (3 pts.) at  $153^\circ$  for about 90 min. With a higher proportion of  $\text{S}$ , up to about 40 per 100 of (I), a hard rubber substitute is obtained. R. G.

**Rubber derivatives.** RUBBER PRODUCERS RES. ASSOC., G. MARTIN, W. S. DAVEY, and H. C. BAKER (B.P. 476,269, 24.8.36).—Brominated rubbers (>70%  $\text{Br}$ ) are prepared by adding to natural or artificial latex aq.  $\text{Br}$  or reactants (*e.g.*, bromates) which liberate  $\text{Br}$  *in situ*. D. F. T.

**Rubber derivative.** T. C. MORRIS, Assr. to WINGFOOT CORP. (U.S.P. 2,064,763, 15.12.36. Appl., 27.4.34).—Rubber solution is heated to its b.p. in presence of  $\text{HCl}$  and some  $\text{H}_2\text{O}$ , and the mixture is then treated with an amphoteric-metal halide ( $\text{SnCl}_4$ ). So prepared, the product is sol. in gasoline and has low  $\eta$ . D. F. T.

[Rubber hydrohalide] films. WINGFOOT CORP. (B.P. 479,464, 7.8.36. U.S., 2.10.35).—A thin film

of rubber hydrohalide (I) contains as plasticiser (II) one of 24 named products, preferably  $\text{Bu}$  stearate (imparts high tear-resistance) and  $\text{Bu}_2$  phthalate, which can be used in amounts >5%, *e.g.*, ~10%. The films are formed by the usual method, *e.g.*, forming a cement of (I), incorporating (II), spraying the mixture on an endless belt, and evaporating the solvent. Inhibitors of photochemical disintegration, *e.g.*,  $(\text{CH}_2)_6\text{N}_4$ , may be added. J. W. CR.

**Manufacture of synthetic rubber.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 488,866, 14.1.37).—Synthetic rubber-like materials *e.g.*, the polymericides of butadiene, isoprene, dimethylbutadiene, etc., are stabilised by adding sulphides of alkylated phenols, prepared, *e.g.*, by interaction of an alkylated phenol such as *p-tert.*-amylphenol, *o*-, *m*-, or *p*-cresol with  $\text{S}$ ,  $\text{S}_2\text{Cl}_2$ ,  $\text{SCL}_2$ , etc. N. H. H.

**Polymerisation of halogeno-butadienes.** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 488,213, 4.1.37).— $\text{CH}_2\text{:CH}\cdot\text{CX}\cdot\text{CH}_2$  ( $\text{X} = \text{Cl}$ ,  $\text{Br}$ ) (I) is polymerised in presence of one or more compounds consisting of (I) substituted with hydrocarbon groups, particularly with alkyl groups of  $\text{C}_{1-7}$ , especially one or two  $\text{Me}$ . Polymerisation may be carried out in various ways, especially when (I) is emulsified in  $\text{H}_2\text{O}$  with alkali oleates, alcohol sulphates, alkyl naphthalenesulphonates, or quaternary  $\text{NH}_4$  salts containing long-chain saturated hydrocarbon radicals. Sp. claim is made to the polymerisation of (I) ( $\text{X} = \text{Cl}$ ) in presence of  $\text{CH}_2\text{:CMe}\cdot\text{CCl}\cdot\text{CH}_2$  and/or  $\text{CHMe}\cdot\text{CMe}\cdot\text{CCl}\cdot\text{CH}_2$ . The polymericides retain their pliability on long storage. R. G.

**Compositions containing rubber, rubber substitutes, and synthetic rubbers.** STANDARD OIL DEVELOPMENT Co. (B.P. 479,478, 17.10.36. Appl., 13.12.35).—An elastic, *isoolefine* [*isobutylene* (I)] polymericide of mol. wt. >30,000 (>50,000–500,000) is incorporated (5–15%) in rubber (dissolved in petroleum naphtha). The (I) may be polymerised at  $\approx -50^\circ$  in presence of a catalyst of the  $\text{BF}_3$  type and a volatile solvent. D. F. T.

**Production of a rubber article having a rough sponge-like surface.** M. SAMUEL (B.P. 476,205, 19.11.36).

**Treating yarns etc. with rubber.** Rubber-coated fabric. Treating fabric containing rubber thread. Adhesive fabrics. Leather substitute.—See VI. Synthetic polysulphide plastics. Condensation products. Shaped elastic masses. Rubber chloride varnishes. Printer's blanket.—See XIII. Sheet-rubber bandage.—See XX.

## XV.—LEATHER; GLUE.

**Quality of leather produced from hides of cattle dead of rinderpest.** M. DEMPSEY and M. E. ROBERTSON (J. Soc. Leather Trades' Chem., 1938, 22, 352–357).—Four dried Nigerian hides were converted into sole leather. The microscopical sections of the soaked and limed hides, respectively, and of the finished leather revealed thin fibre bundles and a low to medium weave angle. The finished leather was very thin and flexible, had poor abrasion-

resistance but a comparatively low  $H_2O$ -absorption, and compared favourably with manufacturing bends of poor quality made from normal dried hides.

D. P.

**Loss of hide substance during soaking.** G. REZABEK (J. Soc. Leather Trades' Chem., 1938, 22, 346—352).—Samples of salted hides have been soaked in still  $H_2O$  which was changed every 24 hr. The moisture and hide substance of the soaked samples have been determined at various time intervals up to 5 days and the loss in hide substance was calc. The sol. N in the soak liquors has also been determined. The loss in hide substance was slightly  $>1\%$  per day and the loss during the first day was somewhat  $>$  that during the remaining days. Max. hydration was attained in 2 days.

D. P.

**Determination of proteolytic activity of bating products.** P. CHAMBARD and M. SOUBIRAN (J. Soc. Leather Trades' Chem., 1938, 22, 333—339).—0—5 c.c. of enzyme solution is measured into each of 10 test tubes, and the vol. diluted to 5 c.c. with distilled  $H_2O$  in each case. 5.0 c.c. of a 0.2% gelatin solution buffered at  $p_H$  7.7—7.8 are added and the test-tubes heated at  $40^\circ$  for 1 hr. 5 c.c. of a modified Esbach's reagent (aq. alcoholic citric and picric acids) are then added to each tube; absence of a ppt. indicates that the gelatin has been completely peptonised. The "activity" of the bate is expressed as the no. of g. of gelatin peptonised by 100 g. of sample. The method measures only a simple peptonisation, the  $\eta$  of the gelatin solution being only slightly reduced and the no. of free  $NH_2$ -groups slightly increased.

D. P.

**Combination of hydrochloric acid and sodium hydroxide with hide, tendon, and bone collagen.** J. BEEK, jun. (J. Res. Nat. Bur. Stand., 1938, 21, 117—124).—The three samples of purified collagen took up equal amounts of HCl from a dil. solution saturated with NaCl. Hide and tendon collagen took up equal amounts of NaOH from NaCl solutions, but bone collagen took up more than the other two. This difference was probably due to a loss of amide-N during purification of the bone collagen. It is concluded that the white connective tissues from the three sources exhibit the same behaviour towards acids or bases.

C. R. H.

**The  $p_H$  of chestnut[wood] extracts.** C. MONNET and P. PERRIN (Leather World, 1936, 28, 386—388).—The extract has the lowest  $p_H$  of any common commercial tanning extracts. It gives better penetration and lower tannin fixation than would be deduced from its  $p_H$  val., and has a peptising rather than a pptg. effect on other tannins. The  $p_H$  imparted to leather by chestnut extract is never low enough to cause premature deterioration.

CH. ABS. (e)

**Chrome[-tanning] liquors.** VIII. E. J. SERFASS and E. R. THEIS (J. Amer. Leather Chem. Assoc., 1938, 33, 422—438; cf. B., 1937, 817).—The  $p_H$ , degree of olation, ratio of complex-bound to ionic  $SO_4^{--}$ , relative mol. wt., and the migration under electric potential are reported for boiled and aged  $\frac{1}{2}$ -basic Cr sulphate solutions containing  $\frac{1}{2}$ —3 mols. of  $Na_2SO_4$  per atom of Cr. The  $p_H$  is increased by addition of neutral  $Na_2SO_4$ , and first decreases but

then returns to its initial val. on ageing. Complex-bound  $SO_4^{--}$  which is driven into the complex by boiling is released on ageing, and at equilibrium the amount of complex  $SO_4^{--}$  is independent of the total  $[Na_2SO_4]$ . An increase in the mol. size of the Cr aggregates, with a further increase on ageing, is produced by addition of  $>1$  mol. of  $Na_2SO_4$  per mol. of Cr. Approx. 95% of the OH groups were olated, irrespective of the amount of neutral sulphate present.

D. P.

**Reversibility of basic chrome sulphate tanning.** G. D. MCLAUGHLIN, D. H. CAMERON, and R. S. ADAMS (J. Amer. Leather Chem. Assoc., 1938, 33, 418—422; cf. B., 1937, 704).—When freshly Cr-tanned leather is agitated with  $H_2O$  and untanned calf skin for 6 weeks in a closed system the amount of Cr and acid stripped off the leather and fixed by the pelt is independent of the initial Cr content and basicity of the leather, but is a function of the  $p_H$  of the equilibrium solution. If the Cr-leather has been previously aged, the amount of Cr removed from the leather is diminished.

D. P.

**Aluminium-tannin combination tannage.** C. OTIN and G. ALEXA (J. Soc. Leather Trades' Chem., 1938, 22, 339—346).—Both a higher degree and rate of tannage are obtained when hide powder pretanned with 22% basic Al sulphate solution is tanned with vegetable tanning materials or sulphite-cellulose waste extract than when ordinary hide powder is so tanned. Similar results are obtained with calf skin. Al is considered to combine with the  $CO_2H$  groups of  $NH_2$ -acids, leaving the  $NH_2$  free to combine with vegetable tannin. The Al pretannage should be done separately from the vegetable tanning.

D. P.

**Physico-chemical properties of complex compounds of iron sulphate and sulphite-cellulose extract.** V. N. NIKISCHIN (Trans. VI Mendeléev Congr. Theor. Appl. Chem., 1935, 1932, 2, Pt. 1, 921—923).—Interaction of aq.  $Fe_2(SO_4)_3$  and sulphite-cellulose extract leads to formation, with the lignosulphonic acids, of Fe complexes. At ratios of tannide to  $Fe_2O_3 > 2$  these give no reaction for  $Fe^{+++}$ . Ca is pptd. as  $CaSO_4$ . The  $p_H$  of the complex systems increases with increase in this ratio and of the basicity of the initial Fe salt and the rate of heating of the system. It increases with the age of the system if unheated, and decreases if heated. Adsorption by raw hide powder increases with the above ratio. By evaporating and drying the complex in open vessels, extracts are obtained which are sol. in hot  $H_2O$ , but the amount of adsorbed tannides falls owing to irreversible changes in the structure of the complex.

CH. ABS. (e)

**Drying of total solids [in tan liquor analysis].** W. K. TAFT (J. Amer. Leather Chem. Assoc., 1938, 33, 410—417).—Large variations are obtained in the results of total solids determinations carried out on different days. No relation was found between the  $H_2O$  evaporated and the no. of dishes or the no. of ventilation holes in the oven, respectively. Better agreement was obtained when total solids were dried in an oven through which air of relatively const. humidity was passed at 85,000—100,000 c.c. per hr. Total solids, estimated under extreme atm. conditions,

showed a max. difference of 1.92%, higher moisture contents being recorded in cold dry than in hot moist conditions. D. P.

Use of sulphur monochloride for the condensation reaction in the synthesis of tanning agents of the sulphonic acid type. J. P. BERKMAN and A. J. SAVITZKI (Trans. VI Mendeléev Congr. Theor. Appl. Chem., 1935, 1932, 2, Pt. 1, 912—919).—The product of sulphonation of crude anthracene with  $H_2SO_4$  was treated with 10—20% of  $S_2Cl_2$  at 80—90°. HCl was removed and the product neutralised with conc. NaOH. The rate of adsorption by hide powder of the resulting tanning agent was > that of uncondensed aromatic sulphonic acids.

CH. ABS. (e)

Preparation of sole leather for analysis. L. A. CUTHBERT, G. A. RUFF, and J. F. WAGONER (J. Amer. Leather Chem. Assoc., 1938, 33, 368—375).—Analyses of sole leather prepared for analysis by means of a circular saw and by hand-planing, a Balderston slicer, and the Wiley mill using 2-, 3-, and 4-mm. screens, respectively, are recorded. Greatest loss in  $H_2O$  occurred with the saw and least with the hand plane. Considerably higher uncombined tannin and total  $H_2O$ -sol. figures were obtained for the mill-ground samples.  $H_2O$ -sol. extraction at 35° of samples prepared by the Wiley mill yielded similar results to those given by  $H_2O$ -sol. extraction at normal temp. of samples prepared by other methods. D. P.

Influence of copper and iron salts on behaviour of leather in the oxygen bomb. J. R. KANAGY (J. Amer. Leather Chem. Assoc., 1938, 33, 352—362, and J. Res. Nat. Bur. Stand., 1938, 20, 849—857).—Samples of chestnut- and quebracho-tanned leather containing 0.01—0.78% of Cu or Fe were aged at 100° in an atm. of  $O_2$  at 100 lb./sq. in. for 7 days and the degree of deterioration was measured by the increase in sol. N. The rate of deterioration is accelerated by increasing Cu content and by Fe salts only below  $p_H$  3.5. Considerable deterioration takes place in presence of Cu at  $p_H$  > 3.0. The metal sulphate is more harmful in each case than the corresponding acetate or lactate. The mechanism of the action of Cu and Fe is considered to be catalytic, Fe having no effect at  $p_H$  > 3.5 because it is present in an insol. form. D. P.

Influence of chrome content on acid rotting of retanned leathers. C. W. BEEBE and R. W. FREY (J. Amer. Leather Chem. Assoc., 1938, 33, 338—340).—Six samples of cow hide grain split, tanned with chestnut-wood liquor to a degree of tannage of 54, have been retanned with varying amounts of  $\frac{1}{2}$  basic Cr alum, so as to contain 0.8—2.8%  $Cr_2O_3$ , and neutralised to  $p_H$  4.5 approx. The resulting leathers were artificially deteriorated by means of the gas-chamber method (cf. B., 1935, 1105) for 12 and 18 weeks, respectively, and the resulting loss in tensile strength, decrease in  $p_H$ , and increase in sol. N and in S determined. The rate of deterioration decreases with increasing Cr content up to ~1.6%  $Cr_2O_3$ . D. P.

Determination of formaldehyde in formaldehyde-tanned leather. J. H. HIGHBERGER and C. E.

RETZSCH (J. Amer. Leather Chem. Assoc., 1938, 33, 341—352).— $CH_2O$  may be accurately determined by means of a modified Clausen titration. Approx. 2 g. of finely-divided leather are distilled with 100 ml. of 2N- $H_2SO_4$  in a Kjeldahl distillation apparatus.  $CH_2O$  distils and is collected in a large excess of  $NaHSO_3$ . The distillate is titrated with standard I solution and starch until the excess of  $NaHSO_3$  is exactly neutralised. The  $CH_2O$ - $NaHSO_3$  compound is then dissociated by addition of  $Na_2CO_3$  or borax to bring the solution to  $p_H$  9—10. 5—10% of EtOH is added to retard oxidation. The  $NaHSO_3$  liberated is determined by a second I titration. Collagen alone and, in presence of various salts, chrome- and Syntantanned leathers treated with known amounts of  $CH_2O$  yield 98—100% of the theoretical val. Low results are obtained with  $CH_2O$ -treated vegetable-tanned leathers. Various commercial  $CH_2O$ -tanned leathers contained 0.10—0.58% of  $CH_2O$ . D. P.

Fat-liquoring [of leather]. S. L. PORTER (J. Amer. Leather Chem. Assoc., 1936, 31, 193).—Neutralisation of sulphonated oil should be regulated in accordance with the type of leather to be produced. The following  $p_H$  vals. are recommended: for Cr-tanned glove 6.5, for garment 5.8, and for shoe leather 5.0. More acid oils have better penetrating powers and less acid oils a better lubricating val. CH. ABS. (e)

Determination of joint strength of high-grade glues. E. SAUER and E. WILLACH (Kolloid-Z., 1938, 84, 205—214).—The strength of glued wood joints under tensile stress was measured under varying conditions for surfaces of 1 cm.<sup>2</sup> at right-angles to the grain, all joints being kept under load for 12 hr., beyond which no increase of strength occurred. The strength decreases continuously with increase in the R.H. of the air with which the joint is in equilibrium. For testing purposes the most suitable  $H_2O$  content (55%) is obtained by drying the joint over saturated  $Ca(NO_3)_2$  solution. Max. strength is obtained with glue concns. between 35 and 45%, the optimum figure being higher for hide than for bone glues. Edge effects are unimportant. A comparison of various marketed products is given.

F. L. U.

Determination of viscosity of animal glues. E. GOEBEL (Chem.-Ztg., 1938, 62, 613—615).—Contrary to general belief, the Suhr apparatus used in determining the concn. of glue solutions appears to have been standardised with glue containing 20% of foreign matter (probably  $H_2O$  18, ash 2%), instead of the usual 16.5% ( $H_2O$  15, ash 1.5%). 17% solutions of the higher-grade glue should therefore correspond with the 17.75% solutions as recorded by the apparatus. Results obtained with these two concns. are compared. J. W. S.

Gluing wood.—See IX.

See also A., I., 476, Determining birefringence of fibres.

PATENTS.

Production of tanning agents. MONSANTO CHEMICALS, LTD., and G. W. GLADDEN (B.P. 464,766, 18.10.35).—A tanning agent, almost or quite colourless, is prepared by interaction of a chlorophenol (or



derivative),  $\text{CH}_2\text{O}$ , and  $\text{H}_2\text{SO}_4$ , in mol. proportions 2 : 1 : 2, and partly neutralising the product.

J. H. L.

#### Manufacture of stiffened leather materials.

CELLULOSE CORP. (B.P. 478,043, 30.4.37. U.S., 5.5.36).—A sheet of cellulose derivative (the stiffening material) is rendered soft and mouldable by treating with a liquid composition comprising a solvent medium for the cellulose derivative and an agent (*e.g.*, an alkali,  $\text{H}_2\text{O}$ ,  $\text{HCO}_2\text{H}$ ,  $\text{NEt}_3$ , etc.) for softening leather, and is then pressed on to a sheet of leather. The solvent medium comprises a low-b.p. solvent (*e.g.*,  $\text{COMe}_2$  with or without  $\text{MeOH}$  or  $\text{EtOH}$ ,  $\text{CHCl}_3$ , etc.), and may include higher-b.p. solvents (*e.g.*,  $\text{OH} \cdot [\text{CH}_2]_2 \cdot \text{OMe}$ ) or plasticisers (*e.g.*, arylsulphonamides).

J. W. CR.

**Manufacture of gelatin.** J. V. S. GLASS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 480,712, 27.8.36 and 27.8.37).—Ossein or other gelatin-forming material is treated with a  $\text{Ca}(\text{OH})_2$  suspension containing 0.01—0.5% (0.01—0.1%) of a primary or *sec.* amine (*e.g.*,  $\text{NH}_2\text{Me}$  or piperidine); an alkaline-earth metal halide ( $\text{CaCl}_2$ ) may also be added to the suspension.

D. W.

**Gelatin products and methods of drying gelatin.** INDUSTRIAL PATENTS CORP., Asses. of R. C. NEWTON and F. L. DE BEUKELAER (B.P. 476,128, 30.7.36. U.S., 28.5.36).—Gelatin (I) is produced in the form of hollow spheres ("pearls") by concentrating a solution to slightly < the deterioration point, allowing it to fall in drops on to a bed of dry granular or powdered (I), covering with a layer of dry (I), separating the coated drops, *e.g.*, by sieving, and drying.

I. C. R.

**Moisture-proof gelatin [wrapping material].** W. H. CHARCH, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,065,792, 29.12.36. Appl., 9.8.28).—Gelatin film is treated with 1—2%  $\text{AcOH}$  at 50—90° for 1—10 min. and then coated with a solution of a cellulose derivative, wax, plasticiser, and resin. (Cf. U.S.P. 2,030,962; B., 1936, 228.)

S. M.

**Manufacture of adhesives composed of water-soluble cellulose ethers.** W. J. TENNANT. From HENKEL & Co., G.M.B.H. (B.P. 489,614, 27.1.37).—Adhesives are obtained by causing regenerated cellulose to interact with a halogenated lower aliphatic acid or salt thereof, *e.g.*,  $\text{CH}_2\text{Cl} \cdot \text{CO}_2\text{H}$ ,  $\text{CH}_2\text{Br} \cdot \text{CH}_2 \cdot \text{SO}_3\text{H}$ , in alkaline aq. suspension and in absence of an org.,  $\text{H}_2\text{O}$ -sol. diluent.

S. C.

**Synthetic phenol resin adhesive.** A. H. BOWEN, Assr. to I. F. LAUCKS, INC. (U.S.P. 2,064,410, 15.12.36. Appl., 13.6.35).—An alkali-sol., thermo-setting phenolic resin is intimately mixed with 5—30% of  $\text{Na}_2\text{SiO}_3$  and stirred into  $\text{H}_2\text{O}$  prior to application on, *e.g.*, plywood.

S. M.

**Manufacture of resinous adhesive [for wood etc.].** C. E. ROZEMA and J. H. TIGELAAR, Assrs. to RECONSTRUCTION FINANCE CORP. (U.S.P. 2,066,857, 5.1.37. Appl., 18.10.33).—A phenol and an aldehyde are heated in presence of an alkali, *e.g.*,  $\text{CaO}$ , until the A-stage resin is formed; an aq. solution of a dispersing agent (gum arabic) is added and the mixture treated with acid until an emulsion of the resin is obtained,

which is rendered alkaline with aq.  $\text{NH}_3$  and a protein (blood-albumin) is incorporated. In the text, the emulsion may be further heated and glycerol, diatomaceous earth, and other materials added.

S. M.

**Manufacture of adhesives such as wood glue [from casein].** W. J. TENNANT. From M. H. SPIER (B.P. 475,305, 30.10.36).—To incorporate  $\text{CH}_2\text{O}$ , (a) acid casein (1 kg.), (b)  $\text{NaOH}$ ,  $\text{KOH}$ , or  $\text{Na}_2\text{SiO}_3$  etc. (0.3—1 g.-equiv.), and (c)  $\text{CH}_2\text{O}$  (0.2—1 g.-mol.) are stirred into  $\text{H}_2\text{O}$ , in this order.

S. M.

**Adhesives.** STEIN-HALL MANUFG. Co. (B.P. 474,583, 8.5.36. U.S., 26.7.35).—Adhesives suitable for making corrugated paper-board consist of a suspension of ungelatinised starch in a viscous medium containing gelatinised starch and other constituents, such that gelatinisation of the ungelatinised starch, and consequent increase in  $\eta$ , takes place *in situ* on the paper as this passes through the hot machines. Formulæ are given.

J. H. L.

**Cementing materials together.** E. I. DU PONT DE NEMOURS & Co. (B.P. 458,818, 21.6.35. U.S., 15.3.35).—A solution (generally 10%) of deacetylated chitin (cf. B.P. 458,839; B., 1937, 370) or a salt thereof in aq. acid (*e.g.*,  $\text{AcOH}$ ) is used as a cement for paper, glass, wood blocks, etc., the adhesive being rendered insol. by drying or exposure to  $\text{NH}_3$  gas, aq. alkali, acid anhydride, or  $\text{CH}_2\text{O}$ .

A. H. C.

**Drying of leather [wet tanned hides].** G. W. JOHNSON. From PROCTOR & SCHWARTZ, INC. (B.P. 489,749, 25.11.37).

**Graining or embossing of leather, artificial leather, paper, and similar materials.** VEREENIGD INDUSTRIEEL BEZIT VERITEX N.V. (B.P. 475,293, 4.8.36. Ger., 2.8.35).

**Swollen fibrous matter. Leather-like sheets. Leatherboard.**—See V. Treating leather. Artificial leather. Leather substitute. Adhesives. Adhesive fabrics.—See VI. Cellulose derivative emulsions. Casein products. Hardened protein materials.—See XIII.

## XVI.—AGRICULTURE.

**Recent experiments in land reclamation in Scotland.** W. G. OGG (Proc. Roy. Phil. Soc. Glasgow, 1938, 63, 76—88).—A review.

**Forests of the Illinoian till plain of south-western Ohio.** E. L. BRAUN (Ecol. Monog., 1936, 6, 89—149).—The  $p_{\text{H}}$  of soils under forests of different age, species, and locality is recorded. CH. ABS. (p)

**Soil reactions in forests of Lenkoran and western Georgia from the viewpoint of citrus and tea plant cultivation.** A. I. POTAPOV (Compt. rend. Acad. Sci. U.R.S.S., 1938, 18, 477—478).—The  $p_{\text{H}}$  and acidity of soils of these areas are recorded and characteristic vegetation is noted. Their suitability for tea and citrus culture is discussed. A. G. P.

**Soil complex in relation to plant communities.** I. Oxidation-reduction potentials in soils. II. Characteristic woodland soils. W. H. PEARSALL (J. Ecol., 1938, 26, 180—193, 194—209).—I. Potential drifts occurring in soils during  $E_{\text{A}}$  determinations are largely eliminated by immersion of the sample in

PhMe, especially when the sample is kneaded with a glass rod to expel air. Storage of moist soil samples results in a decrease in  $E_h$ , which is inversely related to the  $H_2O$  content. Base-deficient soils having  $E_h < 320$  mv., but not those of  $E_h > 350$  mv., develop an increased colour in Comber's CNS' test after treatment with  $H_2O_2$ . Nitrates occur in soils of the latter but not of the former category.

II. Types of woodland vegetation are examined in relation to soil- $p_H$ ,  $E_h$ , base status, and  $NO_3'$  content. A. G. P.

Effect of degree of slope and rainfall characteristics on run-off and soil erosion. J. H. NEAL (Agric. Eng., 1938, 19, 213—217).—An experimental technique is described, and the effects of soil and rainfall conditions on rates of erosion are determined for an individual soil. A. G. P.

Corrosion of iron and lead in soils. V. S. DANIEL-BEK (J. Appl. Chem. Russ., 1938, 11, 567—587).—Soil constituents increasing the velocity of corrosion of Fe are, in diminishing order of importance:  $H_2O$ , sol. chlorides, sulphates, nitrates, and humates; sol. carbonates have a protective action, whilst variations in the permeability to air are of only minor importance. In the case of Pb the factors are, in diminishing order of importance: humidity, org. acid content, permeability, and chloride content; other mineral salts have no action. R. T.

Soil biology of the Algerian high plateau. I, II. C. KILLIAN (Ann. Agron., 1936, 6, 595—614, 702—722; 1937, 7, 207—248, 336—369).—I. The life of the micro-organisms is controlled chiefly by the physical conditions of the soil, e.g., dispersion, which is directly related to the permeability to air and  $H_2O$ , and determines also the retentive power of the soil for sol. salts. These accumulate mostly as NaCl, sometimes as  $Na_2CO_3$ , which is then the limiting factor for microbiological life.

II. Physical and topographical properties and climatic conditions are discussed in their relation to the microbiology of these soils. A. W. M.

Microbiology of Appalachian podsol soils. III. Synchronous changes of bacterial numbers in two field soils. P. H. H. GRAY (Canad. J. Res., 1938, 16, C, 145—151; cf. B., 1936, 291).—In two cultivated podsoils the nos. of bacteria and actinomyces were much higher in late spring than in summer. In spring a less selective medium (soil extract-glucose-agar) indicated higher nos. than did a more selective (Thornton's) medium. In summer the two media recorded similar nos. Seasonal fluctuations were much greater in plots pretreated with limestone and/or  $Na_2CO_3$  than in untreated controls. A. G. P.

Qualitative studies of soil micro-organisms. I. Introduction. A. G. LOCHHEAD and C. B. TAYLOR. II. Survey of bacterial flora of soils differing in fertility. C. B. TAYLOR and A. G. LOCHHEAD (Canad. J. Res., 1938, 16, C, 152—161, 162—173).—I. Investigations of the biological as contrasted with the biochemical aspects of soil bacteriology are reviewed.

II. Soil organisms are classified into 8 groups. The indigenous soil flora remains fairly uniform in

soils of particular types and is not greatly affected by the level of productivity or manuring. Predominant organisms are relatively inactive in single culture. Biochemical activities show considerable divergence even among closely related species. The bacterial flora of soils is probably physiologically unstable and very adaptable. Individual species function most satisfactorily only in association with others. A. G. P.

Acidity and exchangeable lime content of good tea-farm soil in Japan. R. KAWASHIMA (J. Agric. Chem. Soc. Japan, 1938, 14, 791—798).—Data are given for the  $p_H$  of aq. and KCl solutions, Daikuhara and hydrolytic acidities, and % of exchangeable CaO for 25 surface soils and 9 sub-soils. 70% of the former have  $p_H > 5.0$ , and 60% have exchangeable CaO  $< 0.1\%$ . The tea plant grows well on an acid mineral soil deficient in exchangeable Ca. J. N. A.

Applications of colloidal chemistry to agricultural analysis. R. MEURICE (Ann. Chim. Analyt., 1938, [iii], 20, 122—124).—In determinations of base-exchange vals. of soils the size of the sample taken may influence results obtained. The possible error involved in calculating manurial requirements per hectare from base-exchange measurements of laboratory samples, by direct proportionality, is indicated. A. G. P.

Determination of the absolute volume of soil. G. GLIEMEROTH (J. Landw., 1938, 85, 245—253).—Comparison is made of the use of air- and  $H_2O$ -pyknometers for this purpose and the method of direct weighing under  $H_2O$ . Results of the pyknometer methods agree closely. A. G. P.

Colorimetric measurement of phosphorus in soil. J. BAEYENS and D. STENUIT (Agricoltura, 1936, 39, 46—58).—Available  $PO_4'''$  in soils shows varied vals. according to the vol. of citric acid (I) used in the determination. Colorimetric determination of  $PO_4'''$  is suitable if much of the (I) is destroyed (method described). CH. ABS. (p)

Determination of adsorbed bases and exchange capacity of soils by the boiling ammonium chloride procedure. W. M. SHAW (J. Assoc. Off. Agric. Chem., 1938, 21, 252—257).—The procedure is described. The vals. for exchangeable Ca and Mg are less consistent than those for  $NH_3$  absorption (= base-exchange capacity). The steps to be taken to minimise the observed differences are discussed, especially the desirability of neutralising EtOH used for washing the  $NH_4Cl$ -treated soil. E. C. S.

Determination of basoid and acidoid contents of soil ampholytoids. A. V. ZNAMENSKI (J. Appl. Chem. Russ., 1938, 11, 674—681).—Theoretical. Freundlich's equation, with  $n = 1$ , is not applicable to exchange adsorption in soils. R. T.

Hydrogen-ion concentration of soils of arid and semi-arid regions. W. T. MCGEORGE (J. Assoc. Off. Agric. Chem., 1938, 21, 246—247).—For the determination of  $p_H$  in soils of low  $H_2O$  content a spear-type glass electrode is advocated. The val. so obtained is a more reliable index of soil condition than one determined on a more diluted specimen. E. C. S.

Direct determination of  $p_H$  of soil in its natural state by the quinhydrone method. III. Determination of soil- $p_H$  in the dry farm. A. ITANO and Y. TSUJI (Ber. Ohara Inst. landw. Forsch., 1938, 8, 83—95; cf. B., 1937, 477).—The  $p_H$  can be determined in soil with as little as 5% of  $H_2O$ , equilibrium being reached in 2 min. In dry soils the difference in  $p_H$  between adjacent areas is more marked than in wet soils. The vals. obtained under natural conditions are < those with fresh or air-dried samples in the laboratory. The  $p_H$  in the neighbourhood of wheat plants is <, and of rice plants >, that of the soil between the plants. E. C. S.

Hydrogen-ion concentration of soils of humid regions. Factors that influence the  $p_H$  value of coastal plain soils and methods for determination. J. B. HESTER (J. Assoc. Off. Agric. Chem., 1938, 21, 247—251).—The  $p_H$  and buffer capacity of colloid, clay, and humus from 3 areas are tabulated. The colorimetric, quinhydrone, and glass electrode methods are compared. The last is the most accurate, but the first has a low initial cost and is sufficiently accurate. E. C. S.

Research work in the fertiliser industry during the second Five-Year Plan, and its perspectives during the third Five-Year Plan. S. I. VOLFKOVITSCH (Bull. Acad. Sci. U.R.S.S., Sér. Chim., 1938, No. 1, 33—38).—A review. R. T.

Use of common salt as fertiliser. I. W. SELMAN (J. Min. Agric., 1938, 45, 237—246).—Various interactions between the soil and NaCl and their bearing on plant nutrition are discussed. A. G. P.

