# BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DEC. 18 and 25, 1936.\*

# I.-GENERAL ; PLANT ; MACHINERY.

Factors affecting the design of heat-transfer apparatus. E. W. STILL (Inst. Mech. Eng., Preprint, 1936, 44 pp.).—Experiments on heat transfer between air, H.O, ethylene glycol, and oils are described and the results analysed and graphed. The Nusselt equation can be applied in cases of turbulent flow of liquids and gases, and a form of it, together with graphs, is given for the solution of design problems for different conditions, including those in which gilled tubes are used.

D. K. M.

Heat transfer in gas-heated hearth furnaces. W. HEILIGENSTAEDT (Gas- u. Wasserfach, 1936, 79, 754-760, 783-788).-The theory of heat transfer is discussed and equations are developed for calculation of heat-transfer coeffs. Such coeffs. were found for an actual furnace (apparatus described) and agreed with calc. vals. Discrepancies between theoretical and observed vals. found by earlier workers are traced to scaling of the charge. This is corr. for and good agreement is then obtained. A simple method for calculating the coeffs. is given. The advantages of gas heating over electrical heating are briefly discussed.

J. W.

Hot-face insulation [of furnaces and kilns]. H. V. ALLEN (Trans. Ceram. Soc., 1936, 35, 437-461).-The theory, practice, and economics of the use of highly refractory insulating brick are discussed in detail.

J. A. S.

J. A. S. Hardness-testing machines. W. HENGEMÜHLE (Stahl u. Eisen, 1936, 56, 1017-1025).-Illustrated descriptions of modern hardness-testing equipment are given, with curves showing the relation between Brinell (steel ball and sintered carbide ball), Vickers, and Rockwell hardness vals. W. P. R.

Resistance to filtration of compressible materials. I. I. M. SECCHI (Chim. e l'Ind., 1936, 18, 514-517).-The relation between the resistance towards filtration of liquids through powdered mica and the extent to which the mica is compressed has been investigated. An empirical quant. relationship is given. 0. J. W.

Distillation efficiency in 3- and 6-mm. fractionating columns. A. Rose (Ind. Eng. Chem., 1936, 28, 1210-1212).-Except at very low rates of boiling, a 6-mm. column gives results as good as, and is easier to control than, a 3-mm. column in the separation of C6H6 and CCl<sub>4</sub>. High efficiency (30 theoretical plates per ft.) is possible, using an unpacked column and a very low rate of distillation, provided that the column is well lagged. For greater rates of distillation a packed column is more efficient. E. G. H.

Evaporation temperature-the factor determining the process of distillation. V. L. GURVITSCH (Azer. Neft. Choz., 1934, No. 11-12, 83-86).-A discussion. Сн. Авз. (е)

Contributed and applicate indication

Compound evaporator, its theoretical basis and practical design. H. CLAASSEN (Z. Wirts. Zuckerind., 1936, 86, 177-192).-Some defects of vertical-tube evaporator units are discussed and the principles of the author's compound evaporator are described. In ordinary evaporators with tubes 1.5-2 m. long, as used in sugar factories, heat transmission is very inefficient in the lower parts of the tubes, owing to the slow rate of flow of the juice. In evaporators of the Kestner type, with tubes 7 m. long, the rapidly moving vapour in the upper half of the tubes does not contain sufficient liquid to keep the tube walls wet enough for rapid heat transmission. With an evaporator unit containing only a single set of tubes it is, in fact, not possible to obtain satisfactory conditions of heat transfer throughout the length of the tubes. In the compound evaporator, two (or more) tube sets, in separate heating vessels, perform successive stages of the work done by a single set in the present types of unit; the first set produces a liquid-vapour mixture which passes, without separation of liquid from vapour, to the space below the tubes of the second set, and so on if > two sets are used. Each set contains more tubes than the preceding one, because with increasing production of vapour the rate of flow would otherwise become excessive. The relative heating surfaces of the sets should be such that the vapourliquid mixture leaving the final set contains  $\lt$  1 vol. of liquid to 300-400 vols. of vapour, and its rate of flow through the tubes should be > 4-5 m./sec. If necessary, to attain these conditions, a portion of the liquid finally separated may be returned to the vapourliquid mixture at an intermediate stage, or part of the vapour may be separated from the mixture in passing from one tube set to the next. The coeff. of heat transmission for a two-stage compound evaporator is calc. to be 50% > that for an equiv. unit of the ordinary type. The design of the steam chamber has special features (cf. B., 1933, 519; 1934, 383). J. H. L.

Methods for reducing the diameter of vacuum towers and increasing the yield of the lower fraction. L. D. NERSESOV, B. B. KAMINER, B. L. GURVICH, and L. A. FOMENKO (Azer. Neft. Choz., 1934, No. 11-12, 81-83).-The diam. of the bubble towers is lowered by recirculating the condensate through the still. CH. ABS. (e)

Velocity distribution of fluids in pipes. E. C. Koo (J. Chem. Eng. China, 1936, 3, 91-109).-Mathematical. C. I.

a

\* The remainder of this set of Abstracts will appear in next week's issue.

*Erratum.*—On p. 863, col. 1, line 17, for Schiebler read Scheibler.

[Plant for] papermakers' cellulose.—See V. Centrifugal purifier for molten metals.—See X. Paints for boiler interiors.—See XIII.

See also A., Nov., 1334, Dynamics of sorption of mixtures of vapours. 1355, Apparatus for dialysis, osmosis, etc.

#### PATENTS.

Rotary furnaces, kilns, dryers, and the like. F. LLOYD and H. LINDARS (B.P. 454,268, 28.3.35 and 6.2.36).—An (externally heated) horizontal or slightly sloping rotary cylinder is provided with an axial shaft having inclined blades all on one side of a diametral plane, but the circumferential length of each blade extends over > one pitch so that all parts of the kiln are swept, or rather scraped, as a working fit is intended. B. M. V.

Solid-fuel furnaces. INTERNAT. FURNACE EQUIP-MENT CO., LTD., and L. BAILLY (B.P. 454,111, 27.6.35).— A step grate and combustion chamber for supplying hot gases to kilns or the like is provided with a superposed drying or distillation chamber. B. M. V.

Generation of heat by combustion of solid fuel. A. VANDEVELDE (B.P. 454,216, 22.12.34).—Fine coal and air are blown to an intermediate level of a bed of solid fuel through a device so shaped and cooled that a mass of clinker forms in the nearest route of the gases passing to the surface of the bed. B. M. V.

Furnaces. J. S. WITHERS. From C. J. H. PENNING (B.P. 454,019, 22.3.35).—Molasses is injected into a second combustion chamber (C) behind the fire bridge which forms the boundary of the combustion chamber for sugar cane or other solid fuel. C is provided with a sloping imperforate bottom to remove slag.

B. M. V.

Furnace apparatus for heat-treatment of metallic or other bodies. J. S. ATKINSON, and STEIN & ATKIN-SON, LTD. (B.P. 454,382, 29.3.35).—Walking-beam apparatus is provided with operating mechanism at each end, which enables a much greater no. of the hot parts to be in tension only. B. M. V.

Providing crucibles and tools for use in contact with molten masses with a protective coating. E. H. E. JOHANSSON (B.P. 454,506, 1.4.35).—Coverings suitable to resist metal or salt melts comprise  $H_3BO_3$ ,  $B_2O_3$ , borates,  $P_2O_5$ , and phosphates which are viscous at the temp. of use. Asbestos, graphite, or the like may be incorporated. B. M. V.

Carrying out chemical reactions. P. E. KUHL, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 2,028,336, 21.1.36. Appl., 5.10.32).—The interaction of two gases (A, B) in a liquid solvent, A being much more sol. than B, is effected in two steps : (1) concurrent and (2) countercurrent to the solvent. E.g., in the removal of NH<sub>3</sub> (A) and H<sub>2</sub>S (B) from a gas mixture, fresh H<sub>2</sub>O is fed to (2) and the liquid from that to (1). B. M. V.

Preservation of perishable articles by freezing. F. O. S. BLAND (B.P. 454,220, 25.2.35).—A liquid freezing medium comprises a mixture of at least one monohydric

and at least one polyhydric aliphatic alcohol and  $H_{2}O$ , e.g., glycerin 2—40 (32),  $H_{2}O$  25—45 (38), EtOH 20—40 (30)%. It is purified by filtration through fuller's earth, MgO, and active C, and is suitable for operation at  $< -30^{\circ}$ . B. M. V.

**Crusher.** J. R. BAKSTAD, Assr. to JEFFREY MANUFG. Co. (U.S.P. 2,028,643, 21.1.36. Appl., 25.11.30).—A fine crusher is constructed with the upper part of each jaw inclined and corrugated and the lower vertical and plane or decreasingly corrugated; the swing jaw is operated at such a speed that several nips are given to the material during its fall. B. M. V.

Operation of a pneumatic pulveriser. B. GRAEM-IGER (B.P. 454,125, 24.12.35).—The air is compressed to such an extent that (after heating) energy in excess of that required for the pulveriser is available, the excess being returned to the compressors by means of a motor. B. M. V.

Flotation machine. R. P. AKINS (U.S.P. 2,028,179, 21.1.36. Appl., 7.11.33).—Sub-aëration alone is relied on to agitate the pulp and transport the tailings; a mechanical agitator close to the pulp entrance may be desirable. B. M. V.

[Rapid] measurement of moisture content of sands, powders, or the like. FERRANTI, LTD., and J. DAWSON (B.P. 454,438, 30.3.35).—A predetermined quantity of the material is confined at a known pressure and an a.c. is passed through to measure the  $\kappa$ .

B. M. V.

Liquid-purification plant. R. B. Morse (B.P. 454,439, 30.3.35. U.S., 11.4.34).—Basins for sedimentation, coagulation, and filtration are arranged concentrically. B. M. V.

Filter leaf. M. F. MORAN (U.S.P. 2,028,466, 21.1.36. Appl., 14.5.34).—Filter leaves are lowered on to and supported by a pair (each) of standards which also form the filtrate outlet. B. M. V.

Steady bearing for centrifugal separators. J. F. THOLL, ASST. to AMER. TOOL & MACHINE CO. (U.S.P. 2,028,730, 21.1.36. Appl., 30.8.32).—The bottom bearing of a suspended-type machine is supported only by a horizontal rod which is adjustably clamped at the far end only. B. M. V.

Apparatus for drying sludge. L. M. KEOUGHAN (U.S.P. 2,028,395, 21.1.36. Appl., 18.3.33).—Horizontal, steam-containing tubes are arranged in vertical tiers in a tank, alternate tiers being reciprocated vertically B. M. V.

Apparatus for heating liquids. R. MORTON & Co., LTD., and G. A. RAWLINS (B.P. 454,453, 9.5.35).—A hood and baffle for placing over the heater in a tank of, *e.g.*, wort is described. B. M. V.

Tube still and furnace construction. J. S. WALLIS and S. B. DE BENCE, ASSIS. to ALCO PRODUCTS, INC. (U.S.P. 2,028,305, 21.1.36. Appl., 31.8.31).—The tubes which are subjected to radiant heat are close to the roof of the combustion chamber, and the convection tubes in a compartment are divided from this chamber by a wall. B. M. V.

Distillation of easily decomposable materials. W. K. LEWIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 2,028,340, 21.1.36. Appl., 17.10.33).—Nondestructive distillation (e.g., in the manufacture of lubricating oils) is effected in a vac. produced by the aspiration of the vapours in a restricted outlet containing a jet of vaporised hydrocarbon of high b.p. and low v.p., fractions of the distillate being condensed before reaching the outlet. B. M. V.

Separation of water contained in a liquid. E. ALTENKIRCH and H. BEHRINGER (B.P. 454,558, 27.3.35). —The mixed liquid is passed in countercurrent contact with a circulatory current of gas to produce partial or complete evaporation of both constituents. The cycle for the gas comprises : evaporation at atm. pressure and a relatively low temp. of liquid, approx.  $60^{\circ}$ , solar heat, e.g., being applied at one or more of these stages, and the cooling of the gas and vapour, e.g., by excess of sea-H<sub>2</sub>O, being effected in a part screened from the heat and out of contact with the liquid, circulation being solely effected thermosiphonically. The process is also applicable to the concn. of (dil.) EtOH. B. M. V.

(A) Resolving, (B) separating, solutions or liquid mixtures into their constituents by cooling and crystallisation. Ges. F. LINDE'S EISMASCHINEN A.-G., and G. A. KRAUSE (B.P. 454,075 and 454,084, [A] 23.3.36, [B] 19.5.36. Ger., [A] 26.3.35, [B] 8.6.35. Addns. to B.P. 429,474; B., 1935, 754).-(A) An exponential formula is given relating the amount of unfrozen conc. solution to be left in terms of the original concn., in order that the moulded body of ice (or solid pure solvent) should be strong enough to withstand centrifuging and yet be porous enough for the concentrate to separate. If the original concn. is very low, previous concentrate or the dry substance may be added at the start of the process. (B) The porous bodies are formed as separate blocks which can be assembled like bricks to form an annulus or other form for centrifuging. B. M. V.

(A, B) Viscosity-responsive devices. (c) Viscosimeter. H. T. BOOTH, Assr. to LUBRICATION CONTROL CORP. (U.S.P. 2,028,186-8, 21.1.36. Appl., 7.2.33).— Devices comprising a friction tube, sharp-edged orifice, pressure gauge, and pressure regulator are described, the apparatus being placed in a by-pass to the main flow, usually between main flow and atm. Various methods of using such devices are claimed.

#### B. M. V.

Purification of air and gases. C. WETHERLL (B.P. 454,441, 30.3.35).—H<sub>2</sub>O and a liquid of lower surface tension, *e.g.*, oil or glycerin, are separately atomised into the atm. of, *e.g.*, a mine. The liquids may be separately atomised into the same stream of motive air in an apparatus like a double scent spray.

#### B. M. V.

Separation of suspended particles. O. BRANTD and H. FREUND (B.P. 454,050, 8.10.35. Ger., 8.10.34). —Aërosols are coagulated by short-wave (sound) vibration generated in the separating chamber which is tuned to resonance. B. M. V.

Charges or cartridges for generation of gas pressures for actuation of mechanical devices. J. M. HOLM, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 454,091, 23.1.35 and 4.1.36).—The charge comprises material capable of self-contained combustion when ignited at atm. pressure and material capable of combustion only at elevated pressure, a suitable mixture being  $\rm NH_4NO_3$  (I), gelatinised nitrocellulose (II), and a hydrocarbon or other C-containing material (III). The ratio (I): (III) is  $\ll 4:1$  by wt., (I) being sufficient to prevent deposition of C but insufficient to oxidise it all to  $\rm CO_2$ , and (II) is 7-20% of the total. The final pressure is 500-1200 lb. per sq. in. after cooling and is developed in 5 sec. B. M. V.

Laboratory equipment. E. H. FREEMAN (B.P. 454,024, 28.3.35).—Portable benches are provided with rapidly made connexions for gas,  $H_2O$ , and other services arranged in a definite way interchangeably to match corresponding groups of services just below the floor of the laboratory. B. M. V.

Halogenated Ph<sub>2</sub>O.—See III. Separation of gases. —See VII. Crystallising sugar solutions.—See XVII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

Origin of the finely-divided or granular opaque matter in splint coals. R. THIESSEN and G. C. SPRUNK (Fuel, 1936, 15, 304—315; cf. B., 1936, 224).—Microscopical examination of Upper and Lower Cedar Grove (W. Virginia) coals shows the existence of transition stages which indicate that the granular opaque matter is derived predominantly from coniferous wood fibres. It is concluded that the splint coals were laid down under conditions favourable to the action of biological agencies which bring about a high degree of decomp. and maceration of the plant material. The transition studies help to interpret structures, *e.g.*, the so-called "bogenstruktur," encountered in other coals. A. B. M.

**Coal-jigging.** A. A. HIRST (Trans. Inst. Min. Eng., 1936, 92, 19—26).—The construction and operation of an experimental 3-compartment Baum washer-box, each compartment being fitted with a separate, automatically controlled extractor for refuse or middlings, is discussed. The various types of float gear for automatic refuse control are described and compared. The advantages of using dirty  $H_2O$  are discussed, together with the considerations involved in maintaining a washing medium of semi-colloidal soft shale or clay.

H. C. M.

Collection and analysis of air-borne dust during the driving of hard headings. J. I. GRAHAM and F. LAWRENCE (Trans. Inst. Min. Eng., 1936, 92, 1-11).-Tables are given showing the quantity and composition of the dust thrown into suspension in the air during driving operations. The concn. of dust in the air was determined, using a modified "sugar-tube" method, a measured vol. of air being drawn through, and its dust thereby removed by a filter pad of pure KNO<sub>3</sub> (20-60-mesh). The dust content varied from 0.0003 to 0.095 grain per cu. ft. of air, and was much less in wet drilling or in drilling damp rock than in dry drilling. The % free SiO<sub>2</sub> in the dust (10 · 1-47 · 6) was, in general, < the average of the strata through which the heading was being driven. H. C. M.

Treatment of [coal-]washery water : practical aspects of flocculation. L. W. NEEDHAM (Trans. Inst.

Min. Eng., 1936, 92, 28–48; cf. B., 1933, 290).—The mechanism of the operation of the various types of flocculating reagents used in the treatment of washery  $H_2O$  is discussed; they include electrolytes, alkaline starch solutions, glue, and gelatin. Recent work on the settlement rates, filtration, and other properties of flocculated solids is described. A résumé is also given of the commercial processes for collection and treatment of slurry. H. C. M.

Improved permissible flame safety lamps. L. C. ILSLEY, A. B. HOOKER, and E. J. COGGESHALL (U.S. BUR. Mines, 1936, Rept. Invest. 3312, 10 pp.).—A detailed description is given of a flame safety lamp for indicating the % CH<sub>4</sub> in mine air, which is independent of the nature of the mine illumination employed. A bimetallic spiral is mounted over the lamp flame, which is adjusted to const. height so that any increase in flame height due to CH<sub>4</sub> causes an expansion of the spiral, indicated by a pointer moving over a horizontal scale. An approx. straight-line scale deflexion of  $1\cdot 5$ — $2\cdot 0$  in. for each % CH<sub>4</sub> is obtained. H. C. M.

Relation of fuel coefficients to the lower calorific values of ash-free fuels. W. BOIE (Arch. Wärmewirts., 1936, 17, 241—242; cf. B., 1936, 724).—Relations are established between the lower calorific val. of an ash-free fuel and the coeffs.  $\rho$ , *i.e.*, (9h + w)/c, and  $\sigma$ , *i.e.*, 1 + 3(h - o/8)/c, where h, w, c, and o are pts. by wt. of H<sub>2</sub>, H<sub>2</sub>O, C, and O<sub>2</sub>, respectively. A simple diagram applicable to any fuel is given. R. B. C.

Coking the Barnsley Seam. II. R. A. MOTT and R. V. WHEELER (Trans. Inst. Min. Eng., 1936, 90, 318-327; cf. B., 1935, 339).-A method devised for determining durain, based on the fact that progressive addition of bright coal (clarain and vitrain) causes a linear decrease in its swelling power, has shown Barnsley Seam slacks to contain 20-50% of durain, the presence of which favourably influences their coking properties. To determine the effect of fineness of grinding on the quality of the coke made, three slacks were crushed so as to yield (a) 60-74% and (b) 85-90% through a  $\frac{1}{8}$ -in. mesh sieve. Comparative carbonising tests made with 500-lb. charges in an 18-in. experimental oven showed that, as rank of coal decreased, the amount of breeze produced increased, the coke becoming more abradable. Fine grinding decreased abradability, the effect being most marked with coal of lowest rank. With slack containing little durain, fine grinding lowered the impact hardness of the coke, as indicated by the  $1\frac{1}{2}$ -in. shatter index ; with slacks rich in durain the effect of fine grinding was the reverse. It is concluded that when the rank of a coking slack containing durain is low the charge should be finely crushed to prevent formation of abradable coke. A standard of fineness of 100% through 1-in. and 90% through 1-in. mesh is recommended for low-rank slacks. With fine grinding of the charge as much as 60% of a non-coking slack of low rank can be blended with one of higher rank for the production of metallurgical coke. Blending of coke dust with a coking slack, though increasing the impact hardness of the coke produced, may render it abradable; addition of tar to the charge tends to counteract this effect.

R. B. C.

Low-temperature carbonisation. P. A. SCHWARZ (Gas World, 1936, 105, Coking Sect., 139—140).— Experiments on the production of smokeless fuel in modified Otto waste-heat ovens (which had become obsolete) are described. The coal carbonised is an 8:1:1 blend of Northumberland non-coking coal, Durham coking coal, and pulverised coke breeze; the coking time is 25 hr., the final carbonisation temp. 500°, and the throughput per oven (exclusive of added breeze) 4 tons of raw coal. H. C. M.

Technical and economic aspects of a recent lowtemperature carbonisation process. ANON. (Chal. et Ind., 1936, 17, 221—228).—The Zuyderhoudt process is described. The retorts are of the externally-heated, vertical type and have truncated sheet-Fe cores through which the carbonisation gases are quickly removed from the decomp. zone. Yields per ton of coal are given. The tar contains a low % of pitch and a high % of light hydrocarbons. R. B. C.

Lurgi process for complete gasification of coal with oxygen under pressure. H. C. MILLETT (J. Inst. Fuel, 1936, 10, 15-21).-A crit. survey is made of the literature dealing with both the theoretical and experimental investigations of the use of O2 in complete gasification. Small-scale experiments on the complete gasification of semi-coke in O2 and steam showed that on raising the pressure from 1 atm. to 18 atm., the % CH<sub>4</sub> in the CO<sub>2</sub>-free gas increased from 2.4 to 19.8, and the calorific val. from 331 to 450 B.Th.U./cu. ft. Similar results were obtained for the high-pressure gasification of such markedly different fuels as lignite and anthracite. A detailed account is given of the construction, and of the results obtained in the operation, of a large-scale experimental unit having an output of 700,000 cu. ft. of gas per day. When gasifying lignite briquettes (3-8 mm. size) in a mixture containing 10 vols. of steam per vol. of O2, preheated to 500° and at a pressure of 20 atm., gas was obtained which, after washing for removal of  $CO_2$ , had the composition  $CO_2$ 2.0,  $C_mH_n 0.8$ , CO 20.4,  $H_2 53.8$ ,  $CH_4 20.0$ ,  $N_2 3.0\%$ . The  $O_2$  and steam consumptions were, respectively, 14.5cu. ft. and 6.9 lb. per 100 cu. ft. of CO2-free gas, the thermal efficiency of gasification being  $93 \cdot 2\%$ . The max. rate of gasification attained, 184 lb. of fuel per sq. ft. of generator cross-section per hr., was determined by the supply of O2 available. An analysis is made of the reactions occurring during gasification, and it is considered that the technical success of the high-pressure gasification process depends on obtaining a satisfactory balance between the rate of production of CH<sub>4</sub> and the rate of gasification of the coke in steam. An account is also given of the Linde-Fränkl process whereby O, can be produced in quantity at materially reduced costs. H. C. M.

Thermal decomposition of humic acids. G. STADNIKOV, K. SISKOV, and A. USCHAKOVA (Brennstoff-Chem., 1936, 17, 381–384).—Samples of humic acid prepared from a Ukrainian brown coal were subjected to heat-treatment (18–60 hr.) in a current of  $N_2$  at 125–375°. The variation in the ester and ether OMe contents of the products shows that up to 275° a proportion of the CO<sub>2</sub>H groups was eliminated, whilst phenolic OH remained unaffected. At 300° there was

a slight loss of the latter, but no further loss occurred up to  $375^{\circ}$ ; elimination of  $CO_2H$ , however, continued progressively over the whole temp. range. It is concluded that temp. has played no especial rôle in the formation of bituminous coals. The Ba(OH)<sub>2</sub> absorption of the samples after heat-treatment fell rapidly as the temp. of treatment rose from 225° to 375°; it fell also on methylation of the original acid with  $CH_2N_2$ (cf. A., 1936, 819). A. B. M.

Mechanical properties of coke and researches on transient fusion of coal. A. GILLET (Rev. univ. Mines, 1936, [viii], 12, 269-281).—Coal undergoes true fusion at a characteristic temp.; fusion is of sp. duration and is followed by re-solidification of the coal before any appreciable % of volatile matter has been lost. The duration of fusion determines the agglutinating index. Laboratory blending experiments are shown to be a useful means of selecting the most suitable coals for the production of metallurgical coke. R. B. C.

Primary reactions of the combustion of carbon. M. K. GRODZOVSKI and Z. F. CHOUCHANOV (Fuel, 1935, 15, 321-328; cf. B., 1935, 389).-The reactions occurring when a bed of C is blown with enriched air have been studied. The size of the visible combustion zone decreases with increasing  $[O_2]$  and velocity of the blow. When C burns at high rates of combustion (high [O<sub>2</sub>] in the blow) CO is obtained as a primary product ; the secondary reaction  $2CO + O_2 = 2CO_2$  can be prevented by sufficiently increasing the blow velocity, so that under these conditions the max. yield of primary CO is obtained. In the  $O_2$  zone the velocity of the reduction of steam by C is greatly increased. High-velocity gasification does not require very high temp., and can take place at 800-900°. Semi-industrial-scale tests have shown that highvelocity gasification can be operated with high efficiency to yield gases suitable for power raising or for synthetic purposes. A. B. M.

Solvents and resin from light oil refining. ANON. (Amer. Paint J., 1936, 21, No. 1, 14).—In the refining of light oils at by-product coke plants, heavy oils (b.p. 180—375°) are obtained which are good solvents for rubber, chlorinated rubber, Thiokol, resinates, and many synthetic and natural resins and are non-corrosive and unaffected by alkalis and dil. acids. An oil-sol. resin is also obtained which appears to be a mixed polymeride of cyclic dienes and aromatic ethylenic compounds. D. R. D.

High-capacity producer for gas production from brown-coal briquettes. R. Möller (Braunkohle, 1936, 35, 417–420).—A mixed-gas producer having a throughput of 7–45 tons/24 hr. is described. The air blast is saturated with  $H_2O$  condensing in the electrical gas-purification system. R. B. C.

Production of hydrogen-rich gases for synthetic purposes by the water-gas process. J. Gwosdz (Brennstoff u. Wärmewirts., 1936, 18, 59-64, 82-85, 96-99).—A comprehensive review. R. B. C.

Intensive purification of gas by active carbon. K. BLUME (Gas- u. Wasserfach, 1935, 78, 785-789, 810-814).—The problem undertaken was to recover benzol from gas and at the same time to remove such

objectionable constituents as H<sub>2</sub>S, HCN, C<sub>10</sub>H<sub>8</sub>, etc. The working of an active-C benzol extraction plant is described, the economy of this method is demonstrated, and the properties of motor benzols so obtained are dis-cussed. The composition of the benzol is not dependent on the extraction process, as previously believed, but is influenced exclusively by the gas production process, principally by the structure of the gas generating oven. In properties benzol extracted by active C differs very little from standard B.V. benzol; thus the sp. gr. are 0.857 and 0.870 respectively, the initial b.p. 73/80° and 75/81° respectively and the final b.p. 158° and 167° respectively. The octane nos. of active-C extracted benzol-benzene mixtures differ from B.V. benzolbenzene mixtures only by 2 or 3 units. The effectiveness of the extraction process as a gas purification process is discussed. Thus the gas is stripped of 95.5% of benzol, 47% of org. S, 48% of HCN, and 100% of C<sub>10</sub>H<sub>8</sub>. E. L. S.

Supervision of a M.A.N. waterless gas holder. E. A. DAVIES (Gas J., 1936, 216, 339–342).—The max. efficient working of a waterless holder depends on making regular determinations of the  $\eta$  of the tar seal, particularly at the holder temp. Introduction of benzol extraction led to a marked increase in the  $\eta$  of the tar, subsequently avoided by the use of dehydrated tar. H. C. M.

Mathematical and graphical solutions for exhaust and flue-gas analyses. R. H. GRUNDY (Engineering, 1936, 142, 486–488).—A graphical method for determining the % excess air from the CO<sub>2</sub> content of the dry flue gases is described. H. C. M.

Continuous tar-distillation process. J. F. KES-PER (Teer u. Bitumen, 1936, 34, 35–36).—The Guignard process is described. The tar is kept flowing in a thin layer on the walls of a horizontal cylindrical vessel by means of an internal rotating drum furnished with scoops. R. B. C.

Continuous tar distillation in Koppers pipe stills. O. EISLER, Z. ZAMRZLA, and M. WEINKOFF (Glückauf, 1936, 72, 184—188).—The pipe-still process for distilling petroleum has been applied to tar. Details of a plant with a throughput of 200 tons/24 hr. are given. The distillation ranges of the various tar fractions are tabulated. R. B. C.

Fine structure of tar pitch. B. G. ŠIMEK (Brennstoff-Chem., 1936, 17, 384-389) .- An X-ray study of the structure of pitch and its  $\alpha$  (C<sub>6</sub>H<sub>6</sub>-insol.),  $\beta$  (C<sub>6</sub>H<sub>6</sub>-sol., light petroleum-insol), and  $\gamma$  (light petroleum-sol.) constituents has been made. Part of the  $\alpha$  possesses a crystal structure similar to that of charcoal, active C, etc. (cf. A., 1935, 285), the remainder being amorphous. The crystallisable org. constituents, e.g., anthracene (I), do not crystallise out from the whole pitch except in particular cases, e.g., pitches produced by air-blowing material rich in (I). Crystallisation from the pitch is inhibited, not by the solubility of the (I) therein, but by the presence of the insol. colloidally-dispersed  $\beta$ . When the latter are removed crystallisation occurs, e.g., in the separated  $\gamma$ . There is no fundamental difference in structure between the  $\beta$  and the amorphous  $\alpha$  constituents which have been formed by condensation of compounds in the tar. A. B. M.

Position and prospects of mineral oil research. L. UBBELOHDE (Oel u. Kohle, 1936, 12, 983—988).—A lecture.

Chemical transformations of the constituents of petroleum. J. von BRAUN (Bull. Soc. chim., 1936, [v], 3, 1921–1941).—A lecture.

Dehydrating and cleaning crude oil. V. P. BAT (Azer. Neft. Choz., 1934, No. 11–12, 114–115).—A method for the preliminary removal of  $H_2O$  and dirt before pumping oil through trunk pipe lines is described. CH. ABS. (e)

Calculation of viscosity [of mineral oils]. H. VOGEL (Oel u. Kohle, 1936, 12, 835-837).—A crit. discussion of results obtained with various technical viscosimeters, and of the calculation of abs. vals.

Oil volatility directly related to oil consumption. L. L. DAVIS and R. D. BEST (Nat. Petroleum News, 1935, 27, No. 26, 70–75).—Volatility has a greater effect on consumption than has  $\eta$ . Volatility is of minor importance where consumption is high, owing to high speed or engine design. CH. ABS. (e)

Oils used in the absorption of natural gasoline. V. I. KARSHEV and A. F. BOBROVA (Chim. Tverd. Topl., 1934, 5, 839—849).—A review of requirements. Crude oil from Barzas sapropelite tar is suitable. CH. ABS. (e)

Stabilisation and desulphurisation of shale gasolines. L. LEV, V. POLOZOV, and E. VIKTOROV (Chim. Tverd. Topl., 1935, 6, 438—448).—Experiments with  $MoS_2 + ZnO$  and  $MoS_2 +$  cement catalysts at 400°/50— 400 atm. are described. In the desulphurisation there is a simultaneous hydrogenation of unsaturated compounds which lowers the  $C_8H_{18}$  no. of the product.

Сн. Авз. (е)

Analysis of gasoline. I. RAPOPORT and E. FIRS-ANOVA (Chim. Tverd. Topl., 1935, 6, 448–459).— Determination of O compounds with  $FeCl_3 + HCl$  yields incorrect results as unsaturated compounds are also attacked. The Br method for unsaturated compounds cannot be used in presence of O compounds.

Сн. Авз. (е)

Normal heptane from shale-oil gasoline. N. A. ORLOV and I. Z. IVANOV (Chim. Tverd. Topl., 1934, 5, 788—790).—The fraction (b.p. 95—102°) was treated successively with 70%  $H_2SO_4$  (I) (20 min.), 80% (I) (30 min.), and 90% (I) (15 min.). Acid sludge was removed and the product treated with 95% (I) (30 min.) and 4 times with 100% (I). The product was washed, dried, treated for 24 hr. with SbCl<sub>5</sub>, washed with HCl and  $H_2O$ , dried and distilled over Na. The fraction of b.p. 98—99° was re-treated with 100% (I).

Сн. Авз. (е)

Micro-analytical method for determining the hydrocarbon groups present in cracked gasoline. I. E. BESPOLOV and V. M. GENERALOV (Azer. Neft. Choz., 1934, No. 11—12, 90—96).—Air is aspirated through a weighed amount of the gasoline and then through a series of absorbents (Br in AcOH for unsaturated compounds, 98—100%  $H_2SO_4 + 3\%$  AgNO<sub>3</sub> for aromatics). CH. ABS. (e)

Preparation of hexyl alcohols from cracked products. S. A. NAZAROV and E. K. REMIZ (Mat. on Cracking, 1935, No. 2, 172—182 ; cf. B., 1935, 1031). The fraction, b.p. 55—75°, yielded on redistillation 15-17% of hexene fraction. Treatment with H<sub>2</sub>SO<sub>4</sub> and hydrolysis yielded CMe<sub>2</sub>Pr·OH and CHEtPr·OH (4·5 and 14·4% respectively, calc. on the total unsaturated compounds in the original fraction). Successive treatments with 75% acid at 0° and with 85% acid at 5—10° gave the best results. CH. ABS. (e)

Combustion qualities of Diesel fuels. G. D. BOERLAGE and J. J. BROEZE (Ind. Eng. Chem., 1936, 28, 1229-1234).-The suitability of a Diesel fuel depends chiefly on its volatility,  $\eta$ , and cetene no., although the engine type determines the optimum val. for each. Low  $\eta$  and high volatility result in good atomisation but poor penetrating power, the reverse being true with highly viscous and difficultly volatile fuels, which may also cause deposition on the walls. Either extreme leads to uneven distribution and hence to "after-burning." A better criterion for volatility is required than the present 65% point for light fuels and Conradson test for heavy. A fuel of low cetene no. may give greater power and economy, although starting is better or running smoother with fuels of high cetene no. Combustion in Diesel engines is mostly of the destructive type, and under certain conditions there is evidence that it may be partly an oxidation process, e.g., according to the hydroxylation theory. E. G. H.

Vegetable oils as substitutes for mineral oil products. M. FREUND (Petroleum, 1936, 32, No. 45, 10-12).--A discussion.

**Extreme-pressure lubricants testing.** G. L. NEELV (J. Soc. Autom. Eng., 1936, **39**, 293—296).— Experiments showed that the Almen, Timken, Floyd, and General Motors testing machines did not rate the film strengths of standard lubricants in accordance with their service vals. More promising results, however, were obtained with the "S.A.E." machine. R. B. C.

Chemical constitution and film strength as determined by the Timken machine : chlorine compounds added to lubricants. B. H. LINCOLN, G. D. BYRKIT, and W. L. STEINER (Ind. Eng. Chem., 1936, 28, 1191—1197).—The removal of polar compounds during solvent-refining processes decreases the oiliness and load-carrying capacity of a lubricating oil; such properties can, however, be improved by addition of org. Cl-compounds. Results are given of the effect on the Timken film strength of the addition of 1% of a large no. of such compounds to a mineral oil. Trichlororesorcinol was the most powerful compound tried, whilst, in general, chlorophenols, chloroamino-compounds, and chlorinated aliphatic esters were effective. E. G. H.

**Experimental burning of acid sludge in the Stalin [oil] refinery.** S. JOSIFOV (Azer. Neft. Choz., 1934, No. 11—12, 96—97).—A satisfactory boiler fuel was made from the org. material obtained in the electric separation of  $H_2SO_4$  from lubricating oil acid sludge and regular fuel oil, mixed in a 1:4 ratio and electrically heated under const. recirculation. CH. ABS. (e)

Rapid measurement of the oxidation of insulating oils in air. A. GEMANT (Trans. Faraday Soc., 1936, 32, 1628—1633).—When oils are oxidised at 150° at const.

H. J. E.

pressure in a small, enclosed space, the curves showing decrease of vol. of the gaseous phase with time vary characteristically with the grade of the oil. Preliminary results for some oils are given. E.S. H.

Distillation efficiency. Vac. towers.—See I. Analysis of oils.—See III. Bituminous roads.— See IX. Gas-pipe manufacture.—See X.

See also A., Nov., 1334, Properties of activated charcoal. 1344, Measurement of flame velocity. 1347, Water-gas reactions at a Pt surface.

## PATENTS.

Purification of coke-oven and like gases. RUHR-СНЕМІЕ А.-G. (В.Р. 454,368-9, 21.12.34. Ger., [A] 24.1.34, [B] 1.12.34).-(A) The residual gases of the process of B.P. 444,315 and 446,493 (B., 1936, 869) are passed over a hydrogenating catalyst, e.g., Ni or Cu at 300-400°, or MoS<sub>2</sub> at 350-500°, whereby the CO, CO<sub>2</sub>, and unsaturated hydrocarbons are converted by the H<sub>2</sub> present into CH<sub>4</sub> and other saturated hydrocarbons. The coke-oven gas used in the process may be diluted with water-gas, by steaming the coke, or with CO or CO2 from other sources. (B) The gas is treated with active C in such manner that the adsorption process is interrupted when appreciable quantities, e.g., about 15% of the amount in the initial gas, of constituents condensing more readily with C<sub>2</sub>H<sub>6</sub> and C<sub>2</sub>H<sub>4</sub> occur in the treated gas. The gas is thereby freed from S compounds and resinifying impurities. A hydrocarbon mixture which liquefies under pressure and is suitable as a motor fuel is recovered from the C. A. B. M.

Production of gaseous fuel mixtures. RUHR-CHEMIE A.-G. (B.P. 446,493, 27.7.34. Ger., 31.7.33. Addn. to B.P. 444,315; B., 1936, 583).—Coke-oven and the like gases treated with active C (*loc. cit.*) at 30 atm. yield a fuel comprising 30—50 vol.-% or, at 18 atm.,  $\leq 20$  vol.-% of C<sub>2</sub>H<sub>6</sub>-C<sub>2</sub>H<sub>4</sub> and other gases, mostly C<sub>4</sub>H<sub>8</sub> and C<sub>4</sub>H<sub>10</sub> and homologues. D. M. M.

Production of hydrocarbons by thermal treatment of carbonaceous materials. COUTTS & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 454,391, 7.5.35).—The materials, e.g., coal, tar, mineral oil, etc., are treated with hydrogenating gases under suitable conditions of temp. and pressure, e.g., 400—  $480^{\circ}/>50$  atm., in presence of catalysts and of graphitic acid (I) or the solid produced by the thermal decomp. of (I). A. B. M.

Treatment of gases evolved in carbonising or gasifying plants. WOODALL-DUCKHAM (1920), LTD. From DIDIER-WERKE A.-G. (B.P. 455,195, 17.3.36).— The off-take pipes from gas retorts or coke ovens are forked, one branch being at any time closed by a flap valve. One line leads to the usual tar etc. recovery plant, and is used for the gas in the first half of the carbonisation period or while it is tarry. The other line takes the tar-free gas from the latter half of the period and by-passes the tar-recovery plant. It is lagged and the sensible heat recovered by means of a heat exchanger. Alternatively, for continuous verticals, two off-takes are provided from different levels, one for tarry gas and one for tar-free gas. D. M. M. Production of aqueous bituminous emulsions. A./S. FOR KEMISK INDUSTRI (B.P. 454,487, 1.3.35. Denm., 29.6.34).—A mixture of clay and  $H_2O$  is allowed to ripen so as to form a suspension of electrically-charged particles of poly- and heteropoly-silicic acids or salts thereof, and bitumen is then added to form a coarsegrained emulsion. This is heated to increase the charge on the particles and disperse the bitumen without mechanical treatment to form a fine-grained emulsion. A. B. M.

Securing sulphur dioxide extract of petroleum. J. T. RUTHERFORD, Assr. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 2,024,476, 17.12.35. Appl., 8.8.32). —Petroleum or shale oil is extracted with liquid SO<sub>2</sub>. The layer containing the unsaturated and aromatic hydrocarbons is separated, freed from SO<sub>2</sub>, and mixed with a petroleum oil of different b.p. (but freed from such hydrocarbons). This mixture is then again extracted with SO<sub>2</sub>. C. C.

**Refining of mineral oil.** C. R. McKay and J. H. SMITH, Assrs. to SINCLAIR REFINING CO. (U.S.P. 2,022,358, 26.11.35. Appl., 26.3.30).—Acid-treated mineral oils are mixed with a metal hydroxide, *e.g.*, Ca(OH)<sub>2</sub>, without fully neutralising and the sludge is separated. Solid absorbent material is added to the heated clarified oil, *e.g.*, at within 28° of the flash point, in presence of steam. Naphtha may be added to the hot oil before the final separation of neutral and decolorised oil from the spent earth. C. C.

Separation of wax from mineral oil by filtration. TEXACO DEVELOPMENT CORP., Assees. W. P. GEE (B.P. 455,272, 12.3.35. U.S., 31.3.34).—Mineral oil is continuously de-waxed in an apparatus designed to enable chilled gas to be continuously circulated, to keep the filtering surface always at the same temp. as the wax, and to enable the washing to be done with a continuous film of the washing media. Use is made also of selective solvents. D. M. M.

Separation of mixtures of oily or waxy substances according to the countercurrent principle. N. V. VEREENIGDE FABRIEKEN VAN STEARINE, KAARSEN, EN CHEM. PRODUCTEN (B.P. 453,936, 19.1.35. Holl., 22.1.34).—Paraffin wax and oil (e.g.) are separated by utilising solid and liquid phases in such no. of stages that the material introduced at any separating stage has a composition intermediate those of the mixtures moving each way from that stage. B. M. V.

Breaking of [water-in-oil-type] petroleum emulsions. M. DE GROOTE, B. KEISER, and A. F. WIRTEL, Assrs. to TRETOLITE Co. (U.S.P. 2,025,804—5, 31.12.35. Appl., 31.12.34. Cf. U.S.P. 2,023,976, 2,023,979—82, 2,026,195, 2,023,993—7, and 2,026,217—9; B., 1936, 868).—A demulsifying agent is used which may consist of a polyketo-fatty acid or an unpolymerised keto-fatty acid, or their salts, preferably derived from the pressure oxidation of castor oil at relatively low temp. with or without addition of small quantities of true drying oils and/or a solvent. D. M. M.

Liquid fuels. P. A. ARNOLD (B.P. 454,376, 26.3.35).— Fuel for internal-combustion engines contains petrol, paraffin oil, heavy oil, EtOH, or mixtures of these, to which is added a small proportion (1 oz. to 2 gals.) of fish-liver oil, *e.g.*, from cod. C. C.

Production of non-knocking motor fuel by catalytic reaction of carbon monoxide and hydrogen. COUTTS & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 454,389, 5.4.35).—The lower-boiling fractions rich in olefines of the hydrocarbon mixture obtained by the catalytic conversion of  $CO + 2H_2$  are separated and subjected to a polymerising treatment under conditions which yield a product boiling within the motor spirit range; this is then remixed with the other fractions. Polymerisation is effected in presence of AlCl<sub>3</sub>, H<sub>3</sub>PO<sub>4</sub>, etc. at 20—250°. A. B. M.

Means for treating the charges of internalcombustion engines. F. J. HAWKINS (B.P. 455,056, 20.5.35).—An electrolytic cell is provided, by means of which a 2:1 (vol.) mixture of  $H_2$  and  $O_2$  is delivered through a back-fire safety device to the induction pipe of the engine in order to enrich or modify the fuel supplied. Automatic cut-out devices may also be incorporated. D. M. M.

Lubricating compositions. A. S. LEVESLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 454,386, 29.3.35).—Lubricating paste particularly suitable for kier valves contains a  $H_2O$ -sol., aliphatic polyhydroxycompound, e.g., glycerol, or a conc. aq. solution thereof, a cellulose derivative sol. in this viscous fluid and in  $H_2O$ , e.g., glycol-cellulose, and an alkali or  $NH_4$  salt of a higher fatty acid or other  $H_2O$ -sol. soap or soap-like material. C. C.

Lubricants and lubrication. C. ARNOLD. From C. F. PRUTTON and A. K. SMITH (B.P. 454,215, 29.12.34). —Halogenoaryl and other esters of thiophosphorus acids, *e.g.*, (o-C<sub>6</sub>H<sub>4</sub>Cl)<sub>3</sub>PS<sub>4</sub>, are claimed in admixture ( $\geq 20\%$ ) with a mineral lubricating oil to prevent rupture of the lubricating film under high bearing pressures, *e.g.*,  $0\cdot 1$ — $2\cdot 0\%$  for crankcase oil, and in motor fuel as uppercylinder lubricant. (Cf. B.P. 453,047; B., 1936, 1139.)

E. J. B.

Lubricants and lubrication. C. ARNOLD. From C. F. PRUTTON and A. K. SMITH (B.P. 454,552, 29.12.34). —Extreme-pressure lubricants are obtained by incorporating into mineral oil a halogenated org. compound ( $\leq$  55% halogen; b.p. > 170°) together with < 20% of another compound containing O and/or S or a compound containing O, S, and Cl (chlorodibenzoyldimethyl sulphide, chlorophenyl mercaptostearate). (Cf. B.P. 453,047; B., 1936, 1139.) S. C.

Wire-rope lubrication. W. D. Hodson (U.S.P. 2,028,155, 21.1.36. Appl., 3.10.32).—A lubricant for application cold by immersion to the strands of a rope just before closing comprises a fibrous base, grease, and a top drier in proportions to produce a plastic adhesive jelly which will set on the surface only to a leathery consistency. *E.g.*, a mixture of asbestos 10, heavy mineral oil 39, Al oleate 1, and blown oil 50% is claimed. B. M. V.

[Hydraulic brake] fluid composition. G. L. DOELLING (B.P. 454,628, 3.4.35).—See U.S.P. 1,997,998; B., 1936, 131.

Oil-cracking system. A. G. PETERKIN, JUN., ASSI. to Atlantic Refining Co. (U.S.P. 2,028,728, 21.1.36. Appl., 3.3.33).

Cracking of hydrocarbons. H. L. PELZER, Assr. to SINCLAIR REFINING CO. (U.S.P. 2,030,393, 11.2.36. Appl., 28.8.31).