Potash in plant metabolism. G. N. HOFFER (Ind. Eng. Chem., 1938, 30, 885—889).—K does not enter into any permanent chemical combination in plant tissues from which it can be completely leached. Published work on the rôles played by K, and symptoms of K starvation in various crops, are summarised. C. I.

Production of potassium sulphate fertilisers from potassium chloride and mirabilite. A. I. ZASLAVSKI, S. S. SINANI, and L. A. SOKOLOVA (Bull. Acad. Sci. U.R.S.S., 1938, Sér. Chim., 47—68).—Equilibrium in the system  $K_2SO_4$ -NaCl- $H_2O$  has been studied at  $-5^\circ$ ,  $25^\circ$ , and  $75^\circ$ , and the fields of crystallisation of  $K_2SO_4$  and glaserite were determined. The possibility of producing  $K_2SO_4$  by exchange of KCl and  $Na_2SO_4 \cdot 10H_2O$  has been investigated; the yield increases from 21.1% at  $75^\circ$  to 38.0% at  $-5^\circ$ . E. S. H.

Application of sulphur in agriculture. II. Effect of sulphur on colloidal constituents of soil. R. CULTRERA and A. C. GALLETI (Annali Chim. Appl., 1938, 28, 244—252).—S added to soil is slowly oxidised to  $SO_4^{2-}$ , formation of which is accompanied by a proportional fall in  $CO_3^{2-}$  and colloidal dispersivity (cf. B., 1938, 200) and increase in sol. salts and  $\sigma$  of the soil. F. O. H.

Boron compounds used as fertilisers. A. V. SOKOLOV (Bull. Acad. Sci. U.R.S.S., 1938, Sér. Chim., 267—281).—Published work is summarised. E. S. H.

Importance of boron in agriculture and horticulture. R. QUARENDON (Chem. and Ind., 1938, 741—745).—The occurrence of B in soils and certain B-deficiency diseases are discussed. A. G. P.

Use of molasses as a manure. R. C. WOODS (Trop. Agric. Trinidad, 1938, 15, 85—86).—Application of 5—10 tons of molasses per acre temporarily checks weed growth by inducing toxic conditions in soil. On soils of high fertility, yields of maize and sweet potato were not appreciably affected. Molasses increases the porosity of soil. A. G. P.

Transformations of fertilisers in soil. G. BARBIER (Chim. et. Ind., 1938, 39, 851—856).—The rôle of ionic exchange and of certain slower chemical interactions between soil and fertiliser in the nutritive action of the latter is discussed. A. G. P.

Acid- and base-forming quality of fertilisers. L. E. HORAT (J. Assoc. Off. Agric. Chem., 1938, 21, 296—304).—Titration curves of fertiliser compounds show that titration is best carried out to  $p_H$  4.3—4.5. Bromophenol-blue and a mixture of bromocresol-green and Me-orange are suitable indicators. A device for prevention of spattering during evaporation is described. E. C. S.

[Determination of] potash [in fertilisers]. O. W. FORD (J. Assoc. Off. Agric. Chem., 1938, 21, 293—296).—Experiences with a modification of the official method, designed to eliminate foaming during boiling of the sample, are described. E. C. S.

[Determination of water-soluble] phosphoric acid [in fertilisers]. (A) Effect of method of filtering. (B) Variation in citrate-insoluble phosphoric acid with time interval between water extraction and citrate digestion. J. R. ADAMS and W. H. ROSS (J. Assoc. Off. Agric. Chem., 1938, 21, 268—271, 271—273).—(A) The kind of filter-paper used has little if any effect, but washing under suction gives lower results than under-gravity washing.

(B) No increase in citrate-insol.  $P_2O_5$  occurs between washing with  $H_2O$  and citrate digestion unless the material contains  $CaHPO_4$  or  $Ca_3(PO_4)_2$ . E. C. S.

[Determination of] citrate-insoluble phosphoric acid in ammoniated [fertiliser] mixtures containing dolomite. W. H. ROSS, L. F. RADER, jun., and K. C. BEESON (J. Assoc. Off. Agric. Chem., 1938, 21, 258—268).—Preliminary results suggest that  $CaHPO_4$  and  $Ca_3(PO_4)_2$  are hydrated to  $3Ca_3(PO_4)_2 \cdot Ca(OH)_2$  when kept at > room temp., the change being more extensive in presence of dolomite. In presence of F fluophosphates may be formed. These products are insol. in citrate, and their proportions in ammoniated superphosphate depend on basicity,  $H_2O$ , F, concn. of reacting components, and time and temp. of storage. E. C. S.

Development of and nitrogen fixation by certain micro-organisms in relation to trace elements and weather. H. BORTELS (Ber. deut. bot. Ges., 1938, 56, 153—160).—The significance of Mo and V in the growth of *Azotobacter* and other soil micro-organisms is discussed. The productivity of certain

semi-arid soils may be due partly to facilitated growth of *Azotobacter*, and partly to retention of Mo by soil due to restricted leaching. A. G. P.

**Chemical analyses as an aid in the control of nitrogen fertilisation.** J. J. YUEN and R. J. BORDEN (Hawaiian Planters' Rec., 1937, 41, 353—383; Int. Sugar J., 1938, 40, 278).—Rapid methods of determining N in soils have been applied in Hawaii, the  $\text{NO}_3^-$  and  $\text{NH}_3\text{-N}$  being found separately. "Fertility levels" for the total N present in the soils were established, as follows: low 0.001%, doubtful 0.001—0.002, medium 0.002—0.004, high >0.004%; these vals. correspond with the following figures for lb. of N per acre-foot of soil: <25, 25—50, 50—100, and >100. It is considered that a satisfactory sugarcane crop calls for the use of 150—250 lb. of N per acre-foot of soil. J. P. O.

**[Determination of] magnesium and manganese in fertilisers.** J. B. SMITH and E. J. DESZYCK (J. Assoc. Off. Agric. Chem., 1938, 21, 277—293).—Determinations were made of acid-sol.,  $\text{H}_2\text{O}$ -sol., and citrate-insol. MgO, MgO sol. in 2%  $\text{NH}_4\text{Cl}$  in mixed fertilisers, and of MgO in kieserite and  $\text{K}_2\text{SO}_4\text{-Mg(OH)}_2$ , all methods giving reasonable consistent results. The Bartlett-Tobey method for  $\text{H}_2\text{O}$ -sol. MgO gave results in agreement with those of Hoffman's method. The val. of these results in relation to availability of Mg to the plant is discussed. A procedure for the determination of acid-sol. Mn is described. E. C. S.

**Growth substances and the rooting of cuttings.** M. A. H. TINCKER (J. Roy. Hort. Soc., 1938, 63, 210—229).—Comparative trials are recorded of the rooting of cuttings of numerous species on treatment with  $\beta$ -indolyl-acetic and -butyric acids,  $1\text{-C}_{10}\text{H}_7\text{-CH}_2\text{-CO}_2\text{H}$ , and  $\text{CH}_2\text{Ph-CO}_2\text{H}$  (I) are recorded. (I) is probably insufficiently active for general use. The influence of the period of treatment is considered. A. G. P.

**Effect on a Cecil clay type of soil of continuous application of mineral plant-food elements.** G. M. YARREN and E. E. RANDOLPH (J. Elisha Mitchell Sci. Soc., 1935, 51, 216—218).—Soil and crop analyses in continuous manuring experiments are recorded. CH. ABS. (p)

**"Manuring" of green malt with organic nitrogen compounds.** M. HAMBURG (Braun. Malzind., 1938, 31, 27—28).—The possible effects of spraying the sprouting barley with urea nitrate on the protein and carbohydrate contents of the sprouts and the wort are discussed. A. G. P.

(A) **New method of tau-saghyz exploitation.**  
(B) **Rubber formation in tau-saghyz.** F. P. MAZANKO (Compt. rend. Acad. Sci. U.R.S.S., 1938, 19, 95—98, 99—102).—(A) Best yields of rubber were obtained by cutting roots below the crown at the stage of transition to the summer resting period.

(B) Rubber can be produced (after root-tapping) in the latex vessels themselves without participation of the leaves. The bearing of these observations on tapping practice is discussed. A. G. P.

**Absorption of mineral nutrients by cane at successive stages of growth.** A. AYRES (Hawaiian

Planters' Rec., 1937, 41, 335—351; Int. Sugar J., 1938, 40, 279—280).—A field of H 109 sugar cane in Hawaii (first ratoons) was sampled at monthly intervals for 14 months, and the stalks were analysed. The amounts of mineral nutrients absorbed by the leaves and stalks of the plant were found to be markedly influenced by the age of the plant, particularly during the early months of growth. Also the principal nutrients were taken up in widely different amounts, e.g., K and Si to a greater extent than N and P, and Ca and Mg in the least amounts. Max. rates of absorption, excepting in the case of Si, were attained at the age of 3 months, although some nutrients were absorbed at a seemingly faster rate than others. After the max. rate was passed, the absorption rates of all the nutrients decreased. J. P. O.

**Utilisation of cane molasses [in soils]. I. Dissolution of bone by fermentation of cane molasses.** T. R. BHASKARAN, S. C. PILLAI, and V. SUBRAHMANYAN (J. Indian Inst. Sci., 1938, 21A, 27—39).—Appreciable dissolution of bone added to molasses solution inoculated with soil organisms takes place during fermentation,  $\text{PO}_4^{''''}$  dissolving more rapidly than other bone constituents. Dissolution increases with the degree of fineness of the bone material, and with sugar decomp. and acid production, but without proportionality, and reprecipn. may ultimately occur; pretreatment of the bone with  $\text{H}_2\text{O}$ , NaOH, or CaO is without favourable effect. Successive fermentations result in almost complete dissolution. Treatment of the fermented liquor with CaO gives a ppt. somewhat resembling the original bone in composition; it is, however, completely sol. in 1% citric acid. The difficulties involved in applying such a fermentation for the production of fertilisers are great, but availability of bone fertiliser etc. could be increased by suitable application of molasses to the land treated. I. A. P.

**Uptake of nutrients by Brussels sprouts.** A. H. LEWIS (J. Min. Agric., 1938, 45, 262—268).—The gross intake of mineral nutrients by Brussels sprout plants is very high, a large proportion being assimilated in the 3rd and 4th months after planting out. The N and P contents of the crop increase until picking of the sprouts is completed, but the intake of K ceases after October. The Ca content increases rapidly to a max. in September and then diminishes considerably owing to leaf-fall. N, P, and K in moribund leaves are translocated before leaf-fall to developing sprouts which are of low Ca content. The true: crude protein ratio in the plant diminishes somewhat and the org. P fraction increases progressively as the season advances. A. G. P.

**Tonka bean.** F. J. POUND (Trop. Agric. Trinidad, 1938, 15, 4—9, 28—32).—Cultivation, manuring, curing, and analyses of the beans are described. A. G. P.

**Nutrition and fertilisation of soft fruits.** T. WALLACE (Imp. Bur. Fruit Prod., Tech. Comm., 1936, No. 6, 52—87).—Effects of various K fertilisers on the yield and composition of gooseberries and red currants are examined. CH. ABS. (p)

**Oxidase and catalase activity of Bartlett pears in relation to maturity and storage.** B. D. EZELL and F. GERHARDT (J. Agric. Res., 1938, 56, 337—346).—Oxidase (I) activity in the pears diminished throughout the growing season. Catalase (II) activity diminished steadily to a min. at the stage suitable for canning and subsequently increased progressively until full ripeness. High (I) content is associated with poor colour in canned fruit. (II) is unrelated to colour in canned pears, but best-quality produce is obtained from fruit picked at min. (II) level. A. G. P.

**Growth, nutrient requirement, and manuring of rhubarb.** J. BECKER (Obst u. Gemüsebau, 1938, 84, 61—63).—Manurial trials are recorded. The high K requirement of rhubarb is emphasised. A. G. P.

**The blueberry.** B. THOMAS and H. W. DOUGALL (J. Min. Agric., 1938, 45, 546—552).—The plant, *Vaccinium myrtillus*, is found growing on dry, peaty, CaO-deficient soils at comparatively high elevation in competition with heather; young spring growth is useful as sheep and grouse feed. Samples taken from May to Sept. showed consecutive decreases in crude protein from 18.6 to 10.1, and increases in fibre from 9.9 to 17.2%, of the dry matter. Over 90% of the crude is true protein. Samples contained >1.0% of CaO and 0.5% of P<sub>2</sub>O<sub>5</sub>. The dry matter of the berries contained protein 9.7, Et<sub>2</sub>O extract 7.2, fibre 12.8, N-free extract 67.4, ash 2.9, P<sub>2</sub>O<sub>5</sub> 0.42, and CaO 0.35%. The digestibility of the protein of summer and autumn samples is <40%. W. L. D.

**Effect of budding [removal of flower head] on vitality of tobacco plants.** A. WENUSCH and R. SCHÖLLER (Z. Unters. Lebensm., 1938, 75, 574—576; cf. B., 1937, 619).—The loss in wt. on drying, to which the "vitality" (cf. *loc. cit.*) is inversely proportional, of budded and unbudded plants is recorded. Plants budded in full flower lose in vitality, whilst those budded before the flowers are formed gain. E. C. S.

**Correlations in plant-tissue composition, decomposition products, and effect on crop rotation with tobacco.** W. S. EISENMENGER (J. Agric. Res., 1938, 56, 309—316).—Effects of a no. of crops and their decomp. in soil on the growth of subsequent tobacco crops are examined. In general, plants containing high lignin and pentosan contents, high C/N ratios, and exhibiting slow tendency to protein decomp. in soil are less suitable for inclusion in tobacco rotations. A. G. P.

**Comparison of early, medium, and late strains of timothy harvested at different stages of development.** M. W. EVANS and L. E. THATCHER (J. Agric. Res., 1938, 56, 347—364).—Yields and protein contents of hays from red clover-timothy mixtures taken at intervals over a 3-year period are recorded. With all strains of timothy best results in respect of gross yield, protein yield, and quality of hay were obtained by cutting in the early flowering stage or immediately before this. A. G. P.

**Ambrosia (ragweed) and Paspalia weed: destruction with sodium chlorate.** H. BARTLETT (Agric. Gaz. New South Wales, 1937, 48, 307—311).

—Successful results of spraying with 10% aq. NaClO<sub>3</sub> are recorded. A. G. P.

**Mineral oils as insecticides.** J. CARROLL (Econ. Proc. Roy. Dublin Soc., 1938, 3, 63—74).—A review. A. G. P.

**Organic compounds as insecticides.** H. G. GUY (Delaware Agric. Exp. Sta. Bull., 1937, No. 206, 60 pp.).—Among numerous compounds examined, the following exhibited toxicity approaching that of arsenicals: PH<sub>4</sub> compounds (notably PPh<sub>3</sub>MeCl and PPh<sub>3</sub>MeI), co-ordinated Cr salts (piperidinium tetra-thiocyanatodiamminochromium), thiazines (phenyl-thiazine), thiuram sulphides, and thiocarbamates (repellent to chewing insects). Numerous toxicity trials and effects of these substances on plants are recorded. A. G. P.

**Field experiments on bunt of wheat.** G. H. STARR (Wyoming Agric. Exp. Sta. Bull., 1938, No. 226, 23 pp.).—Seed-borne smut is best controlled with Ceresan or CH<sub>2</sub>O. Irrigation tended to favour development of smut in the field. The time of sowing is an important factor. A. G. P.

**Oil emulsion and red spider.** P. H. THOMAS and T. D. RAPHAEL (Tasmanian J. Agric., 1936, 7, 19—20).—Emulsions of crude oil were less satisfactory than those of standard oils for spraying infested apple wood. Heavier oils were usually more effective. Emulsions <1:15 gave inferior results. Poor results were obtained with oil-CaO-S combinations. CH. ABS. (p)

**Lime-sulphur and substitutes in New York.** W. D. MILLS (Penna. State Hort. Assoc. Proc. 77th Ann. Meet.; Penna. State Hort. Assoc. News, 1936, 13, No. 1, 10—12, 14).—Bentonite-S sprays were inferior to CaO-S (I) or flotation S (II) in controlling apple scab. Against leaf scab (I) was more effective than (II); both were equally effective against fruit scab. (II) caused less leaf scorch than (I) when applied later than the calyx spray. Cheek scald was greatest with (I) and least with dry-mix S and bentonite-S. CH. ABS. (p)

**New sprays for disease control.** A. LINVILLE (Penna. State Hort. Soc. Proc. 77th Ann. Meet.; Penna. State Hort. Assoc. News, 1936, 13, No. 1, 34—35).—Bordeaux mixture (4:8:100) used for bitter-rot control on apples injured foliage and fruit. Cu<sub>2</sub>O caused russetting when applied at the cluster-apple stage, but had no ill effects on foliage of apples and peaches in interplanted orchards, and controlled sooty blotch nearly as effectively as did CaO-S. CH. ABS. (p)

**Rôle of arsenicals in the Hudson Valley spray programme for apples.** P. J. CHAPMAN (Proc. 80th Ann. Meet. New York State Hort. Soc., 1935, 180—185).—Customary spreading agents probably increase the killing power of Pb arsenate sprays against apple leaf-roller, but are inferior to oil, which acts by improving coverage. Against the apple maggot Pb and Ca arsenates were superior to Mn arsenate or cryolite. CH. ABS. (p)

**Internal cork of apples.** C. G. SAVAGE and H. BROADFOOT (Agric. Gaz. New South Wales, 1937, 48, 387—390, 447—452).—Application of borax

to soil (1 lb. per tree) prevented internal cork. Direct injection into roots gave partial protection.

A. G. P.

**Chemically treated bands for codling-moth control.** A. M. WOODSIDE (Virginia [Blacksburg] Agric. Exp. Sta. Bull., 1938, No. 315, 22 pp.).—Successful tree banding as a supplement to spraying is described.

A. G. P.

**Spray residue as related to codling-moth control.** S. W. HARMAN (Proc. 81st Ann. Meet. New York State Hort. Soc., 1936, 40—51).

**Cyanide fumigation of citrus trees in the eastern Cape Province, South Africa.** B. SMIT (Union S. Africa Dept. Agric. Bull., 1937, No. 101, 39 pp.).—Details of various methods of application of HCN are described.

A. G. P.

**Determination of small quantities of antimony in tartar emetic spray residues [on citrus leaves].** J. DAVIDSON, G. N. PULLEY, and C. C. CASSIL (J. Assoc. Off. Agric. Chem., 1938, 21, 314—318).—Apparatus and procedure for the stain method are simplified. Better and more consistent stains are obtained when  $Pb^{II}$  is present in the generator.  $Sb^V$  is converted into  $SbH_3$  without previous reduction to  $Sb^{III}$ . Sb as tartar emetic is recovered from vegetable material ashed at  $550^\circ$ .

E. C. S.

**Bait traps for control of the oriental peach moth (*Grapholitha molesta*).** M. L. BOBB (Virginia [Blacksburg] Agric. Exp. Sta. Bull., 1938, No. 314, 14 pp.).—A bait solution of lignin pitch 1 pt. in  $H_2O$  20 pts. containing 1 c.c. of terpinyl acetate per quart gave best results. Means of setting-up traps are compared.

A. G. P.

**Sources of raspberry mosaic infection.** L. M. COOLEY (Proc. 80th Ann. Meet. New York State Hort. Soc., 1935, 273—277).—Wild red raspberries infected with mosaic were killed by spraying with  $NaClO_3$  2 lb.,  $NH_4CNS$  (I) or  $Na_3AsO_3$  0.5 lb. per gal. Toxic effects of (I) in soil disappeared after 3—6 months, but the other preps. prevented cropping for 2—3 years.

CH. ABS. (p)

**Insect problems of small fruits.** O. H. HAMMER (Proc. 81st Ann. Meet. New York State Hort. Soc., 1936, 279—283).—Derris or cubé root preps. controlled the gooseberry fruit worm (*Zophodia grossularice*, Riley) on currants and also the imported currant worm (*Pteronius ribesii*, Scopoli). Currants carrying 7—8 times the tolerance limit of Pb and As residues were not effectively cleaned by dipping in 2% HCl for 1.5 min.

CH. ABS. (p)

**Toxicity of *Haplophyton cimicidum* to fruit flies.** C. C. PLUMMER (U.S. Dept. Agric. Circ., 1938, No. 455, 10 pp.).—Extracts of the leaves probably contain an alkaloid which is toxic to fruit flies. Extracts slowly lose their activity on storage.

A. G. P.

**Control of mango anthracnose (*Colletotrichum gloeosporioides*).** R. E. D. BAKER (Trop. Agric. Trinidad, 1938, 15, 12—14).—Repeated spraying of flower spikes and fruit from the flowering period onward gave considerable control of the disease and wastage of harvested fruit was delayed.

A. G. P.

**Pyrethrum as a possible insecticide for control of sugar-cane froghopper.** A. PICKLES (Trop. Agric. Trinidad, 1938, 15, 75—76).—Preliminary trials indicate the probable val. of pyrethrum dust in controlling the adult insects.

A. G. P.

**Mildew or white rust of tobacco.** E. S. MOORE (Farming in S. Africa, 1938, 13, 223, 241).—Early dusting with S protected the crop from mildew, but harvested leaves retained appreciable amounts of S even when a single treatment was given only 1 month after planting.

A. G. P.

**Control of tobacco insects in Tennessee.** S. MARCOVITCH and W. W. STANEY (Tennessee Agric. Exp. Sta. Bull., 1937, No. 160, 14 pp.).—Recommendations are given for control of hornworm, cut-worms, and leaf-hoppers in tobacco.

A. G. P.

**Effect of low concentrations of copper on germination and growth of conidia of *Sclerotinia fructicola* and *Glomerella cingulata*.** M. C. GOLDSWORTHY and E. L. GREEN (J. Agric. Res., 1938, 56, 489—505).—Aq.  $CuSO_4$  containing 0.25 p.p.m. of  $Cu^{++}$  was toxic to conidia of both species, whether in flowing media or in agar cultures. In the latter the toxic effect depended on the ability of the medium to "fix" Cu, notably by combination with alcoholic OH groups or by adsorption. With relatively insol. Cu compounds, toxicity was controlled by the amount of "available" Cu rather than by the actual  $[Cu^{++}]$  maintained. Effects of a no. of such compounds are recorded.

A. G. P.

**Gypsy and brown-tail moths and their control.** A. F. BURGESS and W. L. BAKER (U.S. Dept. Agric. Circ., 1938, No. 464, 37 pp.).—The life history of these insects is described and their control by various spraying and tree-banding methods discussed.

A. G. P.

**Yellow *Monolepta* beetle: control methods.** C. R. WALLACE (Agric. Gaz. New South Wales, 1937, 48, 268—269).—Dusting with undiluted pyrethrum, pyrethrum-nicotine, or pyrethrum-kaolin is recommended.

A. G. P.

**[Use of] tetrachloroethylene emulsion ("Tetram") [as parasiticide].** H. O. MÖNNIG (Farming in S. Africa, 1938, 13, 146).—Use of the emulsion in treatment of internal parasites of sheep and cattle is described.

A. G. P.

**Bionomics and control of bursate nematodes of horses and sheep.** V. Comparisons of lethal effects of some non-nitrogenous fertilisers on the free-living stages of sclerostomes. I. W. PARNELL (Canad. J. Res., 1938, 16, D, 73—88; cf. B., 1937, 1255).—Larvæ in fresh horse manure are killed by addition of kainit 1 in 23, KCl 1 in 17,  $K_2CO_3$  1 in 13,  $K_2SO_4$  1 in 5, superphosphate (20%) 1 in 5, or superphosphate (16%) 2 in 5 pts. of manure. Basic slag, Florida rock phosphate, and CaO had little toxic action.

A. G. P.

**Concn. of phosphorites. Potash fertilisers. Oxidation of P and  $PH_3$ .**—See VII. [Manure from] *Aleurites* seeds.—See XII. New rubber plant.—See XIV. [Sugar-]carbonatation scums.—See XVII. Cattle sprays. Determining Ca and Mg [in soils].—See XXIII.

See also A., I, 471, Determining Se. 472, Micro-determining Zn. III, 769, Intake of nutrients by maize and hemp. 770, Determining K in plant ash.

#### PATENTS.

**Preparation of [phosphatic] fertilisers containing citrate-soluble phosphate.** H. D. ELKINGTON. From F. KERSCHBAUM (B.P. 475,993, 24.12.36).—Fertilisers containing  $P_2O_5$  (99% citrate-sol.) are obtained by heating at  $1000^\circ$ , preferably with a blast in a Dwight-Lloyd apparatus, raw phosphate, *e.g.*, Florida pebble, with  $Na_3PO_4$  or a mixture which yields this on heating, *e.g.*,  $Na_3PO_4 + Na_2CO_3$ , the  $P_2O_5$  ratio being 1 : 0.8. If 10—20% of KCl or  $MgCl_2$  is added a temp. of  $800$ — $900^\circ$  is sufficient. The amount of  $Na_3PO_4$  may be reduced, *e.g.*, to the theoretical  $P_2O_5$  ratio 1 : 0.5, if the raw phosphate is pretreated with  $P_2O_5$ , *e.g.*, in the waste gases from a P blast furnace. I. C. R.

**Treatment of seeds [with mercurial fungicides].** G. KOSSEL and J. R. BOOER (B.P. 486,986, 2.3.37).—The use as fungicidal seed-dressings is claimed of compounds  $R \cdot Hg \cdot NHR'$ , produced by interaction of an alkyl- or aryl-mercuric salt, oxide, or hydroxide with ureas,  $CaCN_2$ , dialkyl- and diarylguanidines, amines, thioureas, and acid amides. Examples are the compounds  $HgEt \cdot NH \cdot CO \cdot NH_2$  ( $HgEt \cdot OH$  and urea) and  $HgMe \cdot NH \cdot CN [(HgMe)_2SO_4$  and  $CaCN_2]$ . H. A. P.

**Immunising of seed grain.** W. P. WILLIAMS. From SCHERING-KAHLBAUM A.-G. (B.P. 487,512, 21.1.37).—The use is claimed as seed-dressings of compounds  $HgRR'$ , in which R is aralkyl or a saturated or unsaturated substituted hydrocarbon residue, and R' a different hydrocarbon radical, optionally substituted, preferably highly diluted (to Hg content 0.7%) with talc etc. Examples are: Hg ethyl-trichloroethylene,  $HgEt \cdot CCl_2 \cdot CCl_2$  (prepared from  $MgEtBr$  and  $CCl_2 \cdot CCl_2 \cdot HgCl$ ),  $HgEt \cdot [CH_2]_2 \cdot OMe$ ,  $HgEt \cdot CH_2Ph$ , and  $Hg([CH_2]_2 \cdot OMe) \cdot CCl_2 \cdot CCl_2$ . H. A. P.

**Manufacture of parasiticial compositions [for plants].** E. I. DU PONT DE NEMOURS & Co. (B.P. 488,428—9, 6.1.37. U.S., 6.1.36).—(A) The discoloration on storage of parasiticial compositions containing thiodiphenylamine (I) is inhibited and their toxicity is preserved by the incorporation of small amounts (1—10%) of quinol or  $\beta\text{-}C_{10}H_7 \cdot OH$ . (B) The parasiticial efficiency of compositions containing (I) is prolonged by incorporating 1—10% of a stable reducing derivative of a S acid of an oxidation stage  $< H_2SO_4$ . Suitable stabilisers are  $NH_4$ , alkali, or amine salts of mercaptans, *e.g.*, thiolbenzthiazole,  $C_8H_{17} \cdot SH$ ,  $C_{10}H_7 \cdot SH$ , thiourea, or of  $H_2SO_3$ ,  $H_2S_2O_4$ , and polythionic acids. N. H. H.

**Pb arsenate.**—See VII. Preps. for protection from insects. Irrigation  $H_2O$ . Treating waste refuse etc.—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

[Cane-juice] subsidiers. H. HORTON (Int. Sugar J., 1938, 40, 310—311).—The view is maintained that turbulence set up by the scum weirs of a cane-

juice subsidier of the standard Queensland type may be more serious than is generally realised. It is not the length of weir that matters so much as the elimination of turbulence, especially at the entry. An "ideal" subsidier is proposed (which in effect is on the lines of a Dorr clarifier), having an outlet well below the scum level, so as to avoid disturbance from this cause. The flow is radial, and the depth of clear juice is made to suit conditions. Entry must be submerged, and any turbulence it does cause is well isolated from the settling zone. The height of scum over the weir can be varied to suit the operator by making the vertical pipe telescopic and varying the level relative to that of the scum weir. J. P. O.

**Trial with saccharate liming.** R. W. G. HESSEY (Int. Sugar J., 1938, 40, 322).—About 30% of the CaO added to the raw juice was dissolved in multiple-effect supply juice. This method of saccharate liming gave a less turbid juice and a better elimination of waxes, which was in turn reflected in the improved filtering quality of the raw sugars shipped for refining. J. P. O.

**Automatic liming [of sugar-beet juice], using the Eisner apparatus.** ANON. (Int. Sugar J., 1938, 40, 312—313).—Over-liming faults can be eliminated by apparatus controlling the flow of the milk of CaO (I) by the rate of flow of the juice itself. In one form of apparatus, the inflowing juice enters at the bottom of the measuring vessel, at which point the (I) is added, the rising juice compressing the air in the air cylinder so as to force the (I) out of the apparatus into the entering juice. By means of this apparatus a const.  $p_H$  so essential in pre-liming can be maintained. In another type of apparatus, the (I) is displaced by the suction created by the outgoing juice in the air cylinder. Where both pre-liming and main liming are controlled by this apparatus, the pressure type is usually applied to the former and the suction type to the latter operation. Both are entirely automatic. J. P. O.

**Drying [sugar-juice] carbonatation scums.** E. RIEGER (Deut. Zuckerind., 1938, 63, 57—60; Int. Sugar J., 1938, 40, 318).—A Büttner rapid-drying installation is in operation at the Stöbnitz sugar factory, in Germany, drying carbonatation mud for sale to farmers as agricultural CaO. It has a capacity of about 8 tons of mud (45%  $H_2O$  content) per hr., and the fuel consumption (crude brown coal) is about 25% of the output. Drying costs per 100 kg. of finished product amount to 0.72 RM. Packing in 4-ply paper bags is costly, but the product is in too fine a state of division to be transported loose. It is usually applied with a fertiliser distributor at the rate of 23—31 cwt. per acre, depending on soil acidity. J. P. O.

[Importance of] stirring during carbonatation [of beet-sugar juices]. O. SPENGLER (Z. Wirts. Zuckerind., 1938, 88, 371—377).—It is pointed out that an essential condition of the correct operation of the author's process (cf. B., 1937, 1110) is an adequate circulation of the air in the tank, otherwise the  $CO_2$  used for stirring is not carried off sufficiently quickly, and a state approaching stagnation may result. An apparatus is described which is provided with a

propeller operating inside an open central cylinder. The tank has suitable openings in the sides for entrance of air, and a chimney at the top through which air and  $\text{CO}_2$  are discharged. J. P. O.

**Brukner's barium chloride method [for second-carbonation beet juices].** O. SPENGLER and S. BÖTTGER (Deut. Zuckerind., 1937, 62, 368; cf. B., 1937, 716, 827).—The authors' two-stage titration method (B., 1933, 405) is simpler to carry out than Brukner's method, and the latter must be vitiated by the presence of buffer substances.

J. H. L.

**Filtration with filter aids [in beet sugar manufacture].** O. WOHRZEK (Deut. Zuckerind., 1937, 62, 667—669).—Kieselguhr filter aids are of great val. in removing colloidal impurities from raw syrups and refinery liquors. Except in prohibitive amounts they are not likely to improve the filtration of carbonation scums or other massive ppts. Raw kieselguhr is variable in quality and some is unsuitable for the sugar industry. The best is of marine origin, from California, and this is the source of Hyflo Supercel, some applications of which are described.

J. H. L.

**"Collactivit": its [sugar-juice] potash-removing power.** H. C. P. GEERLIGS (Tijd. Tech. Ver. Beet. Raff., 1937, 33, 93; Int. Sugar J., 1938, 40, 317).—One of the advantages claimed for "Collactivit" is that it is capable of removing K from the juice, thus making possible a higher yield of sucrose, but actually the amount thus capable of being eliminated, used at the rate recommended, viz., 240 kg. per  $10^6$  kg. of beets, must in practice be inappreciable. Thus,  $10^6$  kg. of roots give 25,000 kg. of molasses containing 8% of  $\text{K}_2\text{O}$ , or 2000 kg. of  $\text{K}_2\text{O}$ ; but of this quantity the normal dose of the C can adsorb only 20 kg., or 1%, a quantity incapable of affecting the sucrose yield to any perceptible extent. J. P. O.

**Clarification of POJ, EK, and Co cane juices in India.** A. N. KAPOOR and D. G. WALAWALKAR (Int. Sugar J., 1938, 40, 283).—POJ 2878, EK 28, and Co 290 and some other of the new cane varieties grown in the Bombay-Deccan district of India give very high yields, but, owing to their low  $\text{P}_2\text{O}_5$  content, their juices are very difficult to clarify. Sulphitation results in very high Ca contents, which causes serious scaling in the evaporators and heaters. It was possible to improve the clarification by adding  $\text{P}_2\text{O}_5$  to the raw juice, by mixing the CaO thoroughly with the juice, by maintaining at  $p_{\text{H}}$  8.0 for some time, and by sulphiting at 70—75° to  $p_{\text{H}}$  6.8—7.0. Settling then became rapid, with the formation of a very compact mud, and the Ca content of the clarified juice was reduced to about 50% of what it had previously been. J. P. O.

**How have the ash content, rendement, and colour of German raw sugars changed since the introduction of improved methods of juice purification?** K. ZABLINSKY (Deut. Zuckerind., 1937, 62, 605).—Average data of départage analyses of representative German first-product raw sugars, for the 11 campaigns 1925/6 to 1935/6, show a small rise in polarisation (97.13 to 97.84) and marked improvement in contents of ash (1.01 to 0.71%) and

org. non-sugars (1.86 to 1.45%) and in colour, all referred to dry substance. The fall in ash content, most marked in respect of  $\text{CaO}$ ,  $\text{SiO}_2$ , and  $(\text{Fe,Al})_2\text{O}_3$ , is attributed to improved control of second carbonation and to preliming. J. H. L.

**Character of German raw sugars.** W. PAAR (Deut. Zuckerind., 1937, 62, 687—689).—The data given by Zablinsky (cf. preceding abstract) are discussed. The rendements calc. from [polarisation — ( $5 \times$  ash %)] range irregularly from 0.23% below to 0.87% above those calc. from [polarisation — ( $1.857 \times$  non-sugars %)]. Results of calculating the factor in the second formula from molasses purities for the corresponding year are also given. J. H. L.

**Clarification of extracts for estimating sugars in plant materials.** A. V. V. IYENGAR (J. Indian Inst. Sci., 1938, 21A, 9—14).—Excess of basic Pb acetate (I) is best removed by K oxalate; with use of the neutral salt (II)  $\text{Na}_2\text{HPO}_4$  is more satisfactory. Whilst (I) is preferable for clarifying sandal-leaf extract, (II) is better for cotton leaf. Clarification may not remove the whole of the nitrogenous compounds, whilst with cotton leaf reducing substances other than sugar may be present. I. A. P.

**Preparing cube jaggery from low-quality sugar-cane juice.** G. G. AYYAR (Madras Agric. J., 1937, 25, 343—345; Int. Sugar J., 1938, 40, 322).—If the low-purity syrup will not crystallise in the ordinary way, it is heated at 122°, then held at 80—90°, and stirred until minute crystals appear, the masecuite being finally transferred to moulds to yield cubes. J. P. O.

**Commercial production of sugar from nipah palm.** V. M. HINCHY (Int. Sugar J., 1938, 40, 301—303).—In the Federated Malay States the nipah palm, once planted, continues to yield indefinitely, so that replanting is unnecessary. The manufacture of sugar from it can continue throughout the year, and no extraction plant is required, the juice dripping from the tapped stalks into earthenware pots. Additions of Cl ("E. C." or "Perchloron") and CaO were found effectively to preserve the juice during the 24-hr. period of collection, transport to the factory being from pots to buckets, thence to large jars, and finally to tank cars on railway lines. In the factory, clarification of the juice was carried out by heating and subsiding, the resulting clear juice being treated with  $\text{H}_3\text{PO}_4$ , reheated, and again subsided. Taylor bags were used for filtration of the muds. After evaporation under vac., the syrup was subsided and then boiled in a coil pan in the usual way, the resulting sugar being light in colour (96° polarisation) and of a small but free grain. The second masecuite cured fairly freely to produce a marketable sugar, and the final molasses was sent to the distillery for conversion into 95% EtOH. J. P. O.

**Utilisation of [cane] molasses as a road-making material.** R. C. SRIVASTAVA (Ind. Tr. J., 1938, No. 1651, 734—735; Int. Sugar J., 1938, 40, 276—277).—Cane molasses is conc. to "string" proof, treated with 1% of  $\text{H}_2\text{SO}_4$  (suitably diluted), and again conc. This mixture is incorporated with a melted mixture of asphalt and coal tar, to which 1%



of  $H_2SO_4$  has been added. It is applied while hot to the road surface previously covered with stone chips. After a time (2—3 months) the coating may begin to peel off, when it is given a seal-coating, this consisting of the above molasses-asphalt-coal tar composition with a further addition of coal tar in the proportion of 1 lb. to 1 gal. J. P. O.

**Instruments in sugar-factory control.** J. EIGENHUIS (Int. Sugar J., 1938, 40, 304—307).—In weighing the cane, instead of using beam weigh-bridges, considerably faster and equally reliable work can be done with a good clock weigh-bridge by noting the nos. and wts. of the cane trucks being slowly pulled over it. Maceration  $H_2O$ , mixed juice, and molasses are each best dealt with by means of the Boulogne scale, whilst the wt. of sugar can be found from the automatic bag-weighing machines, checked by weighing in bulk in the railway trucks. In boiler-house control, an integrating  $H_2O$  meter is used for all feed- $H_2O$  supply, its indications being checked by means of the Boulogne scale. The use of a steam flowmeter is condemned. For the control of liming, an automatic electrometric  $p_H$  equipment is desirable, whilst for the multiple-effect evaporator a continuous conductometric Brix recorder may be recommended. Electrical conductivity also forms the basis of the most suitable method of controlling pan-boiling, which in one factory in Queensland has been made automatic. J. P. O.

**Measuring the water consumption at the pans [of a raw cane-sugar factory].** C. W. WADDELL (Int. Sugar J., 1938, 40, 274—275).—A meter is described for measuring the "movement" and "clean-up"  $H_2O$  consumption at the pans. It is capable of measuring the flow of  $H_2O$  through a main serving four pans as a unit with <5% error when the rate of flow was varied from 0.5 to 20 cu. ft./min. Of the total  $H_2O$  evaporated in the boiling-house, 90% was eliminated in the multiple-effect evaporator and only 10% in the vac. pans. A correlation was found between the grain size and the  $H_2O$  consumption under local conditions where the pans had to be forced at top speed to keep up with the crushing rate. When a small average grain was boiled, less movement and clean-up  $H_2O$  were necessary at the pan station than when a large grain with a correspondingly smaller total crystal surface area was boiled. J. P. O.

**Steam-jet evacuators in the sugar industry.** M. STUNTZ (Wärme, 1938, 61, 159—162).—Progress in the use of steam-jet pumps for high vac. suggests the use of steam-jet compressors instead of mechanical pumps in the sugar industry. The possibilities are discussed with reference to improvements in the heat balance. R. B. C.

**Routine photometric control of colours and  $p_H$  values.** F. TÖDT and K. HÖPPNER (Z. Wirts. Zuckerind., 1938, 88, 385—430).—It is shown how photoelectric cells may be utilised for the construction of a continuous colorimeter. Methods of photoelectric colorimetry using Se cells have been successfully worked out for routine  $p_H$  determinations, especially as applied to the control of the first and

second carbonatation, using indicators such as phenolphthalein,  $\beta$ -naphthol-violet, and alizarin-yellow.

J. P. O.

**Relation between extinction coefficient and Stammer degrees.** A. BRIEGHEL-MÜLLER (Deut. Zuckerind., 1937, 62, 605—606).—Absorption data given by Landt and Hirschmüller (B., 1937, 964) confirm earlier results by the author (Z. Zuckerind. Czechoslov., 1931, 55, 403), who, however, differs from the authors named regarding corrections for reflexion and for absorption of sucrose and  $H_2O$ . The Stammer colorimeter is not out of date if used with monochromatic light (Hg 436) and adjustable light source (*ibid.*, 1937, 61, 197); the Stammer degree may then be taken as 10 times the extinction coeff. The application of a correction for reflexion is a matter of definition of the Stammer degree. J. H. L.

**Lubricating greases in sugar-mill practice: their properties and applications.** K. E. MASON (Int. Sugar J., 1938, 40, 307—309).—Four different types of grease are classified and described. For cane truck axle boxes a crude petroleum jelly or CaO-base grease is used and for  $H_2O$ -pump shafts and spindles a heavy, under- $H_2O$  graphite grease. Medium-consistency CaO-base greases are used also for shredders, main crushing-engine bearings, and pusher gear and soda-base greases for the ball bearings of centrifugal machines. In Oliver filters use of an extreme-pressure lubricant is advised for the gear drive. Only the ultra-powerful Pb-S type of lubricants can be used for hypoid-equipped axles. Several mills in Queensland have for some years been using "compounds" made of viscous petroleum residuals for the lubrication of rollers and gears, since these offer greater resistance than ordinary mineral oils to the washing action of  $H_2O$  and sugar juice which may splash on the mill-roller journals. J. P. O.

**Gum from *Euphorbia*.**—See V. **Determining MgO in sugar-defecation slime.**—See VII. **Activated C from bagasse.**—See XI. **Absorption of nutrients by cane.** **Control of N fertilisation [of sugar cane].** **Utilising cane molasses.** **Control of sugar-cane froghopper.**—See XVI. **Yeast growth of beet molasses.** **Determining CHAcMe-OH in liquors.**—See XVIII. **Analysis of caramel colour.**—See XIX.

#### PATENTS.

**[Cane] sugar juice extraction and clarification.** E. M. COPP, Assr. to PETREE & DORR ENGINEERS, Inc. (U.S.P. 2,054,556, 15.9.36. Appl., 25.10.33).—In a defecation process of cane-juice purification the sludge obtained from the limed juice by subsidence is filtered off and the filtrate and washings are used for macerating the cane in the mills. J. H. L.

**Extraction and purification of sugar juices.** A. T. SCOTT, Assr. to SHARPLES SPECIALTY CO. (U.S.P. 2,059,971, 3.11.36. Appl., 2.11.33).—Raw juice extracted from the cane or other material at <50° is made distinctly alkaline with CaO at <50° and pptd. impurities are removed centrifugally. It is then made slightly acid, *e.g.*, with  $SO_2$ , heated to 80°, and tempered with CaO to ppt. reaction products. J. H. L.

**Purification of sugar juices.** G. E. G. VON STIETZ, Assr. to SHELL DEVELOPMENT Co. (U.S.P. 2,067,362, 12.1.37. Appl., 16.5.34).—In a liming and carbonatation process carried out below the usual temp., the consumption of CaO and the Ca content of the purified juice are reduced by use of  $\text{NH}_3$  or  $\text{NH}_4$  salts and appropriate  $p_{\text{H}}$  vals. In a typical application, raw beet juice at 55–65° is treated with 0.1% of  $(\text{NH}_4)_2\text{SO}_3$  and 0.4% of CaO, settled, and filtered, treated with 0.05% of  $(\text{NH}_4)_2\text{SO}_4$  and 0.4% of CaO, carbonatated to  $p_{\text{H}}$  11, filtered, further carbonatated to  $p_{\text{H}}$  8, and then adjusted to  $p_{\text{H}}$  9 by boiling or by addition of  $\text{NH}_3$  and filtered after 10 min. J. H. L.

**Sugar-carbonatation process and apparatus.** R. M. WHITE (U.S.P. 2,064,135, 15.12.36. Appl., 6.6.33).—In a continuous process the juice, contained in a carbonatation vessel divided into two compartments communicating at the top and the bottom, flows upwards in one compartment and downwards in the other, owing to propellers in the lower communication passage. Defecating agent is introduced into the upwardly, and gas into the downwardly, flowing stream, carbonatated juice is withdrawn from the bottom, and raw or partly defecated juice is introduced just in front of the propellers so that it mixes thoroughly with the juice already present. J. H. L.

**Production of refined sugar, table syrup, or vinegar from sugar-cane juice and other saccharine matter.** L. C. A. FEURTADO (B.P. 470,889, 14.10.36).—In the clarification of the raw juice an agent, "Nicksilfor," is used, containing Baceeda glucoside powder (from the bark of the bastard cedar tree, *Guazuma tomentosa*, H.B. & K.). Kieselguhr and other known agents may also be added, and the heated juice, after passing through subsidors, is evaporated and boiled for sugar, or partly inverted by boiling with fermented juice and conc. for table syrup, or inverted with HCl and fermented for vinegar. Apparatus is claimed. J. H. L.

**Refining of sugar solutions, particularly solutions containing dextrose.** J. E. POLLAK. From INTERNAT. PATENTS DEVELOPMENT Co. (B.P. 471,845, 12.2.37).—The sweet- $\text{H}_2\text{O}$  obtained in washing out bone-char filters is treated with activated carbons and filtered before being returned to working, to remove from it impurities which the wash-waters have extracted from the bone char with the sugar. J. H. L.

**Manufacture of dextrose.** W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 2,065,724, 29.12.36. Appl., 2.2.34. Renewed 14.9.35. Cf. U.S.P. 1,976,361; B., 1935, 1065).—In the crystallisation of pure glucose in vac. pans, increased yield is attained by lowering the temp. in the later stages, by introducing fresh solution more rapidly than its  $\text{H}_2\text{O}$  is evaporated, and eventually by introducing  $\text{H}_2\text{O}$  and boiling for some time further. Supersaturation is ultimately reduced almost to zero. J. H. L.

**Production of crystalline dextrose.** INTERNAT. PATENTS DEVELOPMENT Co. (B.P. 489,937, 30.1.37. U.S., 14.12.36).—The mother-liquor ("first greens") from the first crystallisation (cf. B.P. 232,160; B.,

1925, 935), after appropriate dilution, is submitted to a mild acid reconversion whereby the concn. of glucose is substantially increased without markedly increasing the total concn. of reducing sugar. The liquor is then adjusted to  $p_{\text{H}}$  5.1, filtered, clarified, conc., and submitted to a second crystallisation (which occupies 7–10 days instead of 18 as hitherto). Crystals separated from the massecuite by centrifuging may be washed for low-grade sugar, or preferably returned via a melter to fresh converter liquor at the refining stage prior to the first crystallisation. J. H. L.

**Quick-hydrating anhydrous dextrose.** T. A. BRUCE, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 2,058,852, 27.10.36. Appl., 23.12.35).—A form of cryst. anhyd. glucose (I) which can be converted into sensibly dry (I) hydrate in 1–8 min. by admixing 8% or less of  $\text{H}_2\text{O}$  or aq. flavouring extract is prepared by so treating ordinary cryst. anhyd. (I) that the facets of the crystals are covered with submicroscopic nuclei of (I) hydrate crystals, as shown by their impaired lustre. Four methods of carrying out the process are described. J. H. L.

**Clarification of sugar [glucose] solutions.** J. E. POLLAK. From INTERNAT. PATENTS DEVELOPMENT Co. (B.P. 484,993, 9.11.36).—In the filtration of converted starch liquors through Sweetland, Vallez, or bag filters, slop (maize residues from wet starch manufacture) is used instead of kieselguhr as a filter aid. J. H. L.

**Manufacture of maple products of intense flavour.** G. S. WHITBY (U.S.P. 2,054,873, 22.9.36. Appl., 21.4.34).—Maple syrup is evaporated below its normal b.p. (e.g., in vac.) to minimise loss of flavour, and afterwards heated in a closed vessel above its normal b.p. to develop latent flavour. J. H. L.

**Manufacture of crystallised sugar.** G. LAMBINON (B.P. 479,585, 7.6.37. Belg., 8.6.36).—An emulsion of raw beet or cane juice with 0.3–3% of mineral or vegetable oil is evaporated to syrup without further purification, and boiled for cryst. sugar. The massecuite may be diluted and cooled to 20° before draining, and impurities may be pptd. from the mother-syrup by EtOH or MeOH, separated, and utilised as fodder. J. H. L.

**Manufacture of a sugar composition.** J. A. JESSOP (B.P. 470,031, 20.5.36).—Sucrose is mixed at ~21° with 5% of a 40–70% solution of lactic acid, and the mixture may be maintained at this temp. for 7 days. The plastic product is unfermentable by yeast and may be incorporated with dough in bread-making. J. H. L.

**Manufacture of high-grade saccharine products and refining raw sugars.** J. P. IOANNU, Assr. to PENNSYLVANIA SALT MANUFG. Co. (U.S.P. 2,059,110, 27.10.36. Appl., 6.3.35).—Raw saccharine liquors are purified by addition of an oxidising and decolorising agent such as a hypochlorite or peroxide, a sol. Al salt to ppt. and flocculate impurities, and an agent which stabilises the flocculum and yields an alkaline hydrolytic product. The treated liquors, having  $p_{\text{H}}$  6–9, are filtered. J. H. L.

**Utilisation [as feeding-stuff] of certain waste products of beet-sugar manufacture.** E. W. JAMES and K. HAMPEL (B.P. 470,797, 22.2.36).—Before the drying of plain or molassed pulp a small proportion of "lime sludge" (carbonation cake) is incorporated with it. If desired, phosphate cake and salt may also be incorporated. Apparatus is described. J. H. L.

**Continuous digestion of starch-containing raw materials and the like.** A. LOGINOV (B.P. 474,320, 11.11.36).—The material, mixed with H<sub>2</sub>O, is pumped through a succession of closed chambers in each of which it is intimately mixed with live steam injected under pressure. Each chamber comprises a conical hopper surrounded by a steam compartment, both delivering together under pressure on to a conical surface in a lower, mixing compartment. Several forms of apparatus are described and claimed. Digestion is stated to be much more rapid than in autoclaves, and without any caramelisation. J. H. L.

**Manufacture of [maize] starch.** R. E. GREENFIELD, Assr. to A. E. STALEY MANUF. CO. (U.S.P. 2,058,683, 27.10.36. Appl., 23.9.32. Renewed 19.9.35. Cf. U.S.P. 2,023,999; B., 1936, 1227).—In the scheme claimed, H<sub>2</sub>O is used in two main circulating systems, one for the mill house, as far as the stage at which the mill starch is filtered before tabling, and the other for the tabling and gluten separation. These are controlled independently, in respect of vol., temp., SO<sub>2</sub> content, etc., and the first is replenished from the second. J. H. L.

**Production of modified dextrins.** DISTILLERS Co., LTD., and P. D. CORPOCK (B.P. 489,379, 28.1.37).—Gum powders sol. in cold H<sub>2</sub>O without lumping are obtained by heating dextrins for a short time (3–15 min.) at a temp. (70–130°) < the charring point and preferably under slight pressure. Small amounts of preservatives, e.g., β-C<sub>10</sub>H<sub>7</sub>·OH or BzOH, bleaching agents, e.g., Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, and/or improvers, e.g., borax or Na<sub>3</sub>PO<sub>4</sub>, may also be incorporated in the product. S. C.

**Revivifying charcoal.**—See II. **Removing salts from [sugar] solutions.**—See VII. **Adhesives.**—See XV.

## XVIII.—FERMENTATION INDUSTRIES.

**Chemical engineering in the fermentation and distilling industries.** R. GILMOUR (Soc. Chem. Ind. and Inst. Chem. Eng., Advance proof, Sept., 1938, 18 pp.).—Processes involved in the production of malt and grain whisky, gin, industrial spirit, abs. EtOH, and bakers' yeast (Vienna yeast, air yeast, and yeast grown with inorg. N) are outlined and appropriate plant is described. Developments in still construction are discussed. The use of stills of modern type for the production of malt whisky and gin would appear to be undesirable, owing to the effects on the character of the products. I. A. P.

**Bacterial population of grains used in a distillery.** C. S. BORUFF, R. I. CLAASSEN, and A. L. SOTIER (Cereal Chem., 1938, 15, 451–456).—A sampling and bacteria-counting method is described 4 N (B.)

which is quick, simple, and suitable for distillery grains, flour, and similar materials, and makes possible a bacteriological standard for those materials. Comparison of results between different laboratories indicates sufficient accuracy. E. A. F.

**Production of new yeast types by crossing.** Ö. WINGE (Woch. Brau., 1938, 55, 265–267).—The spores of a single sporulated yeast cell may give rise to colonies of types differing markedly from one another and from the original. Due to this segregation of characters, a pure culture of yeast ceases to be such if spore formation occurs and the spores germinate in the medium. Further, by fusion from spores of differing yeasts, hybrid types have been obtained with transmission of both morphological peculiarities and enzymic characters. I. A. P.

**[Yeast] growth-factor content of beet molasses.** R. ILLIES (Z. Spiritusind., 1938, 61, 259–260, 267–268; cf. B., 1938, 95).—Using a described method of assessment, based on that of Nielsen (A., 1936, 381), it appears that the contents in growth factor B<sub>1</sub> of the various samples of molasses tested showed very small differences, but the behaviour of a sample somewhat poorer than the others in B<sub>1</sub> was reproducible. I. A. P.

**Control of amount of extract from malt by determination of barley extract.** T. CHRZĄSZCZ and S. GŁOWACKI (Woch. Brau., 1938, 55, 281–285).—The "extract" of barley is affected by the fineness of grind achieved, which itself may be influenced by the H<sub>2</sub>O content; the result also varies with different methods of determination. Types of available method are reviewed, and 7 methods have been applied to 6 different barleys, each being examined with grinds giving 70, 80, and 90% of meal, the results being critically discussed. The Lüers-Miller method gives results for barley extract closely agreeing with those for the extract of the corresponding malt. The Rothenbach-Schultze barley method is least influenced by the grind and might serve for assessing the max. yield of extract possible from a malt. Increasing barley protein causes increasing deviations between barley extract and that of the malt. I. A. P.

**Proteolytic activity of malt.** S. LAUFER (J. Assoc. Off. Agric. Chem., 1938, 21, 160–164; cf. B., 1937, 966).—Further experiences with the method of determination described previously (cf. *loc. cit.*), depending on the change in  $\eta$  of gelatin solutions, are reported. The results are consistent (deviation > 4%) for any one malt infusion. The temp. of incubation is lowered from 40 to 34°. E. C. S.

**Limit attenuation.** F. HELLE (Woch. Brau., 1938, 55, 257–259).—Working with sterilised apparatus with avoidance of subsequent infection, and the use of properly designed stirring apparatus, are of the first importance. Fermentation of wort from the pitching or fermenting vessel at 28–30° for 24 hr. is preferred. Use of high concn. of added yeast (e.g., 8%), though shortening the fermentation time, leads to less accurate results. I. A. P.

**Rapid determination of limit attenuation in wort and beer.** E. SCHILD (Woch. Brau., 1938, 55, 273–275).—Various conditions for carrying out

this determination are critically discussed, with illustrative experiments. Appropriate apparatus is described. I. A. P.

**Brewing process investigations by means of ultracentrifugal sedimentation, diffusion, and electrophoresis.** H. LUNDIN (Woch. Brau., 1938, 55, 241—245, 246, 249—252, 259—261).—Apparatus and procedure are described and an extensive literature is critically reviewed. Results of application of these methods to variously treated barley extracts, malt, wort, and beer are outlined. I. A. P.

**Why pasteurise [beer]? G. OSGOOD** (J. Inc. Brewers' Guild, 1935, 21, 300—311).—Use of Seitz filters for beer and the technique of filling under sterile conditions are discussed. CH. ABS. (p)

**Analytical methods applicable to beer.** E. SINGRUEN (J. Assoc. Off. Agric. Chem., 1938, 21, 318—325).—The determination of  $r_H$ , air, colour, and turbidity, and of acidity by electrometric titration, are reviewed. E. C. S.

**Beer foam determination.** O. C. ORNEAS (Brewers' Digest, 1938, 21, No. 6; Woch. Brau., 1938, 55, 285).—Foam formation is determined by pouring under standard conditions, the foam produced being expressed as vol.-% of beer used. Foam formation increases with  $CO_2$  content for a given beer, but not necessarily for different beers. Foam stability is likewise determined by pouring and the foam measured volumetrically, the method otherwise resembling that of Blom (B., 1937, 829). For beers deficient in  $CO_2$ , streaming  $CO_2$  is recommended instead of pouring. Pasteurisation caused a slight decrease in foam stability and quality. I. A. P.

**Gravimetric estimation of  $\alpha$ -hop resin.** G. HAGUES and A. W. D. HARTLEY (J. Inst. Brew., 1938, 44, 375—383).—Quant. pptn. of the Pb salt of  $\alpha$ -resin is not always attained by the usual standard methods. A type of pptn. curve is described which, it is claimed, must be obtained in cases of quant. pptn. Relatively large errors are obtained with old English hops, but an increase in accuracy is given by low-temp.  $Et_2O$  extraction with subsequent dissolution of the resins at  $0^\circ$  followed by filtration. I. A. P.

**Physical chemistry of alcoholic fermentation.** O. G. DE LIMA (Rev. Chim. Ind., 1938, 7, 188—193).—A survey of the literature. F. R. G.

**[Determination of] alcohol by use of the ebullioscope.** P. VALAER (J. Assoc. Off. Agric. Chem., 1938, 21, 175—177).—In view of the comparatively inaccurate results obtained by Joslyn *et al.* (cf. B., 1937, 486) and the lack of uniformity in the pattern of apparatus available, the method is considered not suitable for further investigation. E. C. S.

**Report of Sub-Committee of Standing Committee for Analysis of the Institute of Brewing formed to investigate methods for estimation of arsenic in brewing materials.** A. E. CASE, H. HERON, and A. J. C. COSBIE (J. Inst. Brew., 1938, 44, 359—361).—After a consideration of the available methods, modifications of the Marsh-Berzelius and Gutzeit tests were selected for further examination,

the advantages, disadvantages, and limits of accuracy of each being considered. The Gutzeit test with  $HgBr_2$  as sensitising agent was considered to provide the most satisfactory results, stable factitious standards for stain comparison being produced by means of  $H_2O$ -colours. I. A. P.

**Estimation of arsenic in brewing materials.** A. E. CASE (J. Inst. Brew., 1938, 44, 362—374; cf. preceding abstract).—Apparatus, technique, and the necessary reagents for application of the recommended Gutzeit procedure to coal, sugar, malt, hops, beer, and finings are described, the importance of details of procedure being emphasised. I. A. P.

**Direct determination of iron in malt beverages.** P. P. GRAY and I. M. STONE (Ind. Eng. Chem. [Anal.], 1938, 10, 415—417).— $Fe^{III}$  in beer is reduced to  $Fe^{II}$  by adding  $NaHSO_3$ . The sample is then treated with 2:2'-dipyridyl in dil. AcOH, and the orange colour developed is compared with a series of standards. The presence of 5 p.p.m. of Al, Cr, Co, Cu, Pb, Mn, Ni, Sn, or Zn does not interfere. E. S. H.

**Manufacture of alcohol from potatoes. V. Pretreatment of potatoes on a semi-industrial scale.** K. SATOH, K. HAYASHI, and A. MATSUMI. VI. Pressed juice. T. YOSIMACHI and Y. OTAKU. VII. Acclimatisation of yeast in acid-sugar mash. T. YOSIMACHI (J. Agric. Chem. Soc. Japan, 1938, 14, 854—858, 859—867, 868—872; cf. B., 1938, 715).—VI. In view of the yield from raw or boiled potatoes and the content of utilisable substances, pressed juice can be used as a supplementary source of fermentative substances and as an adjuster for the  $p_H$  of the mash.

VII. The acid-sugar mash is not easily fermented, but by growing 25 generations of *Sacch. thermantitonus* on the mash containing rice bran extract, the final strain reduced the time of fermentation to 46 hr. J. N. A.

**Importance of azeotropic mixtures in alcohol dehydration.** S. RIEBER (Rev. Chim. Ind., 1938, 7, 227—229; cf. B., 1938, 349).—Polemical. The process of the Soc. Anon. des Distilleries des Deux-Sèvres is more economical for the conversion of sucrose into anhyd. EtOH than the Drawinol method, the steam consumed being 280 kg. compared with 574 kg. per 100 l. of EtOH. F. R. G.

**Determination of acetylmethylcarbinol in fermentation liquors.** G. L. STAHY and C. H. WERKMAN (Iowa State Coll. J. Sci., 1936, 10, 205—211).—Commerical  $CHAcMe \cdot OH$  (I) may be purified by washing with anhyd.  $Et_2O$  and filtering. The Lemoigne-van Niel method of determination gives consistent results equiv. to 84% of the (I) present. Oxidation of 1 mol. of (I) with  $K_2Cr_2O_7-H_3PO_4$  yields 2 mols. of AcOH. Oxidation by  $IO_3^-$  produces 1 mol. of AcOH from 1 mol. of (I). (I) reduces Fehling solution and must be allowed for in sugar determinations. CH. ABS. (p)

**Determination of acetaldehyde in wines.** M. A. JOSLYN and C. L. COMAR (Ind. Eng. Chem. [Anal.], 1938, 10, 364—366).—The methods of Ripper (B., 1900, 280) and of Jaulmes and Espezel (B., 1935, 920) and the  $NH_2OH$  procedure give comparable

results for pure MeCHO solutions. In presence of 12% of EtOH the first method gives the lowest results although in no case is recovery complete, whilst the second method alone is accurate in presence of sulphites. F. N. W.

**Determination of hydroxymethylfurfuraldehyde in port wine.** Its rôle in adulteration of this wine. J. C. BOTELHO (Ann. Chim. Analyt., 1938, [iii], 20, 203—205).—Hydroxymethylfurfuraldehyde is absent from genuine port wines and its presence is an indication of adulteration with caramel or boiled grape juice. E. C. S.

**[Determination of] volatile acids in wine.** M. A. JOSLYN (J. Assoc. Off. Agric. Chem., 1938, 24, 166—174).—Collaborative analyses, using several new modifications of the Hortvet apparatus, show that the error due to CO<sub>2</sub> may be obviated by titrating the distillate while hot. Possible reasons for the lack of concordance in the results obtained are discussed. E. C. S.

**Determination of arsenic in wine.** C. BERTIN (Ann. Falsif., 1938, 31, 215—218).—Martin and Pien's method is considered trustworthy, especially for the determination of < 5 p.p.m. of As. E. C. S.

**Determination of reduced dry extract in liqueur wines.** H. LAGANNE (Ann. Falsif., 1938, 31, 221—224).—The expression  $e = [2.062(q - s) + 1]/2.663$ , where  $e$  = reduced extract,  $q = 2.663(d' - 1)$ ,  $d'$  = the  $d$  of the residue after removal of EtOH, and  $s$  = % of sugar, has given entirely satisfactory results with natural sweet wines and liqueur wines. Expressions suggested by previous workers are criticised. E. C. S.

**"Manuring" of green malt.**—See XVI. **Spirits from potatoes.** Analysis of caramel colour.—See XIX.

See also A., III, 757, *l*-Alanine dehydrogenase. 761, Prep. of pure dihydrocozymase. 762, Influence of O<sub>2</sub> on alcoholic fermentation.

#### PATENTS.

**Production of yeast, especially compressed yeast, and spirit.** A./S. DANSK GÆRINGS-IND. (B.P. 481,045, 22.2.37. Denm., 26.2.36).—Fermentations of different types are carried out simultaneously as to part of their durations, and portions of the yeast of each are separated and mutually interchanged once or at desired intervals. At least one fermentation of the series produces EtOH, at least one is aerated, wort is added during at least one, and at least two are of different durations. I. A. P.

**Preparation of hop extract.** L. A. (FRHR.) VON HORST (B.P. 490,129, 3.2.38. Ger., 4.2.37).—After preliminary drying of green hops, preferably in a vac., the grinding and solvent extraction (Et<sub>2</sub>O and/or EtOH etc.) of the hops, the evaporation of solvent from the extract, the dilution (e.g., with hop tannin extract), and the packing, storing, and transport of the extract are all carried out in an atm. of CO<sub>2</sub>. I. A. P.

**[Alcoholic] fermentation process.** DEUTS. REICH REICHSMONOPOLVERWALTUNG F. BRANNTWEIN,

and W. KARSCH (B.P. 480,570, 7.6.37).—After intimate admixture with yeast in a preliminary vessel (A) the continuously supplied fermentable liquid (e.g., molasses solution) is withdrawn from below through an ascending pipe in A to a fermenting vessel fitted with helical guide plates so arranged, e.g., that the liquid flows downward from above; the rate of flow is adjusted so that fermentation is complete when the liquid leaves this vessel. The fermented wash passes to a separator, the liquid passing to the still and the yeast (after any desired treatment for removal of foreign organisms) being pumped back to A, thus achieving a continuous process. I. A. P.

**Production of brewer's pitch.** M. BARTHEL & Co., CHEM. FABR. G.M.B.H. (B.P. 488,685, 23.9.37. Ger., 28.11.36).—Resin, e.g., fir-root or scraped pine resin, is distilled under high vac., when high-m.p., deleterious oxy-acids remain behind. The distillate, either alone or mixed with a similar distillate prepared from old brewer's pitch, is used for the production of a brewer's pitch of much greater permanence than that from resin not so treated. D. M. M.