Cracking of hydrocarbon oils. F. E. Edson, Assr. to Standard Oil Development Co. (U.S.P. 2,027,355, 7.1.36. Appl., 14.12.32).

Apparatus for expansion of hydrocarbons for cracking and distillation. C. STILL (U.S.P. 2,030,771, 11.2.36. Appl., 27.7.33. Ger., 5.8.32).

Conversion of hydrocarbons. N. GOODWIN and R. W. POINDEXTER, Assrs. to INDUSTRIAL PROCESS Co. (U.S.P. 2,027,862, 14.1.36. Appl., 26.11.28. Renewed 22.1.34).

Conversion of hydrocarbon oils. G. D. WHITE, Assr. to TEXAS Co. (U.S.P. 2,029,601, 4.2.36. Appl., 30.4.34).

Conversion and hydrogenation of heavy petroleum oils. R. H. McKEE, Assr. to U.S. Hydrogenation Corp. (U.S.P. 2,028,795, 28.1.36. Appl., 23.9.32).

Coking of hydrocarbon oils. L. A. MEKLER, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 2,029,783, 4.2.36. Appl., 28.12.31).

Coking of liquid residuums. C. J. PRATT, Assr. to J. P. DEVINE MANUFG. Co., INC. (U.S.P. 2,028,166, 21.1.36. Appl., 6.8.31. Renewed 24.9.34).

Oil distillation. J. S. WALLIS, ASST. to FOSTER WHEELER CORP. (U.S.P. 2,030,485, 11.2.36. Appl., 3.12.30).

Distillation of oil. A. G. PAGE, ASST. to UNION OIL CO. OF CALIFORNIA (U.S.P. 2,029,501, 4.2.36. Appl., 26.9.28).

**Pressure-still header.** S. H. Edwards, Assr. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 2,026,674, 7.1.36. Appl., 10.1.34).

**Return bend for cracking stills.** C. E. ARD, Assr. to F. C. FANTZ (U.S.P. 2,030,493, 11.2.36. Appl., 25.1.33).

Firing of oil stills. C. H. THAYER, Assr. to SUN OIL Co. (U.S.P. 2,029,956, 4.2.36. Appl., 15.8.35).

Fractionation of petroleum mixtures. G. B. COUBROUGH, Assr. to LUMMUS Co. (U.S.P. 2,030,281, 11.2.36. Appl., 22.12.33).

Combined rectification and refrigeration process. P. D. BARTON, ASST. to ALCO PRODUCTS, INC. (U.S.P. 2,028,432, 21.1.36. Appl., 19.3.34).

Electrical dehydrator [for petroleum emulsions]. (A) L. DILLON, (B) H. F. FISHER, ASSTS. to UNION OIL CO. OF CALIFORNIA (U.S.P. 2,029,362 and 2,029,527, 4.2.36. Appl., [A] 23.10.33, [B] 8.5.33).

Making of gasoline. O. BEHIMER, Assr. to TEXAS Co. (U.S.P. 2,027,014, 7.1.36. Appl., 21.11.18. Can., 3.12.21).

Treatment of petroleum. J. B. BARNES, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 2,029,752, 4.2.36, Appl., 3.12.31). **Treatment of hydrocarbon oil.** V. STAPLETON, Assr. to TEXAS CO. (U.S.P. 2,026,803, 7.1.36. Appl., 15.7.33). W. W. GARY, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 2,030,365, 11.2.36. Appl., 1.2.33).

Treatment of hydrocarbon oils. L. DE FLOREZ, Assr. to TEXAS CO. (U.S.P. 2,027,334, 7.1.36. Appl., 27.4.33). R. J. DEARBORN and W. M. STRATFORD, Assrs. to TEXAS CO. (U.S.P. 2,028,100, 14.1.36. Appl., 18.10.30).

Dewaxing. D. E. PAYNE, Assr. to STANDARD OIL Co. (U.S.P. 2,026,651, 7.1.36. Appl., 31.8.31).

Apparatus for hydrolysing acid sludge. H. W. THOMPSON and J. T. RUTHERFORD, ASSIS. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 2,026,714, 7.1.36. Appl., 13.1.33).

Production of lubricating oil. U. B. BRAY and C. E. SWIFT, Assrs. to UNION OIL CO. OF CALIFORNIA (U.S.P. 2,026,729, 7.1.36. Appl., 28.11.32).

Generating heat by combustion of solid fuel. Distillation of materials.—See I. Polymerides of olefines. Chlorination. Undecoic acid. Sulphofatty body. Ethers etc. of paraffin wax. Oxidised fatty acids. Halogenated Ph<sub>2</sub>O. Hydrogenation.— See III. Producing H<sub>2</sub>.—See VII. Apparatus for interaction of CO and H<sub>2</sub>.—See X. AlCl<sub>3</sub> synthesis of resins. Prepared resin. Paracoumarone resins. —See XIII. Germicide etc.—See XXIII.

## III.—ORGANIC INTERMEDIATES.

Determination of fumigants. VI. Purity of commercial ethylene oxide in cylinders. W. B. BROWN (J.S.C.I., 1936, 55, 321–325 T; cf. B., 1936, 77). —Two methods for the determination of the purity of  $(CH_2)_2O$  are described, a correction being applied when dissolved  $CO_2$  is present. Two methods for the determination of the relative proportions of  $(CH_2)_2O$  and  $CO_2$ in cylinders of fumigant are described. The composition of the fumigant may be calc. from the results.

Production of synthetic acids under low pressure. G. JUCHNOVSKI and S. ROCHLIN (Maslob. Shir. Delo, 1934, 10, No. 9—10, 8—10).—The optimum temp. for the oxidation of petrolatum by air was 110—115°. Ca naphthenate was a better catalyst than the Mg salt. The reaction time is greatly reduced by working at 3—4 atm. Higher pressures increase the amount of OH-acids formed. CH. ABS. (e)

Wetting agents frothing in acid media. S. SABETAY (Bull. Soc. chim., 1936, [v], 3, 1918–1919).— Priority of prep. of materials frothing in acid solution is claimed (B., 1924, 681; 1928, 199). Condensation of 1 or 2 mols. of  $N(C_2H_4:OH)_3$  with 1 mol. of lauroyl chloride in CHCl<sub>3</sub> yields compounds which froth even in presence of large quantities of HCl. J. W. S.

Chemistry of naphthalene derivatives. H. H. HODGSON (J. Soc. Dyers and Col., 1936, 52, 369-373).— An account is given of work already published in various papers and its bearing on the electronic structure of  $C_{10}H_8$  derivatives. S. C.

Infra-red absorption spectra of anthracene hydrocarbons. II. Application to analysis of constituents of oils. P. LAMBERT and J. LECOMTE (Ann. Off. nat. Comb. liq., 1935, **10**, 1077—1092; cf. A., 1936, 406).—Infra-red absorption spectra of 9:10-diisobutyl- and -diisoamyl-, 10:10-diisobutyl-, and 9:9:10:10tetraisobutyl-9:10-dihydroanthracene have been determined between 6 and 20  $\mu$ . A comparison of the spectra of these compounds with those obtained previously for anthracene and its tetra-, hexa-, and dodecahydro-derivatives shows that the absorption curves closely follow the modifications in chemical structure. The method can be applied to the identification of hydrocarbons in, e.g., lubricating oils. R. B. C.

 $n-C_7H_{16}$  from gasoline. Hexyl alcohols [from petroleum etc.]. Solvents from light oil.—See II. Skin troubles from solvents.—See XXIII.

See also A., Nov., 1347, Electrochemical study of  $\alpha$ -OH-acids. 1359, Prep. of MeF. Determination of EtOH. 1360, Fluorination of org. compounds. 1361, Prep. of  $\gamma$ -keto-*n*-hexoic acid. 1363, Colorimetric determination of COMe<sub>2</sub>. 1373, Stilbene dye intermediates.

#### PATENTS.

Increasing yield in the catalytic synthesis of aliphatic hydrocarbons. STUDIEN- U. VERWERTUNGS-GES. M.B.H. (B.P. 454,948, 10.4.35. Ger., 25.4.34).— The yield of liquid hydrocarbons obtained by hydrogenation of CO over Co catalyst at 180—200°/1 atm. is increased by operating in several stages of graded temp. within the above limits, the unconverted materials being removed after each stage. A. H. C.

[Preparation of] high-molecular alcohols and unsaturated hydrocarbons. W. H. MCALLISTER, Assr. to PROCTER & GAMBLE Co. (U.S.P. 2,022,894, 3.12.35. Appl., 2.12.32).-A H<sub>2</sub>O-sol. salt of a (saturated) aliphatic acid  $C_n$   $(n \not < 6)$  10-15, a low-mol. H<sub>2</sub>O-sol. aliphatic monohydric alcohol 10-30, and an alkali chlorate, perchlorate, bicarbonate, or sulphate 3-8.5% are dissolved in the remaining % of H2O and electrolysed, preferably with stirring and heating to 25-70° (50°) under reflux, at 2.5-30 (5) volts, 0.1-11.5 (3-8) amp. [c.d. 0.35-50 (3-8) amp./sq. dm.], using an anode made essentially of C which contains a substantial proportion of graphite. Olefines and alcohols  $C_{n-1}$  are formed, the latter partly as easily hydrolysable esters, and both are readily sulphonated by the usual agents. Yields of > 50% are claimed. E. J. B.

Manufacture of polymerisation products from olefines. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 453,854, 18.3.35).—Olefines are polymerised, either singly or mixed, by heating with stirring at  $> 50^{\circ}/(> 1 \text{ atm.})$  with a compound of BF<sub>3</sub> containing at least one H capable of being removed by dissociation, *e.g.*, BF<sub>3</sub>,2H<sub>2</sub>O, BF<sub>3</sub>,2EtOH, BF<sub>3</sub>,2AcOH, to yield liquid and/or resinous products. Examples of the treatment of C<sub>2</sub>H<sub>4</sub>, C<sub>3</sub>H<sub>6</sub>, butenes, and butadiene are given. E. J. B.

Polymerides of olefines. STANDARD OIL DEVELOP-MENT Co. (B.P. 455,114, 9.12.35. U.S., 8.12.34).— Olefines  $\langle C_5$  are converted into valuable polymerides by treating them in the liquid phase in presence of a solvent, e.g.,  $C_3H_8$ , at  $\langle -20^\circ (\langle -40^\circ \rangle)$ , with  $H_2SO_4$  or oleum kept liquid by dilution with a solvent containing S (CS<sub>2</sub>, SO<sub>2</sub>, S<sub>2</sub>Cl<sub>2</sub>, etc.). The examples describe the polymerisation of *iso*-C<sub>4</sub>H<sub>8</sub> at  $-36^\circ$  and  $-80^\circ$  and the working up of the products to give polymerides suitable for incorporation into lubricating oils. Suitable apparatus is described. S. C.

**Preparation of ethylene oxide.** E. C. BRITTON, G. H. COLEMAN, and B. MATE, ASSTS. to DOW CHEM. Co. (U.S.P. 2,022,182, 26.11.35. Appl., 12.5.34).— $(CH_2)_2O$  is produced by slowly adding 2.2 equivs. of alkali or alkaline-earth hydroxide or carbonate in H<sub>2</sub>O to well-agitated CH<sub>2</sub>Cl·CH<sub>2</sub>·OAc at 40—150° (100°). S. C.

Manufacture of halogen derivatives of methane. J. P. BAXTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 454,577, 2.4.35).—Chlorofluoromethanes,  $CCl_xF_{4-x}$ , are prepared by passing  $CS_2$  in a current of  $N_2$  through a molten mixture of  $SbCl_5$  with a F-derivative of Sb at 120°, washing, and fractionally condensing the exit gases at —75°. F may also be introduced as HF in the stream of  $CS_2$ . A. H. C.

**Preparation of 1: 1-** $[\alpha\alpha$ -]**dichloroethane.** CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 454,128, 11.2.36. Ger., 22.2.35).—C<sub>2</sub>H<sub>3</sub>Cl in a solution or suspension of AlCl<sub>3</sub> (1.5) in a chlorinated solvent, preferably CHMeCl<sub>2</sub> (100 pts. by wt.), is treated with gaseous HCl at  $< 35^{\circ}$ (0°). The mixture of HCl and C<sub>2</sub>H<sub>3</sub>Cl obtained by passing C<sub>2</sub>H<sub>2</sub> and HCl over HgCl<sub>2</sub> may be used. The yield is almost quant. E. J. B.

Purification of chlorinated hydrocarbons. E. C. BRITTON, G. H. COLEMAN, J. W. ZEMBA, and E. C. ZUCKERMANDEL, ASSTS. to DOW CHEM. CO. (U.S.P. 2,025,024, 24.12.35. Appl., 2.11.33).—The impurities formed on exposure to light of chlorinated aliphatic hydrocarbons (stable in absence of light), which give a ppt. with benzidine, are removed by treatment with a N base (primary amines, sec. amines with > 1 aryl group attached to the N, tert. alkylamines, and saturated ring bases). The examples describe the purification of C<sub>2</sub>Cl<sub>4</sub> by passing NH<sub>3</sub> through it or by mixing with aq. NH<sub>3</sub> or refluxing with NH<sub>2</sub>Ph. E. J. B.

Chlorination of aliphatic hydrocarbons. E. C. BRITTON, G. H. COLEMAN, and B. C. HADLER, ASSTS. to Dow CHEM. Co. (U.S.P. 2,018,345, 22.10.35. Appl. 1.2.33). —The olefines or chloro-olefines formed as by-products in the chlorination of  $C_3H_8$ ,  $C_4H_{10}$ , and  $C_5H_{12}$  (at  $\geq 200^\circ$ , generally at 300—600°), which are difficultly separated from the alkyl chlorides, are converted into readily separable saturated compounds by combination with the HCl also formed at  $\leq 200^\circ$  (in presence of a catalyst, e.g., kaolin at 70—80°). H. A. P.

Chlorination of ethylene hydrocarbons. M. BERLINER, Assr. to TEXAS Co. (U.S.P. 2,022,616, 26.11.35. Appl., 18.5.34).—Lower olefines are treated with  $Cl_2$  in a mixture of  $C_3H_5Cl_3$ ,  $C_3H_4Cl_4$ ,  $C_4H_7Cl_3$ , and  $C_4H_6Cl_4$  at about 5° to give increased yields of dichlorides. Suitable apparatus is described. S. C.

Chlorination [of aliphatic hydrocarbons]. B. GALLSWORTHY, ASST. to TEXAS CO. (U.S.P. 2,022,619, 26.11.35. Appl., 19.1.33).—Petroleum hydrocarbons (lubricating oils and waxes) are treated with  $Cl_2$  (or HCl-HOCl) in  $H_2O$  at 65—94°/25—500 lb. per sq. in. Apparatus is described. S. C.

[Manufacture of] halogenated acyclic hydrocarbons containing fluorine. H. W. DAUDT, M. A. YOUKER, and H. H. REYNOLDS, ASSTS. to KINETIC CHEMICALS, INC. (U.S.P. 2,024,095, 10.12.35. Appl., 20.6.33. Renewed 8.4.35).—Fluorination of halogenated acyclic compounds is so effected in presence of a catalyst that the F content of the catalyst remains substantially const. *E.g.*, a mixture of SbCl<sub>3</sub> and SbCl<sub>5</sub> is treated with HF, and a mixture of CHCl<sub>3</sub> and HF run in at 65—70°, the F content of the catalyst being kept between 2 and 4%; the products are CHCl<sub>2</sub>F and CHClF<sub>2</sub>. The prep. of CCl<sub>2</sub>F<sub>2</sub> and of CCl<sub>3</sub>F from CCl<sub>4</sub> is also described. P. G. C.

Manufacture of fluorinated organic compounds. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 453,830, 12.3.35).—HF is caused to react additively with an org. compound  $\ll C_{10}$  which contains one or more double linkings, but no triple linking, e.g.,  $C_{12}H_{24}$ , oleic or undecenoic acid, in an inert solvent (CH<sub>2</sub>Cl<sub>2</sub>, CCl<sub>4</sub>). Fluorinated compounds suitable for use as textile auxiliaries, insecticides, etc. are readily obtained. (Cf. B.P. 406,284, B., 1934, 393.) E. J. B.

Manufacture of tertiary alcohols. R. Z. MORAVEC and W. ENGS, ASSTS. to SHELL DEVELOPMENT CO. (U.S.P. 2,019,762, 5.11.35. Appl., 20.2.33).—The product of absorption of a *tert*. olefine  $(C_4H_8)$  in an aq. polybasic inorg. acid  $(H_2SO_4)$  is neutralised with a sufficient concn. of a base  $(NH_3)$  to form two phases ; the alcoholic phase is then separated and purified (by distillation).

H. A P.

Drying of alcohols and esters. B. T. BROOKS, Assr. to STANDARD ALCOHOL CO. (U.S.P. 2,022,274, 26.11.35. Appl., 15.9.31).—Esters and alcohols  $> C_2$  are dried by contact with conc. aq. solutions of NaOH, KOH, CaCl<sub>2</sub>, or ZnCl<sub>2</sub> containing < 60% (50%) of H<sub>2</sub>O. Suitable plant is described. S. C.

Vinyl ester. G. A. PERKINS, ASST. to CARBIDE & CAR-BON CHEMICALS CORP. (U.S.P. 2,021,698, 19.11.35. Appl., 25.1.33).—Paraldehyde and the anhydride of a lower aliphatic acid, when heated in presence of a non-volatile acid, best 0.5% of  $H_2SO_4$ , give vinyl esters. Most of the unchanged aldehyde and anhydride are recovered. Apparatus for continuous reaction and simultaneous fractionation of the product is described. R. S. C.

**Production of esters of polyglycerols.** B. R. HARRIS (U.S.P. 2,023,388, 3.12.35. Appl., 21.11.34).— Wetting and anti-spluttering agents are obtained by heating glycerol with a little NaOH, thereby obtaining a mixture of polyglycerols, the average mol. wt. of which varies according to the conditions, and then partly esterifying this mixture. The properties vary with the degree of "polymerisation" and esterification. R. S. C.

Manufacture of esters of glycols. G. H. COLEMAN and G. V. MOORE, Assrs. to DOW CHEM. Co. (U.S.P. 2,021,852, 19.11.35. Appl., 5.5.32).—(CH<sub>2</sub>Cl)<sub>2</sub>, NaOAc, and 0·1 mol. of a base (alkyl- or cyclohexyl-amine) at 150—250° (175—200°) give good yields of the diacetate. Other esters and those of other glycols are similarly obtained. R. S. C.

Wetting agent. B. R. HARRIS (U.S.P. 2,023,387, 3.12.35. Appl., 31.10.32).—Compounds containing hydrophilic and long-chain (lipophilic) groups are claimed as wetting agents and as agents for preventing spluttering of hot fat. Examples are the salts of pure or crude glycerol, glycols, hydroxycarboxylic acids, sugars, etc., esterified partly by  $H_2SO_4$  and partly by fatty acids. Mixtures with semi-oleins may have improved properties. Among the claims are mixtures of mono-olein disulphate and mono-oleyl diethylene glycol or of monolaurin sulphate and mono-octoin. R. S. C.

Manufacture of acetals of polyvinyl alcohol. I. G. FARBENIND. A.-G. (B.P. 454,013, 18.2.35. Ger., 17.2. and 25.8.34).—The acetals (I) are prepared in known manner from polyvinyl alcohol or its esters, and an aldehyde or ketone in presence of an acid catalyst, and the product, after removal of the solvent (if this is of comparatively high b.p.), is dissolved under pressure in a liquid that is normally a gas, or preferably the condensation is carried out in solution in the latter, *e.g.*, MeCl, Me<sub>2</sub>O, or preferably SO<sub>2</sub>, as this acts as a catalyst. The solution is injected into a bath of a non-solvent for (I) (H<sub>2</sub>O, EtOH-H<sub>2</sub>O), whereby the solvent volatilises and (I) is obtained as a fine, dry powder. E. J. B.

**Preparation of aluminium ethylate [ethoxide].** CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 454,480, 26.3.36. Ger., 9.5.35).—Al(OEt)<sub>3</sub> is prepared in 95% yield without the aid of catalysts by the action of Al on a slight excess of EtOH at 120° (autoclave), followed by distillation in vac. A. H. C.

Manufacture of organic sulphur compounds. COUTTS & CO., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 454,675, 12.6.35. Cf. B.P. 446,992 and 450,559; B., 1936, 972).—Vinyl sulphides or sulphoxides afford sulphonic acids when heated for several hr. in H<sub>2</sub>O or aq. alcohols at 70—130° with bisulphites of Na, K, Ca, or Mg. Among the examples, octadecyl vinyl sulphide, m.p. 33—34°, with NaHSO<sub>3</sub> (8 hr./100°) affords a cryst. pulp sparingly sol. in cold H<sub>2</sub>O; p-tolyl vinyl sulphide, b.p. 91—93°/9—10 mm., affords a sulphonic acid, decomp. 165°; dodecyl vinyl sulphoxide with NaHSO<sub>3</sub> affords white crystals, decomp. 205—210°, sol. in H<sub>2</sub>O, having high foaming power and soap-like character.

K. H. S.

Production of mercaptans and like organic sulphur compounds. CHEM. REACTIONS, LTD. From DEUTS. GOLD-U. SILBER-SCHEIDEANSTALT VORM. ROESSLER (B.P. 453,859, 19.3.35).—Mercaptans, thioethers, etc. are obtained by heating aliphatic or cycloaliphatic carboxylic acids, esters, or salts with  $H_2S$  and  $H_2$  in presence of the sulphide of a heavy metal of group I, VI, or VIII (Mo polysulphide) to 150—400° (250—350°). The  $H_2S$  may be replaced by S,  $CS_2$ , CaS,  $(NH_4)_2S$ , or  $Na_2S$ , which give  $H_2S$  during the reaction. The examples describe the prep. of EtSH and PrSH from  $EtCO_2Et$ ,  $C_{18}H_{37}$ 'SH and  $C_{18}H_{35}$ 'SH from oleic acid, and similar products from stearic acid, olive oil, and naphthenic acid. S. C.

Production of mercaptans and thio-ethers. W. M-LEE (U.S.P. 2,020,421, 12.11.35. Appl., 23.12.32).—The reactions,  $\text{RCO}_2\text{R}' + \text{H}_2\text{S} \Rightarrow \text{R'SH} + \text{RCO}_2\text{H}$  and  $\text{RCO}_2\text{R}' + \text{R''SH} \Rightarrow \text{R'SR''} + \text{RCO}_2\text{H}$ , in which R'=sec.- or, best, tert.-alkyl, occur at room temp. in presence of acid catalysts. Other esters, particularly H sulphates, and crude products from olefines and acids may be used. Crude tert.-amyl and Bu<sup>Y</sup> H sulphates are quoted as examples. R. S. C. Production of mercaptans of high mol. wt. COUTTS & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 454,668, 16.5.35).—Saturated and unsaturated aliphatic and cycloaliphatic alcohols containing  $\measuredangle$  C<sub>12</sub> are converted into mercaptans by passing in the vapour phase with H<sub>2</sub>S over dehydrating catalysts. Octadecyl, m.p. 34—35°, b.p. 170—171°/1·5 mm. (from octadecyl alcohol over ZrO<sub>2</sub>/320° or active C/300°), octadecenyl, b.p. 151—165°/1 mm. (sperm alcohol over Al<sub>2</sub>O<sub>3</sub>/300°), dodecyl, b.p. 95—100°/1 mm. (dodecyl alcohol over TiO<sub>2</sub>), and cetyl mercaptan, b.p. 140—155°/ 0·8 mm. (cetyl alcohol over ZrO<sub>2</sub>/320°), and α-thiol-Δ<sup>n</sup>octadecene, b.p. 159—161°/1·5 mm. (octadecane-αµ-diol over bauxite/320°), are described. K. H. S.

Preparation of organic acids. J. C. WOODHOUSE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,019,754, 5.11.35. Appl., 28.2.31).—An aliphatic alcohol, ether, or ester is caused to interact with CO in presence of an absorbent oxide (for gases), *e.g.*, of Si, Al, Mg, Ti, Zr, or W (at 250—500°/25—900 atm. in the vapour phase). The prep. of AcOH is described as example. H. A. P.

Concentration of aliphatic acids. BRIT. CELANESE, LTD. (B.P. 455,325, 7.10.35. U.S., 5.10.34).—In the concn. of AcOH from cellulose esterification processes by extraction methods, gummy substances deposited in the apparatus are removed by periodically treating the latter with the vapour of a solvent for the gums (usually conc. AcOH). Apparatus is described. S. C.

Preparation of organic acids. J. C. Woodhouse, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,025,676-7, 24.12.35. Appl., 25.10.32).-(A) A saturated monohydric aliphatic alcohol that volatilises without decomp., or a compound that will readily produce it on hydrolysis (esters, ethers, halides, or amines), is passed with CO, (preferably) H<sub>2</sub>O vapour, and a (poly-) halogenated aliphatic monocarboxylic acid, an ester thereof, or an acyl halide, preferably of the acid predominantly formed in the reaction, at 200-400°/25-900 (350-700) atm. over an absorbent surface (SiO<sub>2</sub> gel, active C) if desired. Inert gaseous diluents (N2, H2) may be used. E.g., CO 85, MeOH 5, H<sub>2</sub>O 5, H<sub>2</sub> 5, and CH<sub>2</sub>Cl·CO<sub>2</sub>H 2 pts. by wt. passed over active C at 325°/700 atm. produce chiefly AcOH. (B) A (poly-) olefinic hydrocarbon and excess of CO and steam are caused to react by a similar catalyst and under the same conditions as in (A) to yield predominantly the corresponding acid with one more C atom. E.g., CO 95, C2H4 5, H<sub>2</sub>O 25, and C<sub>2</sub>H<sub>4</sub>Cl·CO<sub>2</sub>H approx. 2 pts at 325°/700 atm. over active charcoal yield chiefly EtCO<sub>2</sub>H. The prep. of PrCO<sub>2</sub>H and BuCO<sub>2</sub>H is also claimed. E. J. B.

Synthesis of organic aliphatic acids. A. T. LARSON, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 2,022,244, 26.11.35. Appl., 18.8.33).—Aliphatic monocarboxylic acids are obtained by interaction of an olefine, CO, and  $H_2O$  in the liquid phase in presence of a catalyst which increases the solubility of CO (Cu, Hg salts) and an acid (HCl,  $H_2SO_4$ ,  $H_3PO_4$ ) at 150—365° (200—300°). The examples describe the prep. of EtCO<sub>2</sub>H from C<sub>2</sub>H<sub>4</sub> and various acid–Cu catalysts. S. C.

Preparation of aliphatic carboxylic acids. A. T. LARSON, ASST. to E. I. DU PONT DE NEMOURS & CO

(U.S.P. 2,020,689, 12.11.35. Appl., 25.10.32).—An olefine, C<sub>n</sub> (1·5—10·0 vol.-%), steam, and CO are passed with a small amount of a mono- or poly-halogenated hydrocarbon (to activate the reagents) at 200—400° (275— 375°)/25—900 atm. over active C. Inert gaseous diluents may be used to control the exothermic reaction. The carboxylic acid C<sub>n+1</sub> usually predominates. Eg., CO 95, C<sub>2</sub>H<sub>4</sub> 5, steam 25 pts., and CCl<sub>4</sub> 0·1 pt. by vol. at 325°/ 700 atm. yield 67% of EtCO<sub>2</sub>H. The prep. of PrCO<sub>2</sub>H and BuCO<sub>2</sub>H is also claimed. E. J. B.

Manufacture of polymerisable compounds [acrylic acid]. TRIPLEX SAFETY GLASS CO., LTD., and J. WILSON (B.P. 455,087, 13.3.35).—CN·CH<sub>2</sub>·CH<sub>2</sub>·OH is distilled in CO<sub>2</sub> with H<sub>2</sub>SO<sub>4</sub>. S. M.

Composition of matter [undecoic acid]. M. DE GROOTE and B. KEISER, Assrs. to TRETOLITE Co. (U.S.P. 2,020,998, 12.11.35. Appl., 31.12.34).—An acid with 6—16 C and I val. 120—190 (e.g., sardine oil) is oxidised by air at 150—250° until the I val. drops to 0.5—0.66of its original val. and then distilled at 270—330°/vac. The crude undecoic acid obtained may be used to break petroleum emulsions. R. S. C.

Sulpho-fatty body. M. DE GROOTE and B. KEISER, Assrs. to TRETOLITE Co. (U.S.P. 2,020,999, 12.11.35-Appl., 31.12.34).—The Na<sub>2</sub>,  $(NH_4)_2$ , or Na  $NH_4$  salt of the crude acidic product described in U.S.P. 2,020,998 (preceding) is claimed as a wetting agent and as an agent to break emulsions. R. S. C.

Manufacture of ethers and esters of paraffin wax. E. I. DU PONT DE NEMOURS & Co. (B.P. 453,522, 13.3.35. U.S., 13.3.34).—Halogenated paraffin wax is caused to interact with a metal derivative of an aliphatic or ar- or cyclo-aliphatic alcohol or a salt of an aliphatic, araliphatic, cycloaliphatic, or aromatic monocarboxylic acid. Examples are an acetate, b.p. 240—260°, stearate, m.p.  $31-34^\circ$ , and "lauryl" ether derived from a "monochlorinated" paraffin wax of b.p. 210—240°/3 mm. (calc. as  $C_{24}H_{50}$ ). The products are used as plasticisers and as ingredients of cosmetics, polishes, and textile assistants. H. A. P.

Manufacture of esters of phosphoric acid. CELL-ULOID CORP. (B.P. 455,014, 13.4.35. U.S., 13.4.34).— Esters of  $H_3PO_4$ , particularly neutral esters, are made by passing a mixture of the appropriate phenol or alcohol with POCl<sub>3</sub>, PCl<sub>5</sub>, or PCl<sub>3</sub> + Cl<sub>2</sub> through a series of retorts each heated to a higher temp. than that of those preceding. (C<sub>6</sub>H<sub>4</sub>Me)<sub>3</sub>PO<sub>4</sub> is made by heating cresylic acid and POCl<sub>3</sub> thus at 150°, 225°, and 300° or cresylic acid and POCl<sub>3</sub> at 50°, 75—100°, and 100—120° and hydrolysing the dichloride. The apparatus (figured) may be worked under vac. or inert gases passed during operation. K. H. S.

Manufacture of isopropyl esters of aliphatic acids. G. H. COLEMAN, ASST. to DOW CHEM. Co. (U.S.P. 2,021,851, 19.11.35. Appl., 9.3.32).—Pr<sup> $\beta$ </sup> esters are obtained by heating C<sub>3</sub>H<sub>8</sub> and an anhyd. aliphatic ester (1 mol.) in presence of H<sub>2</sub>SO<sub>4</sub> (0.05 mol.) at 75—250° under sufficient pressure to keep the reactants liquid. The prep. of Pr<sup> $\beta$ </sup> acetate, propionate, and butyrate is described. R. S. C.

[Preparation of the] oxidised product of waterinsoluble hydroxylated fatty acids. M. DE GROOTE and B. KEISER, Assrs. to TETROLITE Co. (U.S.P. 2,025,803, 31.12.35. Appl., 12.11.34).—Linseed or perilla oil is treated according to U.S.P. 1,745,221 (B., 1930, 519), the sulphated fatty acid hydrolysed to the hydroxylated acid, and the product oxidised by air-blowing at 150° for some hr. The products serve to resolve petroleum emulsions of the  $H_2O$ -in-oil type. K. H. S.

[Preparation of] ester(s) of hydroxycarboxylic acids. B. R. HARRIS (U.S.P. 2,025,984, 31.12.35. Appl., 8.1.34).—Hydroxycarboxylic acids containing at least one OH are esterified with aliphatic acids ( $\langle C_5 \rangle$ ). E.g., mucic acid is esterified with palmityl chloride and citric acid with stearyl chloride, both in  $C_5H_5N$ . K. H. S.

Production of fatty acid esters of s-dialkylethylene glycols. V. L. HANSLEY, Assr. to E. I. DU PONT DE NEMOURS & Co., INC. (U.S.P. 2,025,684, 24.12.35. Appl., 4.10.34).—Na-C<sub>5</sub> $H_{11}$ ·OH reduces acyloins with > 12 C mainly to the (?) meso-form of the glycol. Hydrogenation (Ni preferred) gives mainly the (?) dl-form at 175-250°/ 10-75 atm., but at 100° much of the meso-form is also obtained. The Na reduction may be effected in one reaction from the appropriate ester without isolation of the acyloin. Mixtures of esters or acyloins may be used. Esters of the glycols are also claimed. The following are described : dl-tetracosane-µv-diol, m.p. 123-124° (diacetate, m.p. 44-45°, -stearate, m.p. 28-30°, and -propionate, m.p. 35-36°), and mixtures thereof with the meso-form, m.p., e.g., 60-80°, 70-90° (diacetate, m.p. between 29° and 38°); dl-hexadecane-θι-diol, m.p. 129-130°; dl-octacosane-50-, m.p. 121-123° (diacetate, m.p. 51.5-52°), and dl-hexacosane-or-diol (diacetate, m.p. 65-66°), and lower-melting mixtures thereof with the corresponding meso-forms. R. S. C.

Preparation of esters of unsaturated acids. E. F. IZARD, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,020,685, 12.11.35. Appl., 21.10.33).—An alkyl or aryl halogenoformate is heated at 35—150°/1—100 atm. with an olefine in presence of halides of, e.g., Fe, Al, Bi, or B, to yield an ester of a halogenated saturated aliphatic acid, which is subsequently dehydrohalogenated by, e.g., NaOH-EtOH to yield the corresponding unsaturated ester. E.g., CICO<sub>2</sub>Me (from MeOH and COCl<sub>2</sub>) and C<sub>3</sub>H<sub>6</sub> yield CH<sub>2</sub>:CMe·CO<sub>2</sub>Me. E. J. B.

Sulphonation of higher aliphatic carboxylic acids and their esters. HANSAWERKE LÜRMAN, SCHÜTTE & Co., Assees. of H. GRABBE (B.P. 453,679, 17.10.35. Ger., 18.10.34).—Aliphatic carboxylic acids  $\measuredangle$  C<sub>10</sub> and their esters, particularly of glycerol, are sulphonated in  $\lt$  3 stages, with increasingly large quantities of progressively stronger sulphonating agent, the reaction in each case being allowed to cease before commencing the next. Sulphonation proceeds smoothly and without side reactions or gross decomp. Inert diluents may be used, and condensation with, e.g., alcohols or aromatic hydrocarbons may be effected simultaneously. E.g., coconut oil 10 pts. and 7.5% of glycerol are treated with (a) 100%  $H_2SO_4 5$ , (b) 10% oleum 5, (c) 20% oleum 10, and (d) 30% oleum 25 pts. The products are recovered in known E. J. B. manner.

Manufacture of cyclic ketones. I. G. FARBENIND. A.-G. (B.P. 453,518, 13.3.35. Ger., 31.3.34).—A  $\gamma$ -hydroxycarboxylic acid, its lactone, or the corresponding

unsaturated acid is heated with a dehydrating catalyst. E.g., by heating  $\gamma$ -methyl- $\alpha$ -heptylbutyrolactone, b.p. 150—152°/6 mm., with "Frankonite" (hydrated Al Mg silicate) at the b.p. a ketone (? 1-heptyl- $\Delta^1$ -cyclopentenone), b.p. 105—110°/4 mm. (semicarbazone, m.p. 196— 197°), is formed. Other examples describe the prep. of ketones from  $\gamma$ -ethyl- $\alpha$ -heptyl-, b.p. 110—115/5° mm. (semicarbazone, m.p. 155—160°),  $\gamma$ -n-amyl-, b.p. 90—95°/ 3—4 mm. (semicarbazone, m.p. 198—199°), and  $\gamma$ -heptylbutyrolactone, b.p. 100—105°/5 mm. (semicarbazone, m.p. 193—194°), and allylbutylacetic acid (or  $\gamma$ -methyl-  $\alpha$ -butylbutyrolactone), b.p. 90—95°/4 mm. (semicarbazone, m.p. 200—201°). H. A. P.

Preparation of diketen. G. H. LAW, ASST. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 2,019,983, 5.11.35. Appl., 14.10.32).—Gases produced by pyrolysis of COMe<sub>2</sub> (at 650°) are rapidly cooled (to -10°) and the resulting dil. solution of keten in COMe<sub>2</sub> (4-10%) is allowed to warm up slowly to room temp. If more conc. solutions are used the initial temp. must be lower. H<sub>2</sub>O must be rigidly excluded. H. A. P.

Preparation of acylated polyalkylenepolyamines. I. G. FARBENIND. A.-G. (B.P. 454,723, 6.4.35. Ger., 7.5.34).—Aliphatic or alicyclic acid amides ( $>C_8$ ) are heated to 150—180° with an excess of a polyalkylenepolyamine, or a mixture of such bases and the acylated amine is isolated by concn. in vac. Examples are the reactions of the amides of stearic, oleic, and ricinoleic acids with triethylenetetramine or mixtures containing it. A. H. C.

Manufacture of sulphonylethylamines. COUTTS & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 453,780, 18.3.35. Addn. to B.P. 442,525; B., 1936, 443).—Sulphonylethylamines are obtained by interaction of  $C_2H_2$  at 50-250° (100-180°) with mixtures of sulphinic acids or their salts and compounds containing one or more NH groups (NH3, primary and sec. amines or OH-amines, piperidine, carbazole, etc.), in presence of a basic catalyst (oxides, alkoxides, phenoxides, or naphthoxides of alkali metals; ZnO, CdO, and/or HgO). The examples include the prep. of  $\beta$ -p-toluenesulphon-The examples include the prop. of p-p-toluenesulphon-triethylamilde (oxalate, m.p. 160°);  $\beta$ -p-toluenesulphon-diethylanilide, m.p. 71—72°;  $\beta$ -p-toluenesulphonethyl-piperidide (hydrochloride, m.p. 226°); bis- $\beta$ -(p-toluene-sulphonyl)ethylamine (hydrochloride, m.p. 200—201°); m-bis-(B-diethylaminoethanesulphonyl)benzene (dioxalate, m.p. 143—144°);  $\alpha\beta$ -bis-( $\beta'$ -diethylaminoethanesulphonyl)ethane, and the compound from p-toluenesulphinic acid, C<sub>2</sub>H<sub>2</sub>, and NHPh·NH<sub>2</sub>. S. C.

Preparation of tert.-butyl-m-xylene. W. V. WIRTH, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,023,566, 10.12.35. Appl., 23.2.34).—The yield and quality of  $1:3:5-C_6H_3Me_2Bu^{\gamma}$  from m-xylene and Bu^Cl are raised by using < 2% of AlCl<sub>3</sub> (on the wt. of Bu^Cl), and effecting the reaction at  $< 50^{\circ}$ . P. G. C.

Oxidation of hydrocarbons. F. J. DE REWAL, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 2,022,845, 3.12.35. Appl., 11.8.31).—Hydrocarbons (I), particularly of the  $C_{10}H_8$  and anthracene series, are oxidised in the vapour phase by treatment at 300—400°, preferably at > 1 atm., with N oxides  $\leq 3$  (23—25)% of the total gas vol., and  $O_2$  (air) so that there are  $\leq 2$  mols. of available O per mol. of (I). The exit gases are fractionally cooled so that only the oxidation product of (I) separates out. The catalyst gases are purified and recycled with more (I). Inert gaseous diluents (steam) may be used, and formation of  $NO_2$ -compounds is inhibited. *E.g.*, 90% yields of anthraquinone are obtained from anthracene. Apparatus is described. E. J. B.

Production of light-coloured liquid chlorinated naphthalene. E. R. HANSON and S. BROWN, ASSTS. to HALOWAX CORP. (U.S.P. 2,025,742, 31.12.35. Appl., 2.4.34).—Crude liquid chlorinated  $C_{10}H_8$ ,  $d^{150} \gg 1.25$ , is distilled at pressures  $\gg 100$  mm. Hg in presence of a base (CaO), and the fraction which develops no cloudiness at  $-5^{\circ}$  and has colour lighter than 2 (of the Amer. Paint & Varnish Manufrs.' Assoc. glass standard) is collected. K. H. S.

Halogenated diphenyl oxide. E. C. BRITTON, W. C. STOESSER, and G. G. GOERGEN, ASSTS. to DOW CHEM. Co. (U.S.P. 2,022,634, 26.11.35. Appl., 3.7.33).—Products containing 5—10 halogen atoms are obtained by treating Ph<sub>2</sub>O or its lower halogenated derivatives with Cl<sub>2</sub> or Br at elevated temp. (150°) in presence of a catalyst (FeCl<sub>3</sub>, AlCl<sub>3</sub>). The products may be used as heat-transfer or -storage agents, for electrical insulation, and for mixing with lubricating oils; some of them are suitable as plastics. S. C.

Catalyst [for] and process of hydrogenating organic compounds. A. M. MUCKENFUSS, ASST. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,021,567, 19.11.35. Appl., 6.11.31).—Crude or pure polycyclic aromatic hydrocarbons are hydrogenated at high temp. and pressure in presence of Na, which reacts with the hydrocarbon, RH, to form NaR, which is the actual catalyst and is relatively resistant to poisoning by S. The hydrogenation of  $C_{10}H_{s}$ , anthracene, acenaphthene, and hubricating oil is described. R. S. C.

Manufacture of musk xylene [2:4:6-trinitro-5tert.-butyl-m-xylene]. W. V. WIRTH, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,023,565, 10.12.35. Appl., 1.2.34).—1:3:5:2:4:6-C<sub>6</sub>Me<sub>2</sub>Bu $\gamma$ (NO<sub>2</sub>)<sub>3</sub> is prepared by adding 1:3:5-C<sub>6</sub>H<sub>3</sub>Me<sub>2</sub>Bu $\gamma$  to a HNO<sub>3</sub>-H<sub>2</sub>SO<sub>4</sub> mixture (containing 12.6 to 50% of HNO<sub>3</sub> by wt.) at 20—40°, and finally raising the temp. to 70—112°. P. G. C.

Manufacture of aliphatic-aromatic compounds [long-chain alkylbenzenes and their sulphonic acids]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 453,778, 15.3.35).—Acid chlorides ( $> C_5$ ) are condensed with aromatic hydrocarbons in presence of AlCl<sub>3</sub> and the resulting ketones hydrogenated to the corresponding hydrocarbons by treatment in the liquid phase with H<sub>2</sub> (50—100 atm.) and a hydrogenation catalyst (Ni) at  $\Rightarrow$  140°. The products are sulphonated to give wetting, emulsifying, and foaming agents. The examples include the prep. of *laurophenone*, b.p. 174°/3 mm., n-dodecylbenzene, b.p. 138°/2 mm., [sulphonic acid and its Na and N(C<sub>2</sub>H<sub>4</sub>·OH)<sub>3</sub> salts], and mixed hydrocarbons and sulphonic acids from hardened palm-nut oil fatty acids. S. C.

Preparation of arylamines. H. A. LUBS and J. E. COLE, ASSTS. to E. I. DU PONT DE NEMOURS & CO. (U.S.P.

2,025,876, 31.12.35. Appl., 11.4.31).—5-Chloro-3-thiol-o-toluidine and 4-chloro-2-thiolaniline are obtained simultaneously with 5-chloro-o-toluidine and 2:  $4-C_6H_3Cl_2\cdot NH_2$ , respectively, by alkaline hydrolysis of 2-(5'-chloro-o-toluidino)-4-methyl- and 6-chloro-2-(2': 4'-dichloroanilino)-benzthiazole. K. H. S.

Production of arylamides. H. A. LUBS, E. F. HITCH, and M. A. DAHLEN, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,025,116, 24.12.35. Appl., 22.8.33).— Amides of alkylthiolanilines and hydroxy-aromatic acids are claimed as dye intermediates. By condensation by PCl<sub>3</sub> in PhMe are obtained 2-hydroxy-3-naphth-p-, m.p. 237—238°, and -o-methylthiol-, m.p. 134—135.5°, and -4-ethoxy-2-methylthiol-anilide, m.p. 117—118°, and -5-chloro-3-methylthiol-o-toluidide, m.p. 199.5—200.5°. R. S. C.

Desulphonation of diaminodiphenylamine-2-sulphonic acid compounds. L. M. LAUER, ASST. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 2,022,889, 3.12.35. Appl., 9.2.35).—The acids are desulphonated by 60—80%  $H_2SO_4$  at 60—130° (100—110°); e.g., 4:4'-diaminodiphenylamine is obtained from the 2-sulphonic acid (1 pt.) and 76%  $H_2SO_4$  (4 pts.) at 100—110° (1·5 hr.). R. S. C.

Manufacture of [aryl] aminoalkyl sulphones. Courts & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 453,443, 18.3.35).—Benzenesulphinic acids are caused to interact with halogenoalkylamines or their salts (in neutral or slightly acid aq. solution). *E.g.*,  $Cl^{-}[CH_2]_2$ ·NEt<sub>2</sub>,HCl, p-C<sub>6</sub>H<sub>4</sub>Me·SO<sub>2</sub>Na, and NaOH in EtOH at the b.p. give p-tolyl  $\beta$ -diethylaminoethyl sulphone (oxalate, m.p.165—166°); similarly, p-NHAc·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>Na gives p-acetamidophenyl  $\beta$ -diethylaminoethyl sulphone and some ethylenebis-p-aminophenyl sulphone, m.p. 286° (inhibited by NHEt<sub>2</sub>). Other examples are 2-acetamido-panisyl, m.p. 102—103° (free amine, m.p. 75°), and -p-tolyl, 2-nitro-p-tolyl, m.p. 71—72°, and 4-acetamido-o-tolyl  $\beta$ -diethylaminoethyl sulphone. H. A. P.

Manufacture of amino-sulphones. Courts & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 454,728, 10.5.35).—Amino-sulphones are made by the interaction of alkylenebisaryl sulphones with dialkylamines, a mol. of arylsulphinic acid being eliminated.  $(CH_2 \cdot SO_2 \cdot C_6 H_4 Me-p)_2$  and NHEt<sub>2</sub> in EtOH (6 hr./ 140—150°) afford *p*-tolyl  $\beta$ -diethylaminoethyl sulphone (see preceding abstract). Similarly are obtained the corresponding  $NBu^a_2$ -compound, an oil (oxalate, m.p. 173°), and m-acetamido-, cryst., and m-nitro-p-tolyl  $\beta$ -diethylaminoethyl sulphone, m.p. 71—72°. K. H. S.

Refining of alkylphenols [aryl alkyl ethers]. R. SCHULER, ASST. to STANCO, INC. (U.S.P. 2,022,256' 26.11.35. Appl., 1.12.33).—Alkylphenols containing  $\measuredangle C_4$  in the side-chain are extracted from aryl alkyl ethers and other by-products with liquid NH<sub>3</sub>. The example describes the separation of (mixed) tolyl *sec.*-hexyl ethers from commercial cresol. S. C.

Production of  $\alpha$ -naphthol. W. J. COTTON, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 2,025,197, 24.12.35. Appl., 11.6.34).— $C_{10}H_8$  is added slowly with stirring to 3—4 times its wt. of > 93 (98—100)% H<sub>2</sub>SO<sub>4</sub>, at 15—30° (20—25°), and the 1- $C_{10}H_7$ ·SO<sub>3</sub>H so formed is caused to crystallise out (as dihydrate) immediately the solution becomes saturated with it, for  $\measuredangle$  3 hr. after the addition of the C<sub>10</sub>H<sub>8</sub> has ceased, by seeding, the acid conen. being controlled at  $\bigstar$  80% H<sub>2</sub>SO<sub>4</sub>. The product, containing  $\end{Bmatrix}$  2% of the 2-isomeride, is washed with cone. H<sub>2</sub>SO<sub>4</sub> at 20—25°, after a total reaction time of 2—40 (10—15) hr., and fused with alkali in an inert atm. at 275—310° (280—285°) for 5½—6 hr. in known manner. E. J. B.

Chlorodihydroxyalkylbenzenes. W. E. AUSTIN (U.S.P. 2,023,160, 3.12.35. Appl., 14.3.34).—Chlororesorcinol (from resorcinol and S<sub>2</sub>Cl<sub>2</sub> at 70°), Bu<sup>y</sup>OH, HCl, and ZnCl<sub>2</sub> at 105° give chlorotert.-butylresorcinol, b.p. 135—140°/5 mm., cryst. Chlorotert.-amyl-resorcinol, cryst., b.p. 145—150°/7 mm., -quinol, cryst., b.p. 148— 150°/4 mm., and -pyrocatechol, b.p. 135—140°/3 mm., and chlorodiisobutyl-resorcinol, m.p. 103—104°, and -pyrocatechol, cryst., b.p. 150—165°/5 mm., are similarly prepared and have germicidal properties. R. S. C.

Manufacture of hydroaromatic amino-alcohols and derivatives thereof. A. G. BLOXAM. From Soc. CHEM. IND. IN BASLE (B.P. 454,042, 30.5.35). - Acylaminophenols and their derivatives are reduced with H<sub>2</sub> and a base-metal catalyst in an org. solvent, and the products hydrolysed if desired. E.g., p-OH·C<sub>6</sub>H<sub>4</sub>·NHAc with H<sub>2</sub>-Ni in EtOH at 180° gives trans- (I), m.p 164°, and cis-4-acetamidocyclohexanol (II), m.p. 135° (free amines, m.p. 110-111° and 78-80°; hydrochlorides, m.p. 223- $225^{\circ}$  and  $195^{\circ}$ , respectively). Other examples describe the prep. of trans-, m.p.  $72-73^{\circ}$  (N-Ac derivative, m.p. 146°; hydrochloride, m.p. 185°), and cis-2-amino-, m.p. 65° (N-Ac derivative, m.p. 124-125°; hydrochloride, m.p. 175°), and 3-amino-cyclohexanol, m.p. 73° [N-Ac derivative, m.p. 120°; hydrochloride, m.p. 174°; Et ether, b.p. 74–76°/3 mm. (Ac derivative, m.p. 98°)]; the N-Bz, m.p. 224°, N-acetyl-N-methyl, m.p. 98°, N-Me, m.p. 116°, N-acetyl-O-n-propyl, m.p. 120°, b.p. 147-148°/0.01 mm., and N-acetyl-O-ethyl, m.p. 155°, derivatives, and Et, m.p. 60-62°, and Pra ether b.p. 114-116°/4 mm., of (I); the N-acetyl-N-methyl, m.p. 72°, N-Me, b.p. 94 $-100^{\circ}/3$  mm., N-acetyl-O-ethyl-, m.p. 55°, and -n-propyl, b.p. 125 $-130^{\circ}/0.03$  mm., derivatives, and the Et, b.p. 50-60°/5 mm., and Pr<sup>a</sup> ether, b.p. 80-82°/4 mm., of (II); a Bz derivative, m.p. 206°, and Me, b.p. 52-56°/4 mm. (Ac derivative, b.p. 140-148°/5 mm.), and Et, b.p. 55-60°/3 mm. (Ac derivative, b.p. 130°/ 3 mm.), ethers of 2-aminocyclohexanol, and a Me ether, b.p. 59°/5 mm. (Ac derivative, m.p. 67°), of 4-amino-H. A. P. cyclohexanol.

Production of monocarboxylic acids [benzoic acid] and their derivatives. (A) A. O. JAEGER, (B) J. E. JEWETT, ASSTS. to AMER. CYANAMID & CHEM. CORP. (U.S.P. 2,020,505-6, 12.11.35. Appl., 26.7.30).—(A) A salt of a polycarboxylic acid and a strong inorg. base together with a powdered diluent are caused to react in a uniformly heated rotary kiln containing heavy balls (e.g., of metal) and divided into sections by perforated screens that retain only the balls. Agglomeration of the material is avoided. E.g., o-C<sub>6</sub>H<sub>4</sub>(CO<sub>2</sub>)<sub>2</sub>Ca 2·0, Ca(OH)<sub>2</sub> 1·2 mols., and 2/3 of their combined wt. of CaCO<sub>3</sub> at 420—450° give 85—92% yield of Ca(OBz)<sub>2</sub>. (Cf. U.S.P. 1,727,102, ; B., 1929, 845.) (B) The kiln is divided into a larger no. of sections than in the prior patent by baffles attached

alternately to the centre shaft and to the walls, which retain the balls and cause the reagents to take a sinuous path. E. J. B.

Aralkyl trithiocarbonates. R. W. HESS, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 2,021,726, 19.11.35. Appl., 29.3.30).—CH<sub>2</sub>Ph·SH, CS<sub>2</sub>, and conc. NaOH give *Wa benzyl trithiocarbonate*, CH<sub>2</sub>Ph·S·CS·SNa, cryst., which with HCl affords the *acid*, m.p. 5—7°, with KI<sub>3</sub> affords the *disulphide*, (CH<sub>2</sub>Ph·S·CS·S·)<sub>2</sub>, m.p. 86— 88°, and with CH<sub>2</sub>PhCl gives *dibenzyl trithiocarbonate*, m.p. 13—15°. Similar aralkyl compounds are also claimed but not described. R. S. C.

Reaction of sodium with [naphthalene] hydrocarbons. N. D. Scott, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,023,793, 10.12.35. Appl., 29.6.33).—Reaction between  $C_{10}H_8$  (or its homologues) and an alkali metal is effected in a fully alkylated aliphatic di- or poly-hydroxy-compound, preferably in a N<sub>2</sub> atm.; *e.g.*,  $C_{10}H_8$  and Na in (CH<sub>2</sub>·OMe)<sub>2</sub> when treated with CO<sub>2</sub> afford a mixture of dihydronaphthalenedicarboxylic acids (including the 1: 4- and 1: 2-acids). P. G. C.

Production of nucleus-substituted aliphaticaromatic sulphuric acids [textile assistants]. CHEM. FABR. R. BAUMHEIER A.-G. (B.P. 454,183, 9.7.35. Ger., 23.1.35).—Polynuclear compounds are condensed with fatty acids  $\bigstar$  C<sub>10</sub> in presence of " alcohols corresponding to the acids of the natural fats " (having  $\ll C_7$ ) or their SO4 esters, and the products sulphonated ; alternatively, sulphonation may precede condensation. E.g., a mixture of n-C16H33 OH and C10H8 is sulphonated and condensed by means of 25% oleum, castor oil and further oleum are added, and condensation is continued at 50° until the product is H<sub>2</sub>O-sol. In other examples tetralin, β-C<sub>10</sub>H<sub>7</sub>·OH, and β-C<sub>10</sub>H<sub>7</sub>·NH<sub>2</sub> are used as starting materials and alcohols  $\langle C_7$  are used in addition to the higher alcohols. The products are claimed to have wetting, detergent, emulsifying, and fat-splitting properties. H. A. P.

Manufacture of dicarboxylic acid esters. C. W. HAWLEY, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,020,356, 12.11.35. Appl., 16.3.34).—A polycarboxylic acid anhydride, or the acid with a small amount of a condensing agent, is heated with insufficient of an alcohol (b.p.  $\geq 200^{\circ}$ ) to esterify it completely. A slow stream of a liquid or gaseous alcohol is introduced below the surface of the liquid at > the b.p. of the alcohol to complete the esterification and to remove the H<sub>2</sub>O formed as an azeotrope. Simple or mixed esters are readily produced in good (> 90%) yield. The condensation of  $o - C_6 H_4(CO)_2 O$  with  $OH \cdot [CH_2]_2 \cdot OMe$  is described in detail. E. J. B.

[Preparation of  $\alpha$ -]acetotricarballylic esters. Z. ZWILGMEYER, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 2,017,882, 22.10.35. Appl., 18.4.30).—An ester of CHAcNa·CO<sub>2</sub>H is condensed with a halogenosuccinic ester. *E.g.*, Diamyl chlorosuccinate is condensed with CHAcNa·CO<sub>2</sub>Et in abs. EtOH at room temp. to give  $\alpha$ -Et  $\beta\gamma$ -diamyl- $\alpha$ -acetotricarballylate, b.p. 230°/10 mm. (slight decomp.); the  $\alpha$ -Et  $\beta\gamma$ -Bu<sub>2</sub> ester, b.p. 220°/10 mm., is similarly made. The products are used as plasticisers (for cellulose lacquers). H. A. P.

Manufacture of organic compounds containing acid salt-forming groups [textile assistants]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 452,866, 23.2.35).-The compounds claimed have the general formula R·O·R'·X, R denoting an aromatic or hydroaromatic radical carrying as substituent at least one aliphatic radical of  $\ll C_3$  or an araliphatic radical, R' an aliphatic residue the C chain of which is interrupted at least once by O, S, or N, and X an acid saltforming group. They are surface-active and are used as detergents, wetting, dispersing, and emulsifying agents, etc. E.g., p-isohexylphenol is condensed with Cl·[CH<sub>2</sub>]<sub>2</sub>·OH and NaOH in EtOH and the product is further condensed with NaOEt and CH2Cl·CO2Na; similar products are obtained from *p*-isobutylphenol or p-isobutylcyclohexanol, (CH2)20 (2 mols.), and CH<sub>2</sub>Cl·CO<sub>2</sub>Na. Other examples describe the condensation of 2- or 4-OH·C<sub>6</sub>H<sub>4</sub>·CH<sub>2</sub>Ph with  $(CH_2)_2$ S to give the  $\beta$ -thiolethyl ether and of *p*-*n*-dodecylphenol with (CH<sub>2</sub>)<sub>2</sub>NH to give the β-aminoethyl ether; these are condensed with CH2Cl·CO2H or Cl·[CH2]2·SO3H as before. H. A. P.

[Preparation of] chlorobenzoylaminoanthraquinones. A. J. WUERTZ and W. DETTWYLER, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,019,837, 5.11.35. Appl., 22.6.34).—8-Chloro-1-benzamido- and 1:8-dibenzamido-anthraquinone are chlorinated in an inert solvent of high b.p. in the presence of AcOH and an acid-binding agent (NaOAc or KOAc). Thus are produced (at 95—100°) 4-chloro- and 4:5-dichloro-1:8dibenzamido-, and 4:8-dichloro- and 4:5:8-trichloro-1benzamido-anthraquinone. H. A. P.

Manufacture of [hydr]oxyalkylated nitrogen bases. W. W. GROVES. From ACETA GES.M.B.H. (B.P. 454,320, 24.1.35).—Amine hydrohalides or other salts react smoothly with chloroalkylene oxides at room temp. or 50-60°, best in H<sub>2</sub>O, if necessary in presence of an emulsifying agent to assist homogeneity. The products may be sec., tert., or quaternary bases according to the proportions of the reagents and the conditions. Piperidine hydrochloride (0.9 mol.) and epichlorohydrin (I) (1 mol.) give N- $\gamma$ -chloro- $\beta$ -hydroxypropylpiperidine (50%) and di-γ-chloro-β-hydrxoypropylpiperidinium chloride (25%). Tetrahydroxyethylethylenediamine dihydrochloride and (I) (2 mols.) give the diquaternary salt. Glycine and (I) (3 mols.) give the betaine. NH<sub>4</sub>Cl and (I) (4 mols.) give the quaternary salt. CH2Cl·CH(OH)·CH2·O·SO3Na and NH2Bu,HCl (0.33 mol.) or Na Me βγ-oxidopropyl phosphate and piperidine hydrochloride at 40° give materials useful as textile assistants. R. S. C.

Preparation of derivatives of pyridine and quinoline. KODAK, LTD., Assees. of L. G. S. BROOKER and G. H. KEYES (B.P. 454,687, 9.9.35. U.S., 9.9.34).— Mercaptans are allowed to react with quaternary  $C_5H_5N$ or quinoline bases having I at 2. Alkyl, aryl, or heterocyclic thio-ethers are formed. Thus 2-phenylthiolquinoline ethiodide, m.p. 181—182°, is formed from 2iodoquinoline ethiodide and PhSH (corresponding methiodide, m.p. 184—185°). Similarly are obtained 2-p-tolyl-, m.p. 226—228°, 2- $\beta$ -naphthyl- (I), m.p. 221—222°, 2- $\beta$ -naphthyl-6-methyl-, m.p. 220—221°, 2-2′: 5′dichloro-

CL. IV .- DYESTUFFS.

PATENTS.

Manufacture of [dioxazine] dyestuff-sulphonic acids. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 454,302, 25.3.35. Addn. to B.P. 447,899; B., 1936, 878).—The process of the prior patent is applied to diarylaminobenzoquinones prepared from sulphonated amino-diaryls, -diaryl ethers, -diaryl sulphides, or -diarylureas. E.g., the condensation product from chloranil (I) (1 mol.) and 4:4'-diaminodiphenyl-urea-3:3'-disulphonic acid (2 mols.; in aq. EtOH + NaOAc) is heated with  $H_2SO_4$  at 130° to give a blue dye. Similar products are described starting from (I) and  $4: 2-\mathrm{NH}_2 \cdot \mathrm{C}_6\mathrm{H}_4 \cdot \mathrm{O} \cdot \mathrm{C}_6\mathrm{H}_4 \cdot \mathrm{SO}_3\mathrm{H}$  (red),  $4: x-NH_2 \cdot C_6H_4 \cdot S \cdot C_6H_4 \cdot SO_3H$ (blue-red), 4: 3-NH2.C6H4.C6H4.SO3H (red), and sulphonated  $1: 4-C_{10}\tilde{H}_6Ph\cdot \tilde{NH}_2$  (blue-violet); benzoquinone, its Cl2-derivative, and toluquinone may be used instead of (I). H. A. P.

Improving the properties of phthalocyanine dyes. Courts & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 454,858, 5.4.35).—Phthalocyanines which disperse well are obtained by dissolving the dye in a suitable solvent (e.g.,  $H_2SO_4$ ,  $CISO_3H$ , or  $CCl_3 \cdot CO_2H$ ) and treating with an aq. dispersion or solution containing a substratum (e.g.,  $BaSO_4$ , kieselguhr, or a  $SiO_2$  gel stable to acid). If suspended pigment dyes stable to acid are used as substrata, easily dispersable pigments of a variety of shades are obtainable. Instead of substrata, agents capable of forming substrata with the solvent may be used. F. M. H.

C<sub>5</sub>H<sub>5</sub>N etc. derivatives.—See III.

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

Reactivity of the sulphur linkage in animal fibres. II. Action of baryta and caustic soda on human hair. J. B. SPEAKMAN and C. S. WHEWELL (J. Soc. Dyers and Col., 1936, 52, 380-387; cf. B., 1936, 1033).-Human hair treated with Ba(OH), solution at room temp. loses S, with corresponding loss in setting power, the latter being  $\infty$  the fall in S content. This confirms the view that permanent set involves. the formation of ·S·NH· linkings between the peptide chains, by interaction of SOH and NH<sub>2</sub> groups. Determination of the S content of Ba(OH)<sub>2</sub>-treated fibres by Carius' method proved difficult initially, owing to the presence of a non-oxidisable, H<sub>2</sub>O-sol. compound in the tube, due to combination between Ba and the fibre, as shown by the high ash content of treated fibres. It is suggested that this combination is of the form 'S·Ba·S·, which is supported by the reduced tendency of treated fibres to super-contract in boiling NaHSO<sub>3</sub>. Such reduced tendency is, however, progressive with time of treatment, and cannot be attributed to increasing nos. of ·S·Ba·S· linkings, since the ash content of treated fibres is const. Other NaHSO3-resistant linkings are therefore formed, probably of the form 'C·S·C', as is indicated by the reduced contraction of fibres treated with NaOH. Load-extension curves indicate that the no. of linkings built up is small. The possibility of the formation of linkings of the type 'N:CH' in keratin, by alkaline treatment, is discussed, but it is shown

phenyl- (II), m.p. 205—207° (decomp.), 2-p-chlorophenyl-(III), m.p. 203—205° (decomp.), 2-n-propyl-, m.p. 177— 179°, 2-n-butyl-, m.p. 149—151°, 2-1'-benzothiazolyl-thiolquinoline ethiodide, m.p. 180—181°. The methiodides corresponding with (I), (II), and (III) have m.p. 185— 187°, 151—152°, and 185—187° respectively. The products are of use in the prep. of cyanine dyes.

K. H. S.

Nitrogen-containing esters. B. R. HARRIS (U.S.P. 2,023,075, 3.12.35. Appl., 11.1.34).—Compounds,  $CO_2R\cdot CH_2\cdot NR'R''R''Hal$  (R contains  $\lt 4$  C), are obtained by treating  $CH_2$ Hal· $CO_2H$  with a *tert*.-base and alcohol simultaneously or consecutively and are claimed as wetting agents, emulsifiers, etc. The prep. of 28 such compounds, *e.g.*, carbocholesteroxyquinaldinium bromide, is detailed. The products from mono-oleylethylene glycol chloroacetate and NMe<sub>3</sub>, and from monostearin,  $C_5H_5N$ , and  $CH_2Cl\cdot COCl$ , are speciallyclaimed. R.S.C.

Production of cyclical disubstituted [1:5-polymethylene]tetrazoles. Z. Földi, Assr. to CHINOIN Gvögyszer- és Vegyészeri Termékek Gyára R.T. (KERESZTY & WOLF) (U.S.P. 2,020,937, 12.11.35. Appl., 29.8.34. Hung., 2.9.33).—Aliphatic azido-nitriles (from NaN<sub>3</sub> and chloro-nitriles) with H<sub>2</sub>SO<sub>4</sub> or ClSO<sub>3</sub>H at room temp. give dicyclic tetrazoles,  $(CH_2)_n < \stackrel{Q:N}{N \cdot N} N$ , exothermally. Thus are prepared 1:5-trimethylene-, m p. 110°,  $\gamma$ -methyl- $\alpha$ -ethyl-1:5-trimethylene- b.p. about 160°/ 1 mm., 1:5-tetramethylene-, m.p. 115°, and  $\alpha\gamma$ -dimethyl-1:5-tetramethylene-, m.p. 95—96°, -tetrazole and Et 1:5tetramethylenetetrazole- $\delta$ -carboxylate, m.p. 41°, b.p. about 172°/0.5 mm. ( $\alpha$  is the C adjacent to N<sub>(1</sub>)). R. S. C.

Manufacture of ether derivatives of morpholine alcohols. H. L. Cox and T. F. CARRUTHERS, Assrs. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 2,023,872, 10.12.35. Appl., 31.3.33).—N-Alkoxyalkylmorpholines are prepared by alkylating N-hydroxyalkylmorpholines. N- $\beta$ -Ethoxyethyl-, b.p. 199—200°/750 mm., 80—81°/10 mm., and 2:6-dimethyl-4- $\beta$ -methoxy-n-propyl-morpholine, b.p. 40°/12 mm., are specifically claimed. R. S. C.

Preparation of 1-phenyl-3-methyl-5-pyrazolone and related compounds. CARBIDE & CARBON CHEM-ICALS CORP., Assees. of F. JOHNSTON (B.P. 455,094, 13.4.35. U.S., 21.4.34).—See U.S.P. 2,017,815; B., 1936, 1144.

Lubricants.—See II. BF<sub>3</sub>etc.—See VII. Emulsifying agent. Polishing etc. agents.—See XII. Polymerising methacrylonitrile. Hydroabietoyl chloride. Condensation products.—See XIII. Sulphurising organic compounds. S condensation products.—See XIV. Cholenic acid etc. Aminobenzthiazoles.—See XX. Diazo-type layers.—See XXI. Pb salts of dinitrosalicylic acid. Salts of diazoaminotetrazole.—See XXII.

## IV.-DYESTUFFS.

Chemistry of  $C_{10}H_8$  derivatives.—See III. Phosphotungstic lakes.—See XIII.

See also A., Nov., 1373, Stilbene dyes. 1387, Thioindigo syntheses. 1388, Introduction of the CPh<sub>3</sub> group. 1391, N-Piperazinyl dyes. that, whilst such linkings are probably present, they can play no part in the mechanism of permanent set. S. G. S.

Determination of damage in silk. S. R. TROTMAN (J.S.C.I., 1936, 55, 325–327 r).—The test desoribed previously (B., 1935, 540) has been used to determine the damage caused to silk by degumming, dyeing, and exposure to light. Degumming with pancreatin injures the fibroin more than degumming with soap. Dyeing at above 85° and addition to the dye-bath of NaCl or Na<sub>2</sub>SO<sub>4</sub> damages the silk. Alkali increases the resistance of silk to light, and sericin affords partial protection against light. The intense ultra-violet light from a Fadeometer acts in a different manner from sunlight. Exposure to this light causes a marked diminution in the tensile strength of silk. R. C.

Kapok and other [upholstery] filling materials. 1. Determination of the relation between the springing capacity and the pentosan content. J. J. HANSMA (Chem. Weekblad, 1936, 33, 620-624).—The official (Dutch) method of evaluation is based on a microscopical examination and determination of the pentosan content, which is stated to be usually 25% for genuine kapok. The latter method is, however, unreliable if certain adulterants, notably "akon" (akund), are present, as these give high results, and the sp. springing capacity initially ( $D_0$ ) and after 24 and 48 hr. should also be taken into account (cf. following abstract). Data for 48 samples show that a genuine kapok has a pentosan val. of 23.5-29.0%,  $D_0$  being > 30 and  $D_0-D_{48}$ > 4.5.

Apparatus for determination of the springing capacity of kapok and other [upholstery] fillings. J. J. HANSMA (Chem. Weekblad, 1936, 33, 624-625; cf. preceding abstract).—From the arm of a balance is suspended a lacquered tinned-wire basket weighted with approx.  $2 \cdot 5$  kg. of Pb, on the top of the hinged lid of which is soldered a thin Cu plate with four wire legs. After obtaining a balance (using a g.) with the basket immersed in H<sub>2</sub>O (containing 1% of CH<sub>2</sub>O) so that the level reaches a mark on one of the legs of the disc, x g. of sample are placed in the basket, and the wt. required to restore the balance is found immediately (b g.), and after 24 and 48 hr. Then the sp. springing capacity  $(D_0) = (a-b)/x$ . Unlike existing apparatus, the method may be used for small samples (e.g., x = 60 g.). J. G.

Chemistry of coir fibre. II. Investigation into the nature of the lignin complex with a scheme of analysis for arriving at the "rational" lignin content of a given sample and tracing the significant changes taking place during the natural synthesis and senescence of the fibre. S. R. K. MENON (J. Text. Inst., 1936, 27, 241–252 T; cf. B., 1936, 1145).—The HCl method gives lower, and probably better, vals. of the lignin content than does the  $H_2SO_4$ method, but by both methods some acid-sol., lignin-like substance is lost. The incrustants may be removed from the fibre by repeated extraction with aq.  $(NH_4)_2C_2O_4$ , aq. NaOH, and NaOH in 50% EtOH, and the remaining skeletal substance is composed entirely of Cross and Bevan cellulose and lignin (HCl method). This latter is described as the "rational" lignin content and amounts to about 25% of the fibre ; the total lignin (HCl method) is about 35%. A. G.

Fractional dissolution of secondary cellulose acetate and viscosimetric investigation of the solution. M. TANIGUCHI and I. SAKURADA (J. Soc. Chem. Ind., Japan, 1936, 29, 316 B).—Commercial secondary cellulose acetate was fractionated by extraction with COMe<sub>2</sub> (I)-C<sub>6</sub>H<sub>6</sub> mixtures containing increasing proportions of (I). Viscosimetric measurements showed that the form and charge factors of the fractions were all near the val. (2·5) characteristic of spherical particles ;  $(\eta_{sp}/c)_{c+0}$  and sp. vol. increased with diminishing solubility. A. G.

Spinning properties of lyophilic colloid solutions and mechanical properties of the solid threads formed from them. II. H. ERBRING (Kolloid-Z., 1936, 77, 32—36; cf. A., 1936, 1200).—The spinning power of solutions of cellulose acetate in  $\text{COMe}_2$ -EtOH mixtures of different composition has been determined and the tensile strength and extension of the heatcoagulated threads measured. With increasing EtOH content the spinning power at first decreases, passes through a min. coinciding with min.  $\eta_i$  and then rises to a max., immediately after which coagulated thread are impaired by increasing [EtOH] in the solvent. E. S. H.

Stretching of acetate rayon for improving its properties. A. J. HALL (Amer. Dyestuff Rep., 1936, 25, 533-537).—Stretching of the yarn (with the aid of softening agents) improves its tensile strength, its resistance to delustring in boiling aq. liquors and in weighting treatments, and its suitability for use as crêpe yarn. A. J. H.

[Rayon] staple fibres. J. C. WITHERS (Text. Weekly, 1936, 18, 509-512).-New developments are reviewed.

[Manufacture of] paper pulp from cereal straws by a modified sulphate process. B. WINGFIELD, E. R. WHITTEMORE, C. B. OVERMAN, O. R. SWEENEY, and S. F. ACREE (Paper Trade J., 1936, 103, TAPPI Sect., 237-242) .- A small-scale process is described for the conversion of oat, rye, and wheat straws into pulps suitable for the manufacture of typewriting, book, and other grades of paper. The chemical cook is preceded by steeping in boiling H<sub>2</sub>O, and followed by a three-stage bleaching process using NaOCl in low concn. for a long time. Yields of 45-55% of unbleached pulps are obtained, and the yields after bleaching are 38-43% from wheat, 42-46% from rye, and 40-45% from oat straw. Strength data are given. Oat-straw pulp is the strongest but the most difficult to bleach, that from rve the easiest to bleach but brittle, whilst that from wheat is intermediate in both respects. A comparison of the process with sulphate, soda, and sulphite wood-H. A. H. pulp processes are given.

Chemical engineering problems in the manufacture of papermakers' cellulose. J. L. A. Mac-DONALD (Proc. Soc. Chem. Ind., Chem. Eng. Group, 1935, 17, 100-107).—The processing of wood, rags, and esparto, methods of soda recovery, and the washing and bleaching of the pulp are discussed. Processing in multiple stages is desirable where optimum and/or

special pulp qualities are required. Future advances will be in the direction of continuous digestion, and of the recovery of materials of greater val. from the digester lyes. J. G.

Bleaching soda pulp. E. SUTERMEISTER (Paper Ind., 1936, 17, 834—836).—A two-stage process with OCl' in both stages, is usual in present-day practice, although there are advantages in using Cl<sub>2</sub> or HOCl in the first stage, especially with regard to the increased rate of reaction, improved colour and strength, and less fibre degradation. Unlike sulphite pulp, the strength of soda pulp is never as high after bleaching as before. H. A. H.

Bleaching Western hemlock. N. W. COSTER (Paper Ind., 1936, 17, 829-831).—The application of modern two- and three-stage bleaching systems to hemlock pulp is described. Fibre loss is  $\langle 8\%$ , even for pulps of extreme whiteness, and Cl<sub>2</sub> consumption  $\langle 100$  lb. per ton of pulp. Single-stage bleaching is not satisfactory, owing to the highly coloured tannin derivatives present. H. A. H.

Hardwood sulphite bleaching. W. M. OSBORNE (Paper Ind., 1936, 17, 832—833).—Bisulphite cooking and bleaching conditions necessary for such hardwoods as maple, birch, and beech are discussed. It is more important to have a high combined  $SO_2$  in the cooking of hardwood than of softwood, such conditions resulting in better colour and strength, a lower shive content, and less loss when bleached. Two-stage bleaching, with chlorination as the first stage, is highly desirable, the bleaching rate then being very rapid. Alkaline conditions during bleaching are advocated, H. A. H.

Bleaching of old paper pulp. E. G. MILHAM (Paper Ind., 1936, 17, 837–838).—The most efficient bleaching system for old paper pulp is a single-stage, high-density (16-18%) bleacher, operating at 52–60°, using hypochlorite equiv. to 0.8% of active Cl, for 1–3 hr.

H. A. H.

Lime for pulp bleaching. O. L. Cook (Paper Ind., 1936, 17, 815-817).—Desirable characteristics are emphasised. H. A. H.

Calcium hypochlorite for bleaching of pulp. F. CASCIANI and A. HEILBRON (Paper Ind., 1936, 17, 810-814).—A summary of bleaching practice, with particular reference to two-stage processes. H. A. H.

Buffered bleaching [of pulp]. C. T. HENDERSON (Paper Ind., 1936, 17, 820-821).—The use of HOCI (from CaCO<sub>3</sub> and Cl<sub>2</sub>) is considered preferable to the use of gaseous Cl<sub>2</sub> for the first stage of two-stage bleaching of pulp. The physical strength of the final pulp is thereby maintained, its ash content is less, fibre loss is much reduced, and installation and operating costs are lower. H. A. H.

Water considerations in pulp bleaching. DEV. HAMILTON (Paper Ind., 1936, 17, 818-819).-Necessary requirements are briefly discussed. H. A. H.

Use of glycerin in papermaking. G. LEFFINGWELL (Paper Ind., 1936, 18, 463-464).—The various uses are reviewed. H. A. H.

Calculation of yield on sulphite papers. J. STRACHAN (Paper-Maker, 1936, 92, TS 145-146).— Typical losses due to bleaching, washing, and removal of moisture from fibre and clay are given for sulphite writing and printing papers, esparto, and mechanical pulp furnishes. H. A. H.

Production of pressboard from cornstalks. B. WINGFIELD, T. R. NAFFZIGER, E. R. WHITTEMORE, C. B. OVERMAN, O. R. SWEENEY, and S. F. ACREE (U.S. Bur. Stand. Misc. Publ. M 123, Aug., 1936, 10 pp.; cf. B., 1935, 764) .- Methods of prep., removal of extraneous matter, pulping, forming, pressure-drying, and sizing are outlined. By cooking for 0-3 hr. at 140° in H<sub>2</sub>O and refining mechanically in two stages a uniform pulp was obtained, and the resulting boards had a rupture modulus of 2040-7500 lb./sq. in. The best strengths were obtained by pressure-cooking, and the poorest by mechanical treatment. Optimum pressuredrying conditions are 150° and 500 lb./sq. in., 40% of the H<sub>2</sub>O being expelled and the loss in strength being only 20%; this process is justified by resulting economies in steam. Drying-time control is important in order to avoid blistering and wastage of time due to overdrying, and this was secured by the use of an electrical-resistance method. The best sizing results were obtained by impregnation of the finished board with a solution of paraffin wax or of a halogenated C10H8 in petroleum or CCl4, and heating at 150° for 30 min. It is more economical to size in the beater, paraffin emulsion or rosin being preferable to asphalt, pitch, or Na alginate, and giving boards which absorb 20-30% of H<sub>2</sub>O when immersed for 24 hr. Wet-mat sizing by pressure at 130-150°, or surface-sizing, is not recommended, although if bakelite varnish is used in the latter case improved appearance, strength, and sizing val. result. drug rodto bas doged to viJ. G.

See also A., Nov., 1435, Lignin from Spanish Pinus sylvestris.

## PATENTS.

Treatment of fibres or fibrous materials [hair or wool] containing keratin. J. B. SPEAKMAN (B.P. 453,700-1, 10.12.34).-(A) The rigid structure of keratin is relaxed by fission of the S·S linkings by a reducing agent, e.g., Na2S, NaHSO3 (or Na salt of another reducing S-acid), TiCl<sub>2</sub>, cystine hydrochloride, etc., best at  $p_{\rm H}$  4 or 11—14 (not 6, for then S·N linkings are formed); the fibre is then set to the desired shape, washed, treated with, e.g., H<sub>2</sub>O<sub>2</sub>, COMe<sub>2</sub>, etc. to remove the last traces of reducing agent, and (usually) fixed by an oxidising agent which re-forms S·S linkings and thus restores rigidity to the keratin in the desired shape. Applications to hair-waving and the textile industry are described. (B) The keratin is reduced and set as above and the new structure stabilised by reaction with salts, e.g., CuSO<sub>4</sub>, NiCl<sub>2</sub>, which react with the SH and thus prevent distortion of the shape by further reaction. The combined metal may be later used as mordant for R. S. C. a dye.

[Crimped] textile yarns. H. DREYFUS (B.P. 455,022, 4.4.35).—Yarns or staple fibre which have become crimped have the crimp rendered permanent by application to or formation in the fibre of a synthetic resin or water-resistant polymeride, such formation taking place in presence of an oil or plasticiser for derivatives of cellulose. K, H.S. [Catalyst for] esterification of cellulose. R. MÜLLER, M. SCHENK, W. WIRBATZ, and F. MÜLLER, ASSTS. to C. F. BOEHRINGER & SOEHNE G.M.B.H. (U.S.P. 2,024,381, 17.12.35. Appl., 3.2.33. Ger., 8.2.32).— Cellulose (I), after pretreatment, e.g., with AcOH, if desired, is esterified with the acylating agent catalysed by a small amount of a lower aliphatic sulphonic acid containing > one mineral acid radical, e.g., CH<sub>2</sub>(SO<sub>3</sub>H)<sub>2</sub>, CH(SO<sub>3</sub>H)<sub>3</sub>, CCl<sub>3</sub>·SO<sub>3</sub>H, NO<sub>2</sub>·CH(SO<sub>3</sub>H)<sub>2</sub>. The reaction may be carried out in presence of solvents, swelling agents, etc. E. J. B.

Preparation of mixed esters of cellulose. T. F. MURRAY, JUN., and C. J. STAUD, Assrs. to EASTMAN KODAK CO. (U.S.P. 2,024,658, 17.12.35. Appl., 16.7.32). - A cellulose mixed-ester dope (Ac and aliphatic acid radicals  $> C_2$ ) which contains grain or haze is treated with a small proportion of Ac<sub>2</sub>O to eliminate this defect. F. R. E.

Preparation of cellulose acetate isobutyrate. C. J. MALM and C. L. FLETCHER, ASSTS. to EASTMAN KODAK CO. (U.S.P. 2,024,651, 17.12.35. Appl., 21.4.33). —Cellulose, partly acetylated if desired, is treated with AcOH, Ac<sub>2</sub>O, and/or such quantity of Bu<sup>g</sup>OH or Bu<sup>g</sup><sub>2</sub>O ( $\leq 50\%$  of the total acyl content) as will render the esterification bath a non-solvent for the mixed ester formed. F. R. E.

Manufacture of a cellulose ester containing groups of dicarboxylic acids having heterogeneous linkages. C. J. MALM and C. R. FORDYCE, Assrs. to EASTMAN KODAK Co. (U.S.P. 2,024,238, 17.12.35. Appl., 30.7.32).—Cellulosic materials (I), including ethers and esters which contain esterifiable OH, are heated, preferably at 90–110° and in a solvent ( $C_6H_6$ , C<sub>2</sub>H<sub>2</sub>Cl<sub>4</sub>, etc.) if desired, with the anhydride of a dicarboxylic acid, the two CO2H of which are linked by a chain containing heterogeneous linkings (.O., .S., etc.), and a tert. base. Only one of the CO<sub>2</sub>H of each acid reacts with the (I), the other combining with the base, from which the ester may be liberated by dil. mineral acid. > 3 acid radicals combine with each  $C_6 H_{10} O_5$ unit of (I). Materials are obtained which have superior adhesive properties, are sol. in common solvents, and yield salts with metals. *E.g.*, cellulose acetate (35% Ac) 20, C<sub>5</sub>H<sub>5</sub>N 100, and diglycollic anhydride 40 pts. by wt. are heated at 90-100° for 24 hr., and the product is thinned with COMe<sub>2</sub> and poured into 10% aq. HCl; cellulose acetate diglycollate, containing 30.5% of diglycollyl radical, is pptd. E. J. B.

Extraction of cellulose esters. C. J. STAUD and E. C. YACKEL, ASSTS. to EASTMAN KODAK CO. (U.S.P. 2,024,666, 17.12.35. Appl., 28.4.32).—For the removal of degraded products of lowest  $\eta$ , the ester is extracted with mixtures of H<sub>2</sub>O and COMe<sub>2</sub> or AcOH. F. R. E.

**Refining of organic acid esters of cellulose.** D. E. NORTHROP and R. C. BURTON, ASSTS. to EASTMAN KODAK CO. (U.S.P. 2,024,246, 17.12.35. Appl., 5.5.33).— The ester is dissolved in aq.  $COMe_2$  (50—95%) and pptd. therefrom by gradually adding the solution to agitated aq.  $COMe_2$  (50—35%) in which it is insol.; the solution is maintained at substantially the same conc.. throughout the pptn. F. R. E. Preparation of cellulose ethers. RoHM & HAAS Co. (B.P. 455,253, 18.9.35. U.S., 26.10.34).—Degraded or undegraded cellulose is treated first with a conc. solution of a quaternary NH<sub>4</sub>OH, *e.g.*, NHMe<sub>3</sub>·OH, CH<sub>2</sub>Ph·NMe<sub>2</sub>·OH, in H<sub>2</sub>O and then with an etherifying agent, *e.g.*, Me<sub>2</sub>SO<sub>4</sub>, EtCl, CH<sub>2</sub>PhCl, CH<sub>2</sub>Cl·CH<sub>2</sub>·OH, (CH<sub>2</sub>)<sub>2</sub>O. The products are thickening agents and filmproducing substances. S. C.

Manufacture of crêpe threads and crêpe fabrics. BRIT. CELANESE, LTD., and H. DREYFUS. From CELANESE CORP. OF AMERICA (B.P. 453,196, 8.3.35 and 8.4.36. Cf. B.P. 438,589; B., 1936, 142).—Threads composed of cellulose ester or ether filaments are stretched in a softened state and crêpe-twisted while being steamed in presence or absence of moisture during at least the last part of the crêpe twist. The total twist required is  $\leq$ that needed to secure a similar crêping power without a preliminary stretching operation, and diminishes with increase in the amount of stretching. F. R. E.

Manufacture of short-fibre rayon yarns. A. NAI (B.P. 452,229, 14.6.35).—Twisted yarns are moistened with glycerin, sulphonated vegetable or mineral oil, or the like and are cut in an atm. of > 70% R.H. by the action of an abrading device which severs some filaments of the twist. B. M. V.

Increasing the tensile properties of viscose rayon. J. W. HUMPHREY and J. W. PEDLOW, Assrs. to VISCOSE Co. (U.S.P. 2,024,041, 10.12.35. Appl., 12.11.34).—An aralkyl alcohol (I), e.g.,  $C_2H_4Ph$ ·OH, anisyl alcohol, is added to viscose solution, the bulk of (I) being lost from the filaments during spinning. F. R. E.

Centrifugal treatment of viscoses. H. WALCH, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 2,019,944, 5.11.35. Appl., 25.5.34. Fr., 27.5.33).—Viscose is rendered fluid by rapid heating to a temp. < its point of instantaneous coagulation and centrifuged. It is then atomised in a closed space under partial vac. and in absence of CO<sub>2</sub> to free it from gases, and finally cooled at a speed which is controlled according to the "saline index" which it is to be given. F. R. E.

Viscose and cuprammonium cellulose spinning solutions and [soft-lustre] products thereof. R. S. BLEY, Assr. to N. AMER. RAYON CORP. (U.S.P. 2,021,849, 19.11.35. Appl., 8.9.34).—An alkylnaphthalene (1—10 wt.-% of the cellulose), which is insol. in  $H_2O$  and alkali and of b.p.  $> 100^\circ$ , e.g.,  $C_{10}H_7$  Et, is added to the spinning solution. F. R. E.

Textile process. C. DREYFUS (U.S.P. 2,022,410, 26.11.35. Appl., 17.1.33).—A solution of cellulose acetate in a volatile solvent (I) (COMe<sub>2</sub>) is extruded into an evaporative atm., and the formed filaments are withdrawn while containing  $\lt 0.1\%$  (0.3—0.4%) of (I) and immediately formed into fabric. F. R. E.

Manufacture of threads and fabrics made with filaments of cellulose esters or ethers. BRIT. CELANESE, LTD. From C. DREYFUS (B.P. 453,934, 21.12.34).—In order to prepare them for forming into fabric, threads composed of cellulose derivatives are subjected to the action of steam under conditions such that moisture is present, and, as a final twisting operation,

are twisted to a degree insufficient to impart crêping power to the thread. F. R. E.

Treatment of artificial silk. A. HARTMANN, W. HOEFINGHOFF, and K. MEYER-GAUS, ASSIS. to AMER. BEMBERG CORP. (U.S.P. 2,020,057, 5.11.35. Appl., 24.1.34. Ger., 31.1.33).—Filaments are passed in a helical path over a hollow cylindrical roller (R) mounted on a vertical axis and having a perforated surface covered with porous material. Different treatment liquids are applied simultaneously at spaced points to the filaments while on R, which is so made as to keep the various liquids separated. F. R. E.

Dry-spinning artificial textile fibres. E. VIVIANI. Assr. to RUTH-ALDO Co., INC. (U.S.P. 2,022,260, 26.11.35, Appl., 10.4.31. It., 12.4.30).—The contour and hence the lustre of dry-spun filaments is varied and controlled by subjecting them to a transverse current of heated vapour (air) immediately after extrusion, and then to a relatively quiescent vapour of lower temp. in which the solvent evaporates.—F. R. E.

Manufacture of [non-creasing] rayon. E. J. R. BEATTEY (U.S.P. 2,024,962, 17.12.35. Appl., 26.5.33).— A no. of filaments, while still gelatinous, are drawn, with or without a central silk core, through a tapered guide of diminishing bore placed in the coagulating bath, the discharge opening of the guide being < the combined diam. of the filaments after extrusion from the spinnaret. F. R. E.

Manufacture of [delustred] artificial silk. F. HOELKESKAMP, ASST. to AMER. BEMBERG CORP. (U.S.P. 2,022,961, 3.12.35. Appl., 23.8.32. Ger., 23.9.31).— Viscose or cuprammonium cellulose solution is stretchspun to form filaments which are pptd. in presence of a  $H_2O$ -sol. metallic salt (BaCl<sub>2</sub>) and subsequently coagulated with an acid ( $H_2SO_4$ ) which forms an insol. ppt. with the salt. F. R. E.

Manufacture from continuous filaments of a continuous product containing staple fibre. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 453,432-4, 6.3.35).—A bundle of continuous filaments is passed between the surfaces of two rotating rollers at least one of which is a cutting surface, and these are made to press together at one point so as to cut the bundle into staple fibres, which are twisted to a continuous product continuously with their production. Various arrangements of the rollers and methods of driving them are claimed. F. R. E.

Production of artificial thread [of reduced lustre]. H. ALTWEGG and A. EICHLER, ASSTS. to DU PONT RAVON CO. (U.S.P. 2,022,838, 3.12.35. Appl., 19.8.30. Ger., 23.9.29).—A solution of a cellulose derivative containing a non-volatile org. acid (stearic) or anhydride is dry-spun, and the resulting filament of normal lustre is treated with a solution of a substance (BaCl<sub>2</sub>) which reacts with the acid etc. to form an insol. compound, whereby its lustre is reduced. F.R. E.

Production of tubular artificial textile threads. ALSA SOC. ANON., R. PICARD, and R. FAYS (B.P. 454,811, 2.9.35).—Viscose filaments, which are entirely hollow and of denier  $\geq 2$ , are produced by spinning into a bath prepared as in B.P. 255,527 (B., 1926, 912) while the temp. is varied inversely with the desired titre so that the min. temp. T = a - bd, where a = 70 - 75, b = 9 - 11, d = desired titre < 2 deniers. Alternatively, d may be obtained by varying the temp. as above and also the concn., each to a smaller extent than described (*loc. cit.*). F. R. E.

Manufacture of [water- and fire-proofed] regenerated cellulose sheet or film. D. W. LOSEE, ASST. to JOHNSON-LOSEE CORP. (U.S.P. 2,025,000, 17.12.35. Appl., 1.8.33).—Raw cotton cellulose, purified so as to leave a substantial proportion of the natural incrustations therein, is formed into transparent regenerated cellulose sheet, e.g., via cuprammonium cellulose, impregnated with urea phosphate, and coated with a  $H_2O$ -resistant coating (pyroxylin), the wax-like incrustations being sufficient to render the coated sheet waterproof without the presence of added waxes in the coating. F. R. E.

Manufacture of pulp. C. K. TEXTOR, Assr. to NORTHWEST PAPER Co. (U.S.P. 2,022,872-3, 3.12.35. Appl., 16.9.26).-(A) Chips prepared from long-fibred woods are cooked with liquor containing 40-45% (on the bone-dry wt. of wood) of Na<sub>2</sub>SO<sub>3</sub> and 1.5-3.0% of Na<sub>2</sub>CO<sub>3</sub>. The  $p_{\rm H}$  of the liquor should be 7.5-9.0. It is regenerated by concentrating by evaporation and incinerating to Na<sub>2</sub>CO<sub>3</sub>, which is leached out with H<sub>2</sub>O, and sufficient SO2 is passed in to give the required SO<sub>3</sub>: CO<sub>3</sub> ratio. Make-up Na is added as NaOH to the evaporator. An unlined steel digester is used. (B) Digestion similar to the above is used for hardwoods; 30-40% of Na<sub>2</sub>SO<sub>3</sub> and  $1\cdot 3-2\cdot 6\%$  of Na<sub>2</sub>CO<sub>3</sub>, however, are used. It is stated that in both cases the pulp has a good colour and that its strength approaches that of similar kraft pulps. D. A. C.