**[Butyl alcohol-acetone-ethyl alcohol] fermentation processes.** (A) W. A. BURTON and (B) C. F. ARZBERGER. In part, from COMMERCIAL SOLVENTS CORP., to (A) (B.P. 480,770, 23.5.36 and 5.3.37).—*Clostridium saccharo-acetobutylicum liquefaciens* (4 forms; characters and isolation described) liquefies gelatin and produces average yields of solvents of 29—31% on carbohydrate mash containing 6.5—7.5% of sugar in presence of basic materials and nitrogenous nutrients, PO<sub>4</sub><sup>'''</sup> being added if not already present in adequate concn. Maintenance of suitable  $p_{\text{H}}$  is necessary during fermentation, it being arranged that the final  $p_{\text{H}}$  shall be 5.2—6.4, e.g., by addition of sufficient CaCO<sub>3</sub> at the commencement or by periodic addition of NH<sub>3</sub> or aq. NH<sub>3</sub>. Nitrogenous nutrient may take the form of degraded proteins mixed, e.g., with NH<sub>4</sub> salts. I. A. P.

**Devices for rectifying the fermentation vapours in manufacture of alcohol.** V. SEDLÁK (B.P. 488,141, 31.8.37).—An arrangement of pipe coils for cooling-H<sub>2</sub>O and for preheating the ingoing mash, and also a diffuser through which the heating steam is injected, are claimed. B. M. V.

**Microbiological preparation of 2 : 3- $[\beta\gamma]$ -butylene glycol.** M. A. SCHEFFER, Assr. to N. V. NEDERL. GIST- EN SPIRITUSFABR. (U.S.P. 2,064,359, 15.12.36. Appl., 6.11.33. Holl., 6.12.32).—The accelerating effect of aeration in such fermentation is due simply to lowering of the [CO<sub>2</sub>]. Accordingly, aeration may be dispensed with, a similar effect being achieved by passing gases from other fermentations (e.g., washed gas from BuOH fermentation, containing approx. H<sub>2</sub> 97 and CO<sub>2</sub> 3%), which are substantially free from CO<sub>2</sub>, through the mash. Alternatively, the mash is maintained under vac., this allowing collection of the fermentation gases (CO<sub>2</sub>, H<sub>2</sub>) without admixture with extraneous gases. I. A. P.

**Fatty acids.**—See III. **Vinegar from sugarcane juice etc.**—See XVII. **Treating waste refuse etc.**—See XXIII.

## XIX.—FOODS.

**Grain conditioning.** O. HÜBSCH (Mühle, 1938, 75, 611—614).—A no. of tests to determine whether baking quality could be improved by conditioning gave negative results. E. A. F.

**Technique of experimental [wheat] conditioning.** G. MUELLER (Mühle, 1938, 75, 611—612).—A table of extensograms gives the conditioning temp. needed for any wheat, other factors being const. To eliminate errors due to different responses of different wheats to temp., the temp. obtained from the table is corr. according to the result of a conditiometer test. E. A. F.

**Practical experience in the conditioning of wheat and rye.** F. PROHAZKA (Mühle, 1938, 75, 599—602).—Conditioning of both wheat and rye improves the milling and baking quality. For the latter purpose exact control is necessary. The conditions required are determined on a laboratory conditioner. Temp. must be controlled, preferably by electric thermometers, and the H<sub>2</sub>O content determined after wetting as well as before. Curves showing conditioning characteristics of three wheats are given. E. A. F.

**Are baking and milling quality [of wheat] influenced by conditioning?** BOULANGER (Mühle, 1938, 75, 603—606).—Conditioning experiments on low-quality Karachi and Spanish wheat are described. Great improvements were obtained. E. A. F.

**Outer and inner gluten.** HOPF (Mühle, 1938, 75, 662).—The different behaviour of the outer and inner gluten of wheat in conditioning may be due to the different amounts of heat they receive. Heat conduction takes place through the H<sub>2</sub>O, not through the grain substance itself. E. A. F.

**Distribution of gluten content, gluten quality, and diastatic activity among wheat grains of different sizes.** E. BERLINER (Mühlenlab., 1938, 8, 105—116).—The gluten content and quality and diastatic activity of wheat are affected both by the size of the grain and by its soundness. A large no. of determinations show that errors in testing wheat varieties due to variation in these factors between samples can be eliminated only if the sample consists of <40 g. of grain. In sound wheat large grains contain the most gluten, but of poorest quality; where wheat-bug attack has occurred the smallest grains have the poorest gluten. E. A. F.

**Preparation of flour containing 80% of the grain from Argentine wheat intended for export.** H. D'ANDRÉ (Rev. Soc. Brasil. Quím., 1938, 7, 82—84).—Flour milled to retain 80 kg. instead of the usual 70 kg. out of 100 kg. of Argentine wheat contains only 1.30 kg. of bran, but also 8% of husk particles rich in N, which, owing to their absorptive power, give a higher yield of bread which is more satisfying. F. R. G.

**[Wheat testing by] the Berliner method.** K. KŘTINSKÝ (Mühlenlab., 1938, 8, 115—120).—The relation of the Berliner swelling figure to other wheat properties is investigated. This figure is a good measure of changes brought about by ageing, conditioning, or BrO<sub>3</sub>' treatment, and of gluten quality.

It shows no definite relation to gluten content or to baking quality. E. A. F.

**Water absorption of wheat flour.** J. KULZYCKI (Mem. Inst. Nat. Polon. Econ. Rur. Pulawy, 1934, 15, 50—57).—A direct relation is established between the % increase in wt. of bread and the quantity of H<sub>2</sub>O added. H<sub>2</sub>O absorbed by flour is assumed to be that absorbed by gluten. It is proposed to add sufficient H<sub>2</sub>O to satisfy the absorption capacity (determined experimentally) of the gluten., CH. ABS. (p)

**Viscosity and its relation to flour evaluation.** M. C. MARKLEY (N.-W. Miller, 1938, 194, No. 7, 47—51).—Measurement of the  $\eta$  of a wheat-flour suspension has no advantages as a test for strong flours, but is a good guide to the suitability of weak flours for various purposes.  $\eta$  depends on the wheat type and on the milling. E. A. F.

**Relationship between diastatic activity (maltose figure) and "gassing power" of experimentally milled flours from some Australian wheats.** R. A. BOTTOMLEY (Cereal Chem., 1938, 15, 509—520).—Poor correlation was obtained between the Blish-Sandstedt and Kent-Jones methods of measuring diastatic activity. The latter is the more accurate, quicker, and the more economical. Gassing power, as measured by the Brabender fermentograph, is not highly correlated with diastatic activity, being influenced also by other factors. It appears to be a varietal characteristic. Flours milled on the Brabender experimental mill show higher diastatic activity and gassing power than do commercial flours. E. A. F.

**Standardisation of Irish soda-bread flour.** C. W. BRABENDER, G. MUELLER, and F. HEIDE (Milling, 1938, 90, 696—700).—The problems of Irish soda bread resemble those of rye bread; a high amylogram is the essential criterion of quality. A low amylogram indicates both poor quality and too great sensitivity to oven temp. The farinogram and extensogram are useful supplementary tests. E. A. F.

**Storage of wheaten flour. III. Changes in the flora and fats and influence of these changes on gluten character.** E. C. BARTON-WRIGHT (Cereal Chem., 1938, 15, 521—541; cf. B., 1937, 608, 832).—Bacteria in flour decrease on storage, this being attributed to the low  $p_H$ . Fungi increase and cause mustiness, their increase also being associated with a fall in oil content. Gluten deterioration, especially in low-grade flours, is due to unsaturated free fatty acids in the oil, those of low mol. wt. producing the most damage. The acid val. does not increase continuously except in sterilised flour, but rises to a max. E. A. F.

**Results with a new recording mixer for use with small samples [of wheat].** J. G. MALLOCH (Cereal Chem., 1938, 15, 423—438).—A recording mixer is described which operates with flour samples containing 7 g. of dry matter. Preliminary study of the curves obtained shows that they differ widely in shape and are a good guide to flour quality. There is a break in the curve, the position and sharpness of which are characteristic of the flour and related to its

colloidal properties. Ground wheat gives curves closely related to those of flour. E. A. F.

**Effects in dough of glutathione and papain.** W. P. FORD and A. M. MAIDEN (J.S.C.I., 1938, 57, 278—281).—Farinographic investigations show that glutathione (I) has mainly a rapid dough-softening action, papain a slow one, indicating that (I) probably affects the dough-protein directly and not by activating the flour proteases, as Jørgensen has suggested.

**Plastic properties of doughs.** A. OUGRIMOFF (Bull. École Fr. Meun., 1938, 67—78, 136—141).—Effects of the condition of the wheat and of milling methods on extensimeter results are discussed. Moistening the wheat to 17% of  $H_2O$  increases  $W$ ,  $P$ , and  $G$  and decreases the ash content of the flour. The keeping time and temp. are practically without effect. The method of milling has little influence on the extensimeter results if corrugated rolls are used, but more with smooth rolls. E. A. F.

**Colloidal behaviour of flour doughs. III. Properties of flour-starch-water systems.** M. C. MARKLEY (Cereal Chem., 1938, 15, 438—444; cf. B., 1938, 968).—The properties of mixtures of flour, starch, and  $H_2O$  in varying proportions were studied in the farinograph. There is a sharp change in properties at about 7% gluten content, ascribed to the formation of a protein envelope around the starch granules.  $H_2O$  absorption at 7% gluten content is a measure of the  $H_2O$ -holding power of the gluten independent of its concn. E. A. F.

**Pore formation in dough.** H. KÜHL (Mehl u. Brot, 1938, 38, No. 25, 1—3).—This depends on gas production and gas retention. The former is easily regulated; the latter is improved by conditioning and by adding substances (notably milk products) which modify the colloidal condition of the dough. E. A. F.

**Acidity of bread-dough.** E. GIACANELLI (Annali Chim. Appl., 1938, 28, 275—280).—Addition of small amounts (0.1—0.5%) of lactic acid to the dough improves the quality of the bread, the result being analogous to that obtained by maturation of the flour. F. O. H.

**Photomicrographic studies of dough and bread structure.** S. W. BUTTERWORTH and W. J. COLBECK (Cereal Chem., 1938, 15, 475—488).—Methods of investigating microscopically the structure of dough and bread are described. The min. of prep. should be used, to avoid distortion. There is no apparent difference between doughs at varying stages of fermentation, but the starch grains are squeezed out of the gluten structure on to the surface as fermentation proceeds. E. A. F.

**Various characteristics of mill-stream flours and their relation to loaf volume.** R. H. HARRIS and S. N. WHITE (Cereal Chem., 1938, 15, 489—500).—27 mill-stream flours were subjected to standard basic and malt-phosphate-bromate baking tests, and their  $H_2O$ , ash, gluten, protein, and diastatic activities were determined. The results are examined statistically. The malt- $PO_4'''$ - $BrO_3'$  baking test gives better differentiation for high-gluten flours. Gluten pptd. by  $MgSO_4$  from Na salicylate dispersion

is a fairly const. fraction of the total gluten, and is positively related to loaf vol. High flour quality is associated with low diastatic activity. Flour protein and wet crude gluten are equally closely related to loaf vol. E. A. F.

**Dispersing agents as stabilising media in baking powders.** S. MENDELSON (Food Manuf., 1938, 13, 298—300).—The reagents used as diluents, absorbers of  $H_2O$ , and as having a partitioning effect to prevent intimate contact between the acid and alkaline constituents are usually starches, maize being used up to 50 and wheat and rice up to 25%. Starches are redried before mixing with the inorg. constituents. To avoid uptake of excess of  $H_2O$ ,  $CaH_4(PO_4)_2 \cdot H_2O$  is the best phosphate to use when needed. Addition of steatites or ground rock as a filler is strongly deprecated. W. L. D.

**Practical and simple method of recording the form and porosity of baked products.** C. MATĚJOVSKÝ (Cereal Chem., 1938, 15, 471—474).—A contact print is made of a thin slice of the loaf, kept flat by moistening with glycerin. E. A. F.

**Determination of added potassium bromate in bread manufacture.** J. VIGGIANO and P. CATTANEO (Anal. Assoc. Quím. Argentina, 1938, 26, 1—12; cf. B., 1937, 721).—Br in flour and bread is determined after calcination in presence of  $Na_2CO_3$  by the method of Balatre (A., 1936, 1478). Bread prepared from flour containing 4.7 mg. of Br per kg. and salt containing 3.80 mg. of Br per kg. should contain 3.55 mg. of Br per kg. Higher Br content is held to indicate addition of  $KBrO_3$ . F. R. G.

**Detection and determination of diacetyl and acetylmethylcarbinol in bakery products.** H. SCHMALFUSS and H. WERNER (Z. Unters. Lebensm., 1938, 76, 113—118).—The authors' method as applied to oils etc. (B., 1938, 314) is modified for use with bakery products. The basis of the method is the determination of the least quantity of the material in question the distillate from which will give a definite red coloration with the  $NH_2OH$ - $NiSO_4$  reagent.  $Ac_2$  was not found in any sample of dough or bread examined. A yeast dough contained 5.0, a sour dough 0.67, and bread 0.4 mg.-% of  $CHAcMe \cdot OH$ . E. C. S.

**Application of the neutral wedge photometer to measurement of carotenoid pigments in flour and macaroni products.** V. E. MUNSEY (J. Assoc. Off. Agric. Chem., 1938, 21, 331—351).—From its behaviour in the light petroleum-MeOH separation, in chromatographic analysis, and from its absorption spectrum it is concluded that almost all the carotenoid pigment in flour is xanthophyll. Procedures are described for the standardisation of the neutral wedge photometer for determination of carotene (I) concn., the prep. of the sample, and the extraction of pigment and its photometric determination. Vals. for total colour [as (I)] and (I) in flour, farina, semolina, dried and frozen egg-yolk, and macaroni products are tabulated. E. C. S.

**Study of certain physical properties of bread through the staling process with the use of the electronic cell.** C. A. GLABAU and P. F. GOLDMAN

(Cereal Chem., 1938, 15, 541—551).—The opalescence of starch gels was measured in a photometer using a photo-electric cell. The light transmitted decreased over a period of 10 days. Bread showed similar results, the decrease in light transmission being slower at low temp. and running parallel to other changes associated with staling. E. A. F.

**Storage of rice. XVIII. Relation between varying moisture content and change in quality of milled rice, with special reference to the underdried product.** M. KONDŌ and T. OKAMURA. **XIX. Removal of moisture from hulled rice by heated air.** M. KONDŌ, R. TAKAHASHI, Y. TERASAKA, and S. ISSHIKI. **XX. Unhulled rice stored about one hundred years in a granary.** M. KONDŌ and T. OKAMURA (Ber. Ōhara Inst. landw. Forsch., 1938, 8, 11—34, 35—46, 47—52; cf. B., 1938, 97).—XVIII. Using germinating power as an index of storage conditions, the permissible max. temp. of storage of hulled rice containing 14, 16, 17, and 18.6% of H<sub>2</sub>O were estimated to be, respectively: for  $\frac{1}{2}$  year, room temp. (rising to 33°), 25°, 25°, and 24°; for 1 year, 25°, 20°, 15—20°, and 15—20°; for 2 years, 20°, 17°, 10°, and  $\geq 10^\circ$ ; for  $> 2$  years, 10—15°, 14°,  $> 10^\circ$ , and 5°. Physical, chemical, biological, and culinary properties of the stored rice were studied.

**XIX. Conditions of temp., air flow, and agitation for the successful drying of rice in a commercial drying machine and in a drying chamber are specified. The former is less efficient but more practicable than the latter.**

**XX. Unhulled rice kept for 100 years was much spoiled by *Rhizopertha dominica*; hulled rice was darker in colour and had a peculiar odour, but was edible although unpleasant. Fat, glucose, and dextrin had decreased and, with the exception of lipase activity, the enzyme activity was greatly reduced. Only 8.2% of vitamin-B<sub>1</sub> was retained. Germinating power was entirely lost.** E. C. S.

**Investigations on rice.** A. SREENIVASAN (Current Sci., 1938, 6, 615—616).—A preliminary note on recent work concerning the nature of parboiling and the changes that occur during storage. L. S. T.

**Influence of sea-water on iodine contents of rice, rice-bran, and wheat.** A. ITANO, Y. TSUJI, T. HASEGAWA, and I. MORIYA (Ber. Ōhara Inst. landw. Forsch., 1938, 8, 103—106).—Rice grown on land alongside a sea-H<sub>2</sub>O canal had  $> 3$  times the I content of that grown at a distance,  $> 65\%$  of the I being in the bran. Wheat contained less I than the rice, and the differences were less marked. E. C. S.

**Problems requiring solution in the dairy industry.** G. GÉNIN (Lait, 1938, 18, 610—614).—A review. W. L. D.

**Importance of mastitis to the dairy farmer.** J. G. DAVIS (J. Min. Agric., 1938, 45, 539—546).—A review. W. L. D.

**Density relationships in dairy products.** J. PIEN and G. MAURICE (Lait, 1938, 18, 582—610).—The  $d^{15}$  of milk fat was 0.915 with a coeff. of expansion (16—100°) of  $7.87 \times 10^{-4}$ . Three methods for determining the  $d$  of the non-fatty solids (I) gave vals.

varying from 1.596 to 1.610 as against previously found vals. of 1.600—1.606. The wt. of 1 l. of any milk fraction is = the wt. of fat per l. (B) + the wt. of (I) per l. (e) +  $[1 - (B/0.915) - (e/1.6)]$ . Compared with drying at 100°, this formula gives vals. too high by about 0.2% of the dry matter, but close to those obtained by drying in vac. over H<sub>2</sub>SO<sub>4</sub> at 50°. It is claimed that losses of dry matter at 100° are of this amount. The composition and  $d$  of the (I) are not const., and for (I) of 9.0—9.6%  $d$  ranges from 1.0338 to 1.0360. The relation between  $d$  and ratio of concn. is hyperbolic and solids content can be calc. from  $d$ . The relationship between  $d$  and fat content of cream per kg. is a shallow hyperbolic curve, but linear when calc. on a vol. basis. Tables are given for calculating the fat content of cream from its  $d$ , on both a wt. and a vol. basis. W. L. D.

**Chemistry of the reductase test [for milk].** T. BAUMGÄRTEL (Milch. Zentr., 1938, 67, 233—235).—The mechanism of reduction of methylene-blue by actively-growing organisms in milk is similar to rapid reduction in presence of CH<sub>2</sub>O. The reducing bacterial enzymes utilise the dye as a H-acceptor under all conditions of O<sub>2</sub> tension, but the dye is not bleached until a condition of low O<sub>2</sub> tension is reached, this being attained the more rapidly with high bacterial activity. W. L. D.

**Methylene-blue reduction test [for milk]. II. Hints on technique.** J. G. DAVIS (Dairy Ind., 1938, 3, 257—258; cf. B., 1938, 1092).—Uniformity of technique is important. This can be attained by standardising the milk-sampling methods, the temp. of incubation (37—38°), the test-tubes, and the quality of the solid methylene-blue. H<sub>2</sub>O should be free from nutritive material and Cu. The dye solution should be made up fresh every 14 days and frequent tests for sterility should be made. Observation of samples after 7.5 hr. incubation is not advisable. W. L. D.

**Hygienic control of milk.** G. BARTHÉLEMI (Lait, 1938, 18, 462—473).—Precautions to be taken in the sequence of processes which milk undergoes, and laboratory control, are described. W. L. D.

**Bacteria in milk for manufacture of Gruyère and Emmenthal cheese.** W. DORNER (Lait, 1938, 18, 449—455).—Such bacteria are divided into harmful and harmless or auxiliary groups, and enter milk from air, fodder, dung, the udder, and utensils. Of the harmful organisms, *B. amylobacter* causes gaseous fermentation and bulging of cheese during storage, and *B. proteolyticum* secondary protein degradation to give offensive odours. With the exception of *B. coli aërogenes*, pathogenic organisms cause no trouble in manufacture and storage. W. L. D.

**Origin and significance in hygienic control of microflora invading milk.** G. GUITTONEAU, G. MOCQUOT, and A. EYRARD (Ann. Falsif., 1938, 31, 295—298).—Secondary contamination is regarded as due to unhygienic handling and is to a large extent preventable. E. C. S.

**High-temperature, short-time pasteurisation [of milk].** A. W. FARRALL (Dairy Ind., 1938, 3, 248—249).—The modern process consists in heating



milk at 71° for 15—17 sec. and cooling quickly to bottling temp. Temp. regulation is automatic and the designs of machines are such that milk to milk heat regeneration is effected. The system requires more accurate control than the holder process.

W. L. D.

**Protection [of milk] after pasteurisation.** L. C. BULMER (Canad. Dairy & Ice Cream J., 1938, 17, No. 8, 15—18).—Bacterial contamination of pasteurised milk and products occurs during handling in bulk and from the final container. Ice-cream mixes should be frozen immediately in the same place as where pasteurised and the mix should have a short, direct flow to the freezer. Milk bottles should have well-fitting, hooded caps.

W. L. D.

**Milk and nutrition. III. Effect of commercial pasteurisation on nutritive value of milk as determined by experiments on calves.** NAT. INST. RES. DAIRYING and ROWETT RES. INST. I. Introduction. H. D. KAY and (SIR) J. B. ORR. II. Experiment at the National Institute for Research in Dairying. S. BARTLETT, A. G. COTTON, and J. MACKINTOSH. III. Experiments at the Rowett Research Institute. J. A. CRICHTON and W. A. BIGGAR. IV. Summary of available information. S. BARTLETT. V. Concluding observations (Nat. Inst. Res. Dairying, 1938, 4—5, 6—13, 14—24, 25—26, 27; cf. A., 1937, III, 502; B., 1937, 609).—II. Calves from tuberculin-tested cows were paired and one animal of each pair was fed on commercial raw and one on commercially pasteurised milk from the same bulk. Hay and a grain mixture were fed as supplements in the later stages. With the 11 pairs which finished the experiment, no difference occurred in the wt. of food consumed, in the live wt. and appearance of the calves of both groups. Frame development was slightly superior in the raw-milk group. A tuberculin test at the end of the experiment gave positive reactions for 8 of the calves fed on raw and for 1 fed on pasteurised milk.

III. Two experiments were carried out, one on calves of both sexes and the other on bull calves only. The two groups on raw and pasteurised milk were fed as above for 180 days. In this experiment no difference was found in the amounts of food consumed, gain of live wt., body measurements, or in the analysis of the blood of animals of the 2 groups. On a restricted milk allowance in the second experiment, the calves on pasteurised milk consumed the same quantity of milk but less supplements and showed a slight decrease in growth rate which was found not to be statistically significant.

IV. Results from all work done on calves are tabulated.

W. L. D.

**Use of Leithe-Müller serum in refractometry of milk.** P. JAX (Z. Unters. Lebensm., 1938, 75, 546—565).—CuSO<sub>4</sub>-kaolin and Pb(OAc)<sub>2</sub>-kaolin sera are suitable for refractometry. The vals. obtained are constantly > those obtained by the usual Ackermann method, the depression being 0.4 refractometer unit for the CuSO<sub>4</sub>-kaolin and 1.2 units for the Pb(OAc)<sub>2</sub>-kaolin serum. The latter especially is always perfectly clear, allowing a very accurate reading to be made, and can be used without correction in the case

of sour and coagulated milks. Addition of 0—20% of H<sub>2</sub>O can be detected accurately and of 20—40% only approx.

E. C. S.

**Manufacture and properties of sweetened condensed whey.** G. A. RAMSDELL and B. H. WEBB (Canad. Dairy & Ice Cream J., 1938, 17, No. 8, 47—51, 55).—Whey is separated and pasteurised, and sucrose added in amount = the total solids of the whey. The mixture is condensed in vac. to 76% of total solids, cooled to 35°, stirred for 3 hr. to produce small lactose crystals, and packed in air-tight containers. The material kept well at room temp. for 3 months, but showed a slight darkening of colour and increase in  $\eta$  which were avoided by storing at lower temp. The product was whipped to 200% overrun in 4 min. to give a whip which was stable for 15 hr. The  $\eta$  increases with higher whey solids : sucrose ratio, and age-thickening increases as the temp. of pasteurisation exceeds 62°, and with increase in solids content.

W. L. D.

**Utilisation of whey solids.** B. L. SMITH (Dairy Ind., 1938, 3, 295, 303).—Lactose is recovered (yield 2.5%) by evaporation of whey, crystallisation, and refining. Whey is fermented with *B. bulgaricus* and a little yeast at 43° for 48 hr. with addition of small amounts of Ca(OH)<sub>2</sub> to neutralise the lactic acid (I). Protein is coagulated and filtered off, the solution conc., and the Ca lactate decomposed with its equiv. of H<sub>2</sub>SO<sub>4</sub>. The (I) solution is conc. to a syrupy liquid.

W. L. D.

**Chemical composition of [Indian] buttermilk.** S. RANGANATHAN and G. NARASIMHAMURTY (Agric. Live-Stock India, 1938, 8, 421—426).—Three types are in common use: (a) whole milk, boiled, soured, and the cream removed, (b) whole milk from which the cream has been separated, and (c) liquid from the manufacture of butter from cream. The composition can be gauged from the total solids content or from lactometer readings. Various liquid products are diluted, before consumption, with 3—5 times their vol. of H<sub>2</sub>O; the total solids content of 12 samples ranged from 1.2 to 10.9%. There is a small loss of N in fermented products.

W. L. D.

**Fruit syrups for milk shakes.** V. CHARLEY (Dairy Ind., 1938, 3, 254—256, 258).—Pure fruit-juice syrups contain 55% of sucrose and 58 vol.-% of fresh fruit juice. For good keeping quality the acidity must be equiv. to 1.1% of citric acid. To prevent curdling of the milk in hot milk shakes, 1 pt. of saturated aq. Na citrate is added to 40 pts. of syrup (by vol.). The final  $p_H$  of such milk-juice mixtures ranges from 5.8 to 6.0. Reduction of acidity to <0.6% is effected by reducing the juice content, which necessitates strengthening the flavour by adding pure fruit essences.

W. L. D.

**Modern practices in butter-making.** W. WHITE (Canad. Dairy & Ice Cream J., 1938, 17, No. 8, 41—43).—Cream acidity is kept at 0.3% and below as the butter manufactured from such cream keeps well when stored at low temp. Salting should be about 1.7%. Unsalted butter made from cream of high acidity (0.45%) keeps better than when salted.

The NaCl in butter encourages the development of a fishy flavour. W. L. D.

**Butyric acid, total, and residual values of butter fat of samples of butter submitted to the International Dairy Congress, Berlin, 1937.** J. GROSSFELD, E. SCHWEIZER, and H. DAMM (Z. Unters. Lebensm., 1938, 76, 123—127).—Of 245 samples examined, the mean  $\text{PrCO}_2\text{H}$ , total, and residual (cf. B., 1937, 586) vals. were 20.0, 34.8, and 14.8, respectively. E. C. S.

**Significance of mould and yeast counts [in butter].** H. MACY (Dairy Ind., 1938, 3, 281).—High counts are due to poor or unpasteurised cream, recontamination after processing, contaminated  $\text{H}_2\text{O}$ , churn, and handling utensils, and contamination from the wrapper or from the air of the cooling-rooms. W. L. D.

**Parchment-foil liners for butter boxes.** R. W. BROWN (Canad. Dairy & Ice Cream J., 1938, 17, No. 7, 15—18, 24).—To prevent loss of  $\text{H}_2\text{O}$  from, or adsorption of wood flavours by, the surfaces of boxed butter, the wrapping material can consist of Al foil mounted on both sides with vegetable parchment, using glue or wax as adhesive. Butter so wrapped was graded higher than that packed in parchment only. The butter should have a dry finish to prevent the wrapping layers from parting, and the box corners need extra protection. W. L. D.

**Vitamin-A study of ghee. V. Effect of heat and air on vitamin-A.** B. N. BANERJEE and N. N. DASTUR (Agric. Live-Stock India, 1937, 7, 24—34; cf. B., 1938, 840).—The vitamin-A content (determined by the  $\text{SbCl}_3$  method on the unsaponifiable fraction) of ghee (prep. described) is scarcely altered in 10 hr. at  $100^\circ$ , but, once started, destruction of -A goes rapidly to completion. At  $125^\circ$  half the -A is lost in 5 hr., and at  $175^\circ$  complete destruction occurs in 30 min. At  $100^\circ$   $\text{O}_2$  does not markedly accelerate the rate of destruction. Temp. of  $100^\circ$  for 5 hr. or  $125^\circ$  for  $\frac{1}{2}$  hr. do not induce autocatalytic destruction of -A. The process of frying in ghee completely destroys its vitamin content in 10 min. Ghee prepared from the butter of the cow is more resistant to heat than that from the buffalo. J. L. D.

**Recent developments in manufacture of margarine.** A. A. ROBINSON (Oil & Soap, 1938, 15, 203—206).—Developments are reviewed with special reference to the Vogt continuous process and a modification thereof. In the original process, an emulsion of oil, milk, and NaCl is pumped through a crystallising device consisting of two vertical,  $\text{NH}_3$ -cooled tubes in series, each of which is fitted with a concentric agitator shaft which allows an annular clearance of about 0.25 in., and is fitted with a series of knives which ride by centrifugal force against the inner wall of the tube. Owing to the agitation, the chilled material leaves the second tube as a thick, supercooled liquid which solidifies as it is passed without agitation through a horizontal hot- $\text{H}_2\text{O}$ -jacketed tube ("B unit") from which it is extruded as bar, and then cut and wrapped. It is found that a better flavour is obtained if only the oil is passed through the crystalliser and the milk and NaCl are

added in a blender located between the second tube and the "B unit." E. L.

**Ropiness in starters [lactic cultures].** W. M. BOGDANOW (Lait, 1938, 18, 576—582).—A new form of ropiness is due to *S. lactis*, var. *multigenes*, in symbiotic growth with a diplococcus of considerable proteolytic activity which digests the ropy curd completely on prolonged incubation. W. L. D.

**Mammococci in cheesemaking.** C. GORINI (Lait, 1938, 18, 710—711).—These organisms, which sometimes account for 50% of the udder bacteria proliferating in milk, may be harmful in cheesemaking. *S. liquefaciens* has proteolytic properties which persist even when the organism is dead, and may change the character of ripening Parmesan cheese. W. L. D.

**Rôle of albumin in cheesemaking.** G. T. PYNE and L. Ó'DROMA (Econ. Proc. Roy. Dublin Soc., 1938, 3, 75—82).—Addition of albumin (I) did not affect the quality of cheese obtained from pasteurised milk. Coherence of the curd was diminished by addition of heat-coagulable (I) even when the proportion of sol. (I) remained at normal levels. Rennetting time was unaffected by addition of sol. (I) or gelatin. Detrimental effects of pasteurisation on curd quality [in so far as it is related to (I)] depend on pptn. of (I) on the casein micelle rather than on changes in the sol. (I) content of the milk. A. G. P.

**Products of hydrolysis of casein.** E. W. McCHESNEY (J. Elisha Mitchell Sci. Soc., 1935, 51, 226—227).—A waxy solid was isolated from HCl-hydrolysis products of casein. It contained N entirely in  $\text{NH}_2$ -form, was strongly acid, formed Br-substitution products, and gave positive xanthoproteic and diazo tests, but negative tests for tyrosine, tryptophan, and histidine. CH. ABS. (p)

**Determination of  $p_{\text{H}}$  of lactic casein.** J. PIEN and M. WEISSMANN (Lait, 1938, 18, 455—462).—5 g. of casein are thoroughly ground with 50 ml. of  $\text{H}_2\text{O}$  for  $\approx 30$  min. The supernatant liquid is decanted for an electrometric  $p_{\text{H}}$  determination. The H, Sb, or quinhydrone electrode can be used, the last-named being preferred for rapid working. A saturated  $\text{HgCl}$  electrode is used as half-cell and is connected with an agar bridge through saturated aq. KCl to the casein extract. Vals. ranging from 4.1 to 5.0 were obtained for casein samples from industrial sources. W. L. D.

**Detection and determination of cow's-milk cheese added to Liptauer [sheep's-milk] cheese.** A. PÉTER (Z. Unters. Lebensm., 1938, 76, 118—123).—The paracasein from salted rennet cheese prepared from sheep's milk is almost completely sol. in AcOH, whereas that from cow's milk is only 50% sol. Large additions of cow's milk are readily detected, but  $>50\%$  addition escapes detection in fresh cheese owing to the protective action of the dispersed protein. After keeping for 1—2 months this action is lost and less addition of cow's milk can then be detected and determined to within  $\pm 6\%$ . The sour-milk cheeses show no sp. differences, only 30% of the paracasein of either being sol. in AcOH. E. C. S.

**Chemical changes during melting of natural cheese.** M. KVERTON (Lait, 1938, 18, 561—575).—Cheese was melted by heating at 70—80° for 5—15 min. after addition of 10% of H<sub>2</sub>O. Roughly 5% of H<sub>2</sub>O was lost. An average loss of 2.5% of the fat occurs mainly from mechanical causes, but the fat const. are unchanged when Na<sub>3</sub>PO<sub>4</sub> and Na citrate are used as emulsifiers. After melting, the H<sub>2</sub>O-sol. N compounds reach an average of 30%, 70% of which is accounted for by proteose and peptone. Some N is rendered sol. by base-exchange in the paracaseinate to give sol. alkali compounds. Ash increases with the amount of emulsifying agent added. The acids, determined by difference, amount to 1.5% with phosphate and 3.5% with citrate emulsification. The titratable acidity is decreased by 10% after emulsification. W. L. D.