Production of multi-layer paper, cardboard, pasteboard, boxboard, and the like. E. FUES, ASST. to A. W. DELLER (U.S.P. 2,024,635, 17.12.35. Appl., 26.8.32. Ger., 27.7.31).—A suspension of very heavily beaten pulp, mixed with large amounts of loading and a colloid if desired, is coated on the paper web while it is still wet, and the paper is dried and calendered. The suspension, applied similarly, may also be used as the adhesive in making multi-ply papers or boards.

D. A. C.

Paper impervious to moisture, water, and grease. E. H. BUCY, Assr. to ATLAS POWDER Co. (U.S.P. 2,021,172, 19.11.35. Appl., 21.11.33).—A solution of casein in aq.  $NH_3$  is added to a solution in tung or other drying oil of an oil-sol. PhOH– $CH_2O$  condensation product; driers, stabilisers, thinners, and wax may also be incorporated and the composition used for coating paper. S. M.

Manufacture of grease- and moisture-proof paper and similar materials. J. E. SCHOPP (U.S.P. 2,021,947, 26.11.35. Appl., 12.9.33).—Paper is coated with a composition containing modified starch 9.5, casein (or glue etc.) 9.5, NH<sub>3</sub> 0.6, clay 37.8, plasticiser (e.g., glycerin) 14.2, rubber latex 23.7, and shellac 4.7%. D. A. C.

Watermarking composition [for paper]. J. C. BOYER, ASST. to NAT. LISTING EXCHANGE (U.S.P. 2,021,141, 19.11.35. Appl., 1.5.33. Renewed 11.3.35).— An emulsion of a vegetable oil (castor), mineral matter (calcined diatomite), a resin (Canada balsam), solvent

(turpentine), starch, and  $CaCO_3$  in an aq. solution of a slightly alkaline salt, *e.g.*, borax, is used to render the paper translucent. S. M.

**Paper sizing.** L. L. LARSON, ASST. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 2,022,004, 26.11.35. Appl., 1.7.33).—A resin is made by heating a mixture of glycerin (I), o-C<sub>6</sub>H<sub>4</sub>(CO)<sub>2</sub>O (II), and stearic acid (III) at 200° until the acid val. (to KOH) is about 47. The proportions of ingredients are such that the amount of (III) glyceride formed is 65—90% of the mixture [*e.g.*, (I): (II): (III) = 15.6: 20.2: 64.2]. The resin is then emulsified in dil. aq. NaOH and added, in amounts of 0.5—2% of the paper, to the beater furnish or to the dried web. D. A. C.

Decalcomania paper. J. MACLAURIN (B.P. 453,963, 4.6.35).—The backing sheet or adhesive is coloured with a distinctive dye which will not affect its  $H_2O$ -absorption. Control of manufacturing variables is thereby facilitated, and the paper is made proof against counterfeiting. D. A. C.

Oil-proofing composition [for paper etc.]. J. K. DELANO (U.S.P. 2,025,729, 31.12.35. Appl., 21.2.33).— H<sub>2</sub>O-sol. salts (K, Na, Ca) of glycyrrhizic acid or homologous acids are mixed with glue and a carbohydrate (e.g., molasses) rich in glucose and pentosans. K. H. S.

Forming carrier web [for transparent sheet material]. E. M. KRATZ, Assr. to MARBO PRODUCTS CORP. (U.S.P. 2,024,826, 17.12.35. Appl., 29.6.33).— Webs of fine and coarse linen are united by a suitable adhesive. The outer surface of fine material is first treated with pigmented pyroxylin to obliterate weave marks and then alternately coated with clear plasticised nitrocellulose containing Bu stearate and highly polished, whilst the back surface of the coarse webs is treated with pigmented pyroxylin to prevent curling. F. R. E.

Ornamental compound sheet materials. BRIT. CELANESE, LTD., and B. E. M. MILLER (B.P. 454,442, 30.3.35).—A transparent or translucent sheet composed of an org. derivative of cellulose is stuck by means of a suitable adhesive to the pile side of a pile fabric (composed of an org. cellulose derivative pile and a cotton backing), which is suitably dyed, patterned, or crushed to give ornamental effects. F. R. E.

Manufacture of impregnated fibre articles or sheets. G. A. RICHTER and M. O. SCHUR, Assrs. to BROWN CO. (U.S.P. 2,024,600, 17.12.35. Appl., 2.9.32).— Fibrous sheet material, *e.g.*, paper, is impregnated first with a small proportion of cellulose regenerated thereon from viscose so that there is a residual porosity, and then with a solid, heat- or solvent-fluxed, H<sub>2</sub>O-repellent material (asphalt, rosin, rubber, wax) completely enveloping the whole. F. R. E.

**Preservation of vegetable fibre materials.** F. E. CISLAK, ASST. to P. C. REILLY (U.S.P. 2,022,231, 26.11.35. Appl., 17.3.34).—The materials are protected against decay and insects by mixtures of (a) an insol. borate (preferably Cd or Zn borate) or a mixture of salts giving an insol. borate, and (b) acenaphthene, fluorene, anthracene, phenanthrene, carbazole, and/or a tar acid derivative of m.p.  $> 40^{\circ}$ , with or without C<sub>10</sub>H<sub>8</sub>. This addition is made to the fibre paste. R. S. C. Concn. of aliphatic acids.—See III. Treating artificial filaments etc.—See VI. Insulating material. —See XI. Polyvinyl resin films etc. Fibrous composition. Coating paper.—See XIII. Saccharification of cellulose.—See XVII.

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

Developments in hydrogen peroxide bleaching. ANON. (Text. Weekly, 1936, 18, 515–521).—Large-scale methods for bleaching cotton and wool are described, reference being made to recent methods for wool in which very dil.  $aq. H_2O_2$  is dried into the fibre.

A. J. H.

Maintenance and layout of a modern dyehouse. J. MACE (J. Soc. Dyers & Col., 1936, 52, 419-423).— Descriptive.

[Aminoazobenzene as catalyst in] dyeing Prud' homme [prussiate] aniline-black. H. SUNDER and L. A. LANTZ (Sealed Note 2532, 27.4.32. Bull. Soc. Ind. Mulhouse, 1936, 102, 106-109). Report by E. J. MUELLER (Ibid., 110-113).-Addition of 10% (on wt. of NH<sub>2</sub>Ph) of Eumol [(I.G.), a 15% solution of p-PhN<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·NH<sub>2</sub> (I) in NH<sub>2</sub>Ph] to a prussiate NH<sub>2</sub>Phblack padding or printing liquor makes it unsuitable for producing clear white resist effects, but increases the depth of the resulting black (but bronzy) shade 20-25%. Aminoazotoluene behaves similarly to (I). (Cf. B.P. 176,343; B., 1923, 651 A.) A non-bronzy black which allows the production of good white resists is obtained by replacing Eumol by (I) made by adding AcOH, or preferably HCO<sub>2</sub>H, and NaNO<sub>2</sub> to the aq. NH<sub>2</sub>Ph,HCl used in preparing the padding liquor (II). E.g., 1 litre of (II) is made with HCl ( $d \cdot 1657 \cdot 1 \text{ g.}$ ), NH<sub>2</sub>Ph (53·6), 65% HCO<sub>2</sub>H (7·15), NaNO<sub>2</sub> (0·28), NH<sub>4</sub>Cl (6·22), Na<sub>4</sub>Fe(CN)<sub>6</sub> (55), and NaClO<sub>3</sub> (25·9). The padded fabric emerges from the ager black instead of the usual dark green, but it requires afterchroming. MUELLER confirms the useful catalytic properties of (I), but finds Eumol equally satisfactory. It is suggested that (I) accelerates oxidation of the NH,Ph by acting as an O carrier; the catalyst used in Green's NH2Ph-black process is considered to act more slowly, but similarly. The action of conc. H<sub>2</sub>SO<sub>4</sub> on cotton dyed with NH<sub>2</sub>Ph + (I) indicates that the black pigment is mainly phenylazine, whereas it is mainly indamine when (I) is omitted. A. J. H.

Dyeing of aniline-black on silk and half-silk hosiery. G. PEMBERTON (Text. Colorist, 1936, 58, 686, 696).—Methods and the compositions of various oneand two-bath padding liquors are described.

A. J. H.

Dyeing of natural silk and rayon. F. S. TOMLINSON (J. Soc. Dyers & Col., 1936, 52, 373-378).—A review of methods of dyeing silk and union materials. S. C.

Use and abuse of fastness guarantees [for coloured textiles]. J. R. HANNAY (J. Soc. Dyers & Col., 1936, 52, 413-419).—A discussion.

Chemistry of some newer textile assistants. A. J. HALL (Amer. Dyestuff Rep., 1936, 25, 561—565).— The composition and uses of numerous quaternary NH<sub>4</sub>, sulphonium, and phosphonium compounds in dyeing and finishing are described. A. J. H

Wetting agents.—See III. Stretched acetate rayon. [Bleaching] papermakers' cellulose, pulp, and hemlock.—See V.

See also A., Nov., 1342,  $p_{\rm H}$  vals. of some alkaline products.

#### PATENTS.

Method of or apparatus for dyeing, washing, or bleaching yarns or other material. H. E. PARTRIDGE (B.P. 454,678, 22.6.35).—The apparatus consists of a vat containing liquor which may be uniformly heated and agitated by means of air and steam introduced in the lower part of the vat by special devices.

#### A. J. H.

Bleaching of vegetable fibres. I. J. SMITH (U.S.P. 2,020,437, 12.11.35. Appl., 15.3.28).—Cotton, flax, regenerated cellulose, etc. are bleached by treating the wet out but unboiled material with (a) a hypochlorite solution (NaOCl of  $d \ 1.005-1.025$ ) at  $10-50^{\circ}$ , and (b) an alkaline oxidising solution, e.g., aq. Na<sub>2</sub>O<sub>2</sub>, aq. NaBO<sub>3</sub>, at the boil and, if desired, a Cl-neutralising agent, e.g., aq. NH<sub>3</sub>, NaHSO<sub>4</sub>, either at this stage or between (a) and (b). N. H. H.

Dyeing of animal fibres. J. W. LEITCH & CO., LTD., A. E. EVEREST, and J. A. WALLWORK (B.P. 454,729, 16.5.35. Addn. to B.P. 437,049; B., 1936, 189).— Animal fibres are treated with solutions of diazo compounds capable of combining with the fibre, the solution also containing one or more salts of Cr, Cu, Ni, or Co. In an example, wool is treated with diazotised 5-nitro-oanisidine, a wetting-out agent, and  $Na_2CO_3$ ; after coldworking, CuSO<sub>4</sub> is added and the dye-bath raised to 100°. The wool is dyed brown. K. H. S.

Process of dyeing [with sulphur, carbazole, and vat dyes]. P. BOLEN, ASST. to AMER. LECITHIN Co-(U.S.P. 2,020,496, 12.11.35. Appl., 16.5.32).—Lecithin (I) from, e.g., soya beans or skim milk, when dispersed in H<sub>2</sub>O by means of, e.g., K or Na salts of naphthenic or a fatty acid, or by a suitable solvent, e.g., butylcellosolve, and admixed with a S, carbazole, or vat dye before, during, or after the prep. of the vat, prevents premature oxidation during the dyeing process. 0.5— 3.0 wt.-% of (I) on material to be dyed is used, according to the type of apparatus. Examples illustrate the use of (I) in dyeing with Ciba Brown R, Cibanone Red 4B, Cibanone Black B, Pyrogene Deep Black B, Pyrogene Brown 4R extra, Ciba Blue 2RH, and Ciba Yellow G. N. H. H.

Dyeing and printing textile materials [with azo dyes]. M. A. DAHLEN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S. 2,021,911, 26.11.35. Appl., 27.7.32).—Shades generally fast to washing, Cl<sub>2</sub>, and light are obtained by applying a mixture of a diazoiminocompound, R·N:N·R' (where R is aryl, e.g., of the C<sub>6</sub>H<sub>6</sub>, anthraquinone, or carbazole series, containing, if desired, e.g., alkoxy-, alkyl, or halogen but no solubilising group, and R' is a pyrrolidine or piperidine nucleus containing at least one solubilising group, e.g., CO<sub>2</sub>H, SO<sub>3</sub>H), and any "ice colour" coupling component, e.g., arylamides of 2 : 3-hydroxynaphthoic acid, and subsequently treating with dil. acids, e.g., AcOH, HCO<sub>2</sub>H, at 80—90°. Examples are : 2 : 5-C<sub>6</sub>H<sub>3</sub>Cl<sub>2</sub>·NH<sub>2</sub> (I)  $\rightarrow$  Na piperidine-2carboxylate  $\rightarrow$  2 : 3-hydroxynaphthoic 2' : 5'-dimethoxyanilide (brown); (I)  $\rightarrow$  proline  $\rightarrow$  the 2'-methoxyanilide (bright orange), (I)  $\rightarrow$  Na pyrrolidine-2-sulphonate  $\rightarrow$ the anilide (II), and 3-nitro-*p*-toluidine  $\rightarrow$  proline  $\rightarrow$ (II). N. H. H.

Production of dveings and printings [of ice colours] on fibrous materials. Soc. CHEM. IND. IN BASLE (B.P. 453,953, 30.4.35. Switz., 19.5.34) .- A 2:3-hydroxynaphthoic arylamide is coupled on the fibre with a diazotised 3-amino-4-aryloxyphenyl alkyl ketone to give dyeings or prints of increased purity of shade and fastness to kier-boiling compared with those described in e.g., B.P. 279,146 (B., 1928, 8). Examples are: 2-amino-4-acetyl-1:1'-diphenyl ether or its 4'-Cl-derivative  $\rightarrow$  2:3-hydroxynaphthoic 2'methyl-, 2': 5'-dimethoxy-, 4'-methoxy-2'-methyl-, 3': 4'dichloro-, 5'-chloro-2'-methyl-, or 4'-chloro-2'-methoxyanilide (reds),  $\rightarrow$  the 3'- or 4'-methyl-, 3'-chloro-2'-ethoxy-, 2'-methoxy-5'-methyl-, 4'-chloro- 2'-methoxyanilide (scarlet-reds). Similar shades are obtained from 2-amino-4-acetyl-4'-(or -3'-)methyl-1: 1'-diphenyl ether. The amines are prepared by, e.g., treating PhBr with AcCl, nitrating the product, condensing with a phenol, and reducing. [Stat. ref.] N. H. H.

**Printing with [chrome] mordant dyestuffs.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 453,834, 13.3.35).—In printing on textiles (silk and artificial silk) with Cr mordant dyes, addition, in the form of free acid, salts, or esters, of amino-, hydroxy-, or thiolacetic acid or a substitution product in which a H of the NH<sub>2</sub>, OH, or SH is replaced by, *e.g.*, aryl or alkyl, *e.g.*, Na o-, m-, or p-tolyloxyacetate or NHPh·CH<sub>2</sub>·CO<sub>2</sub>H, to a mixture of the dye and gum thickening, prevents coagulation of the latter by the subsequent addition of the Cr mordant. This effect is enhanced in many cases by adding also one or more of the following : a thiocyanate, a cyanate, an acid amide,  $(CH_2)_6N_4$ , or NaOAc. N. H. H.

(A) Textile printing. (B) Printing. H. A. LUBS and J. E. COLE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,024,973-4, 17.12.35. Appl., [A] 23.12.30, [B] 19.12.33).-(A) Alkali or NH<sub>4</sub> salts of unsubstituted anthraquinonesulphonic acids are claimed as assistants to obtain stronger printing with vat dyes. 12 examples illustrate the use of 0.5-10 (1-5)% of Na anthraquinone-2-sulphonate, -2:6- and -2:7-disulphonate, or the mixed sulphonates from the sulphonation of anthraquinone after the removal of the 2-sulphonic acid, in a paste containing 12.5% of solid thioindigoid dye (6:6'-diethoxy- and 6:6'-dichloro-4:4'-dimethylthioindigo are claimed) in the usual type of printing paste. (B) This also includes the salts of sulphonic acids of substituted (Cl-, NO2-, etc.) anthraquinones. 24 examples are given of suitable dyes and pastes.

E. J. B.

Treatment [stretching] of artificial filaments, yarns, foils, films, and the like containing organic derivatives of cellulose. H. DREYFUS (B.P. 454,580, 3.4.35).—The materials are treated with a liquid or vaporised medium containing 25—65 (40—55)% of a neutral org. liquid and  $\gg 10$  (2—5)% of an acidic material, both of which are solvent and/or swelling agents for the material. The latter is stretched during or after treatment; 200–600% extension is attainable. E.g., dioxan, COMe<sub>2</sub>, olefine glycol ethers and/or esters, with AcOH, lactic acid, etc. are used, in a (preferably)  $H_2O$ -miscible solvent (EtOH,  $C_6H_6$ ). They are finally removed by evaporation, or washing with dil. alkali, or, better, with liquids containing swelling agents, and then with alkali, to retain the lustre. E. J. B.

Treatment [shrinking] of artificial filaments, threads, ribbons, and similar materials of organic derivatives of cellulose. H. DREYFUS (B.P. 454,507, 1.4.35).—The threads etc. are treated with a latent solvent, e.g.,  $CH_2Cl_2$ ,  $C_2H_2Cl_4$ , EtOAc, in a (H<sub>2</sub>Oimmiscible) diluent of b.p.  $\Rightarrow$  80° (30—40°) (C<sub>6</sub>H<sub>6</sub>, CCl<sub>4</sub>, ethers, or light petroleum) at  $\Rightarrow$  12°, and under insufficient tension to cause stretching. A shrinkage of 60% or even more is attainable ( $\Rightarrow$  20% is preferred), and extensibility and tenacity are improved, particularly with yarns that have been (steam-)stretched previously. (Cf. B.P. 389,823; B., 1933, 423.) E. J. B.

Treatment of fibrous and textile materials [with phosphatides]. B. REWALD, Assr. to AMER. LECITHIN Co. (U.S.P. 2,020,517, 12.11.35. Appl., 5.10.32. Ger., 11.1.30).—Addition of phosphatides (vegetable lecithin), emulsified when necessary in  $H_2O$ , produces advantages in treating fibres, threads, and fabrics of all kinds : e.g., in dyeing a better penetration of the dye, a greater brilliance, and a softer feel of the material are obtained; in kier-boiling the cotton has a brilliance not otherwise obtained; in wetting out the time is shortened; in cleaning with aq. soaps etc. or org. liquids, e.g., CCl<sub>4</sub>, the effect is increased and the feel and appearance of the material are improved. N. H. H.

**Treatment of textile materials.** H. DREYFUS (B.P. 454,968, 12.4.35).—Lustrous artificial silk in the spun form is delustred by impregnating with the double NH<sub>4</sub> Ti salts of weak polybasic acids (carbonic, tartaric, oxalic, or citric). Wetting and/or swelling agents may be added to the bath. After impregnation, the salts are decomposed in the fibre by steaming alone, or in presence volatile bases, or in an alkaline bath. In examples, cellulose acetate yarn is delustred with NH<sub>4</sub> Ti tartrate, and regenerated cellulose yarn with NH<sub>4</sub> Ti carbonate. Sn may be similarly applied as NH<sub>4</sub> Sn<sup>IV</sup> oxalate.

K. H. S.

Treatment of textile and other materials. L. DE R. FABER and C. J. CARROLL, Assrs. to FABER ENG. Co. (U.S.P. 2,023,013, 3.12.35. Appl., 28.4.31).—Treatment liquids, e.g., for washing, cleaning, scouring, dry-cleaning, degumming, bleaching, dyeing, etc., are aërated so as to yield a foam which is drawn through the material by maintaining a difference of pressure on the two sides thereof. Apparatus is claimed. F. R. E.

Treatment [sizing] of textile materials. BRIT. CELANESE, LTD., D. FINLAYSON, and C. E. STAFFORD (B.P. 454,425, 27.3.35).—Polymerised vinyl esters are hydrolysed by heating (at 30—50°) with an amount of an alcoholic solution of a base (alkali hydroxides  $\Rightarrow 0.1N$ , NH<sub>2</sub>Me, etc.),  $\Rightarrow 20\%$  of the amount theoretically necessary to effect the required degree of hydrolysis, *i.e.*, so that the product contains  $\ll 20\%$  of the max. of ester groups and is preferably readily H<sub>2</sub>O-sol. or -dispersible. The products may be mixed with other suitable materials, e.g., (sulphonated) fats, waxes, and are suitable for sizing org. derivatives of cellulose, being easily applied and readily removed in the usual way. If hydrolysis is stopped before the material is  $H_2O$ -sol., good resists etc. are obtained. E. J. B.

Dressing of textile materials. BRIT. CELANESE, LTD. (B.P. 454,666, 30.4.35. U.S., 4.5.34).—Dressings for application to textile fibres, including artificial fibres (cellulose acetate etc.), comprise a non-drying animal or vegetable oil, *e.g.*, the glyceride of an unsaturated acid (olive oil), to which is added quinol or an alkyl *p*-hydroxybenzoate (0.2-4%) on the wt. of dressing). Deleterious changes in the dressing on storage of the textile are thereby prevented. K. H. S.

Making leather and other fibrous materials impermeable to liquids. B. REICHERT and E. RYDBERG (B.P. 454,549, 16.5.36. Swed., 20.5.35 and 6.5.36).— The material (e.g., leather) is impregnated with a factice consisting of a vulcanisable oil and  $S_2Cl_2$ , and HCl thereby formed is neutralised by treating the leather with a buffered alkaline liquor, e.g., a 0.5—1% aq. solution of NaOH (or NH<sub>3</sub>) containing 5% of borax, NH<sub>4</sub>OAc, or Na<sub>2</sub>HPO<sub>4</sub>. A. J. H.

Proofing of animal fibres against moth larvæ, mould, and rotting. W. LOWE (B.P. 454,458, 26.7.35. Addn. to B.P. 413,445; B., 1934, 925).—A double salt made by evaporating together aq.  $CrF_3$  (4 pts.) and Na Sb fluoride (1 pt.) is used instead of the separate salts as described previously (*loc. cit.*). A. J. H.

Production of effects on textile webs. HEBERLEIN & Co. A.-G., G. HEBERLEIN, and E. WEISS (B.P. 453,836, 14.3.35, 27. and 29.1.36).—Crêpon or patterned effects are produced on textile webs (containing cotton) by treatment with a swelling agent having shrinking or parchmentisation powers, a material which consists of both yarns sensitive and insensitive to swelling agents, e.g., one containing a pretreated yarn and a raw yarn having the natural incrustation thereon, or one locally rendered insensitive by applying, e.g., H<sub>2</sub>O-resisting agents, waxes, resins, esterification agents, by the reserve printing process. Suitable agents are, solutions of inorg. acids, alkalis, and salts, e.g., H<sub>2</sub>SO<sub>4</sub>, H<sub>3</sub>PO<sub>4</sub>, ZnCl<sub>2</sub>, NH<sub>3</sub>-CuO, and caustic lye.

[Device for] dyeing and like treatment of hose and other shaped textile articles. BRIT. CELANESE, LITD. (B.P. 455,194, 16.3.36. U.S., 16.3.35).

[Mechanism for] production of pattern effects on fabrics. LINEN INDUSTRY RES. ASSOC., and H. BOFFEY (B.P. 455,389, 20.5.35).

Lubricating composition [for kier valves].— See II. Org. F compounds. Wetting etc. agents. Ethers etc. of paraffin wax. Aliphatic-aromatic compounds. Compounds containing acid saltforming groups. Hydroxyalkylated bases.—See III. Ornamental compound sheets.—See V. Coloured lacquers.—See XIII.

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

Fauser process for production of ammonium nitrate. G. PASTONESI (Chim. e. l'Ind., 1936, 18, 511—513).—A description of the process and of the improvements effected during the past five years in the manufacture of  $NH_4NO_3$  by this method. O. J. W.

Stability of dilute solutions of sodium hexametaphosphate. L. GERMAIN (Chim. et Ind., 1936, 35, 22-26).-Titrimetric methods are described for analysing commercial mixtures of Na ortho-, pyro-, and meta-phosphate, and are applied to study the change of  $(NaPO_3)_6$  (I) in solution. The power of (I) to dissolve  $Ba_3(PO_4)_2$  increases with temp. and decreases with addition of Na salts. (I) decomposes in H<sub>2</sub>O with a speed increasing with temp. and decreasing with increasing  $p_{\rm H}$ , forming (a) NaH<sub>2</sub>PO<sub>4</sub> and (b) a polymeride of indeterminate mol. wt. which does not dissolve alkaline-earth phosphates. The polymeride hydrolyses much less easily than (I) or the pyrophosphate. The speed of reaction (a) increases with the temp. and acidity, whereas that of (b) increases with temp. but is almost independent of  $p_{\rm H}$ . The neutral Na pyrophosphate has no special stabilising influence. These results are applied to the industrial uses of (I). The higher is the temp. the more frequent must be the renewal of baths of (I), and an alkaline medium (aq. Na<sub>2</sub>CO<sub>3</sub>) and a temp. of  $> 70^{\circ}$  should be used. R. S. B.

Rapid determination of purity of technical gypsum. J. EIDUKS (Latvij. Univ. Raksti, 1936, 3, 1–10).—The powdered sample is heated for 1 hr. at 450–900° to destroy org. matter, covered with  $C_6H_6$  in a test-glass, and matched in reflected light against standards of known  $CaSO_4$  content. The purer is the sample, the lighter is its colour. An accuracy of 1–2% is obtained. H. J. E.

Determination of alum concentrations. L. E. SIMERL and D. S. DAVIS (Paper Ind., 1936, 18, 460-461). —A nomograph is given. H. A. H.

Papermakers' cellulose [lye].—See V. Action of alkali carbonate on Ca salt solutions.—See XVII.

See also A., Nov., 1348, Production of NO by electric arc. 1349, Separation of Y from its earths. Extraction of In from cylindrite etc. 1350, Prep. and determination of F. 1353, Determination of In in minerals.

#### PATENTS.

Manufacture of sulphuric acid by the contact process. C. F. R. HARRISON, A. M. CLARK, C. L. HILTON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 454,327, 28.2.35).—A gaseous mixture containing ≮ 20% of SO<sub>2</sub> and  $\ll 21\%$  of O<sub>2</sub> is passed over a Pt or V catalyst in four stages at a prescribed max. temp., the conversion in the first stage being  $\gg 50\%$  and in each subsequent stage  $\gg 25\%$ . The conversions are controlled in the first stage by proportioning the amount of catalyst and regulating the inlet temp., and in each subsequent stage by proportioning the amount of catalyst and adding an O2-containing gas free from SO2 to the gaseous mixture before that stage and/or extracting part of the heat of the gaseous mixture. The  $O_2$  content of the gases throughout each stage is regulated so that, calc. on a H<sub>2</sub>O basis, it is  $\lt 0.32x - 2.65$  and > 0.57x + 2.96, x being the  $\frac{1}{2}$  ratio of total  $O_2$  added to the total  $O_2$  + total inert gases introduced prior to that stage. The

total conversion of  $SO_2$  per passage through all the stages is 70–90%, and the gases after removal of  $SO_3$  are treated for recovery of the residual  $SO_2$ , which is returned to the process. W. J. W.

Manufacture of hydrogen cyanide. Courts & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 451,609, 28.2.35. Addn. to B.P. 361,004; B., 1932, 101).—A mixture of, e.g.,  $\mathrm{NH}_3$  11,  $\mathrm{CH}_4$  11.5,  $\mathrm{O}_2$  15,  $\mathrm{H}_2$  2, and  $\mathrm{N}_2$  60.5% is passed through four layers of 1—10 (3)% Ir–Pt wire gauze (500 meshes per sq. cm.) heated at 1050°. A. R. P.

Treatment of chlorates to reduce risk of fire. STOCKHOLMS SUPERFOSPAT FABRIKS AKTIEB. (B.P. 451,894, 28.8.35. Swed., 8.5.35).—NaCl and NaHCO<sub>3</sub> and/or Na<sub>2</sub>CO<sub>3</sub> are mixed with the NaClO<sub>3</sub> solution to reduce the danger of ignition of fabrics with which it may come in contact, *e.g.*, when being applied as a weedicide. A. R. P.

Manufacture of [alkali nitrate] salts by means of base-exchangers. NORSK HYDRO-ELEKTRISK KVAEL-STOF-A./S. (B.P. 451,733, 4.10.35. Norw., 10.10.34).---Aq. Ca(NO<sub>3</sub>)<sub>2</sub> is converted into aq. NaNO<sub>3</sub> by passage through vessels packed with granules of an artificial zeolite in which the SiO<sub>2</sub>: Al<sub>2</sub>O<sub>3</sub> ratio is 1:2:2:--5:5(3-4). The zeolite is formed by mixing equal vols. of 7% aq. Na aluminate (Na<sub>2</sub>O: Al<sub>2</sub>O<sub>3</sub> = 1:1.4) and 7% aq. Na silicate (Na<sub>2</sub>O: SiO<sub>2</sub> = 1:3.4), and washing, drying, and granulating the resulting gel. A. R. P.

Recovery of silver from thiosulphate solutions. J. MURRAY (B.P. 451,703, 16.2.35).—Cu and Zn plates insulated from one another and wrapped for the greater part of their length in blotting paper are immersed in the solution; the Ag is deposited on the Cu and the Zn gradually dissolves. A. R. P.

Production of solid magnesium sulphate monohydrate. METALLGES. A.-G. (B.P. 450,987, 18.3.35. Ger., 27.3.34).—Aq. MgSO<sub>4</sub> is evaporated in a multistage evaporator system at temp. rising from 75° to 125°, and then cooled by 5—8° stages in a cooling system by evaporation of part of the H<sub>2</sub>O. Heat-exchange arrangements are provided. A. R. P.

Production of aluminium chloride and certain metals as by-products. C. G. MINER and D. BAIRD (B.P. 451,969, 10.1.35).—Briquettes of  $Al_2O_3$  or bauxite and C are heated at 1000—1450° together with, or in the vapour of, metal chlorides, *e.g.*, ZnCl<sub>2</sub>, FeCl<sub>3</sub>, CuCl<sub>2</sub>, or PbCl<sub>2</sub>. A. R. P.

Recovery of vanadium from substances of poor vanadium content. O. JOCHEM (B.P. 451,981, 21.5.35). — Bauxite is leached with hot aq. NaOH under pressure and the clarified extract is conc. to  $d \cdot 2 - 1 \cdot 4$ , treated with a little Na<sub>2</sub>HPO<sub>4</sub> or Na<sub>2</sub>HAsO<sub>4</sub>, and cooled to obtain crystals of Na<sub>3</sub>PO<sub>4</sub> or Na<sub>3</sub>AsO<sub>4</sub> containing all the V as Na<sub>3</sub>VO<sub>4</sub>. A. R. P.

Separation of gases. GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 454,130, 22.2.36. Ger., 23.2.35).—For the preliminary removal of  $CO_2$ ,  $H_2O$ , and other easily condensible gases, air is chilled in periodic accumulators or continuous recuperators and then bubbled through liquid air or  $O_2$  from which the solids are subsequently removed by filtration or the like. B. M. V. Treatment of zinc muds or sludges. W. BECKER, F. BECKER, and O. ENGLERT (R. ENGLERT & DR. F. BECKER) (B.P. 451,013, 29.10.35).—Residues containing ZnO derived from Zn-dust reduction of org. compounds are extracted with aq. SO<sub>2</sub>, whereby heavy metals remain as sulphides in the insol. material, and the resulting aq. Zn(HSO<sub>3</sub>)<sub>2</sub> is boiled to ppt. ZnSO<sub>3</sub>, which is roasted to ZnO. A. R. P.

Apparatus for production of hydrogen. W. V. HANKS, G. H. FREVERMUTH, and G. POTTS, ASSTS. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 2,028,326, 21.1.36. Appl., 23.1.31).—Vertical tubes for causing hydrocarbon gas and steam to react pass through the roof and floor of a furnace, being arranged in two rows along the sides. A burner is placed at the top between the rows, and exhaust ports are distributed along an arched flue at the bottom centre line. B. M. V.

Purification of [sulphur burner] gases. B. M. CARTER, Assr. to GEN. CHEM. Co. (U.S.P. 2,028,318, 21.1.36. Appl., 3.2.31).—SO<sub>2</sub> gases for the manufacture of contact H<sub>2</sub>SO<sub>4</sub> are cooled gradually (the early stages being by wet cooling if desired) and several condensates are collected, the earlier ones being rejected and the later utilised as H<sub>2</sub>SO<sub>4</sub>. B. M. V.

Manufacture of products from boron fluoride and oxyacids of phosphorus. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 451,359, 4.2.35).—BF<sub>3</sub> is passed into conc.  $H_3PO_4$  or  $H_4P_2O_7$  until a thick viscous oil is obtained. The  $H_3PO_4$  retains the BF<sub>3</sub> up to > 150° and the product is useful as a catalyst in org. syntheses. A. R. P.

Carrying out chemical reactions. Crucibles for molten masses.—See I. Acid-proof pottery.—See VIII. Apparatus for interaction of CO and  $H_2$ .— See X. Perborates by electrolysis.—See XI. Ti pigments.—See XIII. Salts from molasses.—See XVII. Compounds from fermentation residues.— See XVIII.

## VIII.—GLASS; CERAMICS.

Glasses coloured by carbonaceous matter. I, II. Coloration by pure graphite, pure carbon black, and purified sucrose. K. FUWA (J. Soc. Chem. Ind., Japan, 1936, 39, 299–300 B, 300 B).—When graphite, C black, or sucrose is added to glass (SiO<sub>2</sub> 72, CaO 12, Na<sub>2</sub>O 16%) yellow or brownish-yellow shades are obtained, deepening with increasing amounts of C. Most of the C is probably oxidised during the melting, and lighter shades are obtained if a part of the Na<sub>2</sub>O is replaced by NaNO<sub>3</sub>. Replacing part of the CaO by Na<sub>2</sub>O does not affect the shade. When the amount of C is > 2% (0.4% for graphite) variable results are obtained; the C tends to coagulate and may collect on the surface and oxidise. A. G.

Properties of open-hearth furnace chequerbricks. T. SWINDON and J. H. CHESTERS (Trans. Ceram. Soc., 1936, 35, 469–482).—The results of extensive physical tests on  $SiO_2$ , semi- $SiO_2$ , and fireclay bricks are recorded. The last two materials are equally suitable for chequer use. Clean, unglazed bricks may be re-used with unimpaired efficiency. The corrosive materials in the slag on the bricks are mainly  $Fe_2O_3$  (45%) and CaO (10%). J. A. S.

Hot-face insulation.-See I.

See also A., Nov., 1352, Determination of Li in insol. silicates.

#### PATENTS.

Borosilicate glass containing cerium. CORNING GLASS WORKS, Assees. of H. P. HOOD (B.P. 455,082, 30.6.36. U.S., 30.7.35).—A glass containing alkali oxide < 10, B<sub>2</sub>O<sub>3</sub>  $\leq$  5, CeO<sub>2</sub>  $\leq$  2, and BaO + ZnO  $\leq$  7% can be worked satisfactorily without separation of cryst. CeO<sub>2</sub>. J. A. S.

Toughening or hardening of glass or glass objects. H. C. HEIDE. From F. ECKERT (B.P. 454,374, 26.3.35).—The glass is heated to near the softening point and cooled in stages : (1) rapid chilling to approx. (above or below) the lower annealing temp., (2) slow cooling. If the glass is 5—6 mm. thick (1) should take 5—15 sec. and may be subdivided into different rates; a slight reheat may be applied between (1) and (2). B. M. V.

Manufacture of porcelain resistant to the passage of X-rays. L. BROWN, Assr. to LENOX, INC. (U.S.P. 2,018,600, 22.10.35. Appl., 15.5.35).—A mixture of PbO 40—85, KNO<sub>3</sub> 1—5, and SiO<sub>2</sub> 14—59% is fused, ground to a fine powder, and fritted with a porcelain mixture containing  $ZrO_2$  and  $Al_2O_3$  to produce a product containing 32—66% of PbO, e.g., PbO 33.75, SiO<sub>2</sub> 31.2,  $Al_2O_3$  18,  $ZrO_2$  16, and K, Na, Fe, Ca, and Mg oxides  $\gg 1\%$ . A. R. P.

Ceramic products composed mainly of silica, magnesia, and alkalis. A. MALINOVSZKY, ASST. to MALINITE CORP. (U.S.P. 2,025,762, 31.12.35. Appl., 3.10.32).—Claim is made for a porcelain-like mass consisting of SiO<sub>2</sub> 4—14 (4·39), Al<sub>2</sub>O<sub>3</sub> 1—3 (1·46), MgO 0·6—8 (1·35), and Na<sub>2</sub>O 1 mol. and made, *e.g.*, by firing at 1050° a mixture of talc 49, kaolinite 6, and sodalite  $45^{\circ}_{0}$ . A. R. P.

Production of acid-proof pottery. K. TLSCHER (B.P. 454,475, 17.1.36).—A basic material, being the porous rock formed by atm. action on the clay of waste heaps discarded from coal or lignite mines, is bound with an alkali silicate. B. M. V.

Refractory articles. CARBORUNDUM Co. (B.P. 454,599, 5.4.35. U.S., 19.4.34).—Parts for the construction of a glass furnace are composed substantially of  $\beta$ -Al<sub>2</sub>O<sub>3</sub> formed by molten casting. B. M. V.

**Refractory articles.** CARBORUNDUM Co. (B.P. 455,236, 15.4.35. U.S., 17.4.34. Addn. to B.P. 409,315; B., 1934, 579).—A fusion-cast refractory material comprised of  $Al_2O_3 \ll 80$ ,  $SiO_2 \gg 15$ , and oxides or carbonates of Ca, Mg, Ba, Na, or K 1—10% is particularly resistant to molten glasses, including F-glasses. J. A. S.

Manufacture of porous bonded abrasive articles. CARBORUNDUM Co. (B.P. 454,251, 26.3.35. U.S., 29.3.34). —A mouldable mixture is formed of abrasive grains, bond, viscous pore-forming artificial resin, which may be the same as the bond, and a gas-generating material  $(H_2O_2)$ . The gas is allowed to form in the mould under such pressure that the bubbles are substantially spherical

while the walls of the bubbles are setting. The article is then dried and cured. B. M. V.

Insulators.—See XI. Condensation products.— See XIII.

#### IX.—BUILDING MATERIALS.

Influence of the alkalinity of the mixing water on the setting time of various hydraulic cements. P. ERCULISSE (Compt. rend. 15e Congr. Chim. Industr., 1935, 1, 441—448; Road Abs., 1936, 3, No. 475).—The strength of blast-furnace slag cement is increased. Higher strengths than normal are also obtained by using NaOH solution for gauging a white cement composed principally of 3CaO,SiO<sub>2</sub>. T. W. P.

D. A. Abrams' theory [of concrete]. G. KATH-REIN (Zement, 1936, 25, 682—686, 698—704).—The shape as well as the grading of the particles of the aggregate is of importance in evaluating the optimum composition of concrete. Eight granular materials were passed through a series of sieves and the size and shape distribution of the particles analysed in each sieve fraction and in the original materials. A system of classification of particle shapes is suggested. G. H. C.

Latvian clays as raw material for production of clinker. J. EIDUKS (Latvij. Univ. Raksti, 1936, 3, 25—143).—A comprehensive review and discussion of the Devonian, Tertiary, and Quaternary clays of Latvia. CH. ABS. (e)

Fillers-their employment in bituminous road construction. E. NEUMANN (Proc. Soc. Chem. Ind., Chem. Eng. Group, 1935, 17, 9-22) .- Points dealt with are the evaluation of fillers, e.g., in terms of the standard sieve test and the d in the shaken state and after compression at 800 kg./sq. cm., particle-size gradation (by H<sub>2</sub>O- or air-elutriation), and surface-area measurements; the relation of the type of filler to adsorption of bitumen; the properties of bitumenfiller mixtures (and particularly the cohesive power of the bitumen as given by the ring-and-ball test); the influence of the structure of the binder; and the construction of asphalt and tar carpets on the mortar theory. Adsorption and absorption have a greater influence on stiffening than has filler fineness, the  $\eta$ rise due to increasing additions of filler being directly oc adsorption, although blast-furnace slag is an exception; the  $\eta$  of tar-filler mixtures is thus relatively that of the corresponding bitumen mixtures. The bitumen requirement of a sand-asphalt mixture may be calc. from the amounts of bitumen in the bitumen-filler mixture, and required to coat the sand particles with a film 5 µ thick. J. G.

Structure of the middle lamella [of wood]. A. J. BAILEY (Paper Ind., 1936, 18, 379—381).—The results of experimental work on the structure of the middle lamella of Douglas fir and the effects of chemical digestion (sulphite process and acidified dioxan) are reported. Sulphite digestion similar to commercial practice except for a lower temp. did not completely dissolve these lamellæ, but the resistant segments were dissolved by NaOC1. New morphological features, and hiatus in the secondary wall of the summer-wood tracheids accompanied by segmented middle lamellæ (the ends of the lamellar segments coinciding with the hiatus), have been discovered. Several photomicrographs are given. H. A. H.

Properties of home-grown oak. ANON. (Dept. Sci. Ind. Res., 1936, Forest Products Res. Rec. 11, 14 pp.).—A comparison is made between the strength properties of oak (seasoned, preserved, etc.) from different localities in the United Kingdom and also between home-grown and American oaks. J. P. O.

#### PATENTS.

Manufacture of cement. C. H. BREERWOOD, Assr. to VALLEY FORGE CEMENT CO. (U.S.P. 2,028,313, 21.1.36. Appl., 24.7.34).—The composition of inferior argillaceous limestone or the like is adjusted by wet-dressing, elements of the system being a hydro-separator, rake classifier delivering three products, a shaking table, and final collecting thickener. B. M. V.

High-silica cement. F. A. B. FINKELDEY (U.S.P. 2,028,386, 21.1.36. Appl., 24.1.34).—To hot (700—1050°) Portland cement clinker are added siliceous and calcareous material, mixing being effected until the latter is calcined and the former activated, after which  $H_2O$  is sprayed on to hydrate the former. The siliceous material is approx. 24% and the calcareous material  $\Rightarrow$  6% of the whole. B. M. V.

[Manufacture of] coloured plates from mixtures of hydraulic binding agents and fibrous substances. G. SCHLESS (B.P. 455,343, 27.4.36. Ger., 26.4.35).—Portland cement, asbestos, and pigment are mixed with about 5% of H<sub>2</sub>O added, e.g., as a fine spray. The mixture is loosened in centrifugal mills, pressed into shapes, and laid on moist plates for setting. Fused, bauxite, and other cements may be used, and quartz or corundum added to improve wear-resistance. The plates are free from blooming and are coloured uniformly. T.W.P.

Manufacture of artificial stone. N. V. S. KNIBBS and A. P. PEHRSON (B.P. 455,039, 12.2. and 13.4.35).— Siliceous or argillaceous materials (e.g., shales, clays, etc.) are mixed with quicklime (I) or mixtures of (I) with  $Ca(OH)_2$ , a compound of an oxy-acid of S (e.g.,  $CaSO_4$ ), and  $H_2O$ . After allowing to hydrate, the mixture is hardened by means of steam under pressure. Gasgenerating agents (e.g., Al powder) may be added during mixing, and the degree of porosity may be adjusted by control of temp. during hydration.

T. W. P. Manufacture of decorative fibrous sheet material and plaster board. G. C. TYCE, V. LEFEBURE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 453,648, 14.2.35). —The sheets are made by superimposing a sheet of resin-impregnated paper on a layer of unimpregnated paper or cloth. The lower layer is backed with unimpregnated absorbent paper, and the whole is treated by means of heat and pressure so that the resin flows to cement the layers. Various modifications in manufacture are claimed, together with methods for using the sheets on boards. T. W. P.

Manufacture of glued-up plywood. W. F. LEICESTER (U.S.P. 2,028,287, 21.1.36. Appl., 2.1.31).— Glue liquefiable by heat (e.g., casein-CaO) is applied in the moist state to wood and dried immediately to prevent the liquid soaking into the wood; the parts are then pressed together while hot. B. M. V.

**Production of building material.** A. SINGER (B.P. 453,905, 18.7.35).—Rough block wood is treated with steam of low superpressure (e.g., 0.5 atm.) and Pb(OAc)<sub>2</sub>. The latter is introduced into the steam chambers as a fine spray. The product is converted into wood wool, and used with cement and H<sub>2</sub>O to make slabs. T.W.P.

Adhesive.-See XV.

# X.—METALS ; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Economic possibilities of the Poços de Caldas plateau. O. BARBOS (Bol. Min. Agric., Brasil, 1936, 25, 69—101).—The plateau has important mineral resources, viz., magnetite, bauxite, ochre, Mn, and Zr. L. A. O'N.

Injection of solid and liquid materials in blastfurnace hearths. R. KREIDE and J. ROLL (Stahl u. Eisen, 1936, 56, 1177—1179).—In order to eliminate obstructions in the working of blast furnaces, solid and liquid materials have been injected through the blast tuyères. This also effects an improvement in the physical and chemical properties of the slag. For this purpose sand has proved useful, and for furnaces producing basic Bessemer pig Fe the injection of sand below the tuyères increases the SiO<sub>2</sub> content of the slag without affecting the metal. Good results were obtained in the case of local temp. reduction by addition of Fe–Si or, in severe cases, of crude tar or oil. W. P. R.

Viscosity of open-hearth furnace slags. F. HARTMANN (Arch. Eisenhüttenw., 1936—7, 10, 45— 52).—Tests made on slags during the course of the open-hearth process failed to establish a definite relation between the basicity and  $\eta$  of the slag. Increase in the SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, MnO, CaO, and FeO contents during the refining process sometimes increases and then decreases  $\eta$ . Addition of CaF<sub>2</sub> makes the slag more fluid.

#### A. R. P.

Cementation of grey cast iron with beryllium. J. LAISSUS (Rev. Mét., 1936, 33, 494–497).—Grey cast Fe heated in powdered Fe-Be alloy at 800—1150° for 2—10 hr. is case-hardened by the diffusion of Be. Three diffusion zones are evident, viz., a solid solution of Fe-C-Be + eutectic, a eutectic + Fe-Be compound, and a hypereutectic zone. Surface hardness of 1500 Brinell has been obtained, but no unusual resistance to corrosion results from the "cementation" by Be.