**Selecting non-milk products for ice-cream mix.** W. J. CORBETT (Canad. Dairy & Ice Cream J., 1938, 17, No. 8, 24—28).—The sugar content varies from 13.0 to 16.5%. A 12% fat mix should have 14% and a 15% mix 15% of sugar. Sucrose (I) and anhyd. glucose (II) have the same sweetening effects for equal wts. in the mix. A (I) : (II) ratio of 3 : 1 gives the best results. The gelatin can be reduced by 0.1% when using (II) to avoid a soggy body. Honey of 80% total solids is best used when replacing 75% of the (I). 0.2—0.5% of gelatin, according to its strength, is used and a gelatin test is advisable. A mixture of 0.2% of pectin and 0.3% of gelatin is satisfactory and 0.3% of Na alginate is equal in stabilising power to 0.35% of gelatin. Whipping properties are improved by adding 0.25—0.40% of dried or 0.5—0.7% of frozen egg yolk. W. L. D.

**Frozen pack fruits for ice cream.** M. A. JOSLYN and W. C. COLE (Canad. Dairy & Ice Cream J., 1938, 17, No. 7, 49—53).—Such fruit should be of such a nature as to flavour and colour the ice cream, not too high in tannin or acidity, and free from oxidising enzymes. Fruits are stored in a frozen condition with sucrose, since glucose from maize starch discolours the fruit. Flavour is preserved best at < -18°. The fruit:sugar ratio is 2 : 1. The mixes should contain 15% of most fruit packs but only 12% of raspberries. The sugar content of the final mix is 14%. W. L. D.

**Compounding ice-cream mixes.** L. J. HYNES (Dairy Ind., 1938, 3, 319—320).—Methods of calculating the amounts of sugar, gelatin, cream, and whole milk, to make a mixture containing fat 10, milk solids-not-fat 10, sucrose 14, and gelatin 0.5%, are given. W. L. D.

**Manufacture of French ice cream.** L. J. HYNES (Dairy Ind., 1938, 3, 279—280).—The mix contains milk 40, cream (50% of fat) 24, eggs 20, sugar 12%, flavoured with vanilla. The mix contains 12% of fat. The eggs, cooked at 82°, are whipped with sugar and added to the cold milk mix. Freezing is done slowly and an overrun of 33% is possible. The eggs can be replaced by 4% of dried egg-yolk. W. L. D.

**Problems in ice-cream manufacture.** J. H. ERB (Dairy Ind., 1938, 3, 271—273, 280).—Uni-

formity and conservation of flavour depend on selection of the raw materials and wholesomeness of the fat. A common defect is oxidised flavour due to the fat itself, or to oxidising catalysts in the mix. The development of this flavour can be delayed by adding 0.3—0.5% of fine oat flour to the mix. Factors affecting overrun are enumerated. Lecithin added to chocolate-bar coatings lessens H<sub>2</sub>O absorption and brittleness. W. L. D.

**Decomposition in eggs.** J. CALLAWAY, jun. (J. Assoc. Off. Agric. Chem., 1938, 21, 179—182; cf. B., 1937, 973).—The proposed method of testing (cf. B., 1938, 721) gives results agreeing closely with those obtained by the official method and is recommended for tentative adoption. E. C. S.

**Rotting of hen eggs.** T. FUJIMI (Arb. med. Fak. Okayama, 1935, 4, 572—582).—Non-fertile eggs, injected with *B. coli* and *Strept. albus*, showed diminution of histidine, but not of arginine or lysine, in the coagulable protein in a 4-week period under aerobic or anaerobic conditions. CH. ABS. (p)

**[Determination of] water-soluble nitrogen and crude albumin-nitrogen in dried eggs.** F. J. McNALL (J. Assoc. Off. Agric. Chem., 1938, 21, 182—184).—Collaborative results with dried whole eggs and yolks show good agreement, those for dried whites being less good owing to the difficulty of getting the material into solution. E. C. S.

**Food poisoning. Plants, fungi, and fish.** E. B. DEWBERRY (Food Manuf., 1938, 13, 271—276).—Various poisonous plants and the entry of some of them into foods are described. Poisonous fungi are classified according to their physiological effects, the genus *Amanita* being most virulent, due to their content of phallin and muscarine. Certain fish secrete substances causing gastro-intestinal troubles, and roe-poisoning is common. Shell fish, especially mussels, are liable to be toxic, this being manifested in three forms: erythematous, paralytic, or bacterial food poisoning. Methods of purifying oysters by treatment with sterilised sea-H<sub>2</sub>O are described. W. L. D.

**Meat extracts produced by Brazilian canneries.** J. S. FERNANDES (Rev. Soc. Brasil. Quím., 1938, 7, 65—72).—H<sub>2</sub>O, total solids, colour, ash, free acids, free NH<sub>3</sub>, P<sub>2</sub>O<sub>5</sub>, Cl', and composition of the proteins are recorded for a no. of samples of meat extracts from canneries in Rio Grande do Sul and São Paulo. The vals. are within the limits of tolerance of the international standards. F. R. G.

**Evaluation of meat cubes.** K. KACL and F. FINK (Z. Unters. Lebensm., 1938, 75, 529—546).—Meat cubes are evaluated by means of their total creatinine (I) content. Errors are inadvertently introduced into the determination of this val. by the usual methods owing to the presence of interfering substances which are determined as (I). These substances are removed, for the most part, by treatment with Zn(OH)<sub>2</sub>, the small remaining error being allowed for in a correction factor = 0.82% of (I) in the dry substance of the meat extract when the given procedure is adhered to. Adaptations of a no. of methods to the photometer are described. E. C. S.

Removal of sap from potato pulp by a hydraulic filter-press. L. W. JIRAK (Z. Spiritusind., 1938, 61, 217, 227—228, 235—236).—The described hydraulic filter-press of Hoffmann and Briggen separates 80% of the potato sap from pulp in undiluted form and free from starch and fibre, the filter residue containing only 46—47% of H<sub>2</sub>O. The action is uninterrupted. The use of the apparatus in the prep. of potato flakes and products and the recovery of sap by-products is critically discussed. I. A. P.

Increasing the protein content of potatoes. M. PLATZMANN (Z. Spiritusind., 1938, 61, 263).—The unfavourable distilling properties of potatoes rich in protein are probably due to insufficient use of PO<sub>4</sub>''' and over-manuring with N. By analogy with the known facts of sugar-beet culture, balanced manuring should lead to more efficient use of the N with probable avoidance of these difficulties. I. A. P.

New process for production of dried potatoes and spirits from fresh potatoes in drying plant and distillery. E. LÜHDER, B. LAMPE, and W. KILP (Z. Spiritusind., 1938, 61, 223—226).—By a preliminary pressing of the washed and pulped potatoes, approx.  $\frac{2}{3}$  of the total sap is obtained as press-juice. A saving in drying costs is achieved, and the residual potato material is readily converted into a dry, stable condition for the avoidance of storage loss. Low-temp. slow drying yields a product suitable for distillery mashing with max. EtOH yields, and relatively low temp. suffice in the mash and lead to a spent wash of enhanced fodder val. Mashing may be carried out with or without added press-juice, appropriate  $p_H$  regulation being applied where necessary. I. A. P.

Storage of West Indian mangoes. C. W. WARDLAW and E. R. LEONARD (Imp. Coll. Trop. Agric. Trinidad, Low Temp. Res. Sta. Mem., 1936, No. 3, 47 pp.).—Changes in  $p_H$  and titratable acidity of mangoes during late growth and storage are recorded. Temp. effects are examined. Mangoes are best stored at 8-9°, preferably wrapped in tinfoil. I. A. P.

"Spanish hops" as a herb. C. GRIEBEL (Z. Unters. Lebensm., 1938, 75, 568—572).—Microscopical characters indicate that the herb used, e.g., as flavouring in tinned anchovies, is a mixture of *Origanum hirtum* and *Majorana onites*. E. C. S.

Effects of storage conditions on *Alternaria* in lemons. H. S. FAWCETT, L. J. KLOTZ, and H. W. NIXON (Calif. Citrog., 1936, 21, 118, 143—144).—Fruit stored in cellars with natural ventilation was more liable to breakdown than that stored in air-conditioned refrigerators. The effect is due to the greater uniformity of temp. and R.H. and smaller accumulation of deleterious substances, e.g., CO<sub>2</sub>. I. A. P.

Vitamin-C content of German apples. W. RUDOLPH (Z. Unters. Lebensm., 1938, 75, 565—566).—Varieties contained 1.2—25.8 mg.-% of -C. I. A. P.

Vitamin-B<sub>2</sub> content of prunes. N. F. WITT and E. E. POE (Fruit Prod. J., 1936, 15, 274—275).—On a dry-matter basis, prunes contain 807 Sherman units per lb. I. A. P.

Spray-residue problem of currants. P. J. CHAPMAN and G. W. PEARCE (N.Y. State Hort. Soc. Proc. 80th Ann. Meet., 1935, 250—265).—Currants having As spray residues are cleaned by immersion for 2—3 min. in 1% HCl at >32.2°, provided the residue is >4 times the tolerance limit. Pb is more difficult to remove than As. Jelly from currants carrying 3 times the tolerance limit of As and 5 times that of Pb usually contains < the limit of both. More residue remains in the pomace from cold-pressed than from preheated pulp. Stems carry much of the spray residue, and their removal before pressing materially reduces the Pb and As contents of jelly. I. A. P.

CH. ABS. (p)

Vapour-pressure gradients, water distribution in fruit, and so-called infra-red injury. O. F. CURTIS (Amer. J. Bot., 1937, 24, 705—710).—A steep temp. gradient (5—10°) across an apple results in distillation of H<sub>2</sub>O from the warm to the cool side, causing withering and other symptoms of "radiation injury" on the warm side. A. G. P.

Proportion of copper present in tomato purée. T. COCKBURN and M. HERD (Analyst, 1938, 63, 482—486).—The Cu is determined by the Callan-Henderson method (A., 1930, 53) after destroying the org. matter by a special process. Wide variations occur both in concns. of purées and in their Cu content. Some contain 55 p.p.m., though it has been suggested that >25 p.p.m. should be permitted. I. A. P.

Magnitude of variation in density of home-made fruit juices. T. ROETTGEN (Z. Unters. Lebensm., 1938, 76, 132—151).—The records of 10 years' observation of the  $d$  of apple and pear juices are tabulated and correlated with the weather. The mean  $d$ , total acid, and yield of juice for the whole period were: for apples, 53.3° (Oechsle), 7.5%, and 33.6 l./50 kg.; for pears, 61.6°, 6.1%, and 38.0 l., respectively. I. A. P.

Flash-pasteurisation of apple juice. C. S. PEDERSON and D. K. TRESSLER (Ind. Eng. Chem., 1938, 30, 954—959).—Juice from acid apples may be flash-pasteurised at <71° or heated by the holding method at <68° for 15—20 min. Some juices acquire a cooked flavour if heated at >79° for 1 min. Presence of air is undesirable, and in absence of air it is unnecessary to kill all organisms since mould spores will not develop and surviving bacteria cannot grow in the juice. NaOBz and SO<sub>2</sub> in small quantities lower the effective temp. for holding-pasteurisation, and malic acid also facilitates the killing of bacteria. The canning of deaerated, flash-pasteurised, cloudy apple juice is described. I. A. P.

Elimination of fraud in respect of fruit juices. J. PIEN and H. MEINRATH (Ann. Falsif., 1938, 31, 282—290).—Given a knowledge of the dry extract, mineral, and "protein" (N × 6.25) content of the normal juice, determination of these quantities in any specimen will enable adulteration to be detected. Orange juice should have as a min. dry extract of 130, minerals 3.15, and "protein" 7.80 g./l. I. A. P.

Fruit jellies. IX. Rôle of pectin. 5. Enzymic hydrolysis of starch in presence of pectin, in pectic extracts, and in apple pomace. G. L.

BAKER (Delaware Agric. Exp. Sta. Bull., 1936, No. 204, 88 pp.).—Pectin retards the activity of diastase. In presence of pectin diastatic activity is optimal at 30° and  $p_{H}$  3.2—3.6. The hydrolysis follows the course of a multimol. reaction. Commercial diastase preps. contain pectase, and pectin decomp. proceeds simultaneously with amylolysis, to extents which are more marked at 40° than at 30°. The action of diastase preps. on apple extracts and pomace is recorded, and the bearing of the results on commercial processes is shown. A. G. P.

[Apricot and peach] cordials and liqueurs. J. B. WILSON (J. Assoc. Off. Agric. Chem., 1938, 21, 177—178).—No  $\gamma$ -undecolactone was found in apricot or peach cordial kept for 19 months. E. C. S.

[Examination of] canned foods. V. B. BONNEY (J. Assoc. Off. Agric. Chem., 1938, 21, 244—246).—Kertes' method for the determination of EtOH-insol. solids (cf. B., 1938, 723) is shown by collaborative analysis to be trustworthy and is recommended for official adoption. E. C. S.

[Determination of] colouring matters in foods. C. F. JABLONSKI (J. Assoc. Off. Agric. Chem., 1938, 21, 186—190; cf. B., 1937, 976).—Further reports of collaborative analyses of mixtures of Ponceau SX and 3R are tabulated, many results being close to the theoretical vals. E. C. S.

[Foodstuff] preservatives. [Determination of saccharin.] W. F. REINDOLLAR (J. Assoc. Off. Agric. Chem., 1938, 21, 184—186).—200 p.p.m. of saccharin were added to commercial apple butter. The amounts recovered by the official method varied from 160 to 235 p.p.m., those by the new method (cf. A., 1937, II, 268) from 195 to 229 p.p.m. Improvements in each method are specified. E. C. S.

Determination of boric acid in food products. B. I. GUTERMAN (Proc. Inst. Sci. Res. Food Ind. Leningrad, 1935, 2, 72—84).—The methods of Jørgensen and of Beythien-Hempel give inaccurate results with meat products containing  $PO_4^{''}$ . Heberbrand's colorimetric method is satisfactory in presence of  $PO_4^{''}$ . CH. ABS. (p)

[Determination of] metals in foods. H. J. WICHMANN (J. Assoc. Off. Agric. Chem., 1938, 21, 190—198).—The reports on As, Sb, Cu, F, Pb, Hg, Se, and Zn (cf. following abstracts) are reviewed. E. C. S.

[Determination of] lead [especially in spray residue]. P. A. CLIFFORD (J. Assoc. Off. Agric. Chem., 1938, 21, 212—220; cf. A., 1937, I, 426).—The results are recorded of collaborative analysis of "strip solutions" from unsprayed apples to which known amounts of Pb and interfering substances were added. Colorimetric and electrolytic methods were used, good agreement being obtained with each. Interference by silicates was most troublesome in the former method, but was overcome by taking a smaller aliquot. E. C. S.

[Determination of] fluorine in foods. D. DAHLE (J. Assoc. Off. Agric. Chem., 1938, 21, 208—212; cf. B., 1937, 975).—Collaborative results obtained with the peroxidised Ti, the  $Th(NO_3)_4$ , and Armstrong's

microtitration methods are reported. The deviations are large, and possible sources of error are discussed. E. C. S.

Arsenic introduced into the organism in certain beverages and foods. P. MANCEAU, H. GRIFFON, and R. NICOLAS (Ann. Falsif., 1938, 31, 262—281).—Cribier's method (B., 1924, 113), using the apparatus of Griffon and Buisson (A., 1934, 47), is shown to give trustworthy results when applied to the residues obtained by wet combustion of org. matter provided these are not rich in salts. The results of numerous analyses of wine, beer, lemonade, milk, bread, preserves, and chocolate are tabulated. The quantities of As found were minute. E. C. S.

Volatile constituents of roasted coffee. W. R. JOHNSTON and C. N. FREY (J. Amer. Chem. Soc., 1938, 60, 1624—1627).—Distilling roasted coffee at about 0.005 mm. gives  $Ac_2$ ,  $CHAcMe-OH$ , furan, furfuraldehyde, furfuryl alcohol,  $MeCHO$ ,  $C_5H_5N$ ,  $H_2S$ , a ketone (2:4-dinitrophenylhydrazone, m.p. 258—259°), and other compounds. This work and that of Prescott *et al.* (B., 1937, 975) make it probable that the products isolated by Staudinger and Reichstein (cf. B.P. 246,454 and 260,960; B., 1926, 1028; 1928, 347) are primary constituents of the coffee. R. S. C.

Determination of caffeine in coffee. G. SCOTTI (Boll. Chim. farm., 1938, 77, 403—405).—A modified method is described in which coffee in  $H_2O$  is treated with  $CaO$ , and the extract purified with  $Al(OAc)_3$ , and then with  $KMnO_4$ , after which caffeine is extracted from the dried product by  $CCl_4$ , and then by  $CHCl_3$ , and weighed. E. W. W.

Brick tea. C. SCHULER (Z. Unters. Lebensm., 1938, 75, 572—574).—The sample contained  $H_2O$  9.89, caffeine 2.19, fat 0.69, crude fibre 16.48, ash 7.38, tannins 8.47, essential oil 0.09, and aq. extract 32.9%. E. C. S.

Analysis of caramel colour. W. R. FETZER (Ind. Eng. Chem. [Anal.], 1938, 10, 349—353).—Methods for determining the  $d$ ,  $p_{H}$ , tinctorial power, ash and Fe contents, and general suitability of caramels for colouring beverages, foods, pharmaceuticals, etc. are described. F. N. W.

Technical developments in confectionery. E. B. BENNION (Food Manuf., 1938, 13, 309—311).—Semi- and fully-hydrogenated fats are being increasingly used as shortenings. Whipped cream can be satisfactorily made without whipping by storage of cream with  $N_2O$  under pressure, the container delivering the whipped product in desired quantity by allowing pressure to force out the cream. Batter emulsions containing butter show max. stability at  $p_{H}$  6.3. W. L. D.

Grasses and forage crops in Jamaica. W. L. BARNETT (J. Jamaica Agric. Soc., 1936, 40, 16—26, 114—120).—Numerous analyses are recorded. CH. ABS. (p)

Silage from grasses and legumes. T. E. WOODWARD and J. B. SHEPHERD (U.S. Dept. Agric. Tech. Bull., 1938, No. 611, 33 pp.).—Good, palatable silage may be prepared from chopped (immature) grass or grass + legumes without supplementary

treatment in the silo. Losses of dry matter and carotene are small. Chopping improves palatability and restricts loss of dry matter. Addition of acid to the grass slightly diminishes dry matter and carotene loss, but lowers palatability. Addition of molasses improves palatability but is not otherwise advantageous. Fresh, chopped, green lucerne similarly produces good silage which is, however, less palatable than that from grass. Partial drying of lucerne before ensilage increases surface spoilage, raises the temp. of fermentation, and improves palatability. Lucerne silage is as effective as lucerne hay (equal dry matter basis) for milk production, but is inferior for body-wt. maintenance. A. G. P.

**Lactic silage due to thermophilic organisms.** C. GORINI (Lait, 1938, 18, 673—681).—Failures in natural silage making are due to presence of air and weak lactic fermentation. Butyric fermentation proceeds under such conditions. Best conditions of making allow the temp. of stack or silo to remain at 60° for a considerable time to allow fermentation by *B. lactis thermophilus* to proceed. This temp. also has a pasteurising effect and destroys organisms responsible for protein degradation and butyric fermentation. W. L. D.

**Stability of carotene in plant tissues.** M. W. TAYLOR and W. C. RUSSELL (J. Nutrition, 1938, 16, 1—13).—Chopped, artificially-dried lucerne hay stored in bags lost 50% of its carotene (I) content during 3 (summer—autumn) months. No further loss occurred during the winter but there was a further 25% decline in the subsequent summer. Milled hay retained practically the whole of its (I) when stored for 20 months in vac. at 0±5° in darkness. Fineness of grinding was of little importance compared with exclusion of light and air. In 5 months A.I.V. maize silage lost 20% of its (I); with lucerne silage the loss was insignificant. Ordinary maize silage dried in a vac. over H<sub>2</sub>SO<sub>4</sub> and stored in a vac. in darkness at 0° lost (I) rapidly. Simultaneously with the loss of (I) in stored hay the residual (I) probably undergoes a change which renders it of inferior biological potency. A. G. P.

**Digestibility and nutritive value of Karroo pasture plants. III. Suurgras or stinkgras. IV. Rivier draarbos. V. Rooigras.** J. P. BOTHA (Farming in S. Africa, 1938, 13, 141, 163, 198—199, 235—237; cf. B., 1938, 975).—III. Analyses and results of digestibility trials are recorded. The grass (*Schmidtia kаланariensis*) is deficient in protein but has a highly digestible carbohydrate fraction. The digestibility coeff. is comparable with that of good meadow or oat hay.

IV. Analyses and digestibility data for *Tripteris pachypteris* and *T. spinescens* are recorded. The nutritive val. is superior to that of lucerne hay.

V. *Themeda triandra* when cut at fortnightly intervals closely resembles good meadow hay in food val. Mature grass is of poor val. A. G. P.

**Value of Aus paddy straw as fodder.** I. CHATTERJEE and M. A. HYE (Agric. Live-Stock India, 1938, 8, 361—366).—This variety, which accounts for 26% of the rice crop of Bengal, is harvested before it is dead-ripe; the straw contains 18% more of

digestible nutrients than the *Aman* varieties. The straw is richer in protein, Ca and P, and vitamins than that of the latter. W. L. D.

**Behaviour of rice kura (bran) as cattle feed.** M. CARBERY and I. CHATTERJEE (Agric. Live-Stock India, 1938, 8, 367—375).—The material contains oil 20, P<sub>2</sub>O<sub>5</sub> 6.0, MgO 2.6, and CaO 0.2%. Feeding at the rate of 1.5 kg. per 1000 lb. live wt. gave positive balances for MgO, K<sub>2</sub>O, Na<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, and N, but a negative CaO balance. The high P intake has no deleterious effect since about 85% is in org. combination as phytin. The protein is not deficient in essential NH<sub>2</sub>-acids. W. L. D.

**The Bettler protein-substitution experiments.** P. EHRENBERG, H. NITSCHKE, and J. MÜLLER (Z. Tierernähr. Futtermittelk., 1938, 1, 33—71).—Effects of replacing oil-cake protein in cow rations by urea or glycine are recorded. Urea gave the better results in respect of milk yield and live-wt. maintenance. A. G. P.

**Wintering beef cows on the range with and without a supplement of cottonseed cake.** W. H. BLACK, J. R. QUESENBERRY, and A. L. BAKER (U.S. Dept. Agric. Tech. Bull., 1938, No. 603, 21 pp.).—Use of the cake as a supplement to winter range increased the body wt. of cows and the wt. of subsequent calves, both at birth and at weaning. A. G. P.

**Feeding-value of artificially dried sweet lupin: effects of feeding to milch cows.** K. RICHTER, J. HERBST, and R. EHINGER (Z. Tierernähr. Futtermittelk., 1938, 1, 24—32).—Sweet lupins, cut at the flowering stage and dried artificially, had relatively high digestible protein contents and starch equiv. Replacement of part of the ration with lupin in amounts which maintained the total milk yield increased the % fat in the milk. A. G. P.

**Feeding value of bulky mangolds in comparison with richer roots and sugar beet for milch cows.** K. RICHTER and J. HERBST (Z. Tierernähr. Futtermittelk., 1938, 1, 16—23).—The sap of mangolds has no special effect in cattle feeding. Replacement of mangolds by an equiv. proportion of sugar beet in cow rations diminished total milk and fat yields. A. G. P.

(A) **Digestibility of the Lampe curly-leaved fodder mallow for sheep.** (B) **Feeding-trials with milch cows.** K. SCHARRER and H. NEBELSIEK (Z. Tierernähr. Futtermittelk., 1938, 1, 1—10, 11—15).—(A) Feeding trials are recorded. Max. digestible protein and min. fibre contents of the mallow were attained just before flowering. The crude nutrient val. and digestibility coeff. of the curly-leaved were > those of the smooth-leaved variety at all corresponding stages of growth.

(B) Addition of first-cut mallow (just before flowering) to a basal ration for cows lowered the milk yield but increased the % and gross yield of milk fat. The second-cut fodder increased the yield of milk and fat. Mallow silage (p<sub>n</sub> 4.2) produced some diminution of milk yield without appreciable change in the % of fat. A. G. P.

(A) **Digestibility of soya-bean straw and soya-bean straw-molasses for sheep.** (B) **Digest-**

ibility of raw and steamed soya beans for sheep. K. SCHARRER and H. NEBELSIEK (Z. Tierernähr. Futtermittelk., 1938, 1, 72—81, 82—88).—(A) In trials with wethers the chopped straw showed higher contents of crude and digestible protein, crude fat, and ash, and higher digestibility coeffs. and starch equiv., than did the straw meal. Crude fibre contents were similar. A mixture of chopped straw and molasses had a higher digestible protein content but a lower starch equiv. than a straw meal-molasses mixture. The straw-molasses mixtures were more rapidly digested than the corresponding straw products alone.

(B) The digestibility coeffs. of the individual nutrient constituents and also the starch equiv. were higher in the steamed material. A. G. P.

Relative value of full and restricted feeding in pig-fattening. M. ST.-PIERRE (Bonne Terre, 1938, 19, 147—209).—Effects of full, three-quarter, and half rations on rate of growth and carcass quality are examined. A. G. P.

Casein-wool.—See V. Corrosion-resistance of tinfoil.—See X. Fat of goat's milk. Deodorising edible oils. Refractive dispersion of ghee etc. Cacao-waste fats. Index of keeping quality of fats etc. Utilising animal carcasses. Report of Fat Analysis Commee.—See XII. Bartlett pears in storage. Tea-farm soil. Citrus and tea plant cultivation.—See XVI. EtOH from potatoes.—See XVIII. Cryolite and health. Dairy waste waters.—See XXIII.

See also A., II, 374, Vitamin-E. III, 741—6, Vitamins. 771, Seeds of *Cajanus* and *Canavalia* sp. Vitamin-C in cereals and pulses.

#### PATENTS.

Treatment of wheat flour or other vegetable flours or meals, or the grains or seeds from which said flours or meals are derived. CARBO-NORIT-UNION VERWALTUNGS G.M.B.H. (B.P. 476,412, 31.3.37. Ger., 3.4. and 25.7.36).—Objectionable odours and tastes are removed by admixture with 0.1 wt.-% of finely-ground active C. E. B. H.

Manufacture of leavened bread. (A) A. K. EPSTEIN and B. R. HARRIS, (B) C. N. FREY, A. S. SCHULTZ, and E. B. BROWN, Assrs. to STANDARD BRANDS, INC. (U.S.P. 2,067,908 and 2,067,911, 19.1.37. Appl., [A] 20.10.30, [B] 3.11.27).—(A) A supply of N, in a form readily assimilable by the yeast, is obtained by adding urea to the bread dough; this liberates NH<sub>3</sub> by the action of urease contained in ground soya bean. Small amounts of CaSO<sub>4</sub> and KClO<sub>3</sub> activate the urease, and the presence of NaCl regulates the rate of production of NH<sub>3</sub>. (B) Maize sugar and pure maltose will produce a desirable crust colour in bread if free NH<sub>3</sub> is present in the dough during baking. This is effected by adding 0.5 lb. of urea per 300 lb. of flour, or a smaller amount of urea in combination with (soya-bean) urease. The p<sub>H</sub> of the dough is controlled by adding a relatively insol. acid substance, e.g., mucic acid or CaH<sub>4</sub>(PO<sub>4</sub>)<sub>2</sub>. E. B. H.

[Leavened] bread manufacture and compositions for use in same. C. N. FREY and A.

SCHULTZ, Assrs. to STANDARD BRANDS, INC. (U.S.P. 2,067,912, 19.1.37. Appl., 13.6.30).—A bread improver contains NH<sub>4</sub>Cl (0.15—0.3), CaSO<sub>4</sub> (0.2—0.4), KBrO<sub>3</sub> (0.0039—0.005), and ground soya bean (~1 lb.) per 300 lb. of flour. E. B. H.

Production of thin egg-white. A. K. BALLS and T. L. SWENSON, Ded. to U.S.A. (U.S.P. 2,073,411, 9.3.37. Appl., 6.3.34).—Egg-white is thinned by mixing with 0.02% of a proteolytic enzyme, e.g., trypsin, and maintaining the mixture at 30—40° (38°) for 4—10 hr. If desired, the white may be first screened and the already thin portion removed before treatment. D. M. M.

Preparation of dried protein products. W. CLAYTON and J. A. MONTGOMERIE (B.P. 479,810, 5.4.37).—In the spray- or drum-drying of protein materials, such as egg-yolk, egg-albumin, milk, or blood, surface denaturation, which results in impaired solubility, is avoided by adding surface-active materials, such as saponin, bile salts, peptone, alkali laurates, sulphonated compounds, and rennet, in concns. of 1:1000—10,000. E. B. H.

Method of analysis [for determining total solids in liquid egg material]. F. J. CAHN and A. K. EPSTEIN (Assee.), M. C. REYNOLDS, and B. R. HARRIS (U.S.P. 2,065,114, 22.12.36. Appl., 11.3.35).—Determination of total solids is obtained by measurement of *n*, but to obtain the material in a form suitable for clear reading it must first be mixed with an electrolyte such as NaCl. E. B. H.

Treatment of fish [for canning]. C. S. ASH and P. BERGLUND, Assrs. to CALIFORNIA PACKING CORP. (U.S.P. 2,064,872, 22.12.36. Appl., 30.7.35).—To prevent fish, e.g., sardines, from sticking together or to the sides of the tin during canning, the cleaned fish, after preliminary brining, are immersed in a hardening brine which contains, in addition to NaCl, 1—5% of Ca chloride, tartrate, acetate, or citrate (or a mixture of these salts) and 0.2—1.0% of AcOH. E. B. H.

Preservation of fish, frogs, and the like. F. J. NEVISON (B.P. 479,562, 12.2.37).—Fish etc. (and insects) are preserved for purposes of decoration or permanent bait by placing them alive in EtOH and leaving them for 1—4 weeks. The fish are then gutted and finally stiffened and sealed with cellulose cement. E. B. H.

Manufacture of fish-liver meal and oil from fish liver. G. JONSSON (B.P. 475,893, 29.5.36).—Fish livers are boiled with open or indirect steam in a jacketed, barrel-shaped vessel (the lower conical part of which is deeper than the upper cone) and the grax (greaves) is discharged into a pressure vessel, whence it is forced by steam into an evaporator or coagulator chamber and thence to a second pressure vessel; from here it is driven to a storage vessel and finally to a press to remove remaining oil. Apparatus is claimed. E. L.

Smoking of foodstuffs. C. THON (B.P. 476,469, 28.1.37. Nor., 28.1.36).—The chamber holding the food product is partly evacuated and smoke then admitted from another chamber in which it has been generated. E. B. H.

**Treatment [blanching] of fruit.** R. B. HARVEY (U.S.P. 2,077,411, 20.4.37. Appl., 23.5.34. Renewed 29.6.36).—The green colour of mature fruits, particularly citrus fruits, is destroyed by immersing the fruit for 3—10 min. in a bleaching liquid containing an olefine or a derivative thereof. A preferred liquid consists of 40 g. of neutral soap flakes dissolved in 100 c.c. of  $H_2O$  and mixed with 10 c.c. of  $OH \cdot [CH_2]_2 \cdot OBu$ . This is emulsified with 20 c.c. of triisobutylene or ethyleneformo- or -butyro-hydrin and diluted to 2 l. It is used at  $43^\circ$  and after immersion the fruits are rinsed, dried, polished, and packed. The green colour is removed in 48—96 hr., disclosing the yellow or orange colour beneath.

D. M. M.

**Manufacture of liquorice mass.** W. L. GEDDES, Assr. to MACANDREWS & FORBES Co. (U.S.P. 2,067,913, 19.1.37. Appl., 7.3.35).—Finely-divided solid particles obtained by drying liquorice extract are moulded under pressure.

E. H. S.

**Stabilisation of green fodder.** N. V. INTERNAT. SUIKER EN ALCOHOL COMP. INTERNAT. SUGAR & ALCOHOL Co. "ISACO" (B.P. 479,437, 4.8.36. Ger., 12.8.35).—Grass (100 kg.) is preserved by addition of conc. HCl (100 g.) and furfuraldehyde (4—10 g.). These ingredients may be added in the form of the spent wash from the prep. of wood sugar (2 l.) after heating with acid.

E. B. H.

**Manufacture of ready-to-eat cereal foods [from bran].** KELLOGG Co. (B.P. 487,678, 18.12.36. U.S., 23.12.35).

**Purifying liquids [e.g., cream].**—See I. **Shortenings.** **Fat-containing powder.**—See XII. **Casein products.** **Hardened protein materials.**—See XIII. **Sugar composition.** **Feeding-stuff from beet sugar.** **Cryst. sugar.**—See XVII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Sterilisation of sodium bicarbonate solutions.** K. SCHULZE (Pharm. Zentr., 1938, 79, 473—474).—The solutions are heated at  $120^\circ$  for 15 min. in a closed bottle. Addition of phenolphthalein indicates the formation of  $Na_2CO_3$  during heating and its complete reversion to  $NaHCO_3$  on cooling.

E. H. S.

**Official sulphur ointments and their assay.** H. M. BURLAGE and C. E. BRADY (J. Elisha Mitchell Sci. Soc., 1935, 51, 241—242).—The sample, containing approx. 0.03 g. of S, is covered with 0.2 g. of KCN,  $H_2O$  and  $COMe_2$  are added, and the mixture is evaporated to dryness at temp.  $>$  the m.p. of the ointment. Evaporation is repeated twice after additions of  $COMe_2$ . The residue is repeatedly extracted with hot  $H_2O$ , the combined extracts being boiled with  $H_3BO_3$  and, when cool, treated with  $H_3PO_4$  and enough aq. Br to produce a yellow colour. The solution is decolorised with PhOH solution, and, after 15 min., excess of KI is added. The mixture is kept for 30 min. in darkness and titrated with  $Na_2S_2O_3$ .

CH. ABS. (p)

**Hydrogenated castor oil in ointments.** II. **Cosmetics.** G. W. FIERO and L. D. LOCKIE (J. Amer. Pharm. Assoc., 1938, 27, 402—404; cf. B., 1937, 186).—Hydrogenated castor oil (m.p.  $86^\circ$ ) is

suitable for borax cold cream when  $N(C_2H_4 \cdot OH)_3$  stearate is used as an emulsifier. The oil gives stiffer creams than does white wax or spermaceti. The prep. of satisfactory creams containing the oil is discussed.

F. O. H.

**Iodine in liquid petrolatum [paraffin]. Its preparation and assay.** S. W. BOWER and L. G. FREEMAN (J. Amer. Pharm. Assoc., 1938, 27, 496—499).—The solubility of I in various liquid paraffins ( $d$  0.845—0.905) is 1.32—1.42%. Prep. by using the oil at  $70^\circ$  is recommended. A method of assay (extraction with EtOH and titration of the extract, diluted with aq. KI, by 0.1N- $Na_2S_2O_3$ ) is described.

F. O. H.

**Assay of chloroform liniment, U.S.P. XI.** S. W. GOLDSTEIN and W. F. REINDOLLAR (J. Amer. Pharm. Assoc., 1938, 27, 400—402).—The liniment is digested with EtOH-KOH in a pressure-bottle at  $100^\circ$ , the acidified ( $HNO_3$ ) digest is treated with  $AgNO_3$  and filtered, and excess of  $AgNO_3$  is determined by titration with 0.1N- $NaSCN$ ; alternatively, the Cl' is determined gravimetrically (cf. Beal and Szalkowski, B., 1933, 764).

F. O. H.

**Micro-determination of alcohol concentration and extract content of pharmaceutical and similar preparations.** W. KERN and G. LÜCHOW (Apoth.-Ztg., 1938, 53, 895—897).—Both determinations are combined in a modification of Widmark's method (A., 1922, ii, 789; 1932, 41). Results comparable with those given by the D.A.B. VI and Pharm. Helv. methods are obtained.

E. H. S.

**Determination of salicylic acid and salicylates in pharmaceutical preparations.** D. PONTE (Boll. Chim. farm., 1938, 77, 457—459).—The general method is to extract the salicylic acid (with salicylates, liberated by acidification) with  $Et_2O$  and to titrate the separated acid in aq. EtOH solution with 0.1N-KOH. Variations of this method (e.g.,  $H_2O$ -distillation of the acid) for various types of pharmaceutical preps. are described.

F. O. H.

**Bleaching of a suspension of egg extract (lecithin and lutein) by the action of sunlight and heat.** L. BRACALONI (J. Pharm. Chim., 1938, [viii], 28, 97—102).—Ampoules of egg extract (cf. B., 1937, 1268; 1938, 392) are bleached by long exposure to sunlight or at  $70^\circ$ , but the particles undergo no microscopical or chemical change; the  $p_H$  is decreased, especially in presence of air. The bleached material can be injected with safety.

J. L. D.

**Datura stramonium.** F. H. EBY, F. M. SCHOLL, and D. J. PHILLIPS (J. Amer. Pharm. Assoc., 1938, 27, 474—476).—Two plants grown in Philadelphia under unsuitable conditions were of normal growth and alkaloid content (e.g., up to 0.8% in the petioles).

F. O. H.

**Stability of ipecac preparations.** S. W. GOLDSTEIN (J. Amer. Pharm. Assoc., 1938, 27, 482—484).—Extracts prepared by percolation with U.S.P. X or XI menstrua show little loss in alkaloid content on keeping for 16 months; a small sediment separates after 2 months. Percolation with 9% AcOH gives less stable extracts than those prepared with aq. EtOH.

F. O. H.



**Assay of tablets of belladonna extract.** D. T. WILSON (J. Amer. Pharm. Assoc., 1938, 27, 398—400).—The powdered tablets are extracted with  $\text{Et}_2\text{O}-\text{NH}_3$ , the latter extract being washed with dil.  $\text{H}_2\text{SO}_4$ , and the washings are treated with aq.  $\text{NH}_3$  and extracted with  $\text{CHCl}_3$ . The  $\text{CHCl}_3$  extract is evaporated and the residue treated with 0.02N-acid, excess of which is titrated with 0.02N-NaOH. F. O. H.

**Colour reaction characteristic of hashish.** P. DUQUENOIS and H. N. MUSTAPHA (Bull. Sci. Pharmacol., 1938, 45, 203—205).—Hashish gives a blood-red colour with  $\text{H}_2\text{SO}_4$  containing a little  $\text{H}_2\text{O}_2$ . Under defined conditions the change is sp. but cannot be applied quantitatively. Diaminophenol,  $o\text{-OAc}\cdot\text{C}_6\text{H}_4\cdot\text{CO}_2\text{H}$ , and brucine give red colours which become yellow and  $\text{NHAc}\cdot\text{C}_6\text{H}_4\cdot\text{OEt}$  gives a red-brown colour. Denigès' reagent gives a rose colour at  $100^\circ$ , also afforded by  $m\text{-C}_6\text{H}_4(\text{NH}_4)_2$ .  $\text{MeCHO}$  in  $\text{EtOH}$  containing vanillin followed by pure  $\text{HCl}$  gives a fugitive green colour which becomes grey, then indigo, and finally slowly violet. The reaction is sp. and can be applied to colorimetric determination. H. W.

**Determination of morphine in opium by the I.P.V method.** C. FASANO (Boll. Chim. farm., 1938, 77, 460, 463).—The yield of pptd. alkaloid is an optimum with contact periods of 24, 18, and 12 hr. for maceration periods of 1, 5, and 10 hr., respectively. F. O. H.

**Detection in quinine and quinidine of small amounts of methoxyl-free cinchona alkaloids.** K. W. MERZ and A. HOFMANN (Arch. Pharm., 1938, 276, 388—393).—For the detection of cinchonine in quinidine, 1 g. of the alkaloid is hydrogenated ( $\text{Pd}-\text{BaSO}_4$ ) and, after removal of the catalyst, the product is hydrolysed with 60%  $\text{H}_2\text{SO}_4$ . 15%  $\text{NaOH}$  is added in small portions to the cooled solution until neutralisation is complete and the ppt. has passed into solution, into which  $\text{CO}_2$  is then passed until the bases are repptd. These are filtered, washed with cold  $\text{H}_2\text{O}$ , and dissolved in the requisite amount of 2%  $\text{HCl}$ . The solution is evaporated on the water-bath and cooled, with continuous stirring. After removal of the ppt., the filtrate is evaporated further, again cooled, and filtered, after which the filtrate is treated dropwise with 1%  $\text{NaOH}$ . If a ppt. is formed after neutralisation, the presence of cinchona alkaloids free from  $\text{OMe}$  is established. In confirmation the ppt. is frequently centrifuged with 1%  $\text{NaOH}$ , dissolved in 5%  $\text{H}_2\text{SO}_4$ , and tested for fluorescence; a scarcely perceptible effect due to minute traces of dihydroquinidine is without importance. The filtrate from the ppt. caused by  $\text{CO}_2$  is acidified with  $\text{H}_2\text{SO}_4$ , evaporated almost to dryness, and cautiously neutralised with  $\text{NaOH}$ ; a ppt. indicates the presence of cinchona alkaloids free from  $\text{OMe}$ . For the detection of cinchonidine in quinine a single crystallisation suffices owing to the free solubility of dihydrocupreine dihydrochloride. H. W.

**Separation, identification, and determination of substances entering into the composition of quinine hydrochloride tabloids by successive extractions.** G. N. THOMAS (J. Pharm. Chim.,

1938, [viii], 28, 111—114).— $\text{H}_2\text{O}$  is removed from the powdered tabloids at  $110^\circ$ ; fat, quinine hydrochloride, and sugar are removed from the dry residue by successive extractions with light petroleum,  $\text{CHCl}_3$ , and  $\text{EtOH}$ . The residue now contains mainly starch and talc and sometimes small amounts of wheat flour and gum arabic. The method is approx. quant. if the extracted material is weighed. J. L. D.

**Determination of ecgonine bases in crude cocaine and in coca leaves.** L. VAN ITALLIE (Pharm. Weekblad, 1938, 75, 909—916).—The conclusions of the League of Nations Committee are reported. Crude cocaine is examined for  $\text{H}_2\text{O}$  content and the ecgonine is determined polarimetrically after hydrolysis with 2N-HCl. As a check the combined acids are extracted from the hydrolysis solution with  $\text{Et}_2\text{O}$ -light petroleum and determined by titration with 0.1N-KOH (phenolphthalein). Coca leaves are passed through a 2-mm. sieve and the  $\text{H}_2\text{O}$  content is determined by drying at  $103\text{--}105^\circ$ . 20 g. are mixed with 20 c.c. of 2N- $\text{Na}_2\text{CO}_3$  and extracted continuously with  $\text{Et}_2\text{O}$  for 8 hr. The bases are extracted from the  $\text{Et}_2\text{O}$  extract with  $\text{HCl}$ , again set free, and then determined by titration with 0.1N-HCl. As a check the combined acids are determined as in the case of crude cocaine. S. C.

**Diphenylamine-sulphuric acid reaction of tobacco.** C. PYRIKI (Pharm. Zentr., 1938, 79, 553—558).—The reaction applied to tobacco extract is not very sensitive; an immediate colour can be obtained only with concns. of  $\text{KNO}_3 > 0.5\%$ ; those  $< 0.05\%$  give negative results. The quality of oriental tobacco and cigarettes is inversely  $\propto [\text{NO}_3^-]$ . Of 52 commercial qualities of cigarettes only 7 gave negative reactions and the largest quantities (0.5% and over) were in the cheapest varieties. Many cigars gave stronger reactions than the tobacco. E. H. S.

**Micro-determination of the alkalinity of tobacco smoke.** V. L. NAGY (Pharm. Zentr., 1938, 79, 505—508).—The smoke from 0.05 g. of tobacco burning in a small glass pipe is drawn through a tube filled with a moist absorbent pad which, when combustion has finished, is washed with hot  $\text{H}_2\text{O}$  and the solution is titrated with 0.01N-HCl. E. H. S.

**Determination of volatile oils in vegetable drugs.** L. GOLDBERG, R. K. SNYDER, E. H. WIRTH, and E. N. GATHERCOAL (J. Amer. Pharm. Assoc., 1938, 27, 385—392).—Official methods, based on extraction of the dried material with  $\text{Et}_2\text{O}$ , are liable to give erroneous results. Distillation methods give more accurate results, but an oven method (heating to  $100^\circ$  to give the total volatile matter, from which is subtracted the  $\text{H}_2\text{O}$  content given by distillation with  $\text{PhMe}$ ) is recommended for simplicity and accuracy. F. O. H.

**Determination of the essential oil content of drugs.** O. MORITZ (Arch. Pharm., 1938, 276, 368—388).—An apparatus for carrying out this determination rapidly and reproducibly is described. Previous methods are inadequate. R. S. C.

**Determination of essential oil, particularly in pine-needle oils.** R. PANTKE (Pharm. Zentr., 1938, 79, 474—479).—For the determination of

essential oil in pine-needle oil and its preps. the method of Strazewicz (B., 1936, 298) is the best. The D.A.B. VI and Peyer (B., 1932, 656) methods are unsatisfactory. E. H. S.

**Resins and their significance in perfumery.** K. BOURNOT (Fette u. Seifen, 1938, 45, 408—410).—The various resins, gums, and balsams used in perfumery are reviewed. E. L.

**Use of bleaching earths in synthesising perfumes and processing essential oils.** C. FUCHS (Fette u. Seifen, 1938, 45, 410—413).—The use of bleaching earths as catalyst in reactions (e.g., condensations, esterifications, ring-closure by dehydration, absorptive separation of fractions such as terpenes) of interest to the perfume industry is reviewed. E. L.

**Seeds of *Nyctanthes arbortristis*.**—See XII. **Tobacco. Blaeberrries.**—See XVI. **Analysis of caramel colour. Determining caffeine in coffee.**—See XIX.

See also A., I, 470, **Determining iodides in complex mixtures.** 473, **Determining Hg in pharmaceutical preps.** 478, **Electron ultra-microscope.** II, 345, **Synthetic perfumes from "leaf alcohol."** 359, **Antipneumococcal activity of some 8-quinolyl ethers.** 361, **Synthesis of ephedrine.** 362, **Purification of sitosterol.** 374, **Synthesis of tocopherol.** 380, **NH<sub>2</sub>-alcohols from carbazole.** 383—4, **Alkaloids.** 386, **Determination and identification of alkaloids.** III, 741—6, **Vitamins.** 771, [Oils from] seeds of *Cajanus* and *Canavalia* sp.

#### PATENTS.

**Preparation of [water-]soluble aromatic amino-compounds of therapeutic value.** G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 487,378, 15.12.36).— $p$ -NH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>-SO<sub>2</sub>-NH<sub>2</sub> (I) is condensed with an aromatic, araliphatic, or unsaturated aliphatic aldehyde and an alkali H sulphite (II) (1 mol. in the case of a saturated, and 2 mols. for an unsaturated, aldehyde). The compounds from (I), PhCHO or CH<sub>2</sub>Ph-CHO, and NaHSO<sub>3</sub> (equimols.), and from (I) (1 mol.), CHPh:CH-CHO, CH<sub>2</sub>:CH-CHO, or CHMe:CH-CHO (1 mol.), and NaHSO<sub>3</sub> (2 mols.) are described. Alternatively, the Schiff base from (I) and the aldehyde may be condensed with (II). (Cf. B.P. 462,765; B., 1937, 620.)

H. A. P.

**Manufacture of hormones [from sterols].** NAAML. VENN. ORGANON (B.P. 487,360, 26.5.37. Holl., 26.5.36).—Unsaturated polycyclic alcohols (of the cyclopentanopolyhydrophenanthrene series) are oxidised to ketones by heating with an Al alkoxide or MgCl<sub>2</sub> and an aldehyde or ketone. E.g., cholesterol is converted into cholestenone by heating with Al(OBu)<sub>3</sub> and COMe<sub>2</sub> in C<sub>6</sub>H<sub>6</sub>; ergosterol, similarly, gives *ergostatrienone*, m.p. 131—132.5°. The method is applicable to crude concentrates. H. A. P.

**Manufacture of (A, B) compounds of the androstane and pregnane series, (C)  $\beta\gamma$ -unsaturated ketones of the cyclopentanopolyhydrophenanthrene series, (D) pregnenediones.** SCHERING-KARLBAUM A.-G. (B.P. 486,596, 486,854, 486,992, and

488,103, [A, B] 5.12.36, [c] 25.3.37, [D] 30.12.36. Ger., [A] 5.5.36, [B] 7. and 11.12.35, [c] 31.3.36, [D] 30.12.35). (A) The dehydration by known methods of unsaturated OH-compounds of the androstane and pregnane series, or removal of acid from their esters (ester groups remaining in the product being optionally hydrolysed), is claimed. E.g., distillation in a vac. of  $\Delta^5$ -androstene-3:7:17-triol tribenzoate gives BzOH and a compound (?  $\Delta^{5,7}$ -androstadiene-3:7-diol dibenzoate), m.p. 211—213°, and  $\Delta^5$ -androstene-7-one-3:17-diol diacetate is converted by 2N-KOH-MeOH at the b.p. into  $\Delta^{3,5}$ -androstadien-17-ol-7-one, m.p. 171—172° (acetate, m.p. 220°). (B) Unsaturated compounds of the androstane or pregnane series having CH<sub>2</sub> adjacent to the double linking are oxidised (to ketones) without protection of the double linking, and the products are optionally reduced. OH in the starting material is protected by esterification before oxidation. E.g., androstanediol diacetate is oxidised by excess of CrO<sub>3</sub> in aq. AcOH to  $\Delta^5$ -7-ketoandrostene-3:17-diol diacetate, m.p. 219—220°, which with Al(OPr<sup>i</sup>)<sub>3</sub> in Pr<sup>i</sup>OH or H<sub>2</sub>-PtO<sub>2</sub> gives the 3:17-diacetate, m.p. 241—242°, of  $\Delta^5$ -androstene-3:7:17-triol (KOH-MeOH hydrolysis) (tribenzoate, m.p. 250—251°), and with H<sub>2</sub>-Raney Ni at room temp. gives *androstan-7-one-3:17-diol diacetate*, m.p. 193—194°. Pregnenediol diacetate is oxidised by CrO<sub>3</sub>-AcOH (5 mols.) at 55° to 7-ketopregnenediol diacetate, m.p. 165—166°. (c) 5:6-Dihalogeno-3-keto-derivatives of the cyclopentanopolyhydrophenanthrene series having at 17 CO, CH-OH (or ester), or CHR (R being a hydrocarbon or substituted hydrocarbon group) are dehalogenated under mild conditions which do not isomerise the  $\beta\gamma$ - to the  $\alpha\beta$ -unsaturated ketone (Zn and EtOH in neutral medium). E.g.,  $\Delta^5$ -cholestene dibromide gives with pure Zn and EtOH, MeOH, or COMe<sub>2</sub>  $\Delta^5$ -cholesten-3-one, m.p. 127° [oxime, m.p. 188° (decomp.)]. Dibromination of  $\Delta^5$ -pregnen-3-ol-20-one (Br-AcOH), oxidation with CrO<sub>3</sub>-AcOH (3 equivs.), and reduction of the product with Zn-MeOH gives  $\Delta^5$ -pregnene-3:20-dione, m.p. 158—160° (oxime, m.p. 203—205°, decomp. 207°). A similar series of reactions with  $\Delta^5$ -androstene-3-ol-17-one gives  $\Delta^5$ -androstene-3:17-dione, m.p. 158° (sinters 140°) [dioxime, m.p. 205° (decomp.)], and with androstene-3:17-diol 17-acetate gives  $\Delta^5$ -androstene-17-ol-3-one acetate, m.p. 147° (sinters 130°), and a product, m.p. 180° (sinters 165—170°). (D) Pregnenediols are oxidised with an agent capable of converting CH-OH into CO (excess of CrO<sub>3</sub> or CuO), preferably after protecting the double linking by a reversible process, e.g., addition of halogens or halogen hydrides. E.g., pregnene-3:20-diol is brominated (Br-AcOH), the product is oxidised (CrO<sub>3</sub>-90% AcOH), and finally debrominated to give *pregnene-3:20-dione*, m.p. 128°. H. A. P.

[Preparation of] derivatives of sexual hormones and of compounds containing a cyclopentanopolyhydrophenanthrene ring system having like biological action. A. J. H. KONGSTED (B.P. 487,229, 12.12.36).—Sexual hormones are converted into H<sub>2</sub>O-sol. glucosides by standard methods; these are claimed to have enhanced physiological activity when administered perorally. E.g., interaction of oestrone, acetobromoglucose, and Ag<sub>2</sub>O

in quinoline at room temp. gives a  $H_2O$ -sol. *tetra-acetylglucoside*, m.p.  $210^\circ$ , hydrolysed by  $NaOEt$  to the *glucoside*, m.p.  $170^\circ$ ; with penta-acetylglucose and  $p-C_6H_4Me \cdot SO_3H$  at  $125^\circ$  a compound, m.p.  $210^\circ$  (?), is formed. Dihydro- $\alpha$ -estrone, similarly, gives a *glucoside* (?  $\alpha + \beta$ ), m.p.  $225-260^\circ$ . H. A. P.

**High-vacuum distillation of materials containing sterols and related compounds.** EASTMAN KODAK Co., Assecs. of K. C. D. HICKMAN and A. O. TISCHER (B.P. 489,623, 29.1.37. U.S., 6.2.36).—Compounds having a cholane nucleus are isolated from natural products by high-vac., short-path distillation and further purified, e.g., by crystallisation. E.g., clarified whale oil is subjected to mol. distillation at  $90-220^\circ/0.001-0.003$  mm.; a small fraction containing squalene and free fatty acids distils first, followed, at  $120-160^\circ$ , by cholesterol (I) and its esters, from which (I) may be separated by hydrolysis and crystallisation. Sitosterol and stigmasterol are similarly isolated from soya-bean oil after removal of lecithin. H. A. P.

**Preparation of disubstituted barbituric acids.** E. LILLY and Co. (B.P. 486,934, 11.12.36).—The prep. is claimed by standard methods of barbituric acids and their salts containing in position 5 a  $\beta$ -methylallyl group and a primary or *sec.* hydrocarbon radical of  $C_{2-6}$ . The following are described: *Et*<sub>2</sub>  $\beta$ -methylallyl-*n*-propyl-, b.p.  $103-107^\circ/3$  mm., -isoamyl-, b.p.  $114-117^\circ/2-3$  mm., - $\alpha$ -methylbutyl-, b.p.  $140-144^\circ/8-9$  mm. (corr.), - $\beta$ -methylbutyl-, b.p.  $135-138^\circ/7$  mm., -isopropyl-, b.p.  $124-127^\circ$  (corr.)/9-10 mm., - $\beta$ -ethylbutyl-, b.p.  $127-133^\circ$  (corr.)/1 mm., -isobutyl-, b.p.  $110-114^\circ/1$  mm., and -*n*-hexyl-malonate, b.p.  $122-130^\circ/1$  mm.; *Et*<sub>2</sub>  $\beta$ -methylallyl-, b.p.  $93-94^\circ/3-4$  mm., and *di*- $\beta$ -methylallyl-malonate, b.p.  $124-126^\circ/5-6$  mm.; 5- $\beta$ -methylallyl-5-*n*-propyl-, m.p.  $173-175^\circ$ , -isoamyl-, m.p.  $143-145^\circ$ , - $\alpha$ -methylbutyl-, m.p.  $141.5-143.5^\circ$ , - $\beta$ -methylbutyl-, m.p.  $142-144^\circ$ , -isopropyl-, m.p.  $163-165^\circ$ , -isobutyl-, m.p.  $179-181^\circ$ , -*n*-hexyl-, m.p.  $127-129^\circ$ , - $\beta$ -ethylbutyl-, m.p.  $148-150^\circ$ , -*n*-butyl-, m.p.  $125-127^\circ$ , -*sec.*-butyl-, m.p.  $139-142^\circ$ , -*n*-amyl-, m.p.  $94-96^\circ$ , and - $\alpha$ -ethylpropyl-barbituric acid, m.p.  $183-184^\circ$ ; 5- $\beta$ -methylallyl-, m.p.  $187-189^\circ$ , and 5:5-*di*- $\beta$ -methylallyl-barbituric acid, m.p.  $207-209^\circ$ ; 5-allyl-, m.p.  $149-151^\circ$ , -phenyl-, m.p.  $203-205^\circ$ , and -cyclopentyl-5- $\beta$ -methylallyl-barbituric acid, m.p.  $157-159^\circ$ . The prep. of Na,  $NH_4$ , and alkylamine salts is described. All m.p. are corr. H. A. P.

**Manufacture of stable solutions of ascorbic acid salts of histidine.** F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 480,503, 16.6.37. Switz., 9.7.36).—Access of air is prevented during the prep. and the final solution is saturated with  $O_2$ -free gas and stored either in an atm. of this gas or in an evacuated container. E. H. S.

**Manufacture of ergot-derived product.** E. H. STUART, Assr. to E. LILLY & Co. (U.S.P. 2,067,866, 12.1.37. Appl., 18.11.36).—Ergot is extracted with liquid  $NH_3$  and the extract may be evaporated or extracted with  $Et_2O$  and this extract treated with a dil. acid solution which is then extracted with  $Et_2O$  in presence of alkali. The base so obtained has similar general physiological properties to the known

ergot alkaloids, but in many vital respects it differs both chemically and clinically. E. H. S.

**Manufacture of aromatic sulphonamide-substituted antimony compounds.** [Medicinals.] A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 487,233, 14.12.36).—Aminobenzenesulphonamides and their substituted derivatives (except those containing acid salt-forming groups directly attached to the  $C_6H_6$  nucleus) are converted by the Bart reaction into stibinic acids which are (optionally) reduced to stibine oxides; the last-named may be converted into  $H_2O$ -sol. complex compounds, e.g., with *o*-dihydroxybenzenesulphonic acids. The products are claimed to exert healing power in protozoan infections. E.g.,  $p-NH_2 \cdot C_6H_4 \cdot SO_2 \cdot NH_2$  is diazotised, and added to  $Sb_2O_3$  in conc.  $HCl$  and glycerol, and the solution is made alkaline; on reacidifying *p*-sulphonamidophenylstibinic acid is pptd. This is reduced by  $H_2SO_3$  in dil.  $HCl$ - $MeOH$  plus I to *p*-sulphonamidophenylstibine oxide, which forms a  $H_2O$ -sol. compound with 1:2:3:5- $C_6H_2(OH)_2(SO_3H)_2$ . Stibinic acids and stibine oxides are prepared similarly from  $p-NH_2 \cdot C_6H_4 \cdot SO_2 \cdot NMe_2$ , 2:1:4:5- $NH_2 \cdot C_6H_2Me_2 \cdot SO_2 \cdot NH_2$ , 4:3:1- $NH_2 \cdot C_6H_3Cl \cdot SO_2 \cdot NH_2$ , *o*-anisidine-5-sulphonamide, m.p.  $142^\circ$  (from *o*- $OMe \cdot C_6H_4 \cdot NHAc$  and  $CISO_3H$ , followed by  $NH_3$  and hydrolysis of the Ac group), and *m*- $NH_2 \cdot C_6H_4 \cdot SO_2 \cdot NH_2$ . H. A. P.

**Manufacture of a physiologically active preparation [callikrein].** F. SCHULTZ, Assr. to WINTHROP CHEM. Co., INC. (U.S.P. 2,069,019, 26.1.37. Appl., 6.11.33. Ger., 12.11.32).—Aq. concentrates of the active principle prepared from urine or pancreas, and preferably free from albumin, are treated with  $COMe_2$  in presence of a non-alkaline electrolyte, e.g., 0.1% aq.  $NaCl$ , at a low temp. and the ppt. is isolated and dried. It contains about 12% N, causes simultaneous decrease of the blood pressure and increase of the blood circulation in the lungs, brain, skin, and muscles, and is stable on storage. E. H. S.

**Protection of insulin preparations from premature destruction in the stomach and intestine.** F. LASCH and E. SCHÖNBRUNNER (B.P. 480,476, 1.1.37. Austr., 7.1.36).—One or more org. dyes, e.g., Congo-red, indigo-carmine, Me-orange, and, if desired, resorption-promoting substances, e.g., saponins, are added to the insulin solution. E. H. S.

**Manufacture of a preparation of the hormone of the anterior lobe of the hypophysis.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 488,030, 29.12.36).—A prep. of this gonadotropic hormone, preferably free from salt, is suspended in an innocuous oil, e.g., olive oil, with addition of wool fat. E. H. S.

**Manufacture of extracts from the anterior lobes of the hypophysis.** I. G. FARBENIND. A.-G. (B.P. 479,764, 10.8.36. Ger., 8.10.35. Cf. B.P. 291,018; B., 1930, 302).—The aq. extract, prepared either by direct extraction of the lobes or after extraction with an org. solvent, is pptd. by addition of sufficient low-mol. wt. alcohol (I) to bring the concn. to  $\leq 70\%$  at  $p_H$  6-8, and the ppt. is

extracted with  $H_2O$  at  $p_H$  8—9. From the extract the active substances are obtained by pptn. with (I) and are redissolved in an aq. solvent, frozen, and dried.

E. H. S.

**Production of dry preparations from entire glands, and more particularly from the animal pancreas.** HAUSER & SOBOTKA A.-G. (B.P. 475,868, 22.4.36. Austr., 9.5.35).—Comminuted entire glands are extracted with  $COMe_2$  or const.-b.p. solvent mixtures containing  $COMe_2$ .  $H_2O$  is removed from the extract by distillation and the solvent is re-circulated until the gland pulp is free from  $H_2O$  and fat. Active substances may be recovered from the extract and added to the dry prep. of the extracted gland.

E. H. S.

**Manufacture of stable therapeutic products from intestinal mucous membrane.** I. G. FARBENIND. A.-G. (B.P. 479,487, 4.8.36. Ger., 1.8.35).—Fresh, moist membrane is treated with a disinfectant and frozen. The ground mass is extracted with a buffer solution and the clear extract treated with a  $H_2O$ -sol. org. solvent to ppt. the active principle, e.g., with  $COMe_2$  at  $-5^\circ$ . The ppt. may be redissolved in the buffer solution and the solution evaporated under reduced pressure.

E. H. S.

**Medicinal compound [for intestinal assimilation].** E. C. MOORE (U.S.P. 2,066,572, 5.1.37. Appl., 23.9.33).—An oil, e.g., sandalwood, is mixed with a resin acid, e.g., abietic, which is capable of emulsifying with the alkaline secretions of the intestinal tract. Extract of liquorice, light  $MgO$ , and glycerin may be added and the mass covered with gelatin.

E. H. S.

**Preparing triturations of fresh plants.** G., F., and H. MADAUS (DR. MADAUS & Co.) (B.P. 488,115, 8.1.37. Ger., 9.1. and 19.11.36).—Fresh medicinal etc. plants are triturated in presence of amyloses and/or polyamyloses, with or without addition of buffer salts or mixtures giving an alkaline reaction, and are then dried at  $>30^\circ$  with air of (preferably) slowly decreasing moisture content. This procedure is said to reduce trituration losses.

E. H. S.

**Antiseptic [sheet-rubber] bandage.** E. FETTER Assr. to H. V. LUCAS (U.S.P. 2,064,898, 22.12.36. Appl., 21.2.36).—Self-adhesive, unvulcanised rubber sheet which contains an antiseptic, e.g., hexyl-resorcinol, distributed throughout the rubber is specified.

E. H. S.

**Manufacture of medicinal depôts, masses for infusion, stiffening agents, plastic fillings, and the like.** CHEM. FORSCHUNGSGES. M.B.H. (B.P. 487,506, 7.1.37. Ger., 8.1. and 22.6.36).—Sterile masses, suitable for insertion into body cavities, which are rigid or gelatinous at room temp. but fluid at  $30-60^\circ$ , are obtained by heating a solution or paste of polyvinyl alcohol capable of dissolving or swelling in  $H_2O$  containing a compound of Cr or Fe, and/or a dye of the Congo-red group, or  $NaOAc$ , to  $>100^\circ$  in a closed vessel. Resorption can be accelerated by adding an org. acid (lactic) or retarded by treatment with ultra-violet or X-rays.

S. C.

**Production of a porous absorbent [medical] product.** N. V. KONINKLIJKE PHARMACEUT. FABR.

v./H. BROCADES-STHEEMAN & PHARMACIA (B.P. 487,660, 4.2.37. Holl., 7.2.36).—A porous, spongy mass suitable for use as a tampon is obtained by swelling animal sinews etc., consisting of elastin or collagenous matter, in, e.g., an acid medium, teasing the product into fibres, inflating it with a gas (air,  $CO_2$ ), and then shrinking it by means of, e.g., gaseous  $NH_3$ , which may be mixed with the inert gas for convenience. Excess of liquid is then decanted off and the product sterilised. The products when used as tampons, suppositories, etc. are readily resorbed.

S. C.

**High-vac. distillation.**—See I. Aminoalkyl esters.—See III. Cotton fibre-like material.—See V. Adhesive fabrics.—See VI. X-Ray contrast media for stomach examinations.—See XI.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Cellulose esters in photographic emulsions.** GYDÉ (Rev. Gén. Mat. Plast., 1938, 14, 107—108s).—A brief review of literature.