W. P. R.

Elastic modulus of cast irons. A. MITINSKI (Rev. Mét., 1936, 33, 498—501).—The modulus of cast Fe is a function of the graphite content, and for a white Fe its val. is 25,000. The true modulus is not obtained from test-pieces of 30 mm. diam., as the cooling conditions may not represent the conditions of a larger or smaller casting, and some definite relation between the ruling thickness of the casting and the diam. of testpieces is necessary to obtain a working val. of the modulus. W. P. R.

Deformation and re-orientation in the surface working (lathe cutting) of iron. M. RENNINGER (Metallwirts., 1934, 13, 889—892; Chem. Zentr., 1935, i, 2243).—X-Ray examination shows that the deformation of the cut surface is the deeper the slower is the rate of cutting, and affords evidence that the surface layers acquire a definite orientation which, however, is different from that produced by rolling. A. R. P.

Production and properties of steels made from carbonyl iron. E. K. OFFERMAN, H. BUCHHOLTZ, and E. H. SCHULZ (Stahl u. Eisen, 1936, 56, 1132-1138).-Unalloyed steels were made by sintering carbonyl Fe mixtures containing varying proportions of C and O. The Fe powders employed contained 1.0-1.4% C and 1.0-1.4% 0, 1.6% C and 4.3% 0, and 1.9% C and 1.1% O. Sintering times of 5 hr. and temp. up to 1050° were used. The production of high-C steels by sintering is more difficult than of low-C steels, and segregated zones occur if sintering conditions are not correct. Compared with commercial steels of the same C content the sintered steels had lower strength properties after normalising and quenching, and the pearlite had a strong tendency to spheroidise and grain growth easily occurred. There was no difference in bendingfatigue properties,  $\kappa$ , and magnetic properties, and the sintered steels showed improved welding properties.

W. P. R.

Influence of phosphorus on properties of basic unalloyed steel. A. RISTOW, K. DAEVES, and E. H. SCHULZ (Stahl u. Eisen, 1936, 56, 889-899, 921-927).— The basic Bessemer process and the basic pig-andscrap method of steel-making lead to over-oxidised steel and very inefficient use of the Mn if efforts are made to keep the P content low. In general, the mechanical properties of unalloyed steels are not adversely affected by medium P content, and some properties, *e.g.*, wearand corrosion-resistance, weldability, and machinability, are slightly improved, whereas too low a P content may result in non-metallic inclusions, poor wear-resistance in rails, and excessive sticking in thin sheets during rolling. W. P. R.

Dephosphorisation of steel in coreless induction furnace with alkaline slags. H. SIEGEL (Stahl u. Eisen, 1936, 56, 1179—1184).—The irregular behaviour of FeO-CaO slags is caused by their low fluidity; addition of Na<sub>2</sub>CO<sub>3</sub> decreases the  $\eta$  and dephosphorisation becomes more regular. The reaction proceeds further at 1580° than at 1600° and the resulting slags are richer in P. W. P. R.

Influence of the rate of running through in lead patenting on the strength properties of steel wire. A. POMP and H. RUPPIK (Stahl u. Eisen, 1936, 56, 899-903).—A 0.66% C steel was patented at various quenching and Pb-bath temp. and using three different speeds of travel through the bath. The speed, furnace temp., and furnace size determine the length of time during which the wire was above the A3 temp. The temp. of the wire was measured with a thermocouple welded in position. For fixed bath and quenching temp. the speed of running through the bath has a marked influence on the results of the bend and torsion tests, the best vals. being obtained with medium rates of travel. W. P. R.

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Nickel alloy steels and cast irons in railway locomotives. A. E. Moss and H. OKUDA (Japan Nickel Rev., 1936, 4, 416-448).—The mechanical properties of a wide range of Ni steels are discussed, and the reduction in wt. of locomotives by their use is indicated. The maintenance of toughness at low temp. is a property made use of in locomotives working in very cold climates. W. P. R.

Modern views on the influence of usual alloying elements on malleable cast iron. R. STOTZ (Giessereipraxis, 1935, 56, 27–29, 51–53; Chem. Zentr., 1935, ii, 113).—A review of the influence of C, Si, Mn, P, and S. J. S. A.

Hardenable cast steel containing copper. E. Söhnchen and E. PIVOVARSKY (Giesserei, 1935, [ii], 22, 96-100; Chem. Zentr., 1935, ii, 115).—The pptn.hardening of cast steel containing about 1% Cu, and the influence of Cu on the physical and chemical properties of the steel, are reviewed. J. S. A.

Notch-sensitivity of steel under alternating stresses. R. MALÄNDER (Tech. Mitt. Krupp, 1935, 3, 108—111; Chem. Zentr., 1935, ii, 1430).—Tests on numerous steels with a tensile strength of 38—124 kg./sq. mm. show that the notch-sensitivity increases with increase in tensile strength and that alloyed steels have little superiority over plain steels of equal strength. The notch-sensitivity no. of Thum and Buchmann is shown to be useless in characterising small differences in the quality of steels. No relation could be established between notch-toughness and the notch-sensitivity no. A. R. P.

Welding alloy steels. A. B. KINZEL (Trans. Amer. Soc. Mech. Eng., 1936, 58, 511-513).—The effects of welding heat on metal adjacent to the weld, and the intensity of the internal stresses to be expected with joints of different designs and various welding practices, are discussed. R. B. C.

Arc-welding of structural alloy steels. W. L. WARNER (Trans. Amer. Soc. Mech. Eng., 1936, 58, 516-526).—Data showing the effect of welding heat on the chemical and physical properties of metal in the vicinity of the weld are given. R. B. C.

Preventing corrosion. W. Swallow (Auto. Eng., 1936, 26, 409-410).—Methods of preventing corrosion in steel body work are discussed. R. B. C.

Corrosion - resisting properties of ferrous materials used in gas-pipe manufacture. J. E. FLETCHER (Gas World, 1936, 105, 154-155).—The special claims of wrought Fe are put forward, and its resistance to corrosion by pitting is illustrated by comparison with ingot Fe and mild steel. The constitution of the slag and its condition are valuable in conferring on wrought Fe its characteristic mode of corroding by general wastage rather than by pitting. W. P. R.

Centrifugal purifier for molten metals. E. VROONEN (Rev. Mét., 1936, 33, 502-509).—The purifier consists of a tube 6 m. long and of conical section. It is inclined at 35° and the speed of rotation is 450 r.p.m. It effects very intimate mixing of slag and metal at entry and the centrifugal separation of the two. Data are given showing an elimination of 50-60% of S and 10—40% of Si by using a slag containing 0.5% of Na<sub>2</sub>CO<sub>3</sub> in contact with cast Fe. W. P. R.

Copper-alloy welding. I. T. HOOK (Trans. Amer. Soc. Mech. Eng., 1936, 58, 535-539).—Practical hints are given on the brazing and welding of Cu alloys and on the joining and surfacing of ferrous and other metals with the latter. R. B. C.

Nickel in brass-foundry practice. D. M. CURRY (Metal Ind., N.Y., 1936, 34, 330-332).—The use of Ni in brasses, bronzes, and nickel-silvers is described. L. S. T.

Effect of iron impurities on the annealing of high brass. W. A. GIBSON and J. H. Doss (Metal Ind., N.Y., 1936, 34, 333-335).—With brass  $(64-66\cdot5\%)$  Cu) having Pb and Fe as the only impurities, Fe contents > 0.09% are not harmful to annealing. Between 0.09 and 0.118% Fe marked discontinuities in annealing properties occur. L. S. T.

Influence of annealing on the structure, Brinell hardness, and tensile properties of cold-rolled brass (Cu 67%, Zn 33%). L. LOSKEWICZ [with, in part, G. GOUSCHLBAUER, M. NOSOWICZ, Z. HAVTO, and B. PODCZASKI] (Rev. Mét., 1936, 33, 473-488).—The influence of annealing 70/30 brass for 0.25, 6, and 200 hr. at 200-750°, after cold-rolling, on the microstructure, X-ray structure, and mechanical properties has been studied. It is pointed out that the primitive structure and mechanical properties before rolling have a certain influence on the final product. The refinement of grain size occurs at the same temp., independently of initial grain size, but depending on the amount of cold-rolling ; complete refinement, however, requires a longer time for the initially coarse-grained material. W. P. R.

Alloys in the zinc bath. G. A. BRAYTON (Metal Ind., N.Y., 1936, 34, 336-337). L. S. T.

Nickel alloys in Diesel locomotives and in railcars. K. TAREMURA (Japan Nickel Rev., 1936, 4, 449-471).—Suitable ferrous and non-ferrous Ni alloys are described. W. P. R.

Non-ferrous alloys in railway engineering. K. KAWADA (Japan Nickel Rev., 1936, 4, 475-492).— The properties of pure Ni, Ni-Cu alloys, nickel-silvers, and bearing metals containing Ni are given and discussed in relation to their use for this purpose. W. P. R.

Electric welding of monel and nickel. F. G. FLOCKE and J. G. SCHOENER (Trans. Amer. Soc. Mech. Eng., 1936, 58, 541-546).—The technique employed in the metallic-arc and C-arc welding of these metals and in the welding of Ni to steel is described. R. B. C.

Determination of small amounts of copper in tin by controlled potential. S. TORRANCE (Analyst, 1936, 61, 688—689).—A solution of the metals as sulphates, free from Cl', is prepared. This is electrolysed as described by Lindsey and Sand (A., 1934, 620, 621) with an auxiliary potential of 0.2 volt for 2—3 min., followed by 30 min. at 0.4 volt. E. C. S.

Metal cleaning. F. E. P. GRIGGS (Canad. Chem. Met., 1936, 20, 258, 260, 316, 318).—A review.

Hindering of corrosion by precautions with corroding medium. E. RABALD (Chem. Fabr., 1936, 9, 473–479; cf. B., 1936, 841).—A review in which the influences of changes in the corroding medium, e.g., the addition of  $CrO_4''$ , are discussed. R. S. B.

Composition of insoluble residues formed by dissolving aluminium-copper-nickel alloys in potash. O. QUADRAT and J. JIRISTE (Rev. Mét., 1936, 33, 489–493).—In Al-Cu-Ni alloys with Cu > 3 and Ni > 4% the compounds NiAl<sub>3</sub>, CuAl<sub>2</sub>, and Cu<sub>2</sub>NiAl<sub>3</sub> may be present. The two binary alloys appear to be attacked by KOH, and the resistance to attack of Cu<sub>2</sub>NiAl<sub>3</sub> depends on the degree of its dispersion in the alloy. In the binary alloys AlNi and AlCu only small amounts of Al are found in the insol. residue.

W. P. R.

Recent developments in the production of lightmetal castings. W. LINICUS (Aluminium, 1936, 18, 401-408).—A review.

Temperature measurement of aluminium ingots. A. VON ZEERLEDER (Aluminium, 1935, 17, 199–201).— Ordinary thermocouples may give errors of up to 20° in measuring the temp. of Al ingots during heat-treatment. Accurate results are obtained with the use of a square block of insulating material fitted with a handle and with stout wires of Ni and Ni–Cr, forming the two elements of the couple, protruding just through the block and there sharpened to points. On pressing these points into the Al ingot so that they penetrate the oxide skin the temp. can be read on the galvanometer in 1-2 sec. A. R. P.

Bending of thick aluminium bars. NICOLINI (Aluminium, 1935, 17, 220—221).—Al bars  $(18 \times 3.5$  cm.) when bent through an angle of 90° showed an increase of 20% in tensile strength and 30% in hardness and a decrease of 57% in elongation in the deformed zone; only a negligible reduction in  $\kappa$  was produced by the bending. A. R. P.

Welding of aluminium alloys. G. O. HOGLUND (Trans. Amer. Soc. Mech. Eng., 1936, 58, 547-552).— The difficulties encountered, and the technique and equipment employed, in the gas, metallic-arc, and electric-resistance welding of Al and its alloys are described. R. B. C.

Spectral-analytical determination of alkali and alkaline-earth metals in aluminium and lead. A. K. RUSSANOV and B. I. BODUNKOV (Z. anal. Chem., 1936, 106, 419–427).—The metal is dissolved in acid, and the solution injected into a controlled  $C_2H_2$ -air flame by means of a Lundegårdh atomiser. The lines are photometered visually by observing the thickness of an absorbing wedge required to cause their disappearance. Subjective errors are eliminated by direct comparison with standard solutions of similar concn. J. S. A.

Electromagnetic control of metallurgical products. B. JOUSSET (Rev. Mét., 1936, 33, 525-526).— The control of the hardness of quenched steel parts such as ball bearings, ball races, etc. can be achieved by measuring their magnetic properties. An apparatus which automatically rejects parts below a min. hardness is discussed. W. P. R.

Electrolytic refining of silver containing palladium. G. HÄNSEL [with K. DORSCH] (Metall u. Erz, 1935, 32, 161—163).—A Ag alloy with Ag 79.8, Au 2.3 Pd 0.6, Pb 0.15, Cu 10, Ni 0.76, Zn 0.16, and Fe 0.2% was electrolytically refined in an electrolyte containing Ag 73 and free  $\text{HNO}_3$  4 g./litre until the composition of the latter had changed to Ag 42 and Cu 70 g./litre. Analysis of the products showed that 83.4% of the Pd remained in the anode slime, 12.5% accumulated in the electrolyte, and 4.1% was deposited with the Ag, chiefly in the later deposit. It is recommended that the Pd be removed continuously from the electrolyte by addition of dimethylglyoxime. A. R. P.

Formation and growth of pits in electrodeposited metals. M. CYMBOLISTE (Trans. Electrochem. Soc., 1936, 70, Preprint 29, 347—362).—The origin of the gas bubbles which produce pits in electroplating, especially in Ni deposition, is discussed, and photomicrographs of different types of pit are given. The adherence of bubbles to the basis metal or to the electrodeposited metallic surface is attributed to a wide variety of possible irregularities in this surface. The deposition of a thin coat of Cu between two coats of Ni is suggested as a method for the elimination of pits. O. J. W.

Hardness-testing machines.—See I. Inks for metal foil.—See XIII.

See also A., Nov., 1332, Heat contents of molten alloys. Conductivities of Cu alloys. Consts. of Sb-Ag alloys. Magnetic properties of Ag-Pb, Ag-Sb, Ag-Bi, and Ni-Cu alloys. Stability of cementite. System Fe-Zn. 1333, Systems Mg-Mn and Ni-Sn. 1342, Electrolytic corrosion protection. 1343, Co-deposition of metals. Passivity of Fe and steel in HNO<sub>3</sub>. 1347, Eu. 1351, Colorimetric determination of  $H_3PO_4$  [in steel analysis]. 1355, Hardness tester.

## PATENTS.

Non-reversing open-hearth furnace. F. H. LOFTUS (U.S.P. 2,028,290, 21.1.36. Appl., 30.1.32).— The waste gases are passed through primary and secondary cleaning chambers where they are cleaned by decrease of speed, and are then passed through hollow tile and metallic recuperators situated below the intake. B. M. V.

Protection of cast iron and steel against oxidation at high temperatures. Soc. ANON. DES HAUTS FOURNEAUX DE LA CHIERS (B.P. 451,472, 9.11.35. Fr., 21.11.34).—The metal is sand-blasted, coated with Al by any known process and then with Cu or Fe by plating or welding, heated to a temp. at which the Al diffuses into the outer layers, cooled, and again sand-blasted to remove the outer Cu or Fe layer. A. R. P.

Flame-machining of metal [e.g., steel]. LINDE AIR PRODUCTS CO., Assees. of W. J. JACOBSSON and M. J. HARRIS (B.P. 451,071, 19.11.35. U.S., 4.12.34).—The flame of the torch is kept at an angle to the direction of movement and inclined away from the cut surface so that the molten metal is oxidised and blown away from the work. A. R. P.

Manufacture of iron-silicon alloys. R. MUL-SCHAERT (B.P. 451,463, 31.7.35).—Pig Fe is desulphurised and dephosphorised by addition of Fe-Mn and treatment with a basic slag containing powdered petroleum

coke, CaO, and BaO, then carburised to 3% C by addition of  $M_4$ Fe(CN)<sub>6</sub>, and finally treated with high-grade Fe–Si to bring the Si content up to 15–16%, and, if desired with 0.1–0.2% B (as Fe–B). A. R. P.

**[Ferrocarbon-titanium] alloys.** TITANIUM ALLOY MANUFG. Co. (B.P. 452,092, 7.2.36. U.S., 20.6.35).— The alloys contain Ti 15—25, C 1.5—5, Si  $\gg$  3, and Al  $\gg$  3%, the % Ti being  $> 4 \times \%$  C; they are prepared by melting ilmenite over the graphite hearth of an electric furnace, then melting steel scrap on this lining, and finally reducing ilmenite with C above the steel bath. A. R. P.

[Alloy] lining of apparatus for use when reacting carbon monoxide with hydrogen at elevated temperature and under pressure. Courts & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 454,428, 29.3.35).—Steel is coated with Zn or Cd to form an alloy by diffusion ; *e.g.*, Cr steel is subjected to O-free Zn vapour at 800°. B. M. V.

Manufacture of [lead-, tin-, or zinc-]coated sheet metal [iron]. WEAN ENG. Co., LTD., Assees. of R. J. WEAN (B.P. 451,771, 11.2.35. U.S., 20.2.34).—Fe strip with or without pickling is passed through a centrifugal abrasive blast to roughen its surface, and then directly through a bath of molten Zn, Sn, or Pb. A. R. P.

Copper alloys. F. R. HENSEL and E. I. LARSEN, Assrs. to WESTINGHOUSE ELECTRIC & MANUFG. Co. (U.S.P. 2,025,662, 24.12.35. Appl., 8.2.35).—The alloy contains Cr 0.08—2.54 (1.5) and Zr or Th  $\Rightarrow$  5 (Zr 2.5)%. A. R. P.

Copper alloy. P. H. BRACE, ASST. to WESTINGHOUSE ELECTRIC & MANUFG. Co. (U.S.P. 2,026,209, 31.12.35. Appl., 14.12.34).—A Cu alloy of good electrical conductivity and hardness contains Zr 0.1—5 (0.8) and Ag 0.05—10 (0.5)%. A. R. P.

**Production of composite [copper-silver-gold** alloy] dental plates. S. Loos (U.S.P. 2,025,759, 31.12.35. Appl., 2.5.32. Austr., 2.4.32).—A Cu matrix is formed from an impression of the patient's mouth, Au leaf is applied to the matrix, the overlapping parts of the Au are stopped off with wax, and the Au surface is plated successively with thin layers of Au, Ag, and Cu, the frequency and thickness of the Au layers decreasing from the outside to the core. The composite plate is then annealed to produce alloying of the layers. A. R. P.

[Nickel-chromium-copper] alloy. D. P. MOORE (U.S.P. 2,026,243, 31.12.35. Appl., 18.1.35).—An alloy for use in deoxidising steel consists of Ni  $33 \cdot 3 - 95$ , Cr  $33 \cdot 3 - 2 \cdot 5$ , and Ca  $33 \cdot 3 - 2 \cdot 5 \$ . A. R. P.

Colouring of copper and copper alloys. H. W. BROWNSDON, L. C. BANNISTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 450,459, 15.1.35).—A green patina is obtained by spraying or brushing the metal with an 8:1 vol. mixture of saturated aq.  $(NH_4)_2SO_4$  and conc. HCl. A. R. P.

Treatment of [zinc] sulphide ores. T. A. MITCHELL, Assrs. to HUGHES-MITCHELL PROCESSES, INC. (U.S.P. 2,025,068, 24.12.35. Appl., 17.11.33).—The ground ore or concentrate is mixed with PbO or PbS and roasted to produce a mixture of ZnO and PbSO<sub>4</sub>. This is treated at 120° with Cl<sub>2</sub> or HCl to convert the ZnO present into  $ZnCl_2$ , which is recovered by leaching with dil. HCl. The  $PbCl_2$  in the residue is removed by leaching with brine. A. R. P.

Condensation of metallic [zinc] vapours. Sr. JOSEPH LEAD CO. (B.P. 451,774, 11.2.35. U.S., 8.3.34). —The vapour is drawn through a bath of molten Zn contained in a condenser with the lower half inclined and provided internally with baffles. By reducing the pressure in the upper half of the condenser, the Zn vapour and CO from the retorts are caused to agitate vigorously the liquid Zn and thus produce efficient condensation. A. R. P.

Manufacture of porous metal bodies. V. CUR-STÄEDT (B.P. 451,818, 21.11.35. Ger., 23.11.34).— Layers of tinned or galvanised Fe, Cu, or brass wire mesh are consolidated into porous bodies by the application of heat and pressure. The products are suitable for bearing metals. A. R. P.

Manufacture of tungsten wire. GEN. ELECTRIC Co., LTD., B. P. DUDDING, and W. R. PITKIN (B.P. 451,600, 7.2.35).—After the swaging operation is finished the wire is drawn rapidly through a SiO<sub>2</sub> tube at 1100° against a stream of  $O_2$  and the surface  $WO_3$  is removed by pickling in 20% aq. NaOH. The wire is then drawn to 0.175 mm., annealed at 1300—1500°, and drawn to finished size. A. R. P.

**Production of hard metal alloys.** G. BOECKER (B.P. 451,272, 26.10.34).—The constituents are placed in a graphite crucible surrounded by a thermit mixture, ignition of which provides the heat necessary for melting the alloy. The mixture may consist of Al and the oxides of some of the metals to be alloyed, the resulting metal regulus then being remelted with the other constituents in the manner described above. A. R. P.

Corrosion - resistant [rhodium-nickel] alloys [e.g., for pen nibs]. MOND NICKEL CO., LTD. (B.P. 451,823, 3.12.35. U.S., 5.12.34).—The alloys consist of Ni with 25—80% Rh. Part of the Ni may be replaced by  $\geq 20\%$  of Co, Fe, or Cu, or by  $\geq 55\%$  of Pd, and part of the Rh by Pt or Ir, but the alloy must contain  $\leq 10$  at.-% Rh. Small amounts of W and/or Mo may be added for hardening. A. R. P.

Treatment of hard metal [carbide] compositions [for crushing]. BRIT. THOMSON-HOUSTON CO., LTD. (B.P. 451,095, 28.3.36. U.S., 30.3.35).—Sintered WC-Co compositions are heated at 1700° for 30 sec. in a closed C pot, whereby they become spongy and readily pulverisable. A. R. P.

Welding metallic tips [hard alloys] to metallic bases [gold pen nibs]. A. H. STEVENS. From PFAN-STIEHL CHEM. Co. (B.P. 451,409, 29.12.34).—The Ir alloy pen-point is just "tacked on" to the Au nib by resistance-welding and the nib is then supported in a  $H_2$ arc so that only the pen point is melted and thus welded securely to the tip of the nib. A. R. P.

Separating gold and other heavy concentrates from finely-divided ore or matrix. M. O. McCov (B.P. 451,810, 7.8.35).—The ore pulp is centrifuged in a basket with an annular bulge in the lower part wherein the Au and heavy minerals collect. A. R. P. Recovery of precious metals from ores or precious metal-bearing deposits. MERRILL Co., L. D. MILLS, T. B. CROWE, and J. C. HAUN (B.P. 451,979, 14.5.35).—Low-grade Au ores are leached with very dil. aq. NaCN and the leach liquor is treated with SO<sub>2</sub>, CO<sub>2</sub>, or NaHSO<sub>4</sub> to  $p_{\rm H}$  6—8·4, whereby the dissolved O<sub>2</sub> is removed and the Au can be pptd. with Zn dust without increasing the [CN'] to the normal val. for pptn. A. R. P.

Protection of magnesium and its alloys. J. M. MICHEL, Assr. to MAGNESIUM DEVELOPMENT CORP. (U.S.P. 2,028,343, 21.1.36. Appl., 30.7.32. Ger., 6.8.31). —Inhibition of corrosion of Mg-containing engine parts by glycol or the like in the radiator system is effected by presence of a minor proportion (< 0.1%) of an alkali fluoride. B. M. V.

Production of magnesium-base alloys. J. STONE & Co., LTD., and A. J. MURPHY (B.P. 451,229,31.1.35).— The alloys contain Mg  $88 \cdot 5$ — $91 \cdot 5$  (90), Sn  $4 \cdot 5$ — $5 \cdot 5$  (5), Zn  $3 \cdot 5$ — $4 \cdot 5$  (4), and Cd  $0 \cdot 5$ — $1 \cdot 5$  (1)%. Heat-treatment comprises annealing at 425— $435^{\circ}$  ( $435^{\circ}$ ) for 14—20 (16) hr., quenching, and reheating at 150— $160^{\circ}$  ( $150^{\circ}$ ) for 14—20 (16) hr. The preferred alloy has a yield point of 10—11 tons/sq. in., a tensile strength of 12 tons/sq. in., an elongation of 1— $3^{\circ}_{0}$ , and a Brinell hardness of 65—75. A. R. P.

Aluminium - base alloys. ALUMINIUM, LTD., Assees. of J. A. NOCK, JUN., and H. R. FRECHE (B.P. 451,148, 3.7.35. U.S., 25.10.34).—The alloys consist of Al with Cu 2—12 (4.4), Sn 0.005-0.1 (0.05), Mg 0.005-0.03 (0.02), Mn 0.1-1 (0.8), Si 0.1-1 (0.8), and Cd 0.05-0.15 (0.1)%. The presence of Cd eliminates the deleterious effects of the Mg during ageing.

A. R. P.

Electrodeposition of metals [silver]. A. R. POWELL, E. C. DAVIES, and JOHNSON, MATTHEY & CO., LTD. (B.P. 450,979, 26.1.35).—Na<sub>2</sub>SiO<sub>3</sub>, or a soap derived from a fatty acid of  $> C_8$  or from a sulphonated alcohol, is added to the Ag-plating nath; *e.g.*, the bath contains Ag [as KAg(CN)<sub>2</sub>] 3, free KCN 6, Na oleate 3 oz./gal., and a small amount of brightener made by digesting a portion of the bath with CS<sub>2</sub> or a mercaptan. A. R. P.

Electroplating aluminium with metal. VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 451,904, 23.12.35. Ger., 24.12.34).—After part of the desired thickness of coating is deposited the metal is removed from the bath and the pores are closed by a chemical, electrical, or other oxidising treatment followed by immersion in boiling  $H_2O$ ; plating is then continued in the original bath.

A. R. P.

[Palladium-silver] alloys. BARER & Co., INC. (B.P. 451,101, 23.10.34. U.S., 25.4.34).—See U.S.P. 1,999,864—6; B., 1936, 796.

Heat-treating furnace. Crucibles for molten masses.—See I. Ag from  $M_2S_2O_3$  solutions. Metals as by-products.—See VII. Electrodes for furnaces [for Al]. Cathode for electrolytic cells.— See XI. Ebonite coating [for metal].—See XIV.

## XI.—ELECTROTECHNICS.

Correlation of the chemical composition, physical constitution, and electrical properties of solid

dielectric materials. W. JACKSON (J. Inst. Electr. Eng., 1936, 79, 565-576).—A summary of recent progress.

Measurement of permittivity and power factor of dielectrics at frequencies from  $10^4$  to  $10^8$  cycles per second. L. HARTSHORN and W. H. WARD (J. Inst. Electr. Eng., 1936, 79, 597-609).—A method and apparatus using capacitance variation in a tuned circuit are described.

Insulating oils.—See II. Welding. Determining traces of Cu in Sn. Control of metallurgical products. Refining Ag containing Pd. Pits in electrodeposited metals.—See X.

See also A., Nov., 1342, Electrolytic corrosion protection. 1343, Co-deposition of metals. 1347, Eu. Electrolytic oxidation of  $\alpha$ -OH-acids. 1348, Production of NO by electric arc. 1355, Apparatus for electrodialysis etc. 1396, Preparing proteins at the isoelectric point.

### PATENTS.

Electrodes for electric furnaces. NORSKE A./S. FOR ELEKTROKEMISK INDUSTRI (B.P. 454,584, 4.11.35. Norw., 6.4.34).—Self-baking electrodes when in use for the manufacture of Al are provided with protecting metal plates, not used as conductors, extending to near the surface of the bath and serving to conduct heat away from the lower part of the electrode E. The plates conform to the shape of the electrodes, but exert no material pressure thereon. B. M. V.

Current-supply contacts for electrodes of electric furnaces. SIEMENS-PLANIAWERKE A.-G. F. KOHLE-FABRIKATE (B.P. 454,359, 30.3.36. Ger., 28.3.35).— Longitudinal grooves are formed in the C and subdivided metallic contacts are individually expanded upwards to make contact with the walls of the grooves. B. M. V.

Electric furnaces. G. H. S. GREENE, and WILD-BARFIELD ELECTRIC FURNACES, LTD. (B.P. 454,574, 2.4.35).—The heat-insulating wall is lined internally with a metallic shell which is extended upwards to form a liquid seal with the lid. B. M. V.

Electrical insulating material [from polymerised styrene]. STANDARD TELEPHONES & CABLES, LTD., and J. K. WEBB (B.P. 454,923, 8.3.35).—Rolls of paper etc. are impregnated with a plasticiser and monomeric styrene and are then polymerised by heating in a closed vessel; the rolls are unwound while soft and excess of polystyrene is removed by passing the material through a solvent, e.g.,  $C_6H_6$ . The product after being calendered can be wound and unwound. S. M.

Manufacture of electrical insulators having high surface leakage resistance. CORNING GLASS WORKS, Assees. of W. W. SHAVER (B.P. 454,448, 15.4.35. U.S., 2.5.34).—Glass or porcelain has its surface glazed with alkaline glass, the outermost surface of which is coloured red with Cu and/or Ag compounds of lower alkali content. The metals are preferably diffused in at 600— 750° from any suitable compound having m.p. < that temp., e.g., by dipping in a bath of fused Ag and Cu chlorides containing  $\leq 5\%$  of CuS. B. M. V. Electric arc welding. W. R. HUME, and HUME STEEL, LTD. (B.P. 451,582, 7.1.35).—The work is reciprocated or oscillated during welding so as to flow the molten metal backwards and forwards and expose the underlying metal to the arc. A. R. P.

Electrolytic condensers. HYDRAWERKE A.-G., and K. GORNIAK (B.P. 454,202, 15.1.36).—The electrolyte comprises alkaline lactates in naturally viscous condition and is suitable for the construction comprising foils coiled up together. B. M. V.

Electrolytic devices. J. B. BRENNAN (B.P. 454,047, 7.8.35. U.S., 7.8.34).—In a condenser comprising a filmed anode, an electrolyte, and an unfilmed cathode, the effective side of the cathode is, and that of the anode may be, roughened by etching, their effective areas, thus increased, being equal. B. M. V.

Electrolysis of aqueous solutions [to form perborates]. N. V. INDUSTR. MAATS. VOORH. NOURY & VAN DER LANDE (B.P. 451,985, 27.6.35. Holl., 6.7.34).— Saturated aq. Na<sub>2</sub>CO<sub>3</sub> and borax are together passed at 2 cu. m./hr. through a hose containing alternate Pt-wire anodes and V2A steel cathodes 5—6 mm. apart. Current at 5—6 volts is supplied to the electrodes and the treated solution is cooled to separate NaBO<sub>3</sub>.

A. R. P.

Cathode for electrolytic cells. A. C. JEPHSON and E. B. CUSTER, ASSTS. to NAT. RADIATOR CORP. (U.S.P. 2,028,285, 21.1.36. Appl., 25.7.34).—A cylindrical cathode rotating about a vertical axis for the manufacture of metal sheets is described. B. M. V.

Separators for electric accumulators. COMP. GÉN. D'ELECTRICITÉ (B.P. 451,628, 11.6.35. Ger., 9.6.34).—The separators consist of slabs of porous rubber coated on both sides with a partly vulcanised mixture of latex and glass wool and covered externally with layers of glass wool. A. R. P.

Separators for electric storage batteries. CHANCE BROS. & Co., LTD., and J. W. JOLLY (B.P. 454,041, 29.5.35).—Glass silk is moulded into a frame of rubber or other acid-resisting material. B. M. V.

Electrodes for electric-discharge devices [containing metallic vapour, e.g., mercury]. BRIT. THOMSON-HOUSTON CO., LTD. (B.P. 455,256, 8.11.35. Ger., 8.11.34).—Graphite electrodes, electrolytically purified by use as anodes in a strong acid, e.g., HCl, and subsequent heating at, e.g., 1500°, are used. J. S. G. T.

Device comprising an electric mercury-vapour discharge tube and a red-fluorescent layer. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 454,348, 24.1.36. Ger., 20.3.35).—The Hg pressure is > 10 (> 20) atm., and the red irradiated layer contains rhodamine.

B. M. V.

**Thermionic electrodes.** GEN. ELECTRIC CO., LTD. From PATENT-TREUHAND-GES. F. ELEKTR. GLÜH-LAMPEN M.B.H. (B.P. 451,989, 22.7.35).—A close-wound, W-wire helix, packed with a paste of W powder and an alkaline-earth oxide moistened with EtOH, is slowly heated to  $\lt$  1700° in a neutral or reducing atm.

A. R. P. Measuring moisture content of sands etc.— See I. Treating the charges of engines.—See II. High-mol. alcohols etc. Halogenated  $Ph_2O.$ — See III. Ag from  $M_2S_2O_3$  solutions.—See VII. Porcelain resistant to X-rays.—See VIII. Ag electrodeposition. Al-plate.—See X. Producing articles of continuous length etc. from latex.— See XIV. Electro-filtration of air.—See XXIII.

## XII.—FATS; OILS; WAXES.

Twitchell reagents. XVII. Properties of sulphocompounds isolated from commercial wetting agents. K. NISHIZAWA and R. KINOSHITA (J. Soc. Chem. Ind., Japan, 1936, 39, 293-294B; cf. B., 1936, 1105).—The sulpho-compounds stable to strong acid at high temp. from four commercial wetting agents, Nekal BX, Igepon T, Gardinol R, and Intrasol, have been isolated and their fat-splitting and emulsifying properties in 1% solution at 95° with the addition of varying amounts of H<sub>2</sub>SO<sub>4</sub> compared with those of the stable sulpho-fatty compound (I) obtained by dropping 50% fuming  $H_2SO_4$  on to the product of castor oil and  $Ac_2O$  in  $Et_2O$ . Nekal BX hydrolysed olive oil well, (I) moderately, and the other three reagents much less, the hydrolysis rising with [H2SO4]. The emulsifying power of the Nekal BX alone was large, and increased with [H2SO4], that of the other products and (I) being low. The results support the view that under the conditions employed sulphuric esters may split and the  $H_2SO_4$  react with the oil at the high temp. to give sulphonation products, which may act catalytically in H<sub>2</sub>O as Twitchell reagents to hydrolyse the remaining oil. **F**. **R**.

**Rapid method of saponification.** W. R. STEET (Analyst, 1936, 61, 687).—Saponification is carried out at  $\gg 134^{\circ}$  by substituting OH·[CH<sub>2</sub>]<sub>2</sub>·OEt for EtOH as a solvent for KOH. The saponification of ordinary fatty material is almost instantaneous. E. C. S.

Bleaching qualities of bleaching earths. E. ERD-HEIM (Petroleum, 1936, 32, No. 40, Motorenbetr., 9, No. 10, 9–12).—The relative bleaching effects of American (bentonite), Polish (Siliton), and Rumanian (Vegetalin) earths on colza and linseed oils have been compared with the effect obtained with German earths (Terrana, Frankonit, Silitonit), using the Lunge–Roth photometer for determining colours. Vegetalin was the most and bentonite the least satisfactory. C. C.

Wool fat. I. Separation of wool fatty acids. II. New carboxy-acids of lano-fatty acid series : lanomyristic, lanopalmitic, lanostearic, and lanoarachic acid. T. KUWATA and Y. ISHII (J. Soc. Chem. Ind., Japan, 1936, 39, 317B, 318-319B).-I. On shaking, the saponification (EtOH-KOH) product of wool fat with petroleum-aq. EtOH, K lanocerotate, was pptd. The mixed acids from the EtOH-sol. salts separated from warm MeOH as cryst. solids. The Mg salts of these on treatment with boiling EtOH gave Mg lanopalmitate as a residue. Separation was then effected by repeated fractionation of esters and use of the different solubilities of the acids in MeOH and the Pb salts in EtOH. Besides a large quantity of fluid, saturated acids, five solid acids were obtained : lanomyristic, C14H28O2, m.p. 58.5-59.5° (amide, m.p. 95.5-97.5°), lanopalmitic, C16H32O2, m.p. 44.5-46° (amide, m.p. 81.5-82.5°), lanostearic,  $\rm C_{18}H_{36}O_2,\ m.p.\ 54\cdot5-56^\circ$  (amipe, m.p. 89–90°), lanoarachic,  $\rm C_{20}H_{40}O_2,\ m.p.\ 56\cdot5-58\cdot5^\circ,\ and\ an\ acid,\ m.p.\ 67\cdot5-68\cdot5^\circ$  (amide, m.p. 102·5–103°), probably  $\rm C_{21}H_{42}O_2.$ 

II. The mol. formulæ of these acids were confirmed by sap. vals., elementary analysis, combustion of the acid Mg salts, and N analysis of the amides. Mixed m.p. with the corresponding fatty acids and amides gave depressions, and it is concluded that in wool fat there is no acid normally found in fats. F. R.

Technical applications of wool fat. W. OBST (Allgem. Oel- u. Fett-Ztg., 1936, 33, 468-469).—A review. E. L.

Analysis of edible fats. J. GROSSFELD (Chem.-Ztg., 1936, 60, 913—917).—Modern analytical methods for the evaluation of individual fatty acids in mixed food fats are discussed. E. L.

Mechanical technology of soaps. II. Storing, weighing, and mixing of soap chips. K. Rössner (Fette u. Seifen, 1936, 43, 193–194; cf. B., 1936, 1105).— Customary plant is described. E. L.

Significance of chemical research for the manufacture of soaps and [modern] detergents. H. P. KAUFMANN (Fette u. Seifen, 1936, 43, 178–183).— A lecture. E. L.

Refining of tall oil. R. H. MCKEE and H. L. BLENGSLI (Paper Trade J., 1936, 103, 227-228).— Objectionable coloured and malodorous constituents resulting from oxidation of the rosin present in tall oil are removed by pptn. with excess of gasoline. Further decolorisation is accomplished by means of fuller's earth, activated C, or furfuraldehyde. The purified product is suitable for soap manufacture. H. A. H.

Stand-oil formation. H. KURZ (Fette u. Seifen, 1936, 43, 184–185; cf. B., 1936, 749).—A reply to Scheiber (B., 1936, 892): Scheiber's theory cannot be reconciled with the fact that the n of linseed oil does not fall during stand-oil formation as is the case with tung oil. E. L.

Catalytic polymerisation of fatty oils. H. I. WATERMAN and C. VAN VLODROP (J.S.C.I., 1936, 333– 334).—The prep. of linseed stand oils at 290° is accelerated by the catalytic influence of HCl and of SO<sub>2</sub>. With HCl hydrolysis occurs and dark oils of high acid val., and containing Cl, are obtained. With SO<sub>2</sub> there is no hydrolysis and light oils of low acid val. and low S content (< 0.1%) are obtained. F. C. B. M.

Iodine values of hydrogenated castor oil. Y. TOYAMA and T. ISHIKAWA (J. Soc. Chem. Ind., Japan, 1936, 39, 300–302b).—The m.p., I vals., and Ac-sap. vals. of the products obtained at definite intervals by hydrogenation of castor oil, using a Ni catalyst at various temp., are given. At more elevated temp. the Ac-sap. val. fell due to reduction of the OH of ricinoleic acid). Figures for the I vals. by both the Wijs and the  $C_5H_5N$  sulphate dibromide method (the latter giving the higher vals.) varied with the period of reaction, due to the presence in the hydrogenation product of the glyceride of  $\lambda$ -hydroxystearic acid, which absorbs halogen by substitution, the absorption increasing with rise in temp. and period of reaction. F. R. Determination of rape or mustard-seed oils, and detection of oils used to adulterate them. S. NEOGI (Analyst, 1936, 61, 597-601).—The oil is saponified by alcoholic KOH, and the solution treated with alcoholic Pb(OAc)<sub>2</sub>. The pptd. Pb salts are dissolved in 50% AcOH in EtOH, and the I val. of the contained erucic acid (I) is determined. Mustard-seed oil has an I val. [in terms of (I)] of  $42 \cdot 8 - 49 \cdot 7$ ; other oils have vals. about 2. Hence adulteration may be approx. determined. J. S. A.

Refractometric determination of oil in coconut and sesamé oil cake. D. C. INGRAHAM and T. H. SIMPSON (Oil & Soap, 1936, 13, 222-224).-The nD method (cf. B., 1928, 902) is excellent for the rapid determination of oil in coconut (I) and sesame cake or meal (II) (accuracy  $\pm 0.14$  and  $\pm 0.12$ , respectively). The sample (2 g.) should be dried for 20 min. at 100° in the case of (I) and preheated to 49° in the case of (II) before grinding with sand and 4 c.c. of Halowax. The curves of d and n plotted against % oil in solution are practically straight lines for concns. from 1% to 23.5% and 20% for (I) and (II), respectively (i.e., covering the range from cake to fresh seed in each case). 1-C10H7Br as solvent gives slightly more accurate results, but is more difficult to measure out on account of its high d. The results tend to be slightly low with very oily samples (e.g., the fresh seed). E. L.

Composition of plum-kernel and beechnut oils. E. DELVAUX (Fette u. Seifen, 1936, 43, 183-184).— The light petroleum-extracted oils of seeds of *Prunus* domestica from Münster (yield 42% of dry seed) and of beechnuts from Sauerland (yield  $42\cdot2\%$ ) had respectively: I val. (Kaufmann) 100·3, 111·9; SCN val.  $81\cdot2$ ,  $79\cdot2$ ; hexabromide no. 0,  $3\cdot37$ ; unsaponifiable matter  $0\cdot4$ ,  $0\cdot27\%$ ; they contained the following fatty acids (% on oil): saturated acids (Bertram)  $5\cdot9$ ,  $11\cdot5$ ; linolenic 0,  $2\cdot8$ ; linoleic  $21\cdot0$ ,  $33\cdot2$ ; oleic  $68\cdot9$ ,  $48\cdot4$ . The accuracy of the thiocyanometric method is endorsed. E. L.

Oil of Pleurogrammus monopterygius, Pallas. Y. TOYAMA and T. ISHIKAWA (J. Soc. Chem. Ind., Japan, 1936, 39, 302–304B).—The characteristics of seven specimens of this oil (hokke oil; cf. B., 1934, 894) have been determined. The Me esters of the acids from a specimen were fractionated and various fractions saponified and examined. Palmitic, oleic, and cetoleic acids have been identified, whilst myristic, stearic, and the acids  $C_{16}H_{30}O_2$  and  $C_{20}H_{38}O_2$  were indicated.

F. R. [Marine] animal drying oils. R. BÜRSTENBINDER (Fette u. Seifen, 1936, 43, 185—186; cf. Toch, B., 1936, 1106).—The properties of "Haco" drying oils, *i.e.*, processed train oils, which are claimed to be particularly suitable for under-H<sub>2</sub>O paints, are described. E. L.

Animal and vegetable oils : viscosity-temperature characteristics. A. R. RESCORLA and F. L. CARNAHAN (Ind. Eng. Chem., 1936, 28, 1212—1213).— Viscosities in centistokes at  $37 \cdot 8^{\circ}$  and  $98 \cdot 8^{\circ}$  have been measured for a large no. of animal and vegetable oils. The acid val., sap. val., d, and n of the oils are listed and the  $\eta$  and d indices calc. E. G. H. Minor constituents of fats and oils. H. A. BOEKENOOGEN (Allgem. Oel- u. Fett-Ztg., 1936, 33, 461-468).—The chemistry of the "unsaponifiable" constituents, vitamins, lipochromes, "mucilaginous impurities," etc. is discussed E. L.

Vegetable oils in fuel oils. Cl-compounds and lubricants.—See II. Wetting agents.—See III. Determining butter fat in foods.—See XIX.

See also A., Nov., 1361, Oxidation of drying oils. 1403, Composition of eel oil. 1428–31, Vitamins. 1435, [Oil of] chili seed.

## PATENTS.

Chemical composition [emulsifying agent]. B. R. HARRIS (U.S.P. 2,022,766, 3.12.35. Appl., 10.11.33).— Glycerol is polymerised by heating (at 200°) with a strong alkali, e.g., NaOH, NaOAc, Na<sub>2</sub>CO<sub>3</sub> (1%), for  $\leq 4$  hr. in a non-oxidising atm. The H<sub>2</sub>O formed distils off, and the resulting polyglycerols are esterified by heating to 250—265° for  $\leq 4$  hr. with a high-mol. fatty acid and/or its triglyceride, and an alkaline catalyst to yield mixed polyglycerides having free OH. The polymerisation and esterification may be conducted simultaneously. The products emulsify readily in H<sub>2</sub>O and may be further purified by washing with brine.

E. J. B.

Manufacture of polishing or like agents for treatment of surfaces. W. J. TENNANT. From HENKEL & Co., G.M.B.H. (B.P. 453,911, 14.3.35).—The manufacture by known means of esters of hydroaromatic compounds having  $\leq 2$  OH and  $\leq C_{10}$  is claimed. Examples are : hardened whale-oil fatty acid ester of a hydrogenated o-cresol-CH<sub>2</sub>O condensation product, hardened rape oil acid esters of 4 : 4'-dihydroxyperhydrodi-phenyl- and -m-tolyl-methane, and a montan acid ester of a hydrogenated cresol-COMe<sub>2</sub> condensation product. H. A. P.

Brushless shaving cream and cosmetic cream. PROCTER & GAMBLE Co. (B.P. 454,658, 5.2.35. U.S., 8.2.34).—A sol. salt of a sulphonated or sulphated aliphatic  $CO_2H$ -free compound ( $\langle C_{10} \rangle$  (e.g., Na lauryl sulphate, or compounds of the type  $R \cdot O \cdot [CH_2]_2 \cdot SO_3M$ or  $R \cdot CO \cdot NMe \cdot [CH_2]_2 \cdot SO_3M$ , where R is a fatty acid radical and M a base metal, NH<sub>4</sub>, etc., is used as emulsifier for a stearic acid–H<sub>2</sub>O cream, which will not form insol. Ca soaps with hard H<sub>2</sub>O. E. L.

Soap cream. F. B. DEHN. From PROCTER & GAMBLE Co. (B.P. 454,660, 4.3.35).—Fats (or fatty acids) hydrogenated so as to contain  $\ll 45\%$  of saturated  $C_{18}$  acids are used as the source of > 50% of the total soap in the cream, in order that the product shall retain a creamy consistency (due to the presence of fine soap crystals) at temp. up to about 38°. E. L.