F. MCK.

**Panchromatic emulsions and ordinary snapshot photography without filters.** O. BLOCH and W. H. DIMSDALE (Brit. J. Phot., 1938, 85, 550—551).—Comparison of pictures of an ordinary subject, taken on normal and extreme types of panchromatic emulsions, without using a filter, shows that for daylight the results are almost indistinguishable, as reflected white light detracts from the purity of colours.

J. L.

**Influence of partial grain dissolution in exposed silver bromide emulsions on developable density.** G. UNGAR (Z. wiss. Phot., 1938, 37, 130—139).—Polemical against Kempf (A., 1938, I, 38). The Ag grain developed for each AgBr grain has a mass independent of the mass of the developing nucleus, and the reduction of the mass of the latter does not affect the photographic density, which is dependent only on the no. of Ag grains developed. On gradual dissolution, the smallest AgBr grains will disappear, and others will have no nuclei left; the no. of grains falls rapidly after their size has been reduced below a certain val. A method of calculating the grain no. from the mean radius is described, assuming that the rate of dissolution  $\propto$  the grain surface area at any given moment. Kempf's results are therefore not to be interpreted as an increase of latent image density towards the interior of the grain.

J. L.

**Relation between contrast and grain number in photographic emulsions.** A. P. H. TRIVELLI and W. F. SMITH (Z. wiss. Phot., 1938, 37, 140—146).—A direct proportionality between  $\gamma$  and no. of grains ( $N$ ) per c.c., as reported by Makarov (unpublished, but recorded by Chibisov and Michailova, Kino Phot. Ind., 1937, No. 3, 24), has been found to hold only within narrow limits and for small grains. Several series of tests have been made which show that  $\gamma \propto \log N$ , other factors being const. At high dilutions (small  $N$ ) better agreement is given by  $\gamma \propto N^1$ . Tests were made with visible light and X-ray exposures.

J. L.

**Critical review of the Russian proposal for standardisation of gelatins for photographic purposes.** A. P. H. TRIVELLI and W. F. SMITH (*Z. wiss. Phot.*, 1938, 37, 123—129).—Makarov's proposal (*Sci. et Ind. phot.*, 1936, 7, 1), based on data by Chibisov and Michailova (*Kino Phot. Ind.*, 1937, No. 3, 24), to standardise gelatins by their power to activate standard emulsion, is based on insufficient data. Calculation of the Bravais-Pearson correlation coeffs. and probabilities of the existence of relations, from data with 11 gelatins, shows that there is a direct relation between  $\gamma$  before after-ripening and increase of  $\gamma$  during after-ripening, but not between the  $\gamma$  before after-ripening and the increase of H. & D. val.; the latter relation was postulated by Chibisov. Experiments with the same gelatin but varying grain sizes of emulsion show that relations also exist between  $\gamma$  and increase of grain size, and between the initial  $\gamma$  and increase of H. & D. val., with varying grain size. In the latter case, increase of sensitivity is greater with greater initial  $\gamma$ ; Chibisov and Michailova found it to be greater with smaller initial  $\gamma$  for different gelatins. The grain size may therefore effect similar results, at least in part.

J. L.

**Tannin in photographic developers.** C. EMMERMANN (*Phot. Ind.*, 1938, 36, 938—939).—The amount of  $H_2O$  absorbed by gelatin (fixed-out kinema film, washed and dried) in 0.5% borax and 0.5% borax + 0.1% tannin solutions has been quantitatively determined. Contrary to previous authors, the tannin causes increased swelling velocity. The softening point of this gelatin was 42.3° in  $H_2O$ , and 41.3° in either of the above solutions. The tannin does not therefore produce any tanning effect. Tests with Champlin-15 developer show that the results obtained are identical with or without the tannin component, and its inclusion in the formula is therefore useless.

J. L.

**Developer tests in connexion with graininess.** A. BOER (*Focus*, 1935, 22, 368—519, 17 pp.).—Fine grain was obtained, with reduced emulsion speed, whenever the concn. of alkali (or of  $COMe_2$  in a  $COMe_2$  developer) was reduced.  $Na_2CO_3$  and NaOH were used.

CH. ABS. (e)

**Very rapid development and fixing of photographic negatives.** A. CHARRIQUO and (MLE.) S. VALETTE (*Bull. Soc. Franç. Phot.*, 1938, 25, 125—131).—16 developers (quinol or genol-quinol formulæ) and 11 fixing baths of varying composition have been tested at 20—50°; comparative results are detailed. Tests of the practical applications of one of these developers (the fastest) and fixing baths at 20—25° allowed development in 40 sec.; another developer at 50° required 10 sec. development time and 50 sec. total processing time.

J. L.

**Physical development of the latent image.** W. REINDERS and M. C. F. BEUKERS (*Trans. Faraday Soc.*, 1938, 34, 912—919).—The special adaptability of the physical development to subjects with intricate and fine detail is emphasised. A theory is evolved which allows the determination of the conditions which the developing solution must fulfil to produce good development of the latent image to be made.

The speed of development is largely dependent on the difference of potential ( $\Delta E$ ) between the Ag potential in the solution and the reduction potential of the developer. In the usual neutral developers  $\Delta E$  is 100—170 mv., which is  $\ll$  in the solutions for rapid chemical development. Solutions in which  $\Delta E = 100$  mv. give a much smaller spontaneous deposit of Ag than solutions with higher  $\Delta E$ . The effect of change in composition in the usual solutions on the reduction and Ag potentials has been studied. The latter may be varied within wide limits by changing the relation  $AgNO_3 : Na_2S_2O_3$ .

W. R. A.

**Desensitisation by silver.** LÜPPO-CRAMER (*Phot. Ind.*, 1938, 36, 891—892).—The theory of the Albert effect, particularly in regard to the author's hypothesis that the desensitisation is produced by Ag nuclei, is discussed. The effect is not similar in type to solarisation.

J. L.

**Standardised sensitometry.** P. K. TURNER (*Brit. J. Phot.*, 1938, 85, 517—518).—It is proposed that sensitometry be standardised by the use of standard light (a lamp at 2366° K. corr. to 5400° K.), accurately calibrated standard step-wedges, exposure by projection of an image of the wedge, and development with any desired (specified) developer at specified time and temp., to provide curves at four different gammas (within 10%). Emulsion speeds are then to be quoted both as inertia and threshold speeds at a given (or standard) gamma. A single-speed definition is considered to be an impossible conception.

J. L.

**Scientific comparison of tone quality in prints.** R. S. POTTER (*Camera*, 1936, 52, 242—244).—The darkest black of the glossy print had ~2.5 times the opacity of the darkest black of the matt print. Waxing of a matt print increases its opacity by ~75%. Both glossy and waxed matt prints show a 17% longer scale of gradation than the matt print.

CH. ABS. (e)

**Mechanism of photographic reversal. I. The Sabattier effect and its relation to other reversal processes.** G. W. STEVENS and R. G. W. NORRISH (*Phot. J.*, 1938, 78, 513—533).—Previous theories are discussed in detail, and a series of experiments is made to show that no explanation hitherto advanced is tenable. Reversal is entirely concerned with grains not first exposed; reversal may be produced with very short first development. Evidence is given for diffusion of Ag during development. It is therefore postulated that metallic Ag acts as a desensitiser by deposition on unexposed grains during the first development, in the neighbourhood of the developed grains; the mechanism is not, however, clear.

J. L.

**Practical importance of a new photographic colour process.** R. WEIZSÄCKER (*Chem.-Ztg.*, 1938, 62, 636—637).—Applications of Agfacolor film are described. New film of 15/10° DIN, film for artificial light, and 16-mm. reversal film of 13/10° DIN are recent improved products.

J. L.

**Chemical after-treatment of Leica negatives.** H. STÖCKLER (*Phot. Ind.*, 1938, 36, 960—962).—After-treatments must be such as do not increase the

grain size. Reduction of contrast is therefore best carried out with "Eugradol," whereby partial Se toning affects only the shadows, and the high lights are then selectively reduced. Intensification of contrast is best with "Fesachrome" intensifier, which employs a mixture of basic dyes of substantially neutral grey final colour, the method being based on that of Lumière and Seyewetz (B., 1937, 1275).

J. L.

**Bromoils and transfers.** I, II. C. H. PARTINGTON (Camera Craft, 1935, 42, 544, 598—604).—Operating details are given. CH. ABS. (e)

**Photogravure positives.** H. M. CARTWRIGHT (Penrose Ann., 1936, 38, 119—122). CH. ABS. (e)

**Photography of altered and faded manuscripts.** N. F. BEARDSLEY (Library J., 1936, 61, 96—99).—A review. CH. ABS. (e)

**Painting it out with oil [in photography].** P. H. JAMESON (Camera Craft, 1936, 43, 20—26).—A solution of I in oils and certain other non-aq. solvents is described. CH. ABS. (e)

**Microphotometer for cotton.**—See V.

## PATENTS.

**Manufacture of photographic film.** G. F. NADEAU, Assr. to EASTMAN KODAK Co. (U.S.P. 2,066,850, 5.1.37. Appl., 24.12.34).—A cellulose acetate film is coated with a layer of gelatin dispersed in  $\text{COMe}_2$ -MeOH with the aid of an org. acid which decomposes at 82—127° into volatile or non-volatile, non-corrosive compounds, e.g.,  $\text{CH}_2\text{Bz}\cdot\text{CO}_2\text{H}$  or  $\text{CHAcMe}\cdot\text{CO}_2\text{H}$ . The sensitive film is then applied to the gelatin surface in the usual way. A. R. P.

**Reversal development of light-sensitive silver halide emulsions.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 489,845, 30.11.36).—In reversal development without intermediate exposure before the second development the layer is treated with an acid bath containing an odourless S compound which produces developable Ag nuclei. The process is applicable to multilayered films containing dye components fast to diffusion and developed by a colour developer. In the example, a multilayer film after exposure is developed and then treated in an intermediate bath of  $\text{H}_2\text{O}$  (1 l.), AcOH (5 c.c.), and *N*-allylthiourea (thiosinamine) (0.4 g.). It is then developed with a colour developer, e.g., *p*- $\text{NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{NET}_2$ , washed, and the Ag image removed, washed, and dried. K. H. S.

**Ripening and hardening photographic emulsions.** KODAK, LTD., Assees. of S. E. SHEPPARD, R. C. HOUCK, and R. H. LAMBERT (B.P. 479,419, 10.7.37. U.S., 10.7.36).—Emulsions are simultaneously hardened and ripened, with elimination of after-hardening effects, by adding a suitable hardening agent to a substantially unripened emulsion, immediately coating the emulsion on a support, drying it, and then subjecting it for a suitable time to "moist incubation" at 60—70° and 50—80% R.H. The hardening is thus more conveniently controllable and increased ripening (emulsion speed) is obtained without higher fog level. J. L.

**Colour photography.** L. O. A. POLLAK (B.P. 475,655, 23.5.36).—An unhardened Ag halide-gelatin emulsion layer containing a  $\text{H}_2\text{O}$ -insol., colour-forming substance not influenced by the Ag image is coated on top of one or two hardened Ag halide emulsion layers, the different layers being sensitised to different colours; alternatively, the second hardened layer may be coated on the back of the support, or on a separate support (exposure being made with the emulsions of the two films in contact). After development (hardening- or colour-developers may be used), the colour is formed in the top layer by coupling etc., the gelatin is selectively hardened according to the image (if this has not already occurred), and a wash-out relief is formed from the top layer by treatment with warm  $\text{H}_2\text{O}$ . The other layers are suitably coloured by, e.g., toning. The dye-coupling in the top layer may be an azo component, a leuco-dye, or a substance capable of pptg. normally  $\text{H}_2\text{O}$ -sol. dyes. Various modifications are described. J. L.

**Colour photography.** KODAK, LTD., and R. E. CROWTHER. From EASTMAN KODAK Co. (B.P. [A] 475,784 and [B] 475,786, 21.2.36. Cf. B.P. 468,560; B., 1937, 1410).—(A) The film used comprises three layers coated on one or both sides of the support, and sensitised to blue, green, and red (the blue on top, but the order of the other two may be reversed). The images obtained after exposure are processed to non-complementary colours, e.g., the blue colour-record to minus red, the green record to minus blue, and the red to minus green; this false 3-colour record is printed on to another similar film and the images are processed to give a true 3-colour record; e.g., in the print from the above example, the blue record is processed to minus green, the green to minus red, and the red to minus blue. Other combinations of false colours are described. In at least one film, at least two of the records are processed by colour production, and at least one by colour suppression. A fourth layer dyed black may be added for the production of a key record or for use as antihalation layer. (B) Films and material similar to the above are characterised in that only two of the records in the original film are processed to false (non-complementary) colours. If exactly the same films, or certain specified colour combinations, are to be used for making the print, then the first print from the original is also a false 3-colour record and a second printing from this is required to obtain a true 3-colour record. A single printing only need be used if the prints from the original false colour records are each processed to colours complementary to the sensitivity of the layer containing the other. At least one record may be processed by colour suppression, and the layer below the top blue-sensitive layer is preferably processed to yellow (positive colour suppression). A fourth, black, layer may be used as before. J. L.

**Colour photography.** KODAK, LTD., Assees. of J. G. CAPSTAFF (B.P. 489,529, 14.9.37. U.S., 28.10.36).—Multi-layer material arranged on one side of a support, with lenticulations formed on the surface of the emulsion, is exposed to the desired picture in continuous tone, by exposing either through the support, or through the lenticular face with the

lenticulations temporarily optically obliterated (e.g., by a covering layer of easily-sol. gelatin), the colour-sensitivities of the emulsions being suitably arranged. The image is converted into a broken-tone colour record by a fogging exposure, before or after the picture exposure, through the (uncovered) lenticulations, using a banded filter of minus colours (in relation to the emulsion sensitivities). On reversal development a normal lenticular colour record is obtained. Optical difficulties arising when making a picture exposure through the lenticulations are thus obviated. J. L.

**Printing of colour photographs.** I. G. FARBENIND. A.-G. (B.P. 479,752, 5.8.36. Ger., 9.8.35).—Impurity of yellows in the prints from negatives produced by dye-development, 3-colour processes is prevented by using a printing light from which the range of  $\lambda$  of about 660—720 m $\mu$ . is excluded by means of a suitable filter prepared from, e.g., penta- or hepta-thiocarbocyanine in an EtOH solution or in a gelatin layer. A heavy glass filter containing a metal salt solution, e.g., of CuSO<sub>4</sub>, may also be used. J. L.

**Photographic diazotype processes.** E. N. MASON & SONS, LTD., and D. J. NORMAN (B.P. 489,214, 25.10.37).—Light-sensitive layers are made containing diazo compounds derived from 4-carboalkoxyamino-2:5-dialkoxyanilines. In the examples, paper is coated with the diazo derivatives of 1:4:2:5-NH<sub>2</sub>·C<sub>6</sub>H<sub>2</sub>(NH·CO·OAlk)(OEt)<sub>2</sub>, Alk = Me or Et, exposed to light under tracings, and developed with solutions containing 1:3:5-C<sub>6</sub>H<sub>3</sub>(OH)<sub>3</sub> to give brown or black lines on a white ground. K. H. S.

**Production of photographic and kinematographic pictures in colour.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 488,048, 13.5.37. Ger., 19.5.36. Addn. to B.P. 465,823; B., 1937, 845).—A dye component (or components) rendered non-diffusible by the presence of a saturated or unsaturated aliphatic chain of  $\leq$  C<sub>5</sub> and (optionally) containing a H<sub>2</sub>O-solubilising group is (are) added to a photographic Ag halide emulsion; after exposure and development to a black-and-white Ag picture the dye component is converted into a dye on the site of the Ag image or at parts free from Ag. Multi-layers may be used for three-colour photography. The components preferred are those giving azo or oxidation dyes. E.g., Na 2-hydroxy-6-undecylbenzoate is added to a AgBr emulsion; after exposing, developing, and fixing, the free Ag is converted into Ag<sub>4</sub>Fe(CN)<sub>6</sub>; this is made to absorb diazotised *p*-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·NHPh, and finally coupling to produce a yellow picture is brought about by immersion in 1% aq. Na<sub>2</sub>CO<sub>3</sub>. For producing blue, red, and yellow images with tetrazotised dianisidine there are used the 3-*p*-stearamidobenzamidobenzoyl derivatives of H- and J-acid, and acetoacet-*p*- $\alpha$ -sulphostearamidoanilide (from  $\alpha$ -bromostearyl bromide, *p*-aminoacetanilide, and Na<sub>2</sub>SO<sub>3</sub>), respectively. H. A. P.

**Reproduction of lenticular films.** A. H. J. DE LASSUS ST. GENIES, Assec. of COMP. FRANÇ. POUR L'EXPLOIT. DES PROC. THOMSON-HOUSTON (B.P. 489,823, 25.10.37. Fr., 5.11.36. Addn. to B.P. 470,801).

[Diaphragm for] production of autotype [half-tone] pictures. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 489,374, 24.12.36).

**Cyanine dyes.**—See IV. [Films from] plastic masses. Photomechanical printing forms.—See XIII.

## XXII.—EXPLOSIVES; MATCHES.

**Verification of the law of combustion of parallel layers of colloidal powders.** H. MURAOUB and G. AUNIS (Compt. rend., 1938, 206, 1723—1726).—The law has been verified by showing that an explosive moulded into a ring of square cross-section burns at the same rate as a cylinder of the same explosive of diameter = the thickness of the ring. A. J. E. W.

**Conditions for initiation and propagation of detonation in solid explosives.** W. TAYLOR and A. WEALE (Trans. Faraday Soc., 1938, 34, 995—1003).—Experiments on the decomp. of thin layers of explosive solids show that percussive forces operating in mechanical impacts are capable of causing decomp. Mass detonation is regarded as the passage through the explosive of a narrow reaction zone, in which decomp. takes place under the action of the force in a pressure wave, which is sustained by the energy of the explosion products. Decomp. is primarily due to the grinding together of the particles of explosive with subsequent production at the interfaces of tribo-chemical action, which provides the stimulus for the decomp. Formulæ are derived for the qual. conditions governing the initiation and propagation of detonation waves in solid explosives. W. R. A.

**Energy measurements indicating three characteristic modes of detonation of trinitrophenylmethylnitroamine (tetryl).** T. C. SUTTON (Trans. Faraday Soc., 1938, 34, 992—994).—Detonation of tetryl takes place with three different energies depending on (a) the strength of the container and (b) the density to which the powder has been compressed to a pellet: the greater is the density the larger is the energy of detonation. W. R. A.

**Stability of cellulose nitrates.**—See V.

See also A., II, 354, Nitration of CH<sub>2</sub>Ph·NO<sub>2</sub> and a new isomeride of C<sub>6</sub>H<sub>2</sub>Me(NO<sub>2</sub>)<sub>3</sub>.

## PATENTS.

**Manufacture of [ammonium nitrate] explosives.** HERCULES POWDER CO., Assecs. of R. W. CAIRNS (B.P. 476,285, 3.5.37. U.S., 11.5.36).—The sensitiveness of these explosives, if they have a large negative O balance, is increased by coating the NH<sub>4</sub>NO<sub>3</sub> with a solution of a nitrate of Na, K, Cu, Pb, Zn, Ca, Ni, Fe, Ca, or Mg, or with a solution of any of these nitrates together with NH<sub>4</sub>NO<sub>3</sub> solution, and then drying. W. J. W.

**Non-detonating blasting explosives.** A. T. TYRE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 479,773, 10.8.36).—A hypophosphite of an alkali metal or, preferably, NH<sub>4</sub>, to which NH<sub>4</sub>NO<sub>3</sub> may be added, is mixed with a mixture of an alkali nitrate (KNO<sub>3</sub>)

and an  $\text{NH}_4$  salt ( $\text{NH}_4\text{Cl}$ ), which will form by metathesis a mixture of  $\text{NH}_4\text{NO}_3$  and a cooling salt.

W. J. W.

**Blasting cartridges and charges.** A. IGNATIEFF, and HEATERS, LTD. (B.P. 480,330, 18.8.36).—A heating composition for a compressed  $\text{CO}_2$  blasting cartridge, which yields a large vol. of gaseous products and little or no residue on ignition, comprises an oxidising agent, e.g.,  $\text{KClO}_4$ , with which  $\text{NH}_4\text{NO}_3$  may be mixed, a fuel (salicylic acid), and 18–43% of non-combustible constituents, such as  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ ,  $\text{MgCl}_2$ ,  $\text{ZnSO}_4$ ,  $\text{NaHCO}_3$ , or  $\text{NH}_4\text{H}_2\text{PO}_4$ , to render the charge capable of rapid combustion when confined, but incapable of self-burning in the open. W. J. W.

**Slow-burning powders for fuses etc.** D. E. PEARSALL, Assr. to ENSIGN-BICKFORD Co. (U.S.P. 2,072,719—20, 2.3.37. Appl., [A] 10.5.35, [B] 11.9.36).—To ordinary black powder are added 9–30% of certain inert substances which delay burning; those claimed are: (A) oxides of Sn, Zr, Zn, Cu, Ti, Fe, Ni, etc. and sulphates of Ba or Ti etc., the preferred composition consisting of  $\text{TiO}_2$  with or without  $\text{BaSO}_4$ ; (B)  $\text{BaSO}_4$  or  $\text{PbSO}_4$ . D. M. M.

**Cotton fibre-like material from straw.**—See V.

### XXIII.—SANITATION; WATER PURIFICATION.

**Air conditioning with particular attention to effective temperatures.** W. L. FLEISHER (J. Inst. Heat. Vent. Eng., 1938, 6, 255–274).—A discussion. R. B. C.

**Air purification.** E. L. JOSEPH (J. Inst. Heat. Vent. Eng., 1938, 6, 170–199).—A review. R. B. C.

**Chemical purification of air [from viscose factories].** C. J. SNIJDERS (Chem. Weekblad, 1938, 35, 598–602).—Apparatus and methods are described for the determination of small amounts of  $\text{H}_2\text{S}$  and  $\text{CS}_2$  in the atm. effluent from viscose factories. In spite of the high  $[\text{CO}_2]$  in the effluent gases the most economical way of removing  $\text{H}_2\text{S}$  is to wash the gases at a high velocity (contact time  $>3$  sec.) in a spray tower with 6%  $\text{NaOH}$  (waste alkali from the dialysis batteries) so that the wash liquor running from the tower contains approx.  $\text{Na}_2\text{S}_2\text{O}_3$  3,  $\text{Na}_2\text{S}$  3,  $\text{Na}_2\text{CO}_3$  4, and  $\text{NaOH}$  0.5%. This liquor can be used in the preliminary bleaching process. S. C.

**Determination of oxygen in air.** A. ROMWALTER (Roy. Hung. Palatine-Joseph Univ. Tech. Sci., Publ. Dept. Min. Met., 1937, 9, 89–92).—A portable modification of the Lindemann-Winkler apparatus employing a P pipette is diagrammatically described. R. B. C.

**Technique for use of the impinger method [of dust sampling].** C. E. BROWN and H. H. SCHRENK (U.S. Bur. Mines, 1938, Inf. Circ. 7026, 20 pp.; cf. B., 1938, 330, 467, 851).—The procedure used by the U.S. Bureau of Mines in determining by the impinger method the no. concn. of dust in industrial atm. known or suspected to contain pneumoconiosis-producing material is described. H. C. M.

**Respirator (Mark IV) for use in dust-laden atmospheres.** J. A. SADD, H. L. GREEN, G.

DAVIES, V. S. NICHOLSON, A. S. G. HILL, and H. M. WATSON (Chem. and Ind., 1938, 781–791).—The mark IV dust respirator consists of a moulded rubber facepiece carrying two filter units, an outlet valve, and harness. The filter consists of long-fibre chrysotile asbestos (20%) and merino wool (80%) carded together. Various materials were tested for penetration by a C smoke penetrometer, though practical tests were made on  $\text{SiO}_2$  dusts. The aim was to obtain 95% protection against particles  $<3 \mu$ . diameter with a resistance  $<1$  in. on a  $\text{H}_2\text{O}$ -gauge and at a rate of flow of 3 cu. ft./min., and this was largely achieved. Penetration through any medium is a max. with particles of range 10–0.2  $\mu$ ., smaller particles being more rapidly pptd. by Brownian movement. C. I.

**Methods for detection of toxic gases in industry. Hydrogen cyanide vapour.** ANON. (Dept. Sci. Ind. Res., 1938, Leaf. 2, 9 pp.; cf. B., 1937, 846).—The most suitable tests for industrial detection of HCN are the Congo-red- $\text{AgNO}_3$  and benzidine- $\text{Cu}(\text{OAc})_2$  reactions, both applied on test-papers over which samples of the air are drawn. The former test is affected by presence of acid or alkaline gases and by tobacco smoke. The second test cannot be conducted in presence of  $\text{Cl}_2$ , Br, or  $\text{SO}_2$ ; HCl must be absorbed by a dry Congo-red test-paper and  $\text{H}_2\text{S}$  by a dry  $\text{Pb}(\text{OAc})_2$  paper before the vapours reach the HCN test-paper. Details are given of the methods of prep. of the test-papers and of conducting the tests so as to permit approx. determination of any [HCN] between 10 and 50 p.p.m. J. W. S.

**Cryolite spray residues and human health.** S. MARCOVITCH, G. A. SHUEY, and W. W. STANLEY (Tennessee Agric. Exp. Sta. Bull., 1937, No. 162, 46 pp.).—Limiting proportions of cryolite,  $\text{NaF}$ , and  $\text{CaF}_2$  in food or drinking- $\text{H}_2\text{O}$  necessary to cause striation in rat teeth are determined. Fluorides ppt. Ca in the body, probably as apatite or fluophosphates of Ca or Mg. Addition of  $\text{Ca}_3(\text{PO}_4)_2$  to  $\text{H}_2\text{O}$  containing  $\text{CaF}_2$  diminishes the sol. F' content. Toxicity of  $\text{NaF}$  in  $\text{H}_2\text{O}$  supplied to plants is lowered by addition of P fertilisers but increased by alkali-metal salts. Injury to public health through use of F-containing insecticides is improbable. A. G. P.

**Testing fly sprays: metal turntable method for comparative tests of liquid spray contact insecticides.** F. L. CAMPBELL and W. N. SULLIVAN (Soap, 1938, 14, No. 6, 119–125, 149).—Suitable apparatus is described. With atomised kerosene-base (pyrethrum) sprays, high spraying pressure or delay in exposure of Petri dishes to the settling mist reduced the quantity of oil and the mean size of droplets deposited during a 10-min. exposure. A. G. P.

**Roaches: relation between the ages of cockroaches and their resistance to liquid insecticides.** V. TUMA (Soap, 1938, 14, No. 6, 109–117, 151).—The toxicity of aliphatic thiocyanate and pyrethrum preps. toward the cockroach, *Blattella germanica*, at different ages is determined. Max. resistance is attained at 17 weeks. Results of tests with roaches 17 weeks old bear no relation to tests made with those of mixed ages. A. G. P.



**Test methods on roaches.** E. N. WOODBURY (Soap, 1938, 14, No. 8, 86—90, 107, 109).—Suitable apparatus and a method for rearing large nos. of cockroaches (*Blatella germanica*) of known age, and a settling-mist method for testing kerosene-base insecticides on these insects, are described. The behaviour of different insecticides and the resistance of the insects of different ages and sexes are studied.

E. L.

**Transport and control of *Aedes aegypti* in airplanes.** E. P. HICKS and S. D. CHAND (Rec. Malaria Surv. India, 1936, 6, 73—90).—Spraying with pyrethrum in kerosene was effective in airplanes, baggage rooms, etc.

CH. ABS. (p)

**Cattle sprays: their composition and application.** E. M. SEARLS and F. M. SNYDER (Soap, 1938, 14, No. 8, 103, 105, 107).—The general composition and methods of application of the insecticidal and fly-repelling sprays used against (a) flies (usually of the non-biting type) frequenting the stables, milking-sheds, etc., and (b) biting flies molesting the cattle in the pastures, are discussed.

E. L.

**Ventilation of percolating filters [in sewage purification].** E. J. HAMLIN and H. WILSON (J. Proc. Inst. Sewage Purif., 1937, Pt. II, 32—57).—The comparative efficiencies of the Prüss enclosed filter and the open percolating filter working under identical conditions to give an effluent of 100% stability, using an extremely conc. septic sewage, are being studied. The enclosed filter is less sensitive to cold winds and operates at 3—4 times the rate of the open filter, but before definite conclusions can be given more work is necessary for which larger filters are to be constructed.

O. M.

**Stream pollution. Factors affecting the aerobic decomposition of sewage-sludge deposits.** H. G. BAITY (Sewage Works J., 1938, 10, 539—568).—The polluting effect on streams of sludge banks of org. matter deposited from the discharge of sewage was investigated and the method and technique are described. From the tests the following conclusions are indicated. The total  $O_2$  demand of a sludge film 0.089 cm. thick was 540,000 p.p.m. on the total dry solids (or 635,000 p.p.m. on the org. content) and was absorbed at an average rate of 632 mg. of  $O_2$ /day/sq. m. of surface area (or 17.6 mg./day/g. of org. solids) and required 36 days for complete stabilisation at 21.2°. The rate of  $O_2$  demand is not related to the content of dissolved  $O_2$  of the supernatant  $H_2O$ , but the oxidation proceeds normally so long as dissolved  $O_2$  is available, and after a period of septicity aerobic activity is resumed when  $O_2$  is again available. The depth of sludge deposit does not directly affect the  $O_2$  demand, but is expressed as  $ax^b = y$ . Aerobic sludge decomp. is a surface phenomena, the  $O_2$  diffusing into the sludge to only about 1 cm., below which anaerobic conditions exist, evolving gases which disrupt the sludge deposit and increase the  $O_2$  demand. Sunlight has no influence on the organisms, but in natural stream conditions algal growths would increase the dissolved  $O_2$ . Temp. rise (within the natural stream limits) causes increased aerobic decomp. In highly saline waters (tidal) the  $O_2$  absorption is  $\ll$  in fresh  $H_2O$ . The bacterial and

protozoal life are not much affected by light, salinity, or sludge depth, but are influenced by temp. O. M.

**Chemical coagulation of sewage. IX. Order of addition of chemicals.** W. RUDOLFS and H. W. GEHM (Sewage Works J., 1938, 10, 450—457; cf. B., 1937, 1413).—Experiments with coagulants on different types of sewage (e.g., fresh, septic, dil., conc., and trade waste) show that the best results are obtained with  $Ca(OH)_2$  and  $Fe^{III}$  (or alum) when the  $Ca(OH)_2$  is added first, due to the formation of a denser and more rapidly settling floc, whilst with acid and  $Fe^{III}$  (or alum) addition of the  $Fe^{III}$  before the acid gave better results. Alum gave more uniform results than  $Fe^{III}$  with the different types of sewage as the  $Fe^{III}$  is liable to reduction by the sewage constituents. The  $Ca(OH)_2$  and acid function more as coagulants than as  $pH$  adjusters, and appear to be used as adjuncts to the coagulants to meet certain transient conditions.

O. M.

**[Sewage-]sludge digestion and inspection of tanks at Rockville Centre, N.Y.** C. G. ANDERSEN (Sewage Works J., 1938, 10, 494—502).—The sludge-digestion plant and its operation are discussed. After 8 years' continuous use the concrete and iron-work inside were found to be in good condition.

O. M.

**Elimination of the detritus dump [in sewage purification].** C. B. TOWNEND (J. Proc. Inst. Sewage Purif., 1937, Pt. II, 58—87).—The dumping of foul detritus and screenings is caused primarily by the standard size of detritus tank being too large (i.e., 1/50th of the daily dry weather flow); it can be remedied by smaller and quicker-flowing grit tanks yielding an inoffensive clean grit, together with comminution and digestion of the screenings. Where sedimentation tanks are mechanically cleaned and sludge is handled by centrifugal pumps, screening is advisable to remove rags, which can be disposed of by incineration or digestion. Grit chambers are constructed either so that a fine control of the flow results in a clean grit, or, if this is impracticable, so that the org. matter is subsequently washed out. If immediately below the grit chambers a Venturi flume records the vol. of flow, it will control the level in the grit chamber corresponding with the vol. discharged. In such a multiple-unit system each chamber, or a group of chambers, may be controlled by a flume so that automatically in dry weather only the min. no. of chambers will operate and shallow depths at low flows be avoided, whilst peak storm flows will be treated by the whole battery. The grit chambers at the Mogden plant, designed to treat a max. flow of  $575 \times 10^6$  gals./day are subdivided into 12 equal units (90 ft. long, 1.5 min. detention period, and having parabolic-shaped ends 15 ft. across) arranged in pairs governed by a flume. This allows a particle to fall the 9 ft. depth at a velocity of 0.1 ft./sec. The grit is removed by dredging, and after washing contains ~ 98% of inorg. matter. The cost of detritus handling is reduced considerably by using grit chambers, due to the smaller quantity to be handled.