Washing media. F. WERTH (B.P. 454,693, 30.9.35. Ger., 28.9.34).—Washing tablets of a size to be used once only, which crumble readily in the hand before washing, consist of soap (with or without sawdust) and a wood-pulp prep. which contains less slimy matter than is present in the pulp as originally manufactured. E. L.

Increasing the vitamin content of vitaminbearing oils. H. P. LOOMIS, ASST. to SILMO CHEM. CO.,

INC. (U.S.P. 2,026,395, 31.12.35. Appl., 29.3.33).— The oils are partly saponified at relatively low temp. by a deficiency of alkali, and the soap formed is extracted by a suitable solvent, e.g., a mixture of  $H_2O$  and EtOH, MeOH, carbitol,  $(CH_2 OH)_2$ , or diacetone, from the remaining unsaponified fat, which retains the unsaponifiable matter and vitamins released from the saponified fraction. E. L.

Denaturant for fats and fatty oils. J. E. MUTH, Assr. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 2,025,954, 31.12.35. Appl., 18.7.33).—An intensely bitter taste is imparted to fatty oils by addition (preferably after dilution with mineral oils or Edeleanu extracts) of crude CH<sub>2</sub>Ph ethers of sugars, mannitol, glycerol, or triethylene glycol etc. E. L.

Preservation of oils, fats, and waxes, and substances containing them. SYLVANIA INDUSTRIAL CORP. (B.P. 453,438, 8.3.35. U.S., 14.3.34).—Rancidity is prevented by enclosing the oil etc. in a transparent container, preferably of non-fibrous cellulosic material ("glassine"), coloured with a (yellow) org. dye in sufficient concn. to transmit  $\geq 25\%$  of light of  $\lambda$  2900— 4700 and  $\leq 50\%$  of light of  $\lambda$  4900—6900 A. H. A. P.

Lubricants.—See II. Polyglycerol esters. Wetting agent. Org. S compounds. Undecoic acid. Oxidised fatty acids. Compounds containing acid salt-forming groups.—See III. Margarine.—See XIX.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

White lead-zinc oxide mixtures in oil paints. J. SACHER (Farben-Ztg., 1936, 41, 1084).—To comply with German regulations for conserving Pb pigments, it is recommended that, apart from the obvious admixture with inerts (BaSO<sub>4</sub>), consideration should be given to combinations of the above pigments. Such paints based on a 70:30 mixture have satisfactory brushing and storage characteristics and durability, and show improvement over "straight" white Pb in colour and hardening rate. S. S. W.

Constitution and behaviour of paints for boiler interiors. H. H. MÜLLER-NEUGLÜCK and G. AMMER (Glückauf, 1936, 72, 577—587; cf. B., 1935, 1151).— Paints for preventing boiler corrosion are discussed. Portions of the inside of a steel boiler were treated with six different types of bituminous (tar) paint, and observations made of their behaviour, during a year's service, when subjected to the action of H<sub>2</sub>O at 231°. In spite of the severe conditions, photographs show that certain of the painted surfaces suffered only slight damage.

R. B. C. Driers and their application. F. FRITZ (Chem.-Ztg., 1936, 60, 921-924).—A review.

Bactericidal power of dried painted surfaces. I. E. VALENTINE (Proc. Soc. Exp. Biol. Med., 1936, 34, 166-170).—Paints containing 4% of halogenated oil and some commercial paints exhibited considerable bactericidal power towards *B. typhosus* even after airdrying and baking at 100-125° for 6 hr., but the effect on *Staph. aureus* was only moderate. W. McC.

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Phosphotungstic [colour] lakes. K. M. RICHARDS (J. Soc. Dyers & Col., 1936, 52, 378–380).—A review of the historical development of Fanal lakes. S. C.

Evaluation of rust-preventive pigments by swelling measurements [in water]. H. WAGNER and E. PABST (Farben-Ztg., 1935, 40, 789-790, 811-813).-The changes involved in the swelling of a paint film are discussed and correlated with rust-preventive properties. Tests on a range of Pb chromates show that the swelling val. (S), *i.e.*, the ratio of the swelling of the paint film to that of the oil contained therein, is of more significance than a simple % H<sub>2</sub>O-absorption of the film; S for neutral and basic Pb chromates, free from sol. and soap-forming components, is 0.2-0.4, corresponding to an effective swelling of 15-25% in 100 days; introduction of soap-forming groups can lower S to 0.15. The relation between adhesion, "under-rusting," and S is examined. S. S. W.

Manufacture of offset inks. A. GRUNDER (Amer. Ink Maker, 1936, 14, No. 3, 25, 27).—The consistency of the inks should not be too susceptible to changes in the  $H_2O$  content. The emulsion should be of the  $H_2O$ -in-oil and not of the oil-in- $H_2O$  type. D. R. D.

Inks for use on metal foil. G. WALLACE (Amer. Ink Maker, 1936, 14, No. 3, 19, 21, 43).—The factors to be considered in formulating and applying such inks are discussed. D. R. D.

Determination of the relative printing strengths of inks by calculation. S. L. KARPELES (Amer. Ink Maker, 1936, 14, No. 3, 15—18).—The printing strengths are deduced from the relative tinting strengths of the inks when mixed with a standard white ink. D. R. D.

Calculation of ink [and paint] grinding losses. K. H. BETTNER (Amer. Ink Maker, 1936, 14, No. 3, 23).— Grinding losses should be calc. on a vol. basis, since the vol. lost in any given mill is approx. a const. for the mill, irrespective of the amount of material ground and (in absence of a volatile solvent or diluent) of the composition of the ink or paint, whereas the wt. lost  $\infty$  its d. D. R. D.

Scientific development of coating materials in the principal industrial countries. O. JORDAN (Angew. Chem., 1936, 49, 815-821).—A lecture.

Use of malic acid to prevent the livering of finishes containing zinc oxide. C. BOGIN and W. L. WAMPNER (Amer. Paint J., 1936, 21, No. 1, 7–9, 58–64; No. 2, 16–20, 54–60).—Malic acid (I) (0.5%)is more effective than citric, tartaric, and boric acid in preventing the livering of ZnO lacquers, particularly those containing lactate solvents or alkyl resins of high acid val. It does not affect the H<sub>2</sub>O-resistance of the lacquers. (I) should not be used with EtOH as it is readily esterified. D. R. D.

Viscosity and plasticity of disperse systems. IX. Plastic-viscous properties of printing colours. M. P. VOLAROVITSCH [with V. S. BORINEVITSCH] (Kolloid-Z., 1936, 77, 93—99; cf. A., 1935, 1318).—The plastic consts. of dispersions of lampblack in varnishes have been determined as a function of concn. up to 25% of C. The influence of induline has been investigated.

E.S.H.

**Resinous derivatives of vinyl alcohol.** G. O. CURME, JUN., and S. D. DOUGLAS (Ind. Eng. Chem., 1936, 28, 1123—1129).—The history, properties, and uses of vinyl resins are described, and the advantages of CH<sub>2</sub>:CH·OAc-CH<sub>2</sub>:CHCl co-polymerides and the variation of their physical properties with mol. wt. are indicated. L. A. O'N.

Constitution of phenol-aldehyde resins. V. Condensation of p-benzylphenol with formaldehyde. A. VANSCHEIDT, A. ITENBERG, and V. BALANDINA (Plast. Massui, 1934, No. 6, 11—17; cf. B., 1936, 1108). —1 mol. of p-CH<sub>2</sub>Ph·C<sub>6</sub>H<sub>4</sub>·OH (I) (prep. described) with 2·5 mols. of CH<sub>2</sub>O in alkaline solution give the oo'-Me<sub>2</sub> derivative (II), m.p. 88—9°. Solutions of (II) in H<sub>2</sub>O or EtOH give a blue colour with FeCl<sub>3</sub>. With PhSO<sub>2</sub>Cl, (II) forms a benzenesulphonate, m.p. 82°, which is oxidised by K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> to the dialdehyde, m.p. 136—137°. In presence of HCl (II) condenses with (I) to form 76% of 4-benzyl-2: 6-bis-(3'-hydroxy-2'-benzylbenzyl)phenol (III), m.p. 178°. (III) is also formed when 7 mols. of (I) and 6 mols. of CH<sub>2</sub>O are heated in presence of HCl. CH. ABS. (p)

Fundamental aspects of thixotropy. J. PRYCE-JONES (J. Oil Col. Chem. Assoc., 1936, 19, 295-337).-Plasticity and thixotropy are different but related properties of disperse systems. The  $\eta$  of a plastic system is a function of the rate of shear and is independent of its past history. As the n of a thixotropic system, as well as being a function of rate of shear, is also dependent on its past history it is essential that  $\eta$  comparisons after different setting times be made at the same rate of shear. An improved electromagnetic thixotrometer is described which can be adapted for observations on "elastic recoil" in colloidal systems. It has been shown that thixotropic gelation is of two kinds, "thixotropy" and "false body." The former appears to be a true gel/sol/gel transformation induced by shear and subsequent rest, whilst the latter appears to be a partial dispersion of a gel under the influence of shear and the re-formation of the gel on removal of shear. Systems in all stages intermediate between these two extreme F. C. B. M. cases can be prepared.

Resins from light oil.—See II. Marine animal drying oils.—See XII. Rubber pigments.—See XIV. Skin troubles from solvents.—See XXIII.

See also A., Nov., 1361, Shellac. 1385, Turpentine from *Pinus sylvestris*. Resin components of ammoniacum.

#### PATENTS.

Grease paint. F. FACTOR, Assr. to M. FACTOR & Co. (U.S.P. 2,025,943, 31.12.35. Appl., 4.12.34).—Mixtures of non-volatile oils (olive, mineral, lanoline) ( $\lt$  35),  $\gamma$ -reducing agents [sulphonated alcohols, stearic acid, N(C<sub>2</sub>H<sub>4</sub>·OH)<sub>3</sub>] (10), pulverulent "texture ingredients" (talc, kaolin, ZnO, TiO<sub>2</sub>) ( $\lt$  20), and suspended pigments are claimed as non-drying, cosmetic grease paints. S. S. W.

Production of titanium pigments. BRIT. TITAN PRODUCTS CO., LTD. From TITAN CO., INC. (B.P. 451,890, 31.7.35).—Aq. Ti(SO<sub>4</sub>)<sub>2</sub> (I) from the digestion of ilmenite with  $H_2SO_4$  is hydrolysed in presence of a gel obtained by treatment of pure aq. (I) with NaOH. [Stat. ref.]

A. R. P.

**Treatment of pigments.** G. F. A. STUTZ and H. A. DEFEW, Assrs. to NEW JERSEY ZINC Co. (U.S.P. 2,024,611, 17.12.35. Appl., 23.1.31).—Inorg. pigments obtained by wet pptn. are heated in a closed chamber at  $\leq 1000$  lb./sq. in. and at the appropriate temp. of saturated steam ; most of the liquid associated with the pigment is volatilised and escapes while the pigment is under pressure, whilst the remainder is removed by a sudden drop in pressure which also serves to disintegrate the mass of dry pigment. S. S. W.

Marking ink. F. H. NEWINGTON, W. KILLNER, and C. S. WRIGHT (B.P. 453,961, 27.3.35).—Non-injurious marking ink which necessitates no treatment of the marked material is made by partly converting freshlyprepared  $Ag_2CO_3$  into the Ag salt of a reducing org. acid (e.g., tartaric) and dissolving the mixed salts in a volatile basic medium such as aq. NH<sub>3</sub> with addition of a gum and a sol. dye. A. H. C.

Granule-coated webs for preventing offset in printing. W. J. TENNANT. From CARBORUNDUM Co. (B.P. 454,575, 2.4.35).—For supporting a printed sheet during drying, a web is coated with granules 0.05-0.20 mm. in diam. at a wt. per unit area intermediate between those of close- and open-coat abrasive sheets.

B. M. V.

Bronzing lacquer. D. J. BURKE, ASST. to COMMERCIAL SOLVENTS CORP. (U.S.P. 2,026,493, 31.12.35. Appl., 18.3.35).—The use of 0.2—2.5% of aliphatic esters or alkali salts of malic acid as anti-gelling agents in nitrocellulose bronzing lacquers is claimed. S. S. W.

Manufacture of coloured lacquers. J. R. GEIGY A.-G. (B.P. 454,029, 5.4.35. U.S., 9.4.34).—Dyestuffs obtained by coupling tetrazotised 2:2'-dimethylbenzidine with phenols, mono- or di-amines containing no  $\cdot CO_2H$  or  $\cdot SO_3H$  are sol. in lacquer solvents, giving coloured lacquers of fast shades. The products obtained by coupling with *p*-cresol, 1-phenyl-3-methyl-5-pyrazolone (yellow),  $1:2:4-C_6H_3Me(NH_2)_2$  (red-brown),  $\alpha$ - $C_{10}H_7\cdot NH_2$ , 3-hydroxy-2'-methyldiphenylamine (orangebrown, and  $2:3-OH\cdot C_{10}H_6\cdot CO_2Et$  (blue-red) are described. A. H. C.

Cellulose derivative composition. G. DE W. GRAVES, ASST. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 2,025,048, 24.12.35. Appl., 7.7.34).—Compounds having the formula  $OH'[C_2H_4]OR$  (R = an alkyl group  $\lt C_8$ , e.g., lauryl) are esterified with org. acids. Hydrogenated coconut oil may be thus treated. The products are used as plasticisers for cellulose derivatives. S. M.

Coating composition [for paper] and its preparation. W. E. B. BAKER, Assr. to STEIN, HALL & Co., INC. (U.S.P. 2,024,123, 10.12.35. Appl., 2.7.32).—Starch is treated with approx. 4 wt.-% of a conc., weak, fatty, monobasic, liquid org. acid (AcOH) in presence of a small amount of strong inorg. acid, the uncombined acid in the mildly dextrinised and esterified sol. starch so formed is neutralised with aq. NH<sub>3</sub>, and 10% (on the starch) of a dry sol. Na silicate is added. This mixture is dissolved in H<sub>2</sub>O, mixed with china clay, BaSO<sub>4</sub>, etc., and applied to paper, reactions liberating H<sub>2</sub>SiO<sub>3</sub> occurring in the mineral filler before and after application to the paper. The composition is very fluid and provides a H<sub>2</sub>O-insol., absorbent coating. S. S. W. Production of siccatives. W. KRUMBHAAR, Assr. to BECK, KOLLER & CO., INC. (U.S.P. 2,025,870, 31.12.35. Appl., 24.10.34).—Hydroxides of Co, Mn, Pb, Ca, or Zn are pptd. from aq. solutions of appropriate salts at  $\gg$ room temp., air being eliminated during pptn. and from the H<sub>2</sub>O used, and the H<sub>2</sub>O-wet paste is kneaded in absence of air with an oily dispersion medium (linseed- or tung oil-stand oil) and then heated at  $< 200^{\circ}$  with resinic (abietic) or naphthenic acid, or soap-forming fatty acids or glycerides. S. S. W.

Manufacture of coloured masses [for lacquers etc.]. Soc. CHEM. IND. IN BASLE (B.P. 455,320, 12.8.35. Switz., 13.8.34).—Nitrocellulose lacquers, varnishes, etc. are coloured by incorporating a complex compound of a metal (at. wt. 48—66) and an arylmethane dye which contains OH ortho to a CO<sub>2</sub>H, e.g., the Ni compound of Naphthochrome Azurine B. S. M.

Treatment of natural gums. BECK, KOLLER & Co., INC., Assees. of W. KRUMBHAAR (B.P. 444,853, 12.4.35. U.S., 5.5.34).—Gum copal is masticated, e.g., between heated rollers, at 80—200° for 20—120 min., to increase its solubility in the usual solvents and its reactivity at low temp. to neutralising and modifying agents. It may be afterwards dissolved and neutralised, or esterified with glycerol; or solvents and/or neutralising agents may be applied during mastication. J. H. L.

Aluminium chloride synthesis of resins. S. C. FULTON and J. KUNC, ASSTS. to STANDARD OIL DEVELOP-MENT CO. (U.S.P. 2,025,738, 31.12.35. Appl., 7.4.32).— A cracked petroleum tar distillate is polymerised with AlCl<sub>3</sub> at 120—180°, cooled, settled, treated with 95%  $H_2SO_4$ , neutralised, and distilled to a resinous residue. S. M.

Prepared resin. C. A. THOMAS, ASST. to MONSANTO PETROLEUM CHEMICALS, INC. (U.S.P. 2,023,495, 10.12.35. Appl., 10.11.30).—A polyhydric alcohol is caused to react with a preformed unsaturated hydrocarbon resin obtained by the polymerisation in presence of catalyst (AlCl<sub>3</sub>) of a cracked petroleum distillate, in such a way that the polymerisation involves the reaction of a diolefine with an olefine and/or an alkylbenzene. Alternatively, these ingredients may be employed independently, e.g., as a mixture of isoprene, amylene, and xylene. S. S. W.

Manufacture of films, threads, sheets, or the like and of [polyvinyl] resins therefor. H. E. POTTS. From SHAWINIGAN CHEMICALS, LTD. (B.P. 454,691, 27.12.34).—A polyvinyl ester (acetate) is hydrolysed and condensed with  $CH_2O$  etc. in presence of an acetalising catalyst ( $H_2SO_4$ ) and a lower aliphatic acid, *e.g.*, glacial AcOH, as solvent. The reaction product is neutralised and the resin pptd. with  $H_2O$ .  $H_2O$  may be added to the reactants in controlled amount. (Cf. B.P. 436,072; B., 1935, 1153.) S. M.

[Increasing colour-stability of] synthetic polymerisates and artificial materials therefrom. W. W. GROVES. From DEUTS. CELLULOID-FABR. (B.P. 454,232, 25.3.35).—Discoloration of synthetic and mixed poly merides of the vinyl chloride group on exposure to heat and light is inhibited by addition of 0.36-4% of substances capable of forming hybrid ions (e.g., aliphatic  $\alpha$ -NH<sub>2</sub>-acids, gelatin, glue, o- or p-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·CO<sub>2</sub>H, sulphanilic acid, etc.). A. H. C.

Preparation of [phenol-formaldehyde] resinous condensation products. E. ELBEL and F. SEEBACH, Assrs. to BAKELITE CORP. (U.S.P. 2,024,212, 17.12.35. Appl., 13.5.33. Ger., 13.5.32).—A B-stage phenol-CH<sub>2</sub>O resin which is insol. in COMe<sub>2</sub> is dissolved in, *e.g.*, *cyclo*hexanol and pptd. by adding a non-solvent, *e.g.*, EtOH, ligroin. The product is sol. in COMe<sub>2</sub> and other volatile liquids. S. M.

Polymerisation of methacrylonitrile. TRIPLEX SAFETY GLASS CO., LTD., and J. WILSON (B.P. [A] 453,521 and [B] 453,578, 13.3.35).-(A) Colourless, glass-clear products, which may be coloured by addition of a dye to the polymerisation mixture, are obtained by interpolymerisation by standard methods of CH<sub>2</sub>:CMe·CN (I) with polymerisable substances other than butadiene and its analogues, e.g., CH2:CH.OAc, CH2:CH.CO2Me, CH2: CMe CO2Me. [Stat. ref.] (B) Polymerisation of (I) is carried out by heating it without a catalyst or plasticiser in a solvent for (I) but not for its polymeride (light petroleum, b.p. 80-100°); the polymeride separates as a white powder which is removed from the surface as formed. It may be milled with a plasticiser and/or solvent before pressing into blocks or sheets. B.P. 422,697 and 437,271 (B., 1935, 263; 1936, 31) are disclaimed.

H. A. P.

Preparation of hydroabietoyl chloride. C. O. HENKE and M. A. PRAHL, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,023,473, 10.12.35. Appl., 21.12.32).—Hydrogenated abietic acid or rosin is treated with PCl<sub>5</sub>, SOCl<sub>2</sub>, etc. The di- and tetra-hydro-derivatives are claimed. S. M.

Production of [resinous] composition [from rosin and maleic anhydride]. I. W. HUMPHREY, Assr. to HERCULES POWDER CO. (U.S.P. 2,025,947, 31.12.35. Appl., 3.9.31. Renewed 16.2.35).—Abietic acid or an alkyl, glycol, or glycerol abietate is heated with maleic anhydride. S. M.

Manufacture of [modified alkyd] artificial resins. HERBIG-HAARHAUS A.-G. (B.P. 455,066, 26.11.35. Ger., 30.11.34).—Partial esters, containing free OH groups, of polyhydric alcohols (glycerol) with polybasic and fatty acids [e.g.,  $o-C_6H_4(CO_2)O$ , stearic] are etherified by heating their mixtures at about 290°. Pigments and fillers may be added. S. M.

Production of paracoumarone resins. K. H. ENGEL, Assr. to BARRETT Co. (U.S.P. 2,024,568, 17.12.35. Appl., 20.7.32).—A tar oil (acid content  $\geq 0.1\%$ ) containing styrene-coumarone-indene constituents is treated at  $\geq 25^{\circ}$  with a solution of H<sub>3</sub>BO<sub>3</sub> in H<sub>2</sub>SO<sub>4</sub> (82–100%). S. M.

[Adhesive] condensation products [hydroxyesters]. BRIT. CELANESE, LTD., and W. H. Moss (B.P. 453,833, 13.3.35).—Condensation products sol. in  $H_2O$ and compatible with both cellulose esters or ethers and gelatin are obtained by condensing polyhydric alcohols (glycol, glycerol, diethylene glycol, mannitol, etc.) with hydroxy-polycarboxylic acids (citric, tartaric) or their acyl derivatives (acetylcitric acid) and aldehydes or, alternatively, condensation products of the OH-acids or polyhydric alcohols with aldehydes [methylenecitric acid (I), glycol acetals]. The condensation is carried out by heating the constituents at about 180° for a suitable time without the addition of condensing agents (cf. B.P. 449,185; B., 1936, 872). In the examples, (I) or citric acid is heated with glycerol etc. The products form adhesives for cellulose acetate foils when dissolved in gelatin solutions. The use of the products in the manufacture of safety glass is claimed. S. C.

Manufacture of condensation products [resins]. W. J. TENNANT. From HENKEL & Co., G.M.B.H. (B.P. 455,008, 10.4.35).-Adhesives, artificial resins, etc. of good fastness to light and of increased strength and durability are obtained by condensing pure or crude diazines or 1:3:5-triazines substituted by  $\lt 2$  NH. (or NHR) and possibly other groups, e.g., OH, with lower aliphatic aldehydes or potential aldehydes. Examples of the bases used are 1:4-diaminophthalazine, 4:6diamino-pyrimidine or -2-phenylpyrimidine, 2:4:6triaminopyrimidine (melamine) (I), 2:4- (II) or 4:6diaminoquinazoline, 3:6-diamino-2-methylquinoxaline, and 4:6-diamino-2-phenyltriazine. When (I) (126), formalin (III) (225), and EtOH (500 pts.) are heated to boiling and then evaporated at 40-50°, an EtOH-sol. resin separates, which hardens when fused. The mixture (30) obtained by heating NH<sub>4</sub>CNS at 270° and then washing with aq. NaOH is boiled with (III) (120) and 30% aq. NaOH (6 pts.) for 2 hr., giving a solution suitable for use as an adhesive or on evaporation a hardenable resin. (I) (126) with paraformaldehyde (IV) (60) and EtOH (500) or MeCHO (134) and H<sub>2</sub>O (200 pts.), when boiled and evaporated in vac., gives hardenable resins. (I) (126), urea (120), and (III) (700 pts.) give a jelly, which on drying sets to a firm, clear mass. (II) (16), (III) (200), and conc. HCl are boiled and evaporated, giving a solution for use as an adhesive. (I) (25), guanidine car-bonate (25), and (III) (120 pts.) or (II) (16), dicyanodiamide (17), and (III) (40 pts.) give clear resins. (1) (50) and (IV) (50) in (·CH<sub>2</sub>·OH)<sub>2</sub> (200 pts.) give a rubbery mass. Accelerating or retarding agents, e.g., alkalis, ZnCl<sub>o</sub>, or diluents may be added during the condensation. R. S. C.

[Plastic] fibrous composition. H. C. FISHER, Assr. to RICHARDSON CO. (U.S.P. 2,023,675, 10.12.35. Appl., 25.5.29).—Asphalt or other  $H_2O$ -insol. binder is worked up with fibrous material, *e.g.*, cotton linters, and the process continued in presence of large excess of  $H_2O$ . A plastic mass is ultimately obtained. S. M.

Manufacture of composite [thermoplastic-containing] materials. BRIT. CELANESE, LTD. (B.P.455,328, 31.10.35. U.S., 9.11.34).—Fabrics, felt, and other base materials are impregnated with glycol, glycerol, or derivatives thereof before being coated with nitrocellulose or other thermoplastic composition. S. M.

Manufacture of moulding compositions. C. STRESINO, ASST. to A. O. SMITH CORP. (U.S.P. 2,026,106, 31.12.35. Appl., 3.8.34).—Wood impregnated with sulphite liquor (60 pts. of wood to 40 pts. of dry sulphite-liquor residue) is ground, pasted with H<sub>2</sub>O, heated with 10% of PhCl for 3 hr. at 200°, and pulverised, giving a black moulding powder. S. S. W.

Plastic composition for dolls' heads. J. O. BARKER, Assr. to Sweets Labs., Inc. (U.S.P. 2,024,124, 10.12.35. Appl., 25.11.32).—Compositions of broken-down rubber

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(35), chemically stable resin (coumarone) (35), nonvolatile mineral oil of high  $\eta$  (10-40), and gum chicle (20-40), retaining plastic properties over long periods and being non-injurious to contiguous rubber sheet, are claimed. S. S. W.

Manufacture or treatment of albuminous artificial masses and articles produced therefrom. DEUTS. HYDRIERWERKE A.-G. (B.P. 454,611, 31.7.35. Ger., 31.7.34).—The elasticity and flexibility of articles made from glue, gelatin, casein, or albumin are improved by incorporating compounds of the type RXR' (R =  $alkyl > C_6$ ; R' = alkyl or cycloaliphatic residue carrying an acid salt-forming group;  $\dot{X} = 0$ , NH, S, SO<sub>2</sub>, SO<sub>2</sub>·NH, CO·NH, NH·SO<sub>2</sub>, NH·CO, CO·O, and O·CO) together with other softening agents. The following are specifically claimed : salts of oleic acid ester of OH·CH<sub>2</sub>·CH<sub>2</sub>·SO<sub>3</sub>H, monoglyceryl dodecyl ether sulphate, β-dodecylamino- and -hexadecylthio-ethanesulphonic acid,  $\alpha$ -cetyl hexyl ether of decane- $\kappa$ -sulphonic acid, and oleyl ethylene glycol ether thiosulphate, with inorg. and org. bases. S. C.

[Mould-inhibitors for] coating and plastic compositions. E. I. DU PONT DE NEMOURS & Co. (B.P. 453,657, 15.3.35. U.S., 17.3.34).—The addition of 0.01-2 (0.01-0.2)% (calc. on film solids) of arylmercuri-salts to paints, lacquers, and synthetic plastics is claimed. Examples of the salts used are Hg Ph chloride, acetate, and oleate. H. A. P.

[Surface-]decoration of articles and apparatus therefor. OXVAR, LTD. From OXFORD VARNISH CORP. (B.P. 455,684, 12.3.36).

Acrylic acid. Ethers etc. of paraffin wax. Acetotricarballylic esters. Halogenated  $Ph_2O$ .—See III. Insulating material. See XI. Plastic rubber derivative. Ebonite coating.—See XIV. Coating for skin protection.—See XX. Printing.—See XXI.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Stability of Abiarana gutta-percha latex. F. K. DANIEL, H. FREUNDLICH, and K. SÖLLNER (Trans. Faraday Soc., 1936, 32, 1570—1576).—This latex is more stable than *Hevea brasiliensis* latex to coagulation by electrolytes or the electric current. The protective, adsorbed substance, which is responsible for the stability of the gutta-percha latex, has been separated. This latex has a protective action on the *Hevea* latex. Sols of the former contain negatively-charged particles, the charge of which can be reversed by H', and ter- and quadrivalent ions. E. S. H.

Applications of [rubber] latex. J. G. Fol (India-Rubber J., 1936, 92, 575–589).—A review is given of commercial (and other) modifications of the latex of *Hevea brasiliensis* and of various methods of its application in manufacturing processes. D. F. T.

Viscosity properties of rubber solutions. I, II. W. PHILIPPOFF (Kautschuk, 1936, 12, 102–105, 124– 131; cf. B., 1936, 1057).—A review. Mathematical consideration is given to the best method for presentation of the experimental results. Various forms of apparatus are described and the relation between the  $\eta$  of rubber solutions and the nature of the solvent, the concn., and the temp., respectively, is summarised. D. F. T.

Pigments for the rubber industry. S. REINER (Farben-Chem., 1936, 11, 405-407).—A brief review.

Chemical structure of natural rubber and of varieties of artificial rubber. J. P. WIBAUT (India-Rubber J., 1936, 92, 609-610, 619-624).—A review of the literature concerning natural rubber, gutta-percha, and synthetic rubber, including polymerised  $\beta$ -chloro- $\alpha\gamma$ -butadiene. D. F. T.

Oil-resistance of rubber. IV. Mechanism of oilresistance of Duprene. S. KAMBARA (J. Soc. Chem. Ind., Japan, 1936, 39, 319—322 B; cf. B, 1936, 161).— Assuming  $(C_5H_8)_8$  and  $(C_4H_5Cl)_8$  for the mol. magnitude of rubber and Duprene, respectively, Debye's formula, applied to the characteristics of their respective solutions, indicates that the mol. polarisation of Duprene is much > that of crude natural rubber. This may account for the greater oil-resistance of the former. The course of the alteration in the  $\eta$  of the respective solutions with increase in concn. suggests that the mechanism of the accompanying association of the mols. is different in the two cases, involving end-to-end coupling in Duprene and lateral grouping in natural rubber. D. F. T.

Solvents from light oil.—See II.

## PATENTS.

Determination of the rubber content of latex. J. S. WARD and S. D. GEHMAN, Assrs. to WINGFOOT CORP. (U.S.P. 2,024,617, 17.12.35. Appl., 21.12.32).—An incandescent filament is viewed through a green filter and an extinction cell containing the dispersion. The extent of movement necessary to bring the extinction cell from the extinction point on one side of the point of max. brightness to that on the other side is an index to the rubber content. D. F. T.

Production of articles of continuous length and constant cross-section from fluid materials such as latex. INTERNAT. LATEX PROCESSES, LTD. (B.P. 454,469, 14.11.35. U.S., 16.11.34).—An electrical device records the length and wt. of material extruded during any time interval, and combines the figures to hold the extrusion process const. B. M. V.

Production of compounded aqueous dispersions of or containing rubber. INTERNAT. LATEX PROCESSES, LTD., D. F. TWISS, A. S. CARPENTER, and A. E. T. NEALE (B.P. 454,738, 21.8.35).—Compounded unvulcanised aq. dispersions of rubber, particularly when compounding has been accompanied by dilution and the d of the ingredients differs considerably from that of the aq. medium, are treated with a creaming agent in a proportion effective with uncompounded latex; the treatment increases the intimacy of association of the rubber globules and the particles of the compounding ingredients. D. F. T.

Manufacture of rubber products, more particularly of porous and spongy masses and articles. J. A. TALALAY (B.P. 455,138, 14.3.35).—A (warm) dispersion of rubber or rubber-like substances is frothed, either alone or in presence of one or more substances (possibly introduced under pressure) capable of giving rise to a gas ( $NH_4NO_2$ ,  $H_2O_2$ , liquid or solid  $CO_2$ ), by <sup>subjecting</sup> it to a reduced pressure, whereby porosity results from the expansion (and vaporisation). The material is then allowed to set (under pressure or vac.) while still in the form of froth. D. F. T.

Plastic rubber derivative. T. F. FORD, Assr. to B. F. GOODRICH CO. (U.S.P. 2,024,987, 17.12.35. Appl., 1.9.33).—A mixture of rubber with a smaller proportion of an aldehyde (trioxymethylene etc.) and a phenol is heated in presence of an aromatic sulphonic acid.

D. F. T.

(A) Plasticisation of rubber. (B) Plastic rubber product. (c) Rubber cement of high rubber content and low viscosity. I. WILLIAMS and C. C. SMITH, Assis. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,018,643-5, 22.10.35. Appl., [A] 16.6.34, [B] 6.7.34, [C] 28.8.34).-(A, B) Unvulcanised rubber intended for moulding or extrusion is softened by (milling with) (A) an unsymmetrically substituted hydrazine [NHPh·NH, (I),  $C_6H_4Me\cdot NH\cdot NH_2$  in absence of > 3% S, and (B) a salt of such a hydrazine [(I) hydrochloride or oxalate]. (c) The  $\eta$  of unvulcanised rubber solutions is reduced by incorporation of an unsymmetrically substituted hydrazine, its salts or derivatives, e.g., hydrazones, aryl derivatives,  $CS_2$  derivatives, or "salts" with phenols or naphthols. According to the examples, the best results are given with the CS<sub>2</sub> derivative of (I), (I) itself or other simple arylhydrazines, and their hydrazones with the lower ketones. H. A. P.

Hard rubber [ebonite] coating. L. B. HAINES, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 2,023,582, 10.12.35. Appl., 30.12.31).—A mixture of substantially 500 pts. of smoked sheet rubber, 180 pts. of S,  $2\frac{1}{2}$  pts. of diphenylguanidine, and  $2\frac{1}{2}$  pts. of thiolbenzthiazole is dissolved in 2500 pts. of benzine; the solution is applied to form a coating which is subsequently vulcanised.

D. F. T.

**Rubber compounding.** A. A. SOMERVILLE (U.S.P. 2,026,442, 31.12.35. Appl., 5.12.34).—Rubber is vulcanised after incorporating a catalytic antioxidant capable of inhibiting atm. oxidation ( $\beta$ -C<sub>10</sub>H<sub>7</sub>·NHPh), an org. O absorber (pyrogallol, quinol) to eliminate O<sub>2</sub> initially present in the compound, and an org. base [N(C<sub>2</sub>H<sub>4</sub>·OH)<sub>3</sub>] non-volatile at the vulcanisation temp. and miscible with rubber, to activate the O absorber. Resistance is imparted both to ageing and fatigue. D. F. T.

Vulcanisation of rubber. WINGFOOT CORP. (B.P. 454,863,6.4.35. U.S., 11.4.34).—An accelerator of the formula R  $\[mathbb{N}]{N}$ C·S·CS·NR'R" (R is an arylene group, R' contains a furan ring, and R" is a univalent, non-alicyclic org. radical), is used, *e.g.*, 6-nitrobenzthiazyl di- $\alpha$ -furyldithiocarbamate, m.p. 93–95°. D. F. T.

Vulcanisation of rubber. W. SCOTT, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 2,024,477, 17.12.35. Appl., 29.9.32).—A S derivative of a diarylamine containing an alkoxyphenyl radical, e.g., of p-OEt·C<sub>6</sub>H<sub>4</sub>·NH·C<sub>10</sub>H<sub>7</sub>- $\beta$ , is incorporated as antioxidant. D. F. T.

Vulcanisation of rubber articles in moulds. CONTINENTAL GUMMI-WERKE A.-G. (B.P. 454,829, 7.2.36. Ger., 31.8.35).—The moulds are passed continuously, on an endless conveyor, through a hot-air furnace and thence through a cooling zone. D. F. T. Manufacture of an accelerator for rubber vulcanisation. C. O. NORTH, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 2,024,470, 17.12.35. Appl., 11.1.28).—An aldehyde derivative of a Schiff's base obtained by the action of  $1\frac{1}{2}$ —4 mols. of an aliphatic aldehyde (PrCHO) with 1 mol. of a primary aromatic amine (NH<sub>2</sub>Ph) is treated with CS<sub>2</sub>. D. F. T.

Accelerator of vulcanisation. L. B. SEBRELL, Assr. to WINGFOOT CORP. (U.S.P. 2,024,605, 17.12.35. Appl., 12.4.33).—Vulcanisation is accelerated by a cyclic mercaptan (thiolbenzthiazole) in conjunction with NH<sub>3</sub>, a primary (e.g., aliphatic) amine, or an alkylaryl sec. amine. D. F. T.

[Preservative] treatment of rubber. L. H. How-LAND, Assr. to UNITED STATES RUBBER Co. (U.S.P. 2,026,386, 31.12.35. Appl., 23.1.35).—A reaction product of an alkali metal (Na) and a ketone–arylamine condensation product ( $COMe_2$ -NHPh<sub>2</sub>) is incorporated as ageresister. D. F. T.

**Preservation of rubber.** IMPERIAL CHEM. INDUSTRIES, LTD., W. BAIRD, and M. JONES (B.P. 454,958, 10.4.35. Addn. to B.P. 434,951; B., 1935, 1058).—A 4:4'-dialkoxy-N-alkyldiphenylamine which may contain nuclear-substituted Me and has no alkyl or alkoxy-group  $> C_6$  is incorporated. Examples are 4:4'-dimethoxy-N-methyldiphenylamine, m.p. 82—84°, and 4:4'-dimethoxy-N-ethyldiphenylamine, m.p. 44—45°. D. F. T.

Treatment of tacky material. [Reclaiming of rubber.] W. C. MACFARLANE, ASST. to XYLOS RUBBER Co. (U.S.P. 2,021,961, 26.11.35. Appl., 3.9.32).—Tacky rubber reclaim is extruded into strips through a straining machine, and these are cut up into small pieces which are immediately dusted to prevent adherence and then passed to refining machines. Apparatus is described.

B. M. V.

Manufacture of [rubber-like] sulphur condensation products. IMPERIAL CHEM. INDUSTRIES, LTD., and R. F. GOLDSTEIN (B.P. 453,850, 18.3.35).—Glycerol  $\alpha\gamma$ -dichloro- or -dibromo-hydrin, a di- or poly-sulphide of an alkali or alkaline earth, and an inert material in a fine state of subdivision, *e.g.*, freshly pptd. Mg(OH)<sub>2</sub>, BaSO<sub>4</sub>, or Al silicate, are heated together with stirring in an aq. medium. The condensation product formed adheres to the inert material and thereby forms a synthetic latex which is easily handled, readily washed, *e.g.*, by decantation to < 0.1% Cl, and is easily coagulated by evaporation, addition of acid, etc. to a resistant, practically odourless rubbery material. E. J. B.

Process for sulphurising organic compounds having unsaturated linkings of an aliphatic character. [Producing rubber substitute.] K. W. POSNANSKY and C. SANDVOSS (ALEXANDER & POSNANSKY) (B.P. 453,921, 21.3.35. Ger., 26.3.34).—S is caused to react with aliphatic compounds containing one or more double or triple linkings at  $> 100^{\circ}$  in presence of H<sub>2</sub>S, basic substances (NH<sub>3</sub>, amines, amides, hydrazines, etc.), or the usual vulcanisation accelerators being added as S-carriers. The reaction takes place at much lower temp. than with S alone. The H<sub>2</sub>S is absorbed and the products are very light-coloured rubber substitutes. The examples include the sulphurisation of castor oil, Et cinnamate, and styrene. The vulcanisation of rubber, gutta-percha, and balata by a similar process is claimed. S. C.

Plastic for dolls' heads .- See XIII.

## XV.—LEATHER ; GLUE.

Origin of "reticulin" in the hide. A. KÜNTZEL and (FRL.) A. SEITZ (Collegium, 1936, 567-570).—The residual tissue obtained by Kaye (cf. B., 1936, 754) is shown to be elastin fibres. There is no ground for assuming the existence of a third chemical substance different from collagen and elastin in the raw hide. D. W.

Theory of mineral tannage. V. Effect of chromium salts on the m.p. and mutarotation of gelatin. A. KÜNTZEL and H. BOENSEL (Collegium, 1936, 576-588; cf. B., 1936, 754) .- Aq. gelatin mixed with aq. Cr alum of certain concns. sets to a jelly which does not melt at < 100°, but continues to show characteristic mutarotation. With increasing Cr content, the mutarotation of the gelatin gels is appreciably diminished. Since the mutarotation of Cr-tanned gelatin gels remains practically unchanged the gelatin micelles must be very little penetrated. The small effect of the Cr salts on the mutarotation is due, not to the formation of complexes, but to the linking up of the primary valency chains, which can only be brought about with aggregated salts forming complexes. D. W.

Ellagic acid deposits in [vegetable-tanned] sole leather. A. KÜNTZEL and A. SCHAEFER (Collegium, 1936, 571-576).—The deposits in sole leather appear under the microscope as black amorphous masses by ordinary transmitted light and as fine crystals by polarised light. Single crystals are often seen between the reticular and papillary layers. The former deposits must be spherical and not between the fibres, but permeating them, which is confirmed by their appearance by reflected light. These deposits are ellagic acid which have cryst. in situ. The deposits can be extracted with  $C_5H_5N$ , but the extraction is tedious. Microscopical examination is preferable. D. W.

New sheepskin leather defects caused by chromogenic micro-organisms. W. HAUSAM [with E. LIEBSCHER] (Collegium, 1936, 561-566).—Well-defined, round, greyish- or brownish-violet spots on some vegetable-tanned sheepskins have been traced to *Dematium* mould. No grain damage has been observed, but the organism can penetrate the leather. D. W.

Determination of sulphuric acid in leather. J. AUSCHKAP (Latvij. Univ. Raksti, 1936, 3, 15–24).— Comparative analytical data obtained by various methods are recorded for the  $H_2SO_4$  content of 7 samples of leather prepared under controlled conditions.

#### H. J. E.

See also A., Nov., 1387, Tannin of Formosan tea leaves.

#### PATENTS.

Shoe polish. H. P. GRIFFIN and R. P. RICHARDSON (U.S.P. 2,028,324, 21.1.36. Appl., 27.6.34),—A composition especially for white fabric includes  $BaSO_4$  and ZnS or lithopone, talc, and  $Na_3PO_4$ . B. M. V.

Manufacture of gelatin product. C. H. EPSTEIN and N. R. GOTTHOFFER, ASSIS. to GRAYSLAKE GLATINE Co. (U.S.P. 2,024,683, 17.12.35. Appl., 5.5.32).— Pigskin or other raw material yielding gelatin is hydrolysed by means of dil. aq. citric or tartaric acid, with or without admixture of a mineral acid. D. W.

Manufacture of vegetable glue. E. H. HARVEY, Assr. to PERKINS GLUE Co. (U.S.P. 2,006,164, 25.6.35. Appl., 6.5.35).—Rapidly binding glues, suitable for veneers, are made from cassava starch and 1—3 pts. of  $H_2O$  by gelatinising in the warm with an alkali and adding a peroxide before or after gelatinisation to lessen the  $\eta$ . J. H. L.

Adhesive. F. H. SHOALS, ASST. to MODERN PANELS, INC. (U.S.P. 2,025,180, 24.12.35. Appl., 11.1.33).—A composition suitable for veneers etc. is prepared in described manner from hydrolysed starch, rubber latex, borax, and NaOH. S. M.

Making leather impermeable to liquids.—See VI-Plywood.—See IX.

## XVI.—AGRICULTURE.

Base status of Scottish soils. I. Effects of lime on five typical soils from north-east Scotland. R. L. MITCHELL (J. Agric. Sci., 1936, 26, 664–678).— The max. effect of CaO on these soils is shown within 6—12 months, the  $p_{\rm H}$  change per ton of CaO per acre varying from 0.5 to 1.0. The liming factor in relation to CaO-neutralisation curves of soils varied from 3.0 to 1.5. The proportion of CaO applied which entered the exchange complex was 50–70%. A. G. P.

Changes in Istrian and Dalmatian red-earth soils due to cultivation. E. BLANCK and H. SCHOR-STEIN (J. Landw., 1936, 84, 193—205).—Analytical data are recorded and discussed. No basis was found for the differentiation of various red-earth types. A. G. P.

Genesis and composition of peat deposits. B. D. WILSON, A. J. EAMES, E. V. STAKER (Cornell Univ. Agric. Exp. Sta. Mem., 1936, No. 188, 13 pp.).

Transport of water through heavy clay soils. III. E. C. CHILDS (J. Agric. Sci., 1936, 26, 527–545; cf. B., 1936, 245).—Experimental data concerning the movement of  $H_2O$  in soil are explicable by a theory of diffusion. A. G. P.

Separation of cyanuric acid from the soil of "Kağamigahara." C. ICHIKAWA (J. Agric. Chem. Soc. Japan, 1936, 12, 898—899).—Cyanuric acid was detected in this soil. W. O. K.

Fertiliser value of some concentrated materials, particularly urea and guanidine and their nitrates and phosphates. A. H. LEWIS (J. Agric. Sci., 1936, 26, 509–526).—In pot cultures  $NH_4$  and urea salts were equally effective sources of N to barley, but urea was superior for mustard. Guanidine (I) salts were slower in action and gave better results on neutral than on acid soils. No direct evidence was obtained of a toxic action of (I). Phospham (PN<sub>2</sub>H) and P<sub>3</sub>N<sub>5</sub> had little manurial val. The latter was slightly the better and both were more effective on neutral than on acid soils. When part or all of the P was applied in chemical combination with N yields were higher than when P was given as superphosphate in mixed fertilisers. In

A. G. P.

all tests the yield and intake of N were directly related to the  $NO_3'$  content of acid soils and to the  $NO_3' + NH_4'$ content of neutral soil. A. G. P.

Spreading of dairy wastes and surplus whey on grasslands. F. H. McDowALL (Canad. Dairy and Ice Cream J., 1936, 15, No. 10, 69-71).—The disposal of dairy waste liquids for irrigating grassland is described. Seven acres can cope with 20,000 gals. daily. The manurial equivs. of whey are calc. W. L. D.

Influence of temperature and available nitrogen supply on growth of pasture in the spring. G. E. BLACKMAN (J. Agric. Sci., 1936, 26, 620-647) .-- With a soil temp of  $5.5^{\circ}$  at 4 in. depth no growth of grass occurred. At 5.5-8.3° N-manured grass grew more rapidly than controls. At higher temp. growth rates were similar. Manured herbage reached the grazing stage earlier than the control. Increased N content of manured grass was apparent 4-8 days after treatment, even when growth did not take place ( $< 2 \cdot 8^{\circ}$ ). Max. N contents (3.9-5.3%) were reached approx. 10 days after growth commenced. Treatment with Ca(NO3)2 increased the NO3' content of grass; (NH4)2SO4 increased the  $NH_3$  and amide contents without increasing the  $NO_3'$  vals. Within 10 days of the application of NH4 or NO3' a large proportion of added N was not recoverable in inorg. form. A. G. P.

Influence of soil and fertilisers on quality of soft winter wheat. E. G. BAYFIELD (Ohio Agric. Exp. Sta. Bull., 1936, No. 563, 77 pp.).—Climate is a dominant factor influencing the strength and quality of wheat. Rainfall during and just preceding heading tends to lower the protein (I) content of the grain. Heavier soils are associated with higher (I) contents. Grain from soils derived from limestone usually contains more (I) than that from soils derived from non-calcareous sandstones or shales. Effects of fertiliser treatment are discussed and the val. of balanced nutrients is shown. Soil reaction affects wheat quality by modifying the availability of nutrients to the plants. A. G. P.

**Composts.** M. M. McCool (Contr. Boyce Thompson Inst., 1936, **8**, 263–281).—Changes in  $p_{\rm H}$  and temp. resulting from composting various leaves, straw, etc. with soil are examined. Leaves produced a lower max. temp. but more prolonged heating than did straw. Addition of CaCN<sub>2</sub> facilitated the decomp. of org. matter. The % N in all composts increased with time. A. G. P.

Soil productivity. F. BERKNER (Bodenk. Pflanzenernähr., 1936, 1, 88–95).—The effect of farmyard manure on the productivity and nutrient content of soils persists in the third season after application. The humus, N, C, and acid-sol. K, P, and Ca contents of soil increase with the amount of farmyard manure given, the abs. amounts declining in the third year after treatment. Neubauer vals. for K and P also increased with the application made. In the third season the general level of the K vals. was <, and that of the P vals.>, those of the second season. A. G. P.

Unproductiveness of certain orchard soils as related to lead arsenate spray accumulations. S. C. VANDECAVEYE, G. M. HORNER, and C. M. KEATON (Soil Sci., 1936, 42, 203-215).—The unproductive soils contained  $4 \cdot 5$ —12  $\cdot 5$  p.p.m. of sol. As<sub>2</sub>O<sub>3</sub>, which adversely affected lucerne and barley crops according to the amounts present. As and Pb were found in considerable proportions in roots, whereas in tops the As content was small and that of Pb negligible. Parallel culture experiments confirmed the injurious action of As.

A. G. P.

Green manure fertiliser study on Norfolk sand. J. E. ADAMS, E. M. ROLLER, and H. M. BOGGS (Soil Sci., 1936, 42, 175-185) .- Winter cover crops following highly manured soya beans, velvet beans, or cow peas as green manure produced a better crop in the following season than when a winter fallow was adopted. Use of a cover crop also permitted economy of fertilisers applied to the green manure and was more effective in increasing yields than the green manure crop itself. A direct but not close relation existed between the C content of the soil and crop yields. This was less marked when the green manure crop was generously fertilised. Introduction of green manure into the rotation lowered the C/N ratio of the soil by increasing the N content. A. G. P.

Effect of delinting cottonseed with sulphuric acid on germination and yield. A. HAMID and N. MOHAMMAD (Agric. Live-Stock India, 1936, 6, 653— 661).—In preliminary trials delinting increased the % and rate of germination and increased the crop yield. A. G. P.

Cottonseed treatment with sulphuric acid. B. G. CHRISTIDIS (J. Agric. Sci., 1936, 26, 648-663).—Acid treatment favours earlier germination of seed, especially when the amount of soil- $H_2O$  is limited, but the yield per acre and ginning out-turn were not significantly affected. A. G. P.

Effect of arrowing (flowering) on sugar-cane crops. K. K. RAO and K. V. G. IYER (Agric. Live-Stock India, 1936, 6, 667—685).—Arrowed canes have a higher sucrose content, which is maintained for the next 3 months. The juice contains more mineral matter and less glucose than that of non-arrowed canes. Deterioration of juice begins at a later stage; growth ceases after arrowing and the final yield is considerably < that from non-arrowed plants.

A. G. P.

Influence of light on growth, sugar formation, and nutriment absorption of sugar beet. W. KRÜGER and C. WIMMER [with O. RINGLEBEN] (Z. Wirts. Zuckerind., 1936, 86, 271-288).-Beets grown in diffused daylight were very much smaller in wt. and lower in sugar content and purity of juice than those exposed to sunlight. Their defective growth was not due to reduced capacity to absorb nutriment, for they contained much higher % of N, P, and K than the others. Light and K are of the first importance for sugar formation, but no evidence could be obtained that they are in any degree mutually replaceable : abundant light does not enable the normal K requirements to be reduced without loss, nor will an extra supply of K compensate in any degree for subnormal illumination. J. H. L.

Chlorophobia in plants. M. MASAEVA (Bodenk. Pflanzenernähr., 1936, 1, 39-56).—The inferior action

of fertilisers containing  $\rm NH_4Cl$  or "Potazote" is due to their high Cl' content. The inhibitory action of Cl' is more marked in light, poorly buffered soils than in clays, and is largely attributed to the increased intake of Cl' and Ca" and the lowered K: Ca ratio of the plants. Cl' prolongs the vegetative period of plants. Chlorophobic plants are generally calciphobic also.

A. G. P.

Use of borax in the control of "internal cork" of apples. Preface. T. RIGG. I. Influence of borax top-dressing on the boron status of soil, fruit, and leaves. H. O. ASKEW, E. CHITTENDEN, and R. H. K. THOMSON. II. Effect of tree injection of borax solutions on the boron status of apple trees. III. Effect of borax sprays on the boron status of fruit and incidence of "internal cork" in apples. H. O. ASKEW and E. CHITTENDEN (J. Pomology, 1936, 14, 227 - 228, 228 - 239, 239 - 242, 242 - 245).-I. Applications of borax (I) rapidly increased the B content of soils to a depth of 18 in. and reduced the incidence of internal cork in apples. Use of 0.5 - 1.0 lb. per tree, or 50-100 lb. per acre broadcast, is recommended. The B content of treated fruit was approx. the same as that of fruit free from "cork" grown on healthy soils. (I) had no definite effect on the size or dry matter content of fruit.

II. Injection of (I) solution into trees gave complete control of internal cork.

III. Spraying with 0.25% aq. (I) with or without Ca(OH)<sub>2</sub> controlled corking. (I) migrated from sprayed to unsprayed portions of trees. A. G. P.

Toxicity of certain chemical agents to *Erwinia* amylovora. G. W. KEITT, J. A. PINCKARD, L. SHAW, and A. J. RIKER (J. Agric. Res., 1936, 53, 307–317).— Hg compounds (notably HgCl<sub>2</sub>, HgEtCl, HgO) were highly toxic.  $Ag_2SO_4$  was even more effective. CaO–S was non-effective. Bordeaux mixture and  $ZnSO_4$ –CaO were lethal to fire-blight bacteria at concns. < those usually adopted for orchard use. A. G. P.

#### Starch in potato.—See XVII.

See also A., Nov., 1422, Biological fixation of N. 1432, Effect of B on growth of certain green plants. 1434, Effect of  $CH_2Ph \cdot CO_2H$  on growth of tomato plants. Determining N in plant tissues, and of small amounts of Cu in plants.

#### PATENTS.

Art of irrigating and fertilising plants. W. O. SWEEK, Assr. to W. T. MCGEORGE, J. S. BENROTH, J. A. SAUNDERS, and H. H. PORTER (U.S.P. 2,028,172, 21.1.36. Appl., 22.6.34).—H<sub>2</sub>O for irrigating is stored at a level above the fields and the energy of the fall is used to generate electricity; at off-peak hr. this electricity is used to oxidise atm. N<sub>2</sub>. The N oxides produced are dissolved in part of the H<sub>2</sub>O, which is then exposed to air to form HNO<sub>3</sub> in excess of the alkali normally present in the H<sub>2</sub>O. B. M. V.

Aërating apparatus for fertiliser and the like. A. J. SACKETT (U.S.P. 2,028,413, 21.1.36. Appl., 6.7.34).—Falling comminuted material is positively beaten into cyclonic motion (and the downward motion retarded by the angle of the beaters) in a no. of successive zones, air being induced into the whirling mass and leaving at the upper part. B. M. V.

Insecticide. L. C. HECKERT, Assr. to Röhm & HAAS Co. (U.S.P. 2,024,098, 10.12.35. Appl., 31.5.29).— Esters, RCNS, in which R contains a negative group, are potent insecticides in solution or as solids or vapours. Compounds with > 1 CNS are also claimed. Examples are benzyl (used in kerosene etc.), Me, amyl, or Ph thiocyanate (used as vapour), and p-CNS·C<sub>6</sub>H<sub>4</sub>·NMe<sub>2</sub> (used as dust with CaCO<sub>3</sub> etc., or as hydrochloride in H<sub>2</sub>O). The odour is masked by methylation of substituent NH<sub>2</sub>. R. S. C.

Insecticidal preparation. H. SCHOTTE and K. Görnitz, Assrs. to SCHERING-KAHLBAUM A.-G. (U.S.P. 2,024,392, 17.12.34. Appl., 12.12.33. Ger., 13.11.31).— The efficiency of rotenone-containing insecticides is increased by admixture of veratrin (I) or material containing it. Suitable dusts are derris root (25), sabadilla seeds (7.5), and talc (90), or rotenone (0.2), (I) (0.3), and kaolin (99.5%). R. S. C.

**Spray composition.** F. F. LINDSTAEDT, Assr. to HERCULES GLUE Co., LTD. (U.S.P. 2,022,246, 26.11.35. Appl., 6.10.31).—The control of scale etc. effected by oil sprays is increased by addition of a carrier incorporated in the oil phase of the spray, which lengthens the life of the oil on the plant, increases the amount of deposit, and permits efficient use of low-viscosity ( $45^{\circ}$ ) oil without injury to the plant. The carrier may be rubber latex with or without an additional emulsifier or a clay; the quantity of clay thus added is increased by admixture with a metallic (e.g., Mg) soap which is sol. in or wetted or dispersed by the oil. *E.g.*, an emulsion (1:50 in H<sub>2</sub>O) of latex 450, sulphonated castor oil 12, NH<sub>3</sub> 12, H<sub>2</sub>O 120, oil to make 7570 c.c. is claimed. R. S. C.

Means for destroying growing bracken. V. H. HOLT (B.P. 455,326, 10.10.35).

Org. F compounds.—See III. Treating chlorates. —See VII. Salts from cane molasses.—See XVII. Fungicide etc.—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

Recent scientific and technical progress in the sugar industry. D. TEATINI (Chim. e l'Ind., 1936, 18, 501-510).—A lecture describing mainly the author's own methods in the sugar industry. O. J. W.

Abnormally high purity of some cane juices. Y. K. R. RAO (Internat. Sugar J., 1936, 38, 389).— Figures are given showing the possibility of error in the determination of the purity of mill juices when the Brix hydrometer is used, due to the presence of emulsified air, thus causing an abnormally high purity val. A normal purity figure is obtained, however, when the solids are determined by means of the refractometer. The degree of emulsification depends, not only on the cane variety, but also on operations such as milling, pumping, and mixing, as well as on whether maceration is hot or cold. J. P. O.

Growth of sugar crystals in vacuum pans. A. L. WEBRE (Internat. Sugar J., 1936, 38, 379-381).— Uniform conditions as regards concn. of syrup and movement of the mass do not obtain in either coil or

calandria pans of ordinary design. Temp. variations of the order of 50° F. may occur, even in carefully controlled operation, leading to the disturbance of the optimum graining supersaturation. It is practically impossible to approach ideal conditions of boiling in vac. pans with natural circulation, by means of inducing adequate circulation the influence of the height of the massecuite on the making of the strike is eliminated completely, and it becomes possible to boil satisfactorily with as great a height as 14 ft. above the top of the heating surface. Sluggish operation at the end of the strike also disappears, and the greatly increased heat transmission makes possible the use of juice vapours at low temp., even under partial vac.; the most important gain, however, is in the uniformity of the size and the shape of the crystal-the best evidence of satisfactory conditions throughout the J. P. O. process of boiling.

Internal structures in [sugar] diffusers for the better percolation of the leach liquor. E. LEHRE (Deuts. Zuckerind., 1935, 60, 282; Chem. Zentr., 1935, ii, 1458).—The greater is the no. of internal structures, e.g., baffles, in the upper part of the diffuser the greater is the no. of hollow spaces and dead angles, so that the output is adversely affected and the leaching nonuniform. To prevent seepage of the leach liquor down the inside walls the greatest possible wt. of beet slices should be charged into the vessel and the charge should be pressed down so that it is forced as tightly as possible against the walls. A. R. P.

Action of alkali carbonate on calcium salt solutions and of carbon dioxide on limed sugar solutions and factory juices. III. O. SPENGLER and G. DORFMÜLLER (Z. Wirts. Zuckerind., 1936, 86, 371-394; cf. A., 1935, 1088).-The highly hydrated CaCO<sub>3</sub> [primary substance (I)] formed in the initial stages of pptn. of CaCl<sub>2</sub> solutions by Na<sub>2</sub>CO<sub>3</sub> is stable only in an alkaline medium, and only under these conditions can its transformation into cryst. hexahydrate be observed. This transformation can be accelerated by rubbing with a glass rod. When CO2 is passed into sucrose solutions containing dissolved CaO until pptn. begins, a considerable quantity of (I) is formed and remains in solution even in the warm. By careful neutralisation at low temp. it may be separated and transformed into hexahydrate. A 10% sucrose solution containing 1.7%of dissolved CaO may retain in solution (I) equiv. to J. H. L.  $1\cdot4\%$  of CaCO<sub>3</sub>.

Influence of different purification processes on the quality of beet-sugar juices with special regard to the ripeness of the beets. O. SPENGLER, S. BÖTTGER, and W. KRÜGER (Z. Wirts. Zuckerind., 1936, 86, 437—472).—In two successive years the same set of purification processes was applied to juices from beets of the same type worked at different parts of the campaign, viz., Aug.—Sept. (unripe), Nov.—Dec. (ripe), and Mar.—Apr. (over-ripe), to ascertain how the degree of ripeness affects the results obtained with different processes. The results were judged mainly from the character and composition of the final thick juice. With the normal method of working (without preliming or other special processes) unripe roots gave thick juices conspicuous for high ash content and low purity; over-ripe roots yielded very dark juice of high Ca content. Several forms of pre-liming were employed. They had little effect on the ash or Ca content of the thick juices; their main benefits (colour and purity) were conspicuous with all classes of beets, but least with the over-ripe. With all the beets, liming for the second carbonatation improved the juice colour, and triple carbonatation (Karlik) improved it more. With ripe and unripe, but not over-ripe, beets very striking improvement in colour, and reduction of Ca content, were produced by the Weisberg sulphitation process and still more by final-juice sulphuring. In all cases some improvement in ash content and very great improvement in colour and (except with over-ripe beets) in Ca content were obtained by thick-juice liming (0.2% CaO on roots) and saturation; errors in thin-juice purification can be, in part, corr. by this procedure. J. H. L.

Chemistry of [beet] juice discoloration. I. O. SPENGLER, S. BÖTTGER, and B. SEELIGER (Z. Wirts. Zuckerind., 1936, 86, 193-239).-Special apparatus was designed for heating sugar solutions at const. temp. With two thin juices from different beets, heated at temp. between 90° and 130°, a temp. rise of 10° increased the rate of discoloration  $2\frac{1}{2}$ - or 3-fold. Experiments were made with pure sucrose solutions buffered to resemble thin juice in  $p_{\rm H}$  val. and alkalinity, the temp. of heating being 90°, 110°, and 130°. At const. temp. the colour increased linearly with the time, and a continuous fall in alkalinity occurred. A slight fall in polarisation was detected in most cases, but in some it appeared to rise again on further heating. A temp. rise of 10° increased the rate of discoloration 3-fold; *i.e.*, the rate was about 81 times as great at  $130^{\circ}$  as at 90°. An increase in the concn. of H ions or sugar lessened the rate of discoloration. In preparing the buffered solutions for these experiments it was found that sucrose decreases the  $p_{\rm H}$  val. of buffer solutions, doubtless owing to its slightly acidic character. Sucrose solutions buffered to the same  $p_{\rm H}$  val. (in the cold) and alkalinity, with different buffers, did not discolour to the same extent when heated under the same conditions ; probably they were not alike in  $p_{\mathbf{H}}$  val. at high temp.

J. H. L.

Laws of reaction of invert sugar and sucrose with hot alkaline copper solutions. O. SPENGLER, F. TÖDT, and M. SCHEUER (Z. Wirts. Zuckerind., 1936, 86, 130-146, 322-331).-Using Cu reagents of different alkalinities, and heating in a boiling water-bath, the authors compared the course of reduction by 10 mg. of invert sugar (I) and by 10 g. of sucrose (II). With (II) the wt. of reduced Cu increased uniformly throughout the whole period of heating, at a rate determined mainly by alkalinity. With (I) the wt. of reduced Cu rose to practically const. vals.; the higher was the alkalinity the lower was the const. val., and the more rapidly was it attained. At  $p_{\rm H}$  vals. of 13, 10.4, and 9 in the reaction mixture, 1 mol. of hexose (III) reduced about 5, 6, and 7 equivs. of Cu, respectively. It is suggested that with Müller's solution at 10-fold dilution, under the conditions employed, the reaction is stoicheiometric, (III) being oxidised to arabonic acid (cf. A.,

1935, 1354). Other matters studied were the influence of [Cu] on the reduction, and procedure for determining the reduced Cu iodometrically. The outcome of this and preceding work (B., 1934, 694, 938, 1029; 1935, 375) is the following method for determining small quantities of invert sugar, e.g., in raw beet sugars. A 10-g. sample, or such smaller quantity as contains  $\geq 30$ mg. of invert sugar, is dissolved in 100 c.c. of H<sub>2</sub>O and without clarification mixed with 10 c.c. of Müller's solution (a cold solution of 68 g. of anhyd. Na<sub>2</sub>CO<sub>2</sub> in 500 c.c. of H<sub>o</sub>O is poured into one of 35 g. of CuSO<sub>4</sub>,5H<sub>o</sub>O in 400 c.c. of H<sub>2</sub>O, and the mixture made up to 1 litre and filtered with some active C) in a 300-c.c. Erlenmeyer flask and heated for exactly 10 min. in a vigorously boiling water-bath, the level of the bath-H<sub>2</sub>O being at least 2 cm. above that of the reaction mixture. After rapid cooling without motion, the liquid is treated with 5 c.c. of 5N-AcOH or -tartaric acid and immediately afterwards with an excess of N/30-I, and when all the Cu ppt. has dissolved, the excess of I is titrated with  $N/30-Na_2S_2O_3$ . The vol. of I consumed is corr. by an occasional blank test on Müller's solution without sugar, and by a blank test on the sugar sample without heating, and 2 c.c. of N/30-I is deducted for the reducing effect of 10 g. of (II) if present. After these corrections have been made each c.c. of  $N/30-I \equiv 1$  mg. of (I). Satisfactory results with raw beet sugars are recorded. J. H. L.

Thermophilic contamination within the sugar factory. F. R. CALTON (Ind. Eng. Chem., 1936, 28, 1235—1238).—In an American granulated beet-sugar factory a study was made of the behaviour throughout the several stages of manufacture of the bacteria causing trouble in canned vegetables. "Flat sours" were eliminated by filtration to the extent of about 96%, but the syrups were later contaminated, notably in the pan-storage tank. Anaërobes, which cause  $CO_2$  and  $H_2$ formation in the tins, were found to be actively propagated within the evaporator steam-chest, and large nos. were developing in the tail-pipe supply system, thus continuously contaminating the diffusion-battery supply  $H_2O$ . J. P. O.

Action of [fermenting] molasses on boiler scale containing gypsum. W. KLP and R. BUSE (Z. Spiritusind., 1936, 59, 379–380).—The  $PrCO_2H$  (I) fermentation process, as used for the removal of boiler scale, has no chemical action on  $CaSO_4$ , this salt being recovered quantitatively from such process carried out in the laboratory in its presence, whilst independently, attempts to demonstrate the production from it of  $H_2S$ or  $SO_2$  failed. The (I), however, decomposes scale carbonates and so (assisted by the fermentation gases) exerts a loosening effect on  $CaSO_4$ , facilitating its removal by washing. Scales composed principally of  $CaSO_4$  and silicates are unaffected by this fermentation process. I. A. P.

Starch content of potato flakes. M. PLATZMANN (Z. Spiritusind., 1936, 59, 361).—Soil conditions and methods of fertilisation necessary for the cultivation of potatoes suitable for flaking are indicated. I. A. P.

Technical evaluation of starch pulp by sieving. S. BAROBIN (Z. Spiritusind., 1936, 59, 351, 359, 367).— Mutter Chemical Apstracts-D.

A method of evaluation is described and critically discussed, wherein the pulp is divided into three portions by sieves of decreasing mesh size, the portions being weighed. Thus, a measure of the efficiency of pulping can be obtained in approx. 20 min. Results are not in close agreement with those obtained by the Saare chemical method, but it is not certain that this fact is to the discredit of the sieve method. I. A. P.

Determination of starch in starch, flour, wheat, and bread by the polarimetric method. F. HERLES (Chem. Listy, 1936, 30, 231-232).—The material is dissolved in 50 c.c. of 60% HCl (1 hr.; 20°), the solution diluted to 100 c.c., and the starch determined polarimetrically, applying corrections for the vol. occupied by the starch, and for presence of other optically active substances. R. T.

Compound evaporator.—See I. Sugar beet. Sugar-cane crops.—See XVI. Fodder yeast. [EtOH from] beet shavings.—See XVIII. Acid ensilage of beet slices.—See XIX.

See also A., Nov., 1363, Quant. differentiation of fructose and mannose. 1364, Prep. of *d*- and *l*-threose. 1365, Iodometric determination of fructose. 1397, Micro-determination of fructose in presence of glucose. 1421, Micro-organisms of sugar-cane juice.

#### PATENTS.

Crystallisers suitable for sugar massecuite. BLAIRS, LTD., and R. T. BINNIE (B.P. 453,966, 6.6.35).— Rotating, spiral cooling tubes in a tank are described.

B. M. V. Processes for continuously crystallising sugar solutions. J. A. PLATTE and G. H. DE VRIES (B.P. 444,534, 26.7.35. Holl., 14.9.34).-Hot saturated syrup is delivered continuously to a compartmented crystalliser, in the first compartment of which grain is formed by controlled cooling, or is introduced. In the remaining compartments countercurrent cooling is so regulated that crystallisation is completed at a const. supersaturation, the no. of cooling elements increasing in successive compartments. Stirring is provided for and the cooling elements impose on the syrup a baffled path. For fine-grained sugars the entire crystallisation can be carried out in a single receptacle, and smaller grain can be produced than in a vac. pan. For large-grained sugars several receptacles in series are necessary. The syrup may be fed at 83° Brix and 100°, from a multipleeffect evaporator in which, to avoid crystallisation, the

**Recovery of sugar [from beet molasses].** F. H. ROGERS. From SPRECKELS SUGAR Co. (B.P. 443,662, 10.1.35).—In a continuous process, a regulated proportion of milk-of-CaO is mixed with a stream of cooled molasses solution, and the mixture passed through a grinding unit, cooled, and filtered. The filtrate is rejected and the ppt. may be used for the purification of raw beet juice. Apparatus is claimed. J. H. L.

usual direction of flow of juice is reversed. J. H. L.

Recovery of sugar and salts from sugar-cane molasses. E. A. VAZQUEZ (U.S.P. 2,000,202, 7.5.35. Appl., 25.10.32. Cuba, 20.7.32).—Warm molasses is extracted with a mixture of 95% EtOH, EtOAc, or

other solvent, and  $H_2SO_4$ , to remove org. acids, wax, colouring matters, etc., which are recovered. The residue is neutralised with CaO and heated with 80—90% EtOH to extract the sugars, which may be used for preparing edible syrup etc. The final residue, consisting largely of salts, is used as fertiliser or otherwise. J. H. L.

**Production of pure white sugar.** I. BLOCH (U.S.P. 2,003,965, 4.6.35. Appl., 14.5.31. Ger., 22.1.31). —Massecuites or raw sugars are mixed with glycol (I) at room temp. or slightly above for about 15 min., and centrifuged. The removal of syrup from the crystals can be thus effected without any dissolution of sugar. The proportion of (I) used is, e.g., 3 litres to 6—8 kg. of massecuite, or 6 litres to 10 kg. of raw sugar. Runoffs containing (I) can be further used in the same way. Further crystallisation of green syrups can be brought about by addition of (I). J. H. L.

Production of malt syrup. H. C. GORE, S. JÓZSA, and C. N. FREY, Assrs. to STANDARD BRANDS, INC. (U.S.P. 2,000,169, 7.5.35. Appl., 11.1.30).—Malt mashes are heated rapidly to 70°, to avoid proteolysis, and after saccharification at this temp. heated at 90° to destroy enzymes, and the filtered wort is evaporated. Avoidance of proteolysis reduces risk of evolution of gas, attributed to interaction of NH<sub>2</sub>-acids with sugar, during storage of the syrup. J. H. L.

Saccharification of cellulose. H. SCHOLLER (B.P. 444,766, 27.9.34. Ger., 27.9.33).-The process of B.P. 367,916 (B., 1932, 443) is modified to lessen destruction of sugar in the pores of the cellulosic material. Each batch of acid liquor is introduced at a temp. lower (by  $10-20^{\circ}$ ) than that of the material at the time, though higher than that of the previous acid batch. After each acid batch has passed through, the material is heated to a temp. above that of the next acid batch. This heating may be effected by injection of steam from below. Acid batches may be introduced from above under pressure, and the first portions of each may be less acid than the later portions. The whole process may be completed in 12 hr., but the sugar formed remains for only a short time in the material. Acid batches, after passing through the material, may remain in the lower part of the apparatus at high temp. to complete saccharification of dissolved polyoses, e.g., J. H. L. cellobiose.

Apparatus for carrying out the saccharification of cellulose material. H. SCHOLLER (B.P. 449,601, 27.9.34. Ger., 27.9.33).—Apparatus for carrying out the process of B.P. 444,766 (preceding abstract), comprises a percolator having means for supply of acid at the upper part, which is cylindrical, the lower part being conical, lined with filter material, and interlined with material forming passages for outlet of filtrate and inlet of steam. Provision is also made for steam to be admitted direct to the cellulosic material from inlets near the point of the cone. An inspection window in the outlet pipe is described. B. M. V.

Method of purification of carbohydrate solutions obtained in the saccharification of cellulosecontaining materials. E. FAERBER, H. KOCH, and H. URBAN, ASSTS. to HOLZHYDROLYSE A.-G. (U.S.P. 1,997,545, 9.4.35. Appl., 3.5.33. Ger., 31.10.31).— The solutions, containing about 20-40% of sugar and 1-5% of acid, are heated sufficiently to ppt. humous colouring matters but not to hydrolyse polymeric carbohydrates completely. J. H. L.

Manufacture of starches. F. H. THURBER, Ded. to U.S.A. (U.S.P. 2,001,925, 21.5.35. Appl., 15.8.32).— Alkaline sulphite or sulphide solutions are added to freshly pulped sweet potatoes or other starch-yielding material, and if necessary to the separated starch in course of purification, in order to remove colouring matters, facilitate purification, and increase yield. J. H. L.

Manufacture of starch. R. E. GREENFIELD and H. R. BAKER, Assrs. to A. E. STALEY MANUFG. Co. (U.S.P. 2,023,999, 10.12.35. Appl., 23.9.32).-In the starch-milk flotation process of germ removal, the separated liquor is employed for steeping additional maize, whilst the fresh H<sub>2</sub>O washings from the starch and germ are returned to the first process as additional starch milk. Subsequently, the separated liquor from the starch-gluten milk and later fresh H<sub>2</sub>O washings are used for washing the fibre and are then returned for starch-milk flotation. Likewise, liquor separated from the gluten is used for taking up succeeding starch-gluten mixtures for tabling, whilst wash-H<sub>2</sub>O from the filtered starch is partly used for taking up tabled starch for filtering and is in part returned to the process. The H<sub>2</sub>O balance is thus improved, and greater control of the sol. materials is possible. I. A. P.

Furnaces.—See I. Vegetable glue.—See XV. Compounds from fermentation residues.—See XVIII.

## XVIII.—FERMENTATION INDUSTRIES.

Recent advances in the fermentation industries. T. CHRZASZCZ and J. JANICKI (Chem. & Ind., 1936, 884– 891).—A review.

Fodder yeast. H. FINK (Z. Spiritusind., 1936, 59, 373-374, 376).—A lecture.

Production of fodder yeast from sulphite waste liquors. H. FINK and R. LECHNER (Angew. Chem., 1936, 49, 775—777).—Sulphite waste liquors from the technical production of sugar from wood form practicable substrates for the growth of yeasts (*Torula utilis*) without any further addition of N. The economics of the problem are discussed. J. S. A.

Dependence of yeast-protein composition on the growth medium. H. LÜERS and M. VAIDYA (Z. Spiritusind., 1936, 59, 365-366, 377-378).-Molasses wort alone, or this with mineral matter, and also NH4 salts, urea, or malt-germ extract as source of N, was used in individual yeast-culture experiments. All the NH<sub>2</sub>-acids investigated (cystine, tyrosine, tryptophan, histidine, arginine) were present in hydrolysates from all the various yeast crops obtained, though the relative amounts of each varied somewhat with the N source. The purine contents of the yeasts were remarkably const., but notable variations in sterol contents which occurred in some crops could not be related to the N source. Urea alone is not a sufficient source of N for yeast multiplication and protein synthesis, but there can be no doubt as to the biological val. of mineralcultivated yeasts. I. A. P.

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Chemical changes in wort composition during sparging. E. SCHILD [with A. LÖTHER and O. GEIGER] (Woch. Brau., 1936, 53, 345-350, 353-357).-Detailed analyses are given of worts from bright and dark brews taken at various gravities from the first to the last runnings. The ratio maltose/dextrin in the extract is modified by sparging in the direction of decreased enzyme activity, and relative increases are found in inorg. and nitrogenous constituents, the increases being due partly to the physical properties of the liquor, and partly to the increased alkalinity of the later runnings. The increases in  $PO_4^{\prime\prime\prime}$  are without danger, but the additional pigmented husk constituents and  $H_2SiO_3$  may interfere with the break. The nitrogenous substances dissolved by the sparging are of undesirable high-mol. form. The detailed results are critically discussed. I. A. P.

Maturing of beer and its acceleration. J. STAUBER (Woch. Brau., 1936, 53, 361-365).—A lecture, with special reference to the use of fibrous clarifying agents composed largely of pretreated asbestos. I. A. P.

Possibility of alcohol loss by the Hesse method of combating foaming [distillery] fermentations. B. LAMPE, R. DEPLANQUE, and E. ROEHRICH (Z. Spiritusind., 1936, 59, 366, 368-369).-The starch in a kilned distillery malt, but not in a green malt, may be almost completely saccharified and fermented during the aftersaccharification at 30° (72 hr.), the protein and diastase contents of the malt being without importance. Increase in the duration of this process to 144 hr. gave a complete conversion of the starch of a green malt rich in protein, but was still insufficient when the malt possessed low protein content. In the Hesse method, therefore, wherein one third of the green malt is added to the cooled mash, there is a possibility of a definite, though small, loss of EtOH. On the first day of fermentation, a mash prepared from sucrose and green malt showed a less rapid fermentation than when the latter was added without mashing, but the difference was equalised on the second day. I. A. P.

Relation between rate of access of air, oxygen and carbon dioxide content of the waste gases, alcohol conversion, and evaporation losses in the Schüzenbach vinegar process. H. KREIPE (Deuts. Essigind., 1935, 39, 189–191; Chem. Zentr., 1935, ii, 1623).—The rate of access of air considerably affects the evaporation in the Schüzenbach process. Since the AcOH bacteria can function satisfactorily at low  $[O_2]$ the air supply should be reduced to a min. sufficient to give good ventilation of the waste gas and control of its  $CO_2$  and  $O_2$  content; the  $[O_2]$  can be safely reduced to 12—15% in the waste gas. A. R. P.

Using dried beet shavings for spirit manufacture. B. LAMPE (Z. Spiritusind., 1936, 59, 360—361).— On a laboratory scale, fermentations of beet-shaving mashes made with cold or hot  $H_2O$ , with or without malt and/or yeast extract, gave satisfactory yields of EtOH (approx. 39 c.c./100 g. of shavings), which were but slightly affected by varying the mashing conditions. A recommended procedure is described. I. A. P.

Detection of diethyl phthalate in whiskies and other alcoholic products. I. SCHWARTZ (J. Amer. Pharm. Assoc., 1936, 25, 749–752).—A 25-c.c. sample, with 50 c.c. of  $H_2O$ , is distilled and 10–22 c.c. of the first 50 c.c. of distillate are evaporated in presence of  $0\cdot 2$ — $0\cdot 3$  c.c. of 5% NaOH. The residue is treated with  $H_2SO_4$  + resorcinol at 165–175°, cooled, diluted, and extracted with Et<sub>2</sub>O. A positive test is indicated by a green fluorescence appearing on slow addition of 5% NaOH to the  $H_2O$ -washed Et<sub>2</sub>O extract. The method is more reliable than that of the U.S.P. X. F. O. H.

Preserving grape juice.—See XIX.

See also A., Nov., 1359, Determination of EtOH. 1418, Inulinase.  $\beta$ -Glycuronosidase. 1419, Cozymase pyrophosphate. Racemiase. 1421, Action of small quantities of Cu in wine-making. Oligodynamic action of metals on yeast. Carbohydrates of yeast.

#### PATENTS.

Stationary [hop] drying kilns. KALOROIL BURNERS, LTD., and A. GOLDBERG (B.P. 454,517, 26.4.35).— Forced-draught apparatus suitable for hop oasts, incorporating the mixing of products of combustion with additional air before entering the oast proper, is described. B. M. V.

Yeast fermentation. D. A. LEGG, Assr. to COMMER-CIAL SOLVENTS CORP. (U.S.P. 2,026,237, 31.12.35. Appl., 8.6.32).—The extent of fermentation of hydrol, and the EtOH yields therefrom, are increased by heating for 30—50 min. at 40 lb./sq. in. with 3% (on the hydrol) of  $H_2SO_4$  or an equiv. mixture of acid with Ca superphosphate after dilution to < 25% sugar concn. After adjustment of the  $p_H$  to 5—6 by addition of, e.g., NaOH, CaO, or NH<sub>3</sub>, fermentation is carried out (at 27°) with yeast acclimatised to hydrol. To supply N deficiencies in the medium to be fermented, the distillery slop from BuOH–COMe<sub>2</sub> fermentation, or maize steep-H<sub>2</sub>O, may replace part of the  $H_2O$  used in the prep. of the hydrol mash. I. A. P.

Alcoholic fermentation. USINES DE MELLE, and F. BOINOT (B.P. 455,339, 9.3.36. Fr., 30.3.35. Addn. to B.P. 420,878; B., 1935, 203).—Prior to yeast fermentation, the mash is heated for  $\lt$  10 min. at 80—140°, with or without addition of acid which is subsequently partly neutralised. The resultant pasteurisation or sterilisation is accompanied by coagulation of undesirable substances, thus favouring the subsequent fermentation process. I. A. P.

Brewing of beer. E. PAULS (B.P. 454,766, 12.3.36. Ger., 12.3.35).—A wort is prepared in such a way that its protein content is low, the ratio maltose/dextrin high, and the extract content is  $\gg 14\%$ . After heavy hopping, the wort is either fermented with a mixture of *Sacch. cerevisia* (low) and an organism capable of fermenting dextrins, *e.g.*, *Schizosacch. Pombe*, or the latter may be used alone or after preliminary fermentation with the former. A beer of low extract content is thus obtained. I. A. P.

Alkali-metal compound recovery from waste organic mixtures [fermentation residues]. G. T. REICH (U.S.P. 2,002,797, 28.5.35. Appl., 21.10.32. Cf. U.S.P. 1,886,045; B., 1933, 729).—K is separated as nitrate or phosphate from conc. residues from the fermentation of molasses by treatment with  $NH_4NO_3$  or  $NH_4$  phosphate. The separated mother-liquor, conc. to  $d \ 1.2 - 1.45$ , deposits further K and  $NH_4$  salts, and is treated on the lines of the prior patent. J. H. L.

Heating liquids [e.g., wort].—See I. Malt syrup. —See XVII.

## XIX.—FOODS.

Ravages of the parasites of wheat. R. GUILLEMET (Compt. rend., 1936, 203, 570–572).—Aq. extracts of *Aelia cognata* (or, in a less degree, of the heads) treble the rate of maltose formation from flour. Dough from the gluten of unattacked wheat loses its consistency a few hr. after adding such extracts; the sol. and  $NH_2$ -N are also increased. Extracts of corpses of several weeks are as active as those from fresh bodies. J. L. D.

Determination of milk solids-not-fat in bread. D. A. MAGRAW and L. E. COPELAND (Cereal Chem., 1936, 13, 541-549).—The lactose (I) content of milk solids-not-fat (II) is nearly const. at 50%; (I) is therefore used as a measure of (II). It is determined by Munson and Walker's method after removing starch and protein. Corrections are made for blanks and losses. The method is accurate to  $\pm 0.50\%$  (II) and entails no exceptionally specialised technique. E. A. F.

Experimental durum milling and macaronimaking technique. D. S. BINNINGTON and W. F. GEDDES (Cereal Chem., 1936, 13, 497-521).-Increasing cultivation of durum wheat in Canada has led to the establishment of a special laboratory containing an experimental mill and purifier, macaroni-processing equipment, and drying cabinet. The colour of the product depends on the processing and can be used to measure variations in this. Colour is measured spectrophotometrically in terms of % brightness, % purity, and dominant  $\lambda$ ; the relation of these factors to absorption, kneading, rest, and pressing is discussed. Colour can be used to differentiate wheat samples only if the processing is standardised; a tentative experimental technique is E. A. F. proposed.

Wooden vessels for milk transport. G. GENIN (Lait, 1936, 16, 841-846).—The staves of wooden vessels used for milk transport do not rise above 17% moisture content (original, air-dry, 8-11%) after being in contact with milk. Unpainted staves after washing with H<sub>2</sub>O alone contain 14-15% of moisture. The construction of such vessels must be such as to afford the means of keeping the insides scrupulously clean. Detergents have little wearing effect on the wood surface. After a short use the tannin of wood has no effect on the taste of milk, and generally the odour, taste, and colour of milk are unaffected. W. L. D.

Effect of alkali carbonates added to milk on the preparation of the calcium chloride serum. A. TAPERNOUX (Lait, 1936, 16, 832-840).-0.6 ml. of 20% CaCl<sub>2</sub> solution coagulated 100 ml. of milk on boiling. Addition of 0.25% of Na<sub>2</sub>CO<sub>3</sub> gave a cloudy serum. To produce a cloudy serum on adding 1.5 ml. of CaCl<sub>2</sub> to 100 ml. of milk, 2.0% of Na<sub>2</sub>CO<sub>3</sub> is needed. Li<sub>2</sub>CO<sub>3</sub> and (NH<sub>4</sub>)<sub>4</sub>H<sub>2</sub>(CO<sub>3</sub>)<sub>3</sub> act similarly. The decrease in coagulating power is due to the pptn. of Ca<sup>--</sup> as CaCO<sub>3</sub>, the effect being quant. Milks which have had developed

lactic acidity neutralised by alkali carbonate behave normally towards  $CaCl_2$  precipitant. Addition of dichromate and formol to milk has no effect on the behaviour of alkalinised milk towards  $CaCl_2$ . W. L. D.

Comparison of methods for determination of added water in milk. H. MOHLER and H. FORSTER (Mitt. Lebensm. Hyg., 1936, 27, 113-124).-Methods compared depend on determinations of fat- or fat- and sugar-free dry solids, n,  $\Lambda$ , f.-p. depression (I), and mol. consts. The formula given for the  $\Lambda$  method takes into account the effects of skimming and of spontaneous development of acidity, and has a mean error of 0.5%. The formula  $100(33\cdot 1-M)/M$ , where M = $R = 15 \cdot 0 + 2 \cdot 2x + 2 \cdot 4y$  gives the most satisfactory results, R being the n of the  $Ca(NO_3)_2$  serum (cf. following abstract), and x and y, respectively, the Cl' and sol. P205 contents as determined by the Ryan-Pyne method (B., 1935, 77). The St. Gallen modification of this method serves only as a sorting test, whilst the original Mathieu-Ferré method (mol. consts.) is the least satisfactory of the three. The bearing on the above methods of Wiegner's rule is discussed. J. G.

Determination of added water in cream. H. MOHLER and H. FORSTER (Mitt. Lebensm. Hyg., 1936, 27, 125—130).—Formulæ are given for the calculation of added H<sub>2</sub>O in cream, using the method already described (preceding abstract); the serum is produced by treating 60 c.c. of sample at 35—40° with 0.5 c.c. of CaCl<sub>2</sub> [or 47% Ca(NO<sub>3</sub>)<sub>2</sub>,4H<sub>2</sub>O] solution, and after shaking and heating on the water-bath for 20 min., cooling, and decanting through a filter. The *n* of cream is usually much > (and never <) that of the corresponding milk or skim milk. J. G.

Citric acid in milk and its determination. L. H. LAMPITT and H. S. ROOKE (Analyst, 1936, **61**, 654—665). —Of numerous methods tested, not all are applicable without previous removal of lactose. In the modifications of Denigès' method, numerical factors proposed by various authors are not applicable to all concess of citric acid (I), and some are incorrect.  $COMe_2$  methods are not trustworthy. A modified procedure for the pentabromoacetone (II) method is recommended, which gives results < 2 mg. low for > 0.11 g. of (I) in presence of milk-serum. The solubility of (II) is 5 mg. per 100 c.c. of reaction liquid at 0°. E. C. S.

Determination of the original f.p. of sour milk. H. J. EVANS (Analyst, 1936, 61, 666-670).—An analysis of the milk is made, and the % of lactose (I) in the fresh milk is calc. by one of two methods (cf. Thorpe, B., 1905, 513; A., 1906, ii, 813). The wt. of sour milk corresponding to 50 ml. of the original milk is ashed by a special method designed to give a final correct acidity. The ash and the (I) (calc. wt.) are dissolved in H<sub>2</sub>O, the solution is adjusted to 50 ml., and its f.p. is determined. In 16 milks allowed to sour, this differed from that of the original milk by  $\gg 0.02^\circ$ , and in most instances by  $\gg 0.01^\circ$ . E. C. S.

Agreement in the analysis of composite and oneday samples of milk. D. MEADE and J. N. LECKIE (Milk Plant Month., 1936, 25, No. 8, 28-30).—The average fat content of three 1-day samples per month was higher than the composite of daily samples for 10-day periods. The 1-day samples showed more variation. Considerable variation occurs in the milk of single herds. W. L. D.

Cheese-yielding capacity of milk. F. H. McDowall (New Zealand Dept. Sci. Ind. Res., Bull. 50, 137-364 ; Dairy Res. Inst. [N.Z.], Publ. 72).-An equitable system of payment for milk for cheesemaking is possible only on the cheese-yielding capacity of milk deduced from vals. for fat and casein content. A comparison of the accuracy of the Walker and A.O.A.C. methods for determining casein and total protein vals. used in conjunction with the Babcock method for fat determination as a method for estimating the cheese-yielding capacity of milk is described. Methods of expressing cheese yield are discussed; various factors influencing the composition of milk and the factors associated with cheese manufacture and ripening which affect cheese yield are described. Sampling of milk and the accuracy of the methods of determination of fat and casein are discussed. Tables are given showing average yields of cheese per lb. of fat for milks of casein/fat ratios of 0.51-0.80. The results fall uniformly on a straight line expressed by the equation : yield of cheese per 100 lb. of milk =  $2.08 \times \text{casein}$  (Walker) +  $1.19 \times$ fat, or  $2.42 \times \text{casein}$  (A.O.A.C.) +  $0.98 \times \text{fat}$ ; or  $1.67 \times \text{total protein} + 1.14 \times \text{fat.}$  Yields of green cheese have also been calc. on similar lines. To avoid the tendency to produce cheese having < 50% of fat in the dry matter, allowance should not be made for milks having a casein/fat ratio > 0.72. The Walker casein/total protein ratio averaged 0.772 (0.770-0.775) and the A.O.A.C. casein/total protein ratio 0.767 (0.733 -0.784). Cheese yields per 100 lb. of milk, on a " casein + fat " basis of calculation, are represented by : (Walker casein)  $1.33 \times (\text{casein} + \text{fat}) + 1.42$ ; (A.O.A.C. casein)  $1.20 \times (\text{casein} + \text{fat}) + 2.29$ . Cheese yield is discussed from the viewpoint of fat content, and the vals. for Walker and A.O.A.C. caseins, total protein, fat, and cheese yield are related to each other. A costed cheese system of payment is described. W. L. D.

Cream quality. A. M. STIRLING (New Zealand J. Agric., 1936, 52, Reprint, 321—330; Dairy Res. Inst. [N.Z.], Publ. 81).—Common faults in New Zealand cream for butter manufacture are: food flavours, mostly that of land-cress; tallowy flavour, from metallic contamination; high acidity and high bacterial count (Breed). Lack of facilities for cream cooling was the chief factor for deterioration of quality. W. L. D.

Cheesy flavours in unsalted butter. H. MACY and E. D. HERREID (Canad. Dairy & Ice Cream J., 1936, 15, No. 10, 65-67).—Several types of bacteria found in some samples of fresh cream are responsible for Cheddar cheese flavour in butter. Starter cultures exert a protective effect against the taint, but pasteurisation is the only effective way of treatment of fresh cream.

W. L. D.

Determination of the butter fat contents of edible fats and of foodstuffs in general. T. VON FELLENBERG (Mitt. Lebensm. Hyg., 1936, 27, 133-157).-Two determinations using Wiegner's method of fractional distillation (B., 1922, 606 A) showed that the sol, fatty acids of coconut fat (I) consist chiefly of octoic acid (68.0 and 63.9%), with decoic acid (29.0 and 33.4%), AcOH (2.5 and 2.4%), and CH<sub>2</sub>O (0.5 and 0.3%); those from butter fat (II) contained only hexoic acid and AcOH. A semi-micro modification of the Kuhlmann and Grossfeld method for the determination of (II) from the PrCO<sub>2</sub>H val. (B., 1926, 447) involves fractional distillation, the first fraction being used to eliminate the error due to (I) and the second to calculate (II). The error for a wide range of mixtures of lard with coconut fat and/or (II) is normally  $\pm 1.0\%$  (2.6—3.4%) in a few cases). J. G.