O. M.

**Influence of oxygen concentration on biochemical oxygen demand [of sewage and efflu-**

ents]. R. POMEROY (*Sewage Works J.*, 1938, 10, 465—481).—The biochemical  $O_2$  demand of diluted sewage and partly oxidised effluents decrease as the dissolved  $O_2$  concn. increases, throughout the entire range (0.8—40 p.p.m.). The rate of  $O_2$  consumption decreases 20% with filtered sewage samples and 37% for partly oxidised effluents when the  $[O_2]$  increases from 6 to 36 p.p.m. Sierp's work on the relative effect of air and  $O_2$  in determining biochemical  $O_2$  demand of activated sludge and sewage, and Heukelekian's work on the effect of  $[O_2]$  on bacterial counts are discussed. O. M.

**Oxidation of sewage.** F. R. O'SHAUGHNESSY and S. J. ROBERTS (*J.S.C.I.*, 1938, 57, 281—285).—A review of the evidence relating to the org. matter which escapes oxidation during sewage purification and its effect on the later stages of purification. Experiments show that in the biochemical  $O_2$  demand test "humus" solids which separate from the liquor are responsible for nitrification. If conditions are such that these solids are present at the start, then nitrification and C oxidation proceed simultaneously.

[Operation of] the activated-sludge process [at Mogden Sewage-Purification Works]. W. T. LOCKETT (*J. Proc. Inst. Sewage Purif.*, 1937, Pt. II, 88—112).—For the year June 1st, 1936—May 31st, 1937, the plant dealt with a daily average of 60,020,000 gals. of sewage, producing a non-putrescible, well-nitrified effluent. The purification (96% on biochemical  $O_2$  demand), the high-density, rapidly-settling sludge, the low proportion of surplus sludge produced to the vol. of sewage treated (0.79%), and the average air pressure ( $6\frac{1}{4}$  lb./sq. in.) are all noteworthy features. The air consumption [1.57 cu. ft./gal. on dry-weather flow (D.W.F.)] and the detention period (6.46 hr.) are above actual requirements for the production of a nitrified effluent, but allow an ample margin of safety. The sludge is low in grease (5.1%  $Et_2O$ -extractible), high in N, and high in protozoal life, with but few filamentous types. High nitrification is found to correspond with high-density sludge and no trouble from "bulking" or "rising" has been experienced. The vol. of sludge returned to sewage (50% D.W.F.) ensures a short detention period for it in the final separating tanks, thereby obviating loss of activity, odour, "rising," and a too high concn. in the re-aeration units for efficient aeration. Also the high  $H_2O$  content ensures substantial dilution of incoming sewage. "Rising" of sludge with nitrified effluents is thought to be due to incompletely activated sludge being mixed with activated sludge in the separating tanks and should therefore be remedied by thorough conditioning of the sludge in the mixed-liquor channels before settlement, elimination of short-circuiting, and reduction to a min. of the period of non-aeration (i.e., during settling). The disadvantages of complete treatment are longer detention periods and greater air consumption, whilst the advantages are a uniformly dense sludge facilitating operation of the plant, freedom from bulking, large reduction in the bulk of surplus sludge, freedom from odours, a nitrified effluent for preservation of river conditions, greater

capacity for overcoming overloads, and a sludge more amenable to dewatering. O. M.

**Clarification stage of the activated-sludge process. VIII. Uptake of soluble organic substances.** R. S. INGOLS (*Sewage Works J.*, 1938, 10, 458—464; cf. B., 1938, 987).—As with  $NH_3$ , the removal of sol. org. substances by activated sludge is brought about by biological or biochemical processes, independently of purely physical phenomena; thus it is shown, using a hydrolysate of casein as representative of sol. N org. compounds, that neither adsorption nor base exchange but purely biological activity occurs. Likewise, using glucose as representative of the labile C compounds, it is shown that its utilisation is due to enzyme action, dependent on adsorption at sp. activated surfaces. O. M.

**Effects of activated carbon in digestion of fresh solids-activated sludge mixtures.** G. E. FLOWER, C. B. BUDD, and C. HAUCK (*Sewage Works J.*, 1938, 10, 441—449).—An attempt is made to explain the widely different reports appearing in the literature on the effect of activated C in sewage treatment on sludge digestion, gas production, drainability of digested sludge, etc. Experiments at Cleveland, Ohio, show that activated C had little effect on the  $p_H$  of digested sludge, that it reduces and controls odour, that a definite optimum dosage exists for the best settlement, and that it may reduce the quantity of precipitants required. Some evidence points to enhanced digestion and gas production. O. M.

[Sewage] sludge drying on open and covered drying beds. L. V. CARPENTER (*Sewage Works J.*, 1938, 10, 503—512).—Drying depends on drainage and evaporation. Drainage is a sedimentation process and can be improved by using shallower sand beds with free under-drainage. Evaporation includes evaporation from sludge and the removal of moist air. Laboratory tests indicate that the higher is the initial  $H_2O$  content, the larger is the % of  $H_2O$  removed by draining, which can be increased by constructing the beds to have bottom-ventilation. The drying on glass-covered beds of 0.5 sq. ft. *per cap.* capacity at 4 different Long Island works of different types of digested sludges (heated separate digestion, activated sludge, and Imhoff sludge) are described. If the sludge were removed at 75% of  $H_2O$ , it could be satisfactorily dried with only 0.25 sq. ft. *per cap.* O. M.

**Photo-electric apparatus for measurement of turbidity in sewages and effluents.** J. M. WISHART (*J. Proc. Inst. Sewage Purif.*, 1937, Pt. II, 115—128).—The intensity of light passing through a sample of effluent etc. is registered by a photo-electric cell on a micro-ammeter. The apparatus is set, using an iris diaphragm with distilled  $H_2O$  as standard, and the difference in transparency between turbid samples and distilled  $H_2O$  is determined. For turbidities the apparatus can be calibrated against fuller's earth, bentonite, etc., but the use of an independent rational standard is proposed. As turbidity depends on particle size, the sewage samples are emulsified for uniformity in working. O. M.

**Water-main contamination traced to jute packing.** G. E. ARNOLD (West. Construction News, 1936, 11, 36—37).—Jute is a source of contamination in new mains. Sterilisation by pressure steam is recommended. Chlorination is unsatisfactory.

CH. ABS. (p)

**Corrosion and the formation of protective coatings in water pipes.** J. KOONJMAN (Gas-u. Wasserfach, 1938, 81, 611—615, 628—633).—The  $\text{Ca}(\text{HCO}_3)_2$  equilibrium is affected by org. matter so that data determined with synthetic hard  $\text{H}_2\text{O}$  are not generally applicable to practical conditions (cf. B., 1929, 266). Analyses of several coatings all show much more Fe than Ca, but the theory that  $\text{CO}_2$  is adsorbed from the solution by  $\text{Fe}(\text{OH})_3$  (A., 1929, 257) is rejected and it is suggested that the discharge of H ions which occurs on dissolution of Fe causes the conversion of  $\text{HCO}_3^-$  into  $\text{CO}_3^{2-}$ , with pptn. of  $\text{CaCO}_3$  in amorphous or cryst. form according to the velocity of the process, the latter form yielding hard, adherent coatings.

A. R. PE.

[Water-pipe] corrosion control studies and operating experiences. I. Engineering and economic aspects of pipe corrosion and its control. T. H. WIGGIN. II. Effectiveness of chemical treatment for control of pipe corrosion. G. D. NORCOM. III. Calcium carbonate units and corrosion control. I. M. GLACE. IV. Correcting corrosivity of zeolite-softened water. D. E. DAVIS. V. Experience with zeolite water-plant. H. M. OLSON. VI. Accomplishments with chloramination. J. W. ACKERMAN. VII. Use of sodium silicate in corrosion control at a rayon plant. C. ALEXANDER (J. Amer. Water Works Assoc., 1938, 30, 1342—1352, 1353—1365, 1366—1374, 1375—1383, 1384, 1385, 1386—1387).—I. Internal corrosion of pipes is often not recognised, as absence of "red  $\text{H}_2\text{O}$ " etc. is taken to indicate freedom from corrosion instead of measurements of the flow coeffs. of the pipes. Treatment of  $\text{H}_2\text{O}$  with  $\text{CaO}$  and  $\text{Na}_2\text{CO}_3$  usually prevents the appearance of "red  $\text{H}_2\text{O}$ ," but not the reduction of the flow capacity of the pipes by tuberculation, which latter results in economic losses. The use of non-corrosive, cement-lined and tar-enamel pipes, or of concrete or asbestos-cement pipes, is suggested. Annual cleaning is necessary to maintain the high flow capacity of the pipes since tuberculation rapidly re-forms.

II. Internal corrosion of pipes has two general effects: (a) dissolution of the Fe from the pipes, thus increasing the Fe content and producing "red  $\text{H}_2\text{O}$ " etc.; (b) tuberculation and rusting of pipes, which may occur with no increase of Fe content, the products of corrosion being retained on the pipe wall. Neutralisation of free  $\text{CO}_2$  with  $\text{CaO}$  to  $p_{\text{H}}$  8.0—8.2 usually prevents "red  $\text{H}_2\text{O}$ " but not tuberculation. Corrosion is favoured by high  $\text{O}_2$  content, high  $\text{CO}_2$  (particularly in soft  $\text{H}_2\text{O}$ ), low  $p_{\text{H}}$ , high Cl<sup>-</sup>, and high-velocity turbulence, and is retarded by hard  $\text{H}_2\text{O}$  (buffer salts), high org. matter, high  $p_{\text{H}}$  (especially in presence of  $\text{CaCO}_3$ ), and presence of rust and protective coatings. Langelier's equation to calculate whether the  $p_{\text{H}}$  of a  $\text{H}_2\text{O}$  is > or < that of  $\text{CaCO}_3$  saturation (*i.e.*, whether it tends to deposit

or dissolve  $\text{CaCO}_3$ ) requires numerous complicated analyses and ignores the  $\text{O}_2$  and org. matter content; for practical purposes, therefore, Hoover's marble test appears to be superior. Alkali treatment may be included to control "red  $\text{H}_2\text{O}$ " or to produce a protective coating ( $p_{\text{H}}$  8.0—9.6), but the old deposits and tubercles are simply coated over and the flow capacity of the pipe is not restored. Observations of the effect of chemical treatment on a newly-cleaned pipe system show that corrosion occurs with no "red  $\text{H}_2\text{O}$ " production or increase of Fe content of the  $\text{H}_2\text{O}$ , and that a coating spreads over the old scale but corrosion continues within the tubercles, and that mechanically cleaned mains corrode much more rapidly than those not cleaned, as chemically induced coatings were absent in the cleaned pipes.

III. The efficiency of  $\text{CO}_2$  removal from  $\text{H}_2\text{O}$  by passage through  $\text{CaCO}_3$  filters depends on the size of the  $\text{CaCO}_3$  chippings, the temp., and the  $\text{CO}_2$  and alkalinity contents, and in practice takes  $\frac{1}{2}$ —3 hr. Many Pennsylvania  $\text{H}_2\text{O}$  supplies (*e.g.*, Dillsburg) high in  $\text{CO}_2$  and having low alkalinity and no hardness are very corrosive and tuberculating even after aeration to 3—5 p.p.m. of  $\text{CO}_2$ , but after  $\text{CaCO}_3$  treatment they cause no "red  $\text{H}_2\text{O}$ " complaints.

IV. The Ca content and  $p_{\text{H}}$  are the factors mostly affecting corrosion by zeolite-treated  $\text{H}_2\text{O}$ , the ionic exchange of Ca for Na tending to make it more corrosive than the raw  $\text{H}_2\text{O}$ ; this can be offset by increasing the  $p_{\text{H}}$ . Waters having a Langelier saturation index of < -0.6 do not produce "red  $\text{H}_2\text{O}$ ," but cause tuberculation and loss of carrying capacity. If this index is above -0.6,  $\text{Na}_2\text{CO}_3$ ,  $\text{NaOH}$ ,  $\text{Na}_2\text{SiO}_3$ , etc. must be added to raise the  $p_{\text{H}}$  and prevent "red  $\text{H}_2\text{O}$ " formation, whilst for protection of the carrying capacity of the pipes a positive index is necessary.

V. A zeolite system should be run undersaturated until the scale within the pipes dissolves, and then  $\text{CaCO}_3$  should be corr. to protect the pipes and maintain their full capacity.

VI. Application of  $\text{Cl}_2$  and  $\text{NH}_3$  to a pipe that previously had to be cleaned frequently has maintained its carrying capacity.

VII.  $\text{H}_2\text{O}$  of low hardness but high in sol. silicates (50—60 p.p.m. of  $\text{SiO}_2$ ) forms excellent protective films.

O. M.

**Developments in the chemistry of rubber and like substances in relation to waterworks practice.** E. A. HAUSER (J. New England Water Works Assoc., 1938, 52, 254—255).—Uses of some known new rubber-like synthetic org. products are discussed, *e.g.*, Neoprene for lining rubber hosepipes for transportation of corrosive liquids, Thiokol for jointing of bell and spigot pipes, "Simplex" couples for jointing "Transite" (asbestos-cement) pipes, and pipelines etc. made from Mipolam (a polyvinyl synthetic resin).

O. M.

**Sterilisation and cleaning of a well with chlorine and Dry Ice.** M. SUTER (J. Amer. Water Works Assoc., 1938, 30, 1130—1132).—A well (250 ft. deep, 6 in. diameter) blocked by org. filth was satisfactorily cleaned by additions of conc. hypochlorite

solutions, stirred by dropping in blocks of Dry Ice (solid  $\text{CO}_2$ ). O. M.

**Pollution and natural purification of Illinois River below Peoria.** W. H. WISELY and C. W. KLASSEN (*Sewage Works J.*, 1938, 10, 569—595).—The effects of pollution from sewage and trade wastes of Peoria and Pekin on the river during July and August, 1936 (dry period), were surveyed. The excessive strong trade wastes from distilleries etc. was  $\approx 10^6$  population, whilst the effluent from the sewage (130,000 population) was  $\approx 12,000$  people and was negligible. The pollution load was calc. from the vol. and concn. of the wastes, and likewise from the flow velocity, dissolved  $\text{O}_2$ , biochemical  $\text{O}_2$  demand, temp., etc. of the river at different points the rate of de- and re-aeration,  $\text{O}_2$  balance, and  $\text{O}_2$  demand of the sludge deposits were computed. The portion under survey had a total re-aeration capacity of about 526,000 lb. of  $\text{O}_2$  per day, whilst the total biochemical  $\text{O}_2$  demand was 667,000 lb. per day. This unfavourable dissolved- $\text{O}_2$  balance was due largely to the presence of sludge deposits, which had an  $\text{O}_2$  demand of about 263,000 lb. of  $\text{O}_2$  per day, and, having accumulated, exerted their greatest demand at the time of low summer flow, and it was computed that had they been absent the  $\text{O}_2$  condition of the river would have been satisfactory. A permissible pollution limit was computed to be 292,000 lb. of  $20^\circ$  biochemical  $\text{O}_2$  demand per day, which should not have caused dissolved  $\text{O}_2$  depletion to  $< 4.0$  p.p.m. provided org. matter capable of creating sludge deposits was very limited in the discharges. To prevent these crit. river conditions the max. removal of settleable org. solids and reduction of non-settleable org. matter will be necessary. Above Peoria the stream was in satisfactory condition although receiving the Chicago wastes. O. M.

**Specifications and tests for powdered activated carbon. Final report of the Sub-Committee.** WATER PURIFICATION DIVISION, and WATER WORKS PRACTICE and AMER. WATER WORKS ASSOC. COMMITTEES (*J. Amer. Water Works Assoc.*, 1938, 30, 1133—1224).—A Committee record of the current information regarding the manufacture, physical and chemical characteristics, and use in  $\text{H}_2\text{O}$  purification of activated C, together with tentative recommendations for specifications, tests, evaluation, purchase, and general precautions, all of which are fully discussed. It is proposed to limit the tests to those of PhOH adsorption, threshold odour, moisture,  $d$ , and fineness, which are described in detail. The PhOH test is for use in evaluation, but may be supplemented by the threshold-odour test wherever local conditions make it necessary. Specimen specifications, contracts, basis of payments, price adjustments, and rejections are appended. O. M.

**Specifications and tests for powdered carbons [for water purification].** L. C. BILLINGS (*J. Amer. Water Works Assoc.*, 1938, 30, 1234—1237).—As the manufacture and use of activated C for such purpose are still developing, the Sub-Committee's report should be included in waterworks' standards only as tentative provisions and amended only as progress occurs. O. M.

**Comparison of tests for activated carbons [for water purification].** W. A. HELBIG (*J. Amer. Water Works Assoc.*, 1938, 30, 1225—1233).—The threshold-odour and phenol-adsorption tests are compared. Direct evaluation of activated C for taste and odour removal on the particular  $\text{H}_2\text{O}$  sample is reliable and more rational than the indirect phenol-adsorption method, as the tastes and odours to be removed are often not phenolic. Several discrepancies of the phenol-adsorption test are discussed. O. M.

**Activated carbon application efficiency [in water purification].** M. C. SMITH (*J. Amer. Water Works Assoc.*, 1938, 30, 1325—1334).—The more efficient use of activated C is discussed, and addition of C before filtration instead of before or after coagulation is suggested; this treatment is found to give a 90% saving in C required and to increase the filter runs at Richmond.

In a discussion, J. R. BAYLIS suggests that the saving effected at Richmond by adding C to the filters is probably due to the amount of odour constituents being reduced by coagulation and sedimentation, and can not be duplicated at many other places. O. M.

**Taking the guesswork out of activated carbon dosage [in water purification].** W. A. HELBIG (*J. Amer. Water Works Assoc.*, 1938, 30, 1320—1324).—Characteristic isotherms of any particular activated C are obtained by plotting the logarithmic vals. for the threshold odour test in Freundlich's adsorption equation  $\log (X/M) = \log K + n^{-1} \log C$ , where  $X$  units of odour are removed by  $M$  units of activated C dosage, leaving  $C$  as the residual odour concn., and  $K$  and  $n$  are consts. depending on the nature of the C, the odour impurity to be removed, and the liquid (*e.g.*,  $\text{H}_2\text{O}$ ) treated. The isotherms can be used to determine the C dosage required to remove a given intensity of odour, for comparisons and selection of carbons, and for maintaining uniformity of supplies. O. M.

**Removal of iron from and two methods of purifying water.** ANON. (*Text. Manuf.*, 1936, 62, 154).—Certain synthetic phenolic and tannin resins remove Ca and Mg as effectively as customary  $\text{H}_2\text{O}$ -softening agents. Resins from aromatic bases (*e.g.*,  $\text{NH}_2\text{Ph}$  resin) remove anions. A prep. which removes Fe is recorded. CH. ABS. (p)

**Laboratory control [of water purification].** C. R. COX (*Water Works Eng.*, 1936, 89, 132—134, 193—195, 253—256, 315—317, 364—366, 437—438, 488—490).—Methods of analysis, of control of chlorination processes, and of elimination of tastes and odours are reviewed. CH. ABS. (p)

**Economics of water purification.** A. L. SHAW and E. S. CHASE (*J. New England Water Works Assoc.*, 1938, 52, 131—150).—The monetary evaluation of waters free from disease, taste, colour, turbidity, corrosivity, hardness, Fe and Mn., etc. is attempted and it is shown that purification is economically justifiable if the total costs of purification of waters are  $<$  the economic losses due to impairments. The economics of  $\text{H}_2\text{O}$ -purification processes are discussed. O. M.

**Improvements in the soap-solution method for determining hardness of water.** K. W. BROWN and P. A. VILLARRUZ (J. Amer. Water Works Assoc., 1938, 30, 1021—1039).—To stabilise and clarify the standard alcoholic soap solution it is set aside for several days in the refrigerator, then filtered therein to remove undissolved colloidal particles, and afterwards standardised so that 6.4 ml. = 20 ml. of standard aq.  $\text{CaCl}_2 = 80$  p.p.m. of  $\text{CaCO}_3$ , and not diluted to 1 ml. = 1 mg. of  $\text{CaCO}_3$ . The effects of hydrolysis (which sets in if the alcoholic concn. is < 24%), temp. (negligible between 15° and 35° if standardised at 20°), acidity (neutralised by NaOH), false (or ghost) end-points, heavy metallic ions (e.g., Cu, Fe, Pb, Sn, etc.), and particularly Ca and Mg are discussed. The soap method for determining total hardness is correct up to 90 p.p.m. of Ca alone, but as Mg alone consumes 1.4 times more soap than does Ca, it gives high results. When Ca and Mg are present together, if the Mg : Ca ratio is < 1 : 3 the results are low and if > 1 : 3 they are high. O. M.

**Direct determination of ammonia in water.** J. C. HARRAL (Analyst, 1938, 63, 597—598).— $[\text{Cl}_2] > 50$  p.p.m. causes no error in determinations of  $\text{NH}_3$  by Kitto's method (cf. B., 1938, 737), but 180 p.p.m. cause complete loss of  $\text{NH}_3$ . Darkening of the  $\text{H}_2\text{O}$  when treated with  $\text{K}_2\text{S}_2\text{O}_8$  and acid is due to the presence of Mn, and in such cases it is advisable to add a crystal of  $\text{H}_2\text{C}_2\text{O}_4$  to the warm solution after treatment with  $\text{K}_2\text{S}_2\text{O}_8$  and to add Rochelle salt before the Nessler reagent. J. W. S.

**Determination of calcium and magnesium by soap titration.** E. L. BREAZEALE and R. A. GREENE (J. Amer. Water Works Assoc., 1938, 30, 1040—1048).—The use and limitations of the direct soap-titration method for determining small amounts of Ca and Mg in  $\text{H}_2\text{O}$  and soils are discussed. Ca is determined in presence of alkaline tartrate, and Mg in presence of alkaline  $\text{NH}_4\text{Cl}$ , care being taken not to over-titrate. Satisfactory results are obtained if the Mg : Ca ratio is < 1 : 2, but at higher ratios high Ca and low Mg results are obtained. This can be overcome by adding excess of standard aq.  $\text{CaCl}_2$  to increase the Ca : Mg ratio to approx. 4 : 1. Presence of relatively large amounts of salts interferes with the separation of Ca and Mg. O. M.

**Micro-determination of calcium in water.** E. L. BREAZEALE and R. A. GREENE (J. Lab. clin. Med., 1938, 23, 845—847).—The Clark-Collip modification of the Kramer-Tisdall method may be applied directly to waters of > 16—20 p.p.m. of Ca. C. J. C. B.

**Palmitate determination of magnesia in water.** H. ATKINSON (Analyst, 1938, 63, 493—494).—The drawbacks to Kitto's method (B., 1938, 737) are that the end-point of the total hardness determination is indefinite and that the Ca tends to be carried down with the Mg in presence of considerable proportions of the latter. It is better to ppt. the Mg from boiling solution within a definite  $p_{\text{H}}$  range (9.5—10.0). By titrating the filtrate with standard acid the Mg can then be determined fairly accurately. T. H. G.

**Determination of lead in drinking water.** H. INGLESON (Analyst, 1938, 63, 546—560).—The most satisfactory filter medium for absorbing Pb is a  $\text{CaCO}_3$ -MgO mixture. After passing a known vol. of  $\text{H}_2\text{O}$  through a filter packed with this mixture, under the conditions of normal domestic use, the absorbent is dissolved as far as possible in a min. of  $\text{HNO}_3$ . The solution is made just alkaline with  $\text{NH}_3$ , and  $\text{K}_2\text{CrO}_4$  is added to ppt. Pb and Cu, which are filtered off. The ppt. is dissolved in dil. HCl and taken to dryness repeatedly with conc. HCl, after which it is extracted with hot  $\text{H}_2\text{O}$ . The residue is added to the material insol. in  $\text{HNO}_3$ , treated with HF to remove Si, and combined with the Pb and Cu solution. These metals are then pptd. with  $\text{H}_2\text{S}$ , the ppts. redissolved in  $\text{HNO}_3$ , and Pb is determined by anodic deposition. J. W. S.

**Bionomics of *Anopheles sundaicus* (*A. ludlowi*) in salt lakes of Calcutta.** S. K. NEOGI (Rec. Malaria Surv. India, 1936, 6, 31—47).—Breeding areas of the mosquitoes include those having salinity of 7.8—2675 pts. of NaCl, and albuminoid  $\text{NH}_3$  0.002—1.2 pts., per 100,000 of solution. Breeding is closely correlated with salinity but not with albuminoid  $\text{NH}_3$  content, and is favoured by presence of algae. No relation between salinity and occurrence of algae is apparent. CH. ABS. (p)

**High-efficiency filter beds for industrial effluents.** W. HUSMANN (Gesundheitsing., 1938, 61, 148—150).—Effluents from coal-hydrogenation plants with high contents of  $(\text{NH}_4)_2\text{S}$  and phenols (typical analysis given) can be satisfactorily purified by means of filter-beds if first diluted with domestic effluents. A loading ratio of mixed effluents : filter material of 1.4 : 1 gives the best results. R. B. C.

**Textile waste disposal.** L. L. HEDGEPEETH and C. B. GRIFFIN, jun. (Sewage Works J., 1938, 10, 534—538).—Textile wastes and their disposal are reviewed. O. M.

**Treatment of waste waters from dairies.** S. H. JENKINS (J. Proc. Inst. Sewage Purif., 1937, Pt. II, 13—31).—Large-scale experiments show that milk washings diluted to a biochemical  $\text{O}_2$  demand of 25 pts. per  $10^5$  can be purified either by double filtration, using two filters alternating as primary and secondary units every three weeks, or by the activated-sludge process after dilution to a biochemical  $\text{O}_2$  demand of 50 pts. per  $10^5$ . Double filtration appears to be more suitable for the milk industry. In like manner admixtures of whey washings (from cheese manufacture) with milk washings can be purified. In this case a different kind of growth (tougher, more leathery and stringy, and more difficult to remove) was noted, due probably to the acids and sugars of the whey. O. M.

**Treatment of dairy waste waters.** G. GÉNIN (Lait, 1938, 18, 711—714).—A review. W. L. D.

**Erratum :** On p. 738, col. 2, line 9, for INGOTS read INGOLS.

**Dehumidification of air.**—See I. Analysis of HCN-air mixtures.—See VII.

See also A., I, 470, Determining  $\text{SO}_2$  in air. 471, Determining P in natural waters.

## PATENTS.

**Dental preparation.** M. SAHYUN (U.S.P. 2,069,157, 26.1.37. Appl., 4.12.33).—Aq. pastes of soap, abrasive, glycerin, and basic and acidic buffers which will resist the acid or alkaline conditions which may be present in the mouth, and maintain  $p_H$  7—9, e.g.,  $Na_3PO_4 + NaH_2PO_4$ , are specified. E. H. S.

**Air-filtering apparatus.** N. DANILS (B.P. 488,152, 19.11.37).—Filter screens are radially arranged and are rotated counter to the flow of air in the top 180° (approx.) in which zone filtering takes place; the dirt mostly drops off in the next 45°, and the remainder is shaken off by a blast admitted through the remaining 45° approx. B. M. V.

**Filtering material for smokes for gas masks.** R. P. HOWES (B.P. 487,777, 19.12.36).—Fine feathers are impregnated with lampblack; they are not felted or stuck together, but may be impregnated with other materials also if desired. B. M. V.

**Emulsifiable solutions of sulphur dioxide [as insecticide sprays etc.].** K. T. STEIK and J. F. MULLER, Assrs. to NAT. OIL PRODUCTS CO. (U.S.P. 2,068,089, 19.1.37. Appl., 27.8.34).—A solution of  $SO_2$  in a mixture of dry mahogany soaps and paraffin or castor oil is stable, non-oxidising, non-corrosive, and can readily be emulsified with  $H_2O$  to form insecticide sprays etc. E. L.

**Preparations for protection from insects.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 488,519, 7.1.37. Addn. to B.P. 473,592; B., 1938, 112).—Preps. for warding off insects are made by incorporating cinnamyl alcohol or its ethers or esters or  $CH_2Ph \cdot OBz$  with a  $H_2O$ -sol. alkaline-earth salt, e.g.,  $SrBr_2$ ,  $CaCl_2$ ,  $MgSO_4$ , in a conc. org. solvent (an alcohol), e.g.,  $EtOH$ ,  $Pr^iOH$ , glycerol, containing, if desired,  $>15\%$  of  $H_2O$ . N. H. H.

**Manufacture of highly concentrated vermin-destroying agents and the like, emulsifiable in water.** DEUTS. HYDRIERWERKE A.-G. (B.P. 487,503, 21.12.36. Ger., 21.12.35).—Clear, highly conc. solutions, emulsifiable in  $H_2O$ , of  $H_2O$ -insol. vermin-destroying agents, viz., vegetable poisons, e.g., rotenone (I), pyrethrins, CNS-compounds, are prepared by dissolving the latter, together with a quaternary  $NH_4$  compound having an alkyl residue of  $<C_6$  attached to  $N^+$ , in an org. solvent which is immiscible with  $H_2O$ , e.g.,  $CHCl_3$ ,  $C_2HCl_3$ ,  $CCl_4$ ,  $C_6H_6$ . An example is (I) (35) and  $NMe_3Cl \cdot C_{16}H_{33}$  (II) (25) are dissolved in  $CHCl_3$  (50 pts.). Other agents claimed are  $NMe_3X \cdot C_{18}H_{37}$  ( $X = Cl$  or  $Br$ ) and the methosulphate from (II). N. H. H.

**Sewage treatment.** E. B. MALLORY, Assr. to LANCASTER IRON WORKS, INC. (U.S.P. 2,078,260, 27.4.37. Appl., 18.2.36).—A septic tank of sheet metal for individual houses is provided with an automatic, aperiodic discharge siphon which empties a minor compartment of the tank. B. M. V.

**Treatment by fermentation of waste refuse and of vegetation.** SOC. EAU ET ASSAINISSEMENT ANC. ETABL. C. GIBAUT, and P. E. G. CHIDAINE (B.P. 490,426, 12.2.37).—The open fermentation cell described and claimed is provided with a false bottom, e.g., a cloth-covered one, which retains even the

smallest particles of refuse but allows air and  $H_2O$  to pass. Air is sucked uniformly downward through the mass of crude refuse or vegetation, and  $H_2O$  (at  $p_H$  7.5—8.0) or waste (sewage) liquor is sprayed over. Purification of sewage liquor occurs when fermentation is started, and such liquor draining off may be returned by spraying until sufficiently purified for discharge. A temp. of 65—70° is maintained. The gases produced may be led to a furnace or its chimney. Towards the end of the process spraying is discontinued but aëration continued to permit dehydration, the product being passed over an arrangement of sieves for separation of mould for fertiliser, inert material, and combustible material (ash is returned later for enrichment of mould). Numerous arrangements of plant for controlled operation are described, and provision is made for continuous operation and for enrichment of the mould with mineral fertilisers. Warm  $H_2O$  may be used for starting in cases of exceptional cold. I. A. P.

**Purification of water.** A.-G. F. HYDROLOGIE (B.P. 487,675, 17.12.36. Switz., 29.1.36).—With the claimed objects of de-acidifying, removing Fe, sterilising, freeing from dissolved metal compounds and from excess of Cl and O, and allowing a  $CaO-H_2CO_3$  protecting coat to be formed in Fe tubes, tanks, etc., the  $H_2O$  is filtered through basic-reacting dolomite, at least part of the  $MgCO_3$  being converted into  $MgO$  rather than  $Mg(OH)_2$ ; the dolomite may first be impregnated with metal salts or mixed with granules of two metals of different electric potential. [Stat. ref.] B. M. V.

**Treatment of [irrigation] water.** N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 479,530, 30.8.36. U.S., 30.8.35).—Addition of  $HPO_3$  or its sol. salts ( $NH_3$ , Na, K) to a natural  $H_2O$  in substantially  $<$  the stoichiometric amount with reference to its Ca content is claimed to prevent or delay pptn. of  $CaCO_3$  when  $NH_3$  is added to the  $H_2O$ , as for irrigation  $H_2O$  to supply  $NH_3$  to the soil. O. M.

**Purification of liquids.** I. WALLQUIST (B.P. 488,151, 17.11.37).—For the treatment of the white- $H_2O$  of pulp and paper mills an endless filter cloth runs over two (or more) pervious drums; the layer of fibres deposited on the first drum from the white- $H_2O$  is left undisturbed until after passage over the second drum, where the  $H_2O$  is re-filtered. B. M. V.

**Removal of phenols from waste aqueous liquids.** G. W. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 479,359, 5.8.36).—Emulsion formation, such as normally occurs during the solvent extraction of waste aq. liquids for recovery of phenols, can be avoided by treating the waste liquor, free from oily and solid substances insol. in  $H_2O$ , before the extraction, with small amounts of an active C, whereby the emulsifying constituents are removed. H. C. M.

**Gas masks.** G. INGRAM (B.P. 487,753, 3.12.37).

**Carbonating water and beverages.** BARNES ENG. (PTY.), LTD. (B.P. 487,554, 9.8.37. Austral., 14.9.36. Addn. to B.P. 451,277).

**Heat exchangers for air conditioning.**—See I. N compounds.—See III.  $O_2$  for respirators.—See VII. Amine resins.—See XIII.