Cheese-ripening studies. (A) Influence of yeast extract on types of streptococci found in starters. B. A. EAGLES, O. OKULITCH, and A. G. CAMPBELL. (B) Influence of configurational relations of the hexoses on sugar-fermenting abilities of lactic acid streptococci. O. OKULITCH and B. A. EAGLES (Canad. J. Res., 1936, 14, B, 311-319, 320-324).---(A) The distribution of the lactic acid organisms propagated in milk enriched with 0.15% yeast extract was markedly different from that of organisms in milk alone. Yeast extract favours the development of a larger proportion of slow, acid-producing strains which are able to ferment sources of C more complex than hexoses. Some new strains of lactic acid streptococci are described.

(B) Transfers of Strep. cremoris into sucrose, maltose, dextrin, and starch did not influence its acid-producing ability, but glucose, mannose, fructose, and salicin caused a marked inhibiting effect on acid production in milk and lactose broth. Galactose and lactose showed a less inhibiting effect. The inhibitory effect of glucose or some of its metabolic products is suggested as the cause of loss of vitality in starters. W. L. D.

Chemistry of Cheddar cheese-making. IV. Lactose and lactic acid in whey and curd ; presence of bound water in curd ; existence of a Donnan equilibrium between curd and whey ; rate of penetration of salt into curd. F. H. McDowall and R. M. DOLBY (J. Dairy Res., 1936, 7, 156-173; cf. B., 1936, 759).-The distribution of lactose (I) and lactic acid (II) in whey, curd, and curd juice is examined together with the effect of NaCl thereon. Conversion of (I) into (II) by starter organisms is almost quant. At the period when cheese is normally hooped very little salt has penetrated into the centre of the curd, but within 12 hr. of hooping, salt distribution is substantially uniform. Bound H<sub>2</sub>O probably exists in curd and a Donnan equilibrium controls the partition of electrolytes between curd and whey. A. G. P.

Cataract-preventing vitamin (flavin) in cheese. P. L. DAY and W. J. DARBY (Food Res., 1936, 1, 349– 355).—American, Swiss, and cream cheese contained 2, 1.5, and 0.36 Bourquin–Sherman units per g. It is calc. that 1 unit of  $-B_2 \equiv 0.004$  mg. of flavin. E.C.S.

Lacticinia. Manufacture of "ricotta." P. ROCHA FILHO (Bol. Min. Agric., Brasil, 1936, 25, 137— 139).—The manufacture of "ricotta" (an Italian product) from the residual whey from Brazilian cheesemaking is advocated. L. A. O'N.

Tubercle bacillus in milk, butter, and white cheese. W. SARNOWIEC (Compt. rend. Soc. Biol., 1936, 123, 370-372). H. G. R.

Gelling of casein. W. KOPACZEWSKI (Lait, 1936, 16, 801—810).—Na caseinate (I) solutions form gels with a large no. of reagents. The gelling action is additive, depending on the gelling power of the acid or base. Certain salts will accelerate the coagulation of acid or alkaline solutions of casein. The rate of coagulation is a function of the concn. of the coagulating reagent. The rate of syneresis of such gels is variable. Addition of CaCl<sub>2</sub> or lactic acid to (I) causes local coagulation, around the surface of the drops, which can be dispersed on shaking. The coagulum obtained by addition of Ca salts can be broken up by shaking, but it again solidifies on setting aside without shaking (thixotropy). The following ions which cannot alone coagulate milk accelerate the coagulation by other ions : Cl, Br, SO<sub>4</sub>, PO<sub>4</sub>, Mg, Cd, La, Ce. The ions Ba, Cu, Fe, Al, and Sn retard the rate of coagulation, whilst Pb, U, and Th have no effect. Blood anticoagulants (NaF, citrate) have no anticoagulant effect when lactic acid is added to casein solutions. W. L. D.

Dried egg-white. A. K. BALLS and T. L. SWENSON (Food Res., 1936, 1, 319-324).—The fermentation necessary to liquefy the thick white before drying is accelerated by addition of trypsin. If it is carried out at 4-6° little bacterial action occurs. In consequence, the product has a higher sugar content than is usual. E. C. S.

Controlling the ripening of the Japanese persimmon. J. V. RAKITIN (Compt. rend. Acad. Sci. U.R.S.S., 1936, 3, 229—234).—Stimulated ripening, as judged by formation of EtOH in the fruit, is merely an acceleration of the natural process of decay. Marked stimulative effects were produced by injection of 12% aq. EtOH and 1% aq. acid, by mechanical injury (incision, squeezing), and by immersion in  $H_2O$  at 30—35°.

A. G. P.

Preservation of grape juice. IV. Pasteurisation of juices or musts prepared from several varieties of grapes. C. S. PEDERSON, E. A. BEAVENS, and H. E. GORESLINE (Food Res., 1936, 1, 325–335; cf. B., 1936, 856).—The nos. of micro-organisms in musts from six varieties of grapes were sufficiently reduced for fermentation with a pure-culture inoculum of yeast by heating to  $54\cdot4-60^\circ$ . In the juices, all organisms were killed at  $73\cdot9^\circ$ , except in that from Californian Muscat grapes, in which bacteria were present after heating to  $76\cdot7^\circ$ . In presence of small amounts of SO<sub>2</sub> the temp. required to kill the organisms was slightly, but not materially, reduced.

E. C. S.

Nitrogenous constituents of dried apricots during browning. C. C. BEDFORD (Food Res., 1936, 1, 337– 339).—Changes in H<sub>2</sub>O-sol. N are slight. Some properties of the brown pigment, which in the crude form contains approx. 1% N, are described. E. C. S.

Determination of boric acid in dried fruit. W. B. BROWN (Analyst, 1936, 61, 671-680).—A procedure is described for ashing this material and determining  $H_3BO_3$  by titration with NaOH in presence of mannitol between the end-points of "Sofnol No. 1" and phenolphthalein. The results of 41 determinations on various examples agreed to within 40 p.p.m. with those of Dodd (A., 1929, 362) for similar material. None of the samples contained > 250 p.p.m. E. C. S.

Effect of home canning and storage on the ascorbic acid content of tomatoes. E. P. DANIEL and M. B. RUTHERFORD (Food Res., 1936, 1, 341-347). -Losses ranged from 21 to 55% of the initial ascorbic acid. E. C. S.

Effect of stage of maturity of snap bean on its composition and use as a food product. C. W. CULPEPPER (Food Res., 1936, 1, 357-376).—The changes in total solids, sugars, N, and tannins of hulls and seeds are recorded. For table use, the best stage is at 20-25 days from flowering; for canning, at 15 days. E. C. S.

Factors influencing the vitamin-C content of vegetables. D. K. TRESSLER, G. L. MACK, and C. G. KING (Amer. J. Publ. Health, 1936, 26, 905-909; cf. B., 1936, 472).—The ascorbic acid (I) content of peas, snap beans, tomatoes, cabbage, and spinach is greatly influenced by the variety, soil, and growing conditions. In tomatoes it increases as they ripen, but in peas it decreases. Snap beans, peas, and spinach lose (I) at room temp., but only slowly at 1-3°, whilst the more acidic vegetables, *e.g.*, tomatoes and rhubarb, lose (I) very slowly even at room temp. C. J.

Isolation and identification of an anaërobic organism producing gas in boiled beef. L. S. McCLUNG and E. WHEATON (Food Res., 1936, 1, 307— 318).—*Clostridium welchii*, although killed during processing, developed sufficiently in the cans before cooking, to produce swells. E. C. S.

Salt curing of herring. G. A. REAY (J.S.C.I., 1936, 55, 309–315 T).—Experimental results show how interchange of salt and  $H_2O$  is affected by conce. of salt, mode of application of salt, and by size and composition of the herring. The salting of herring much more lightly than is now done commercially was investigated. The light cures stored at  $-2^{\circ}$  to  $-5^{\circ}$  differed from commercial cures in their diminished "saltiness," in their greater proportion of  $H_2O$ , and in their "cured" flavour and its slower development, but after some months were palatable, especially if made from rich, fatty fish. Their qualities are discussed in the light of current taste in salt herring.

Rice bran as food for dairy cows. T. BONADONNA (Lait, 1936, 16, 811-832).—The inclusion of 25% or 40% of rice bran in the concentrates fed to groups of dairy cows did not significantly affect milk or butter fat yield. A slight lowering in the amount of volatile fatty acids of the butter fat from cows fed on rice bran was observed. Practical results and considerable economy in cost of ration are grounds for recommending rice bran to be used up to 40% of the concentrate ration. W. L. D.

Nutrition of the bacon pig. I. Influence of high levels of protein intake on growth, conformation, and quality. H. E. WOODMAN, R. E. EVANS, and E. H. CALLOW [with J. WISHART] (J. Agric. Sci., 1936, 26, 546-619).—A detailed investigation of the effect of the protein level in the ration on live-wt. increases, carcase quality, protein utilisation, and bacon grade is described. A. G. P.

Fluorine in animal nutrition. C. H. KICK, R. M. BETHKE, B. H. EDGINGTON, O. H. M. WILDER, P. R. RECORD, W. WILDER, T. J. HILL, and S. W. CHASE (Ohio Agric. Exp. Sta. Bull., 1935, No. 558, 77 pp.).-The toxicity of F compounds to pigs, rats, and chicks was in the descending order, NaF (I), rock phosphate (II), phosphatic limestone, superphosphate, CaF, (III). Rations containing excessive F reduced growth and feed consumption and, in the case of pigs, increased the food requirement per unit gain in wt. Small amounts of F thickened but weakened bones, increased their Mg and F contents, and decreased the CO<sub>3</sub>" content without affecting the total ash, Ca, or P. In pigs excessive F softened the teeth, caused hypoplasia of the enamel, increased the % F, but did not alter the % ash, Ca, Mg, P, or  $CO_3''$ . Effects of F on various other organs are recorded. Rats retained F from (I), (II), and Na<sub>2</sub>SiF<sub>6</sub>, but not from (III). A. G. P.

Acid ensilage of pressed beet slices of high sugar content. A. H. ERDENBRECHER and W. DÖRFELDT (Z. Wirts. Zuckerind., 1936, 86, 289-321).-Trials in earth-covered trenches and in small experimental vessels indicated that so-called sugar press-slices (from the process of G.P. 609,808) containing about 26% of dry matter, of which nearly half is sugar, yield a product superior in appearance and odour to that from ordinary exhausted beet slices containing about 17% of dry matter and 0.6% of sugar. The latter became pappy and their non-sugars underwent profound decomp. The sugar press-slices preserved their shape; acidification was mainly at the expense of the sugar, and ceased before much decomp. of non-sugars had occurred, provided air was not admitted. Close packing and effective exclusion of air are necessary to restrict losses of dry matter; earth silos are not suitable. Addition of salts did not reduce losses, but addition of lactic bacteria and lactic acid proved beneficial. The nutritive val. of the product has not yet been determined. J. H. L.

Nutritive value of the protein of Cajanus indicus. C. D. MILLER and R. C. ROBBINS (J. Agric. Res., 1936, 53, 281—293).—When fed to rats in proportions to give 18% of the protein of the diet pigeon-pea seed was inadequate for normal growth and reproduction. When fed at a level calc. to provide 8% of the protein, addition of cystine did not materially improve the growth, but did so when the meal provided 11% of the protein. The first limiting growth factor of the prepared peaglobulins was tryptophan. A. G. P.

Analysis of edible fats.—See XII. Determining starch in bread etc. Starch in potato flakes. Thermophilic contamination.—See XVII. Fodder yeast.—See XVIII. Oligodynamic effect of metals. —See XXIII.

See also A., Nov., 1405, Rennet test for milk. 1428–31, Vitamins.

## PATENTS,

Manufacture of mill products for alimentary purposes and of paste goods and baked products from such milled products. M. BIENENSTOCK, L. CSÁKI, J. PLESS, A. SAGI, and E. SAGI (U.S.P. 2,025,705, 31.12.35. Appl., 15.8.32. Hung., 31.8.31).—A flour rich in gluten-protein is obtained from the germ of seed kernels of the carob tree (*Ceratonia siliqua*) or of certain *Cercis* sp. Denaturation of the protein during milling must be avoided and the formation of a green coloration during wet separation of the germ is prevented by addition of citric acid. The gluten-rich flour may be employed for a variety of purposes by diluting with a cereal or root starch, bran, casein, etc. Alternatively, the swelling power may be reduced by addition of suitable salts. E. B. H.

Preparation of bread and other baked cereal foodstuffs. E. I. DU PONT DE NEMOURS & Co. (B.P. 454,513, 1.4.35. U.S., 31.3.34).—Cake batters containing an initial  $[H_2O_2]$  of about 1 wt.-% of the flour and subjected to a slow baking process may contain  $\geq 0.5\%$  of undecomposed  $H_2O_2$  when placed in the oven. E. B. H.

Deodoriser [for dairy products]. F. G. CORNELL, JUN. (U.S.P. 2,025,196, 24.12.35. Appl., 9.1.33).— An apparatus is described in which the dairy liquid (milk, cream, etc.) is subjected to vac. treatment and is then discharged at high velocity as a thin stream into which steam is injected. It is claimed that both volatile and non-volatile substances are thereby removed. E. B. H.

Manufacture of margarine. B. R. HARRIS (U.S.P. 2,024,355, 17.12.35. Appl., 14.8.33).—A small amount (e.g., 0.5 - 1.5%) of the hydrophilic material obtained by partly saponifying a fat with alkali, mixing the product to a paste with H<sub>2</sub>O, and finally decomp. the greater part of the soap formed with an innocuous acid (e.g., HC), lactic acid, H<sub>3</sub>PO<sub>4</sub>) is incorporated in margarine to improve the texture etc. and to reduce the tendency to "weep." E. L.

Manufacturing of margarine. A. D. JOVCE, Assr. to DURKEE FAMOUS FOODS, INC. (U.S.P. 2,024,647, 17.12.35. Appl., 5.5.31).—The molten, blended margarine fats (without any pretreatment with milk etc.) are chilled so as to obtain a supercooled, non-cryst., plastic "base" which keeps well and may be worked up with the requisite milk component to form a margarine at any convenient time. E. L.

Production of easily soluble pudding preparations. A. VAN DANTZIG (B.P. 455,261, 10.3.36).—A prepared blanc-mange or custard mix which can be directly dissolved in hot milk is made by mixing fondant, "drop" sugar, custard powder, and fruit paste or other flavouring. A paste or hard form of product is obtained by varying the amount of H<sub>2</sub>O incorporated during mixing. E. B. H.

Seasoning means or condiment. E. BRUST (U.S.P. 2,021,403, 19.11.35. Appl., 18.5.34).—Finely-granulated citric acid is heated with 3 times its wt. of table salt to  $100^{\circ}$  with continual stirring, until all the salt has been "wetted" by the acid. The mass is cooled slowly, the stirring being continued. Into 85—90 pts. of the mixture are stirred powdered paprika (3.5), white pepper (2.5), and granulated sugar (6 pts.). The product is non-hygroscopic. E. B. H.

Manufacture of solidified honey. V. J. HAMPTON (U.S.P. 2,021,450, 19.11.35. Appl., 21.11.32).—A hard,

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CL. XX.-MEDICINAL SUBSTANCES; ESSENTIAL OILS.

candy-like product containing < 2% of  $H_2O$  is prepared from honey by concn. under partial vac. at  $< 85^{\circ}$ , the resulting syrup being allowed to solidify in a dry atm. E. B. H.

Manufacture of soluble coffee. W. A. HEYMAN (U.S.P. 2,022,467, 26.11.35. Appl., 8.1.35).—Perforated containers, *e.g.*, cheese cloth or metal gauze, are filled with granules of roasted coffee and of dried coffee extract in the proportions of 4:1. The extract is rapidly sol. and gives body and colour, whilst the granules supply fresh coffee flavour and aroma. E. B. H.

Manufacture of caffeine-free coffee. J. MacLANG, Assr. to COFFEX A.-G. (U.S.P. 2,023,333, 3.12.35. Appl., 22.3.33. Switz., 4.4.32).—Raw coffee is autoclaved with 16% of H<sub>2</sub>O and extracted with preheated  $(CH_2Cl)_2$ . The solvent is removed with a non-aq. fluid under pressure at a temp. > the b.p. of the solvent. The coffee is then dried under vac. E. B. H.

Temperature-measuring appliance for hot fluid masses [of chocolate]. O. SCHRAMM (U.S.P. 2,025,617, 24.12.35. Appl., 12.10.33. Ger., 17.10.32).—In a chocolate-tempering machine containing a screw conveyor (C), a ring shaped, heat-absorbing element is built into the casing of C and connected with a recording thermometer. A. R. P.

Treatment of chocolate or other semi-liquid or plastic substances. C. B. K. Boggild, M. JACOBSEN, and K. C. S. AASTED (B.P. 454,610, 31.7.35).—Air is used as extracting agent during conching or like process, by circulating the chocolate, at one part of the cycle beating air into it and at another part breaking the emulsion by application of vac. B. M. V.

Confections. B. R. HARRIS (U.S.P. 2,025,985–6, 31.12.35. Appl., [A] 12.2.34, [B] 3.4.35).—Reduction of  $\eta$ , stability of emulsions, etc. in confections such as chocolate or toffee are brought about by addition of  $\frac{1}{2}$ -3% of the product obtained by treating a fatty acid ester of glycerin possessing at least one free OH (e.g., cottonseed oil, cacao butter, oleic acid, monostearin, etc. heated with glycerin) with P<sub>2</sub>O<sub>5</sub>. E. B. H.

Production of [candy] confections. B. R. HARRIS (U.S.P. 2,024,356, 17.12.35. Appl., 24.2.34).—Compounds formed between lypophilic substances (fatty acids or alcohols) and hydrophilic substances (glycerols, polyglycerols, sugars, etc.) are used as emulsifying agents in toffee etc. and to reduce the  $\eta$  of chocolate for enrobing. Examples are monostearylglycerol, the monocetyl ether of glycerol, alkyl derivatives of sugars, etc. E. B. H.

Production of lecithin from vegetable raw materials. S. O. SORENSEN and G. F. BEAL, ASSTS. to AMER. LECITHIN CO. (U.S.P. 2,024,398, 17.12.35. Appl., 4.12.34).—The extraction of undesirable bitter principles is avoided by extracting soya beans with  $C_6H_{14}$  (or other hydrocarbon of b.p.  $< 90^{\circ}$ ) in place of the usual EtOH-benzol mixture. The need for further purification is avoided. E. B. H.

Flavouring material. A. K. EPSTEIN and B. R. HARRIS (U.S.P. 2,023,877, 10.12.32. Appl., 14.12.32).— A butter-like flavour is given to foodstuffs by small additions of  $\alpha$ -diketones with 4—8 C (cf. U.S.P. 1,945,345 —7; B., 1934, 1035, 940), e.g., 5 c.c. of 2% aq. COAcEt to 100 lb. of margarine, Ac<sub>2</sub>-COMeEt or -COAcPr<sup>a</sup> (12:1) for flour, or 0.1% of Ac<sub>2</sub>-COAcEt (3:1), Ac<sub>2</sub>-COAcEt-COAcPr<sup>a</sup> (4:2:1), or Ac<sub>2</sub>-COAcPr<sup>a</sup> (10:3) for shortening. R. S. C.

Vitamins in oils.-See XII.

XX.-MEDICINAL SUBSTANCES ; ESSENTIAL OILS.

Nitrate content of tobacco. A. WENUSCH and E. MOLINARI (Pharm. Zentr., 1936, 77, 689—690; cf. B., 1935, 381).—Oriental tobaccos do not contain nitrates, either before or after fermentation. E. H. S.

Lead content of chewing tobaccos and snuffs. C. C. CASSIL and C. M. SMITH (Amer. J. Publ. Health, 1936, 26, 901-904).—A method for the determination of Pb in tobacco is described which yields a recovery of approx. 90%. Chewing tobaccos examined contained  $3\cdot5$ -85 $\cdot$ 0 p.p.m. of Pb and  $4\cdot1$ -36 $\cdot$ 0 p.p.m. of As<sub>2</sub>O<sub>3</sub>, whilst samples of snuff contained  $12\cdot0$ -131 $\cdot0$  p.p.m. of Pb and  $4\cdot9$ -51 $\cdot0$  p.p.m. of As<sub>2</sub>O<sub>3</sub> which were probably derived from Pb arsenate used as an insecticide on the growing plant. C. J.

Microchemical identification of ephedrine and ephetonine. G. A. W. J. O. E. PARIS (Pharm. Weekblad, 1936, 73, 1526–1527).—dl- and l-Ephedrine hydrochloride give characteristic ppts., which are readily differentiated, with  $K_2C_2O_4$  and  $Na_3VO_4$ . S. C.

Flowers and leaves of Malvae sylvestris, subsp. Mauritanicae, Thellung. E. WALDSTÄTTEN (Sci. Pharm., 1935, 6, 39–41).—The colour intensity and  $\eta$  of the flowers are considerably > those of wild forms of mallow. The leaf drug of the subsp. is, in point of  $\eta$ , = that of mallow leaves hitherto used. CH. ABS. (p)

Bioassays of rodenticides. J. C. MUNCH, F. E. GARLOUGH, and J. C. WARD (J. Amer. Pharm. Assoc., 1936, 25, 744—746).—Analytical data often afford no criterion of the toxicity of animal poisons (e.g., red squill,  $Tl_2SO_4$ ,  $Zn_3P_2$ ); bioassays are then essential, as exemplified by "chemically-pure" strychnine. F. O. H.

Theory of distillation as applied to essential oils. VI. A. L. BLOOMFIELD (Perf. Essent. Oil Rec., 1936, 27, 404—405; cf. B., 1936, 1128).—The distillation of insol. and partly sol. substances in steam and superheated steam is discussed. E. H. S.

Clove tree and its products. I. Determination of essential oil in cloves etc. L. W. RAYMOND (Perf. Essent. Oil Rec., 1936, 27, 393–396).—A laboratory apparatus for the distillation of essential oil-bearing materials, either by boiling with  $H_2O$  or by low-pressure steam, and with or without cohobation, is described. The yields of oil obtained are in good agreement with those obtained in commercial practice, and this is exemplified by the distillations of several samples of cloves, nutmegs, and mace. E. H. S.

True and poisonous star anise. M. WAGENAAR (Pharm. Weekblad, 1936, 73, 1490-1502).—Illicium verum can be differentiated from *I. religiosum* by various botanical differences and by colour reactions with conc. HCl and B-C10H7 OH, FeCl3, NH2Ph dyes, and particularly conc. H2SO4 on an EtOH or COMe, extract. S. C.

## Flavin in cheese.-See XIX.

See also A., Nov., 1359, Determination of EtOH. 1374, Substituted hydroxybenzyl alcohols. 1377, Derivatives of  $\beta$ -amino- $\alpha$ -hydroxy- $\alpha$ -phenylpropionic acid. 1381, Constituents of pyrethrum flowers. 1382, Sexual hormones. Constituents of the adrenal gland. 1389, Urethanes as local anæsthetics. 1394-5, Alkaloids and derivatives. 1398, Identification of vohimbine. 1423, Diphtheria toxin. 1424, Concn. and partial purification of bacteriophage. 1427, "International standard" œstrin preps. Isolation of œstrogenic substance of liquor folliculi. 1428, Concn. and purification of gonadotropic substance in urine. Extraction of cryst. folliculin. 1428-31, Vitamins. 1435, Chinese drugs (Tu Hao; Hseh Tuang seed). Brazilian pipi.

#### PATENTS.

Recovery of hormones. A. J. H. KONGSTED and H. L. P. KJEMS (B.P. 454,778, 5.5.36) .- Aq. liquids containing hormones are treated (at  $p_{\rm H}$  3-7) with a solution of a ferri- or ferro-cyanide and a solution of a metallic salt which forms an insol. ferri- or ferro-cyanide, and the adsorbed hormone is recovered from the ppt. E. H. S.

Synthetic preparation of  $\beta$ - (or 10-)hydroxycamphor. (A, B) ZAIDAN HOJIN RIKAGAKU KENKYUJO, and (B) Y. SAHASHI, K. TAKEUCHI, T. SHIMAMOTO, and T. IKI (B.P. [A] 453,789, [B] 453,812, 29.4.35. [A] Japan, 17.5.34).—(A)  $\beta(10)$ -Bromo- or -chloro-camphor and KOAc in boiling AcOH give  $\beta(10)$ -(I), b.p. 128–130°/4–5 acetoxycamphor mm., hydrolysed to the alcohol (II), m.p. 215°,  $[\alpha]_{\rm p}$  +52° in EtOH, which with  $K_2Cr_2O_7$  gives ketopinic acid. (B)  $\alpha\beta(3:10)$ -Dibromocamphor and KOAc in boiling AcOH give α(3)-bromo-β(10)-acetoxycamphor, b.p. 160-165°/ 6-7 mm., which (a) with Zn dust-AcOH gives (I), or (b) is hydrolysed to  $\alpha(3)$ -bromo- $\beta(10)$ -hydroxycamphor, R. S. C. converted by Zn-AcOH into (II).

[Medicinal] solutions of alkoxyphenols. W. KROPP, Assr. to WINTHROP CHEM. Co., INC. (U.S.P. 2,025,869, 31.12.35. Appl., 7.2.35. Ger., 10.2.34).-The presence of 30-50% of 1-phenyl-2: 3-dimethyl-5pyrazolone-4-methylaminomethane-sulphonates or -sulphinates allows dissolution of 7-25% of alkoxyphenols in H<sub>2</sub>O. The resultant stable solutions are suitable for injection alone or with other drugs, e.g., anti-anæmic preps. Examples are: guaiacol (I) (15) or eugenol (10) in 50% aq. Na 1-phenyl-2: 3-dimethyl-5-pyrazolone-4-methylaminomethanesulphonate (II) (100 pts.); (II) (40) with (a) pyrogallol Me<sub>2</sub> or (b) Bu ether  $(7 \cdot 5)$ , (c) (I) (9), and ephedrine (1), or (d) (I) (10), quinine hydrochloride (5 g.), and aq. liver extract in 100 c.c. of solution. R. S. C.

Production of guaiacol compounds. S. ROSEN-ZWEIG (U.S.P. 2,023,551, 10.12.35. Appl., 11.10.34. Austr., 28.6.33).—Substances for use in pharmacology are prepared by interaction of mono- or di- (I) -guaiacyl phosphate (prepared from guaiacol and POCl<sub>a</sub>), or a salt with an org. base, e.g., quinine, (CH<sub>2</sub>·NH<sub>2</sub>)<sub>2</sub> (II), N(C<sub>2</sub>H<sub>4</sub>·OH)<sub>3</sub> (III). The substance, C<sub>30</sub>H<sub>38</sub>O<sub>12</sub>N<sub>2</sub>P<sub>2</sub>, from (I) and (II) has m.p. 135° and the substance from (I) and (III), m.p. 100°. P. G. C.

Preparation of alkali and alkaline-earth salts of sugar phosphoric acid esters. S. FUNAOKA (U.S.P. 2,024,036, 10.12.35. Appl., 4.9.34. Jap., 26.6.34) .--The prep. of Ca fructose phosphate,  $[\alpha]_D = -23 \cdot 9^\circ$  in H<sub>2</sub>O, from fructose and POCl<sub>2</sub> and its purification are detailed. Thus obtained it is suitable for subcutaneous or intravenous injection in 5-10-c.c. doses of 5% aq. solution. Other sugars may be used. R. S. C.

(A)  $\alpha$ -p-Anisyl- $\beta$ -methyl-, (B)  $\alpha$ -p-hydroxyphenyl- $\beta$ -methyl-, (c)  $\alpha$ -p-hydroxyphenyl- $\beta$ -dimethyl-[p-anisylisopropylmethylamine aminopropane etc.] and its acid addition salts. G. A. ALLES (U.S.P. 2,015,578-80, 24.9.35. Appl., 21.4.33).-(A) B-p-Anisylisopropylamine gives a benzylidene derivative, b.p. 190-195°/10-15 mm., the glassy methiodide of which with HCl-aq. MeOH at 100° gives β-p-anisylisopropylmethylamine (I), b.p. 133°/15 mm. (hydrochloride, m.p. 176-177°; hydrobromide, m.p. 148-149°; H oxalate, m.p. 94°). (B) (I) and boiling 64% HBr give β-p-hydroxyphenylisopropylmethylamine (II), m.p. 161-162° [sulphate, m.p.  $> 285^{\circ}$ ; H (?  $H_2$ ) phosphate, m.p. 190°; H tartrate, m.p. 122—124°], which resembles ephedrine in pharmacological action. (c) (I) and MeI (1 mol.) give a mixture of quaternary *iodide*, cryst., and  $\beta$ -p-anisylisopropyldimethylamine, b.p. 121°/15 mm. (hydrochloride, m.p. 160—162°; hydrobromide, m.p. 143—145°), which with hot 57% HI yields  $\beta$ -p-hydroxyphenylisopropyldimethylamine, m.p. 133-134° [phosphate, m.p. 210°-212°; also obtained from (II)], which, like (I), resembles hordenine physiologically. R. S. C.

Manufacture of esters of polycyclic alcohols. SCHERING-KAHLBAUM A.-G. (B.P. 455,018-9, 8.4.35. Addns. to B.P. 454,632; B., 1936, 1178).-(A) Esters of unsaturated polycyclic alcohols similarly constituted to androsterone are reduced to the corresponding saturated alcohol esters. The examples describe the hydrogenation of dehydroandrosterone propionate (Pt) and benzoate (Ni) to the propionate and hexahydrobenzoate of androstanediol (I). (B) Diesters of (I) are obtained by treating saturated or unsaturated hydroxyketones or diketones of the cyclopentanopolyhydrophenanthrene series with reducing agents (as loc. cit.) and acylating agents (Ac<sub>2</sub>O). The examples describe the prep. of the diacetate, m.p. 159-160°, of (I) from trans-androsterone, androstanedione, and androstenedione.

Production of acyl compounds of polycyclic alcohols of the cyclopentanopolyhydrophenanthrene series. W. P. WILLIAMS. From SCHERING-KAHLBAUM A.-G. (B.P. 454,790, 4.3.35) .- Acyl derivatives of unsaturated cyclopentanophenanthradiols (B.P. 452,716; B., 1936, 1178) are hydrogenated. Thus 3-acetoxy-17-methoxy- and -17-benzoyloxy-androstene afford (PtO<sub>2</sub>; AcOH) the corresponding androstane deriv-R. S. C. atives.

Manufacture of cholenic acid or its lower homologues and their derivatives. A. CARPMAEL. From SCHERING-KAHLBAUM A.-G. (B.P. 453,773, 14.3.35).— Sterols containing at least one double linking in the nucleus are oxidised (CrO<sub>3</sub>-AcOH), preferably after protection of the double linking and OH. *E.g.*, cholesteryl acetate dibromide is oxidised and the product separated as a difficultly H<sub>2</sub>O-sol. Na salt; this is reduced (Zn dust-AcOH) to 3-acetoxycholenic acid, m.p. 183—184° (*Me* ester, m.p. 154—155°; free OH-acid, m.p. 232°). Separation as Ba salts, and conversion into Me esters gives also 3-acetoxynorcholenic acid (*Me* ester, m.p. 125—127°; free OH-acid, m.p. 240—242°), also obtained by CrO<sub>3</sub> oxidation of phytosteryl acetate dibromide. Similarly, from stigmasteryl acetate dibromide there is obtained 3-hydroxybisnorcholenic acid, m.p. 295—302°. H. A. P.

Method of purifying and decolorising cincophen. E. F. GRETHER, Assr. to Dow CHEM. Co. (U.S.P. 2,018,354, 22.10.35. Appl., 28.4.32).—Cincophen is obtained in a dense, permanently colourless form by crystallisation (charcoal) of the  $\rm NH_4$  salt from  $\rm H_2O$  and decomp. thereof by a fatty acid  $\gg C_8$ . R. S. C.

Purification of neoarsphenamine [neosalvarsan]. G. W. RAIZISS and A. I. KREMENS, ASSTS. to ABBOTT LABS. (U.S.P. 2,020,655, 12.11.35. Appl., 19.2.31).— The purity of the neosalvarsan obtained is improved if 7% of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> (or NaHSO<sub>3</sub>) is added to the aq. OH·CH<sub>2</sub>·O·SO<sub>3</sub>Na before the condensation. It probably functions by removal of the CH<sub>2</sub>O present and formed. R. S. C.

Aryl mercuric heterocyclic carboxylates. C. N. ANDERSON, ASST. to LEVER BROS. CO. (U.S.P. 2,022,997, 3.12.35. Appl., 9.1.35).—Salts, RCO<sub>2</sub>HgAr, and similar polycarboxylates are claimed as potent antiseptics of low toxicity. The following are described : *phenylmercury furoate*, m.p. 115°, 2-*phenylquinoline-4-carboxylate*, m.p. 206—208°, *nicotinate*, m.p. 193—195°, 2-*phenyl-6methylquinoline-4-carboxylate*, m.p. 162—163°, and *coumarinearboxylate*, m.p. 205—206°, and *di(phenylmercury) pyridine-2*: 3-*dicarboxylate*, m.p. 163—165°. R. S. C.

Manufacture of complex double compounds of organic heavy-metal mercapto [thiol] compounds. SCHERING-KAHLBAUM A.-G. (B.P. 454,244, 26.3.35. Ger., 29.3.34. Addn. to B.P. 398,020; B., 1933, 988).— Heavy-metal compounds of glutathione (I) or their heavy-metal salts are caused to interact with org. or inorg. compounds having only one SH, or derived salts. *Compounds* are described from Na<sub>2</sub> auroglutathione and Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, Na 1-thioglucose, and Na glutathione (II), from Na auroglutathione and (I), and from Na cuproglutathione and (II). The products are stated to be effective in combating infectious diseases. H. A. P.

Production of ethereal sulphur-containing compounds. F. BOEDECKER (U.S.P. 2,021,539, 19.11.35. Appl., 30.9.33. Ger., 4.7.32).—Essential oils containing S compounds, or the disulphide fractions thereof, with deoxy- (I) or *apo*-cholic acid (II) form odourless cryst. compounds, which with warm dil. NaOH regenerate the components. (I) with asant oil in EtOH gives a *compound*, m.p. about 167—168°, and with the fraction, b.p. 104—135°/11 mm., of asafœtida oil gives a *compound*, m.p. 168—170°. (II) with mustard-seed oil in EtOH gives a compound, m.p. 166-169°. The products are of therapeutic val. R. S. C.

Preparation of aminobenzthiazole compounds [local anæsthetics]. M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 2,019,529, 5.11.35. Appl., 10.9.31).—The following are prepared by standard methods : 1-amino-5-isobut-, m.p. 140° (hydrochloride, m.p. 171°), -5-isoamyl-, m.p. 122° (hydrochloride, m.p. 135°), -5-isoprop-, m.p. 135°, and -5-, m.p. 121°, and -3-n-but-oxyphenylbenzthiazole, m.p. 142—143° (hydrobromide, m.p. 166—167°). They are claimed to have greater anæsthetic action and less toxicity than cocaine. H. A. P.

Topical anæsthetic. S. QUISLING (U.S.P. 2,025,399, 24.12.35. Appl., 29.8.33).—An aq. or aq.-EtOH solution of guaiacol and Na salicylate and/or NaOBz is claimed. E. H. S.

Fungicide [against ringworm]. H. A. BRUSON, Assr. to RESINOUS PRODUCTS & CHEM. Co., INC. (U.S.P. 2,022,185, 26.11.35. Appl., 17.9.31).—The acids, 2:5- $OH \cdot C_6H_3(CRR'_2) \cdot CO_2H$ , in which R' = alkyl and R = Hor alkyl, their Na or K salts or esters are toxic as dusts or ointments against parasites, particularly those of the ringworm type. The acids with  $CRR'_2 = Pr^{\beta}$ , sec.-Bu, and Bu<sup> $\gamma$ </sup> are specifically claimed. R. S. C.

Pharmaceutical preparation and dentrifice. B. R. FAUNCE (U.S.P. 2,025,655, 24.12.35. Appl., 14.5.30).— The sterilised filtrate from a fully fermented mixture of animal bile and glycerin is used as a basis for tooth pastes. E. H. S.

Dentifrice products. T. GIBBS & Co. (B.P. 446,491 20.7.34. Fr., 9.1.34).—The use of finely-powdered mica (e.g., muscovite, lepidolite, or biotite) as an abrasive constituent is claimed. L. C. M.

Manufacture of dentifrice pastes. J. PARERA (B.P. 453,995, 6.2.36).—Transparent or translucent products are prepared from a mixture of cacao-butter soap, glycerin, and  $H_2O$  with addition of aromatic, antiseptic, and colouring materials. D. M. M.

Face and toilet creams. TOKALON, LTD. From TOKALON SOC. ANON. (B.P. 453,440, 12.3.35).--KOH,  $N(C_2H_4 \cdot OH)_3$ , and excess of stearic acid are caused to react either in intimate mixture or in solution. A preferred thickening agent is "starch glycerite." B. M. V.

Compositions [embrocation] for application to the human skin. U.S. INDUSTRIAL ALCOHOL CO. (B.P. 446,594, 31.12.34. U.S., 30.12.33).—Claim is made for thixotropic compositions consisting (approx.) of a 1:1 mixture of EtOH and  $Pr^{\rho}OH$  containing a gelforming agent (e.g., Na stearate)  $\gg$  3 and a gel modifier (e.g., lanoline, beeswax, paraffin or carnauba wax, coconut oil)  $\gg 4^{\circ}_{0}$ . L. C. M.

Coating composition for protecting the skin. O. McDANIEL, Assr. to MOUNTAIN VARNISH & COLOR WORKS, INC. (U.S.P. 2,021,131, 19.11.35. Appl., 22.4.32). —A composition which dries to a soft pliable film is prepared by stirring Na silicate (906) into a hot solution of Na stearate (288) in H<sub>2</sub>O (1600) and adding glycerol (1155 pts. by wt.). S. M.

Alkyl chlorodihydroxybenzenes.—See III. Vitamins in oils.—See XII. British Chemical Abstracts-B.

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# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Lime-fog and its obviation [in photography]. K. KIESER (Phot. Ind., 1936, 34, 1166—1167).—1—3 g. of  $(NaPO_3)_6$  (Calgon) are added to 1 litre of developer; all CaCO<sub>3</sub> deposition is thereby obviated. J. L.

See also A., Nov., 1348, Theory of desensitisation.

# PATENTS.

Production of light-sensitive diazo-type layers. J. HALDEN & Co., LTD., and J. HOLDEN (B.P. 454,665, 30.4.35).—Salts of type 1:3:6:4-OAr CH2 CO NH C6H2 (OAlk)2 N2X are stable, couple very rapidly, bleach permanently, give insol. dyes, and are thus excellent for diazo-type papers, especially those of B.P. 443,955 (B., 1936, 668). Pptn. of dyes in the coating solution may be retarded by addition of colloids. Large amounts of inorg. salts or of acid should be avoided. H<sub>3</sub>PO<sub>4</sub> is the preferred acid. Premature coupling may be prevented by interposition of a waterproofing layer between the diazonium salt and the second dye-component. Small amounts of accelerators may be added. Interaction of CH<sub>2</sub>Cl·CO<sub>2</sub>H first with p-cresol and then with 2:5-dimethoxyphenylene-1:4-diamine gives 4-p-tolyloxyacetamido-2:5-dimethoxyaniline; the diazonium salt (12-15 g.) thereof and H<sub>3</sub>PO<sub>4</sub> (15 g.) in 1 litre of  $H_2O$  are filtered and used to impregnate paper; the dried paper after exposure is developed by phloroglucinol (I) (3 g.) and Na<sub>2</sub>CO<sub>3</sub> (30 g.) in 1 litre of H<sub>2</sub>O, giving brown lines on a white ground. Alternatively, the dried coated paper is dusted with a mixture of dry soap (50 g.), MgO (5 g.), and 2:3-dihydroxynaphth-pphenetidide, which gives violet lines on a white ground. Paper is coated with a solution of the diazonium salt (40 g.) from 4-phenoxyacetamido-2: 5-diethoxyaniline and H<sub>3</sub>PO<sub>4</sub> (10 c.c.) in 1 litre of H<sub>2</sub>O, dried, coated with a 5% solution of paraffin wax in petroleum, dried, and then powdered with a mixture of powdered soap (200), (I) (20), Na lauryl sulphate (20), and French chalk (80 g.). R. S. C.

Light-sensitive layers incorporating diazonium compounds. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 454,814, 7.10.35. Ger., 10.10.34).-Yellowing of finished prints from diazonium-coated, "self-colouring" papers (i.e., paper which is developed by exposure to steam) is avoided by incorporation of a constituent which removes the excess of alkali and forms an insol. hydroxide. Paper, painted with a 1% aq. solution of the ZnCl<sub>2</sub> double salt (I) of diphenyl-4-diazonium chloride and dried, is rubbed with a mixture of (a) solid phloroglucinol (II) and Ca silicate or (b)  $\beta$ -C<sub>10</sub>H<sub>7</sub>·OH and Na aluminate with or without anhyd. Na<sub>2</sub>CO<sub>3</sub>. Paper, painted with a 1% aq. solution of  $NMe_2 \cdot C_6 H_4 \cdot N_2 \cdot BF_4$ and dried, is rubbed with  $Al_2(SO_4)_3$ , CaO (or  $Na_2CO_3$ ), and  $\beta$ -C<sub>10</sub>H<sub>7</sub>·OH; alternatively, the Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> is added to the aq. solution. Paper dusted with a mixture of Na<sub>2</sub>CO<sub>3</sub> (150), Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> (250), talc (100), (II) (50), and (I) gives after exposure a dark blue image with steam. R. S. C.

Projection screens and allied methods. D. F. NEWMAN, ASST. to TRANS-LUX DAYLIGHT PICTURE SCREEN CORP. (U.S.P. 2,024,382, 17.12.35. Appl., 23.10.30. Renewed 28.5.35. Cf. B.P. 376,801; B., 1932, 1056).—A projection screen, preferably for use by projection of pictures from behind the screen, is composed of homogeneous cellulose acetate material, an agent imparting flexibility to the film (e.g., Ph<sub>3</sub>PO<sub>4</sub>, castor oil), a light-diffusing substance (e.g., ZnO, TiO<sub>2</sub>, etc.), and, if desired, a light-filtering agent (e.g., Co-blue). The sheet is prepared by dissolving the materials, some preferably separately, in solvents (e.g., COMe<sub>2</sub> and Et lactate) to obtain a viscous mass, and then casting the sheet in the normal manner. Alternatively, the lightdiffusing agent may be sprayed on the surface of the screen after casting; the surface of the screen may also be roughened or corrugated if desired. J. L.

Photographic developers. A. C. BANFIELD, and BAIRD TELEVISION, LTD. (B.P. 454,794, 5.4.35).—A rapid developer, which also completely hardens the film developed, is composed of  $o - C_6 H_4(OH)_2$  (I), or, preferably, a mixture of (I) and quinol containing  $\leq$  one third (I), with KOH and  $\leq 1\%$  of CH<sub>2</sub>O. The developer is suitably kept in two solutions, one of the (I) and a preservative (K<sub>2</sub>S<sub>2</sub>O<sub>5</sub>), and the other of the NaOH and CH<sub>2</sub>O. This developer does not cause fogging.

J. L.

Photographic developing. GEVAERT PHOTO PROD-UCTEN N.V. (B.P. 454,870, 8.4.35. Austr., 7.4.34) .--4-Keto-2-thio-5-arylidenetetrahydrothiazoles, not containing a basic substituent in the Ph, prevent fogging of deteriorated Ag emulsions when incorporated in the developer at 1:5000-100,000 (20,000 preferred) or as a layer behind the Ag. The colour produced is yellow but is bleached by time or immersion in aq. alkaline  $Na_2SO_3$ ; the  $Na_2SO_3$  may be added to the developer. Fogging does not occur even at 25° or on longer development. 4-Keto-2-thio-5-benzylidenetetrahydrothiazole, cryst., is obtained by refluxing PhCHO (1 mol.) and 4-keto-2-thiotetrahydrothiazole (1 mol.) in AcOH (10 mols.) for 5 hr. Compounds with basic substituents in the Ph react at the CS with alkaline Ag solutions, which may account for their unsuitability for use.

R. S. C.

Art of printing. V. DIETZ, Assr. to E. B. ELLIOTT and M. C. BOYD (U.S.P. 2,023,669, 10.12.35. Appl., 6.1.33. Ger., 11.10.32) .- Prints are made from photographic negatives, stencils, etc. on to dichromated gelatin layers. The unhardened parts are removed by washing in H<sub>2</sub>O, and the film is then dried. The dry film is soaked in an ink-repellent mixture which also swells unhardened (unexposed) parts, e.g., H<sub>2</sub>O 24, glycerin 75, and ox gall 1%, conc. to 75% of solids, together with 2% of NaHCO<sub>3</sub> or 0.25% of  $p-C_6H_4(NH_2)_2$ . The surface of the swollen print is then dried, and inked ; the printing ink also contains ox gall (e.g., 12%) to aid the repulsion of ink from unhardened areas. Prints can then be impressed from the inked surface. Other bile salts than ox gall may be employed; the Na glycocholate is more effective than the Na taurocholate in ox gall. Heavy pressure is not necessary for printing from such inked surface-dried layers, and large nos. of high-quality prints are obtainable. J. L.

Process and solution for treating photographic images. K. C. D. HICKMAN and W. J. WEYERTS, Assrs.

to EASTMAN KODAK CO. (U.S.P. 2,024,644, 17.12.35. Appl., 11.6.32).—A Ag<sub>2</sub>S image is intensified in a bath of a Ag salt (e.g., AgNO<sub>3</sub>), Na<sub>2</sub>SO<sub>3</sub>, and NaHSO<sub>3</sub>, with simultaneous exposure to light; other reducers than Na<sub>2</sub>SO<sub>3</sub> may be used, or a solution of AgNO<sub>2</sub> in NaNO<sub>2</sub>, without a reducing agent, may be employed. If certain compounds providing labile S are incorporated, e.g., alkali thionates, S:CMe·NH<sub>2</sub>, etc., the intensification may proceed in the dark. The Ag<sub>2</sub>S image is preferably formed from the original Ag image in two stages, first bleaching with a powerful oxidising solution relatively poor in Ag precipitant, and then sulphiding in a bath of  $Na_2S$  and a solvent for AgBr, e.g.,  $Na_2S_2O_3$ , thus forming a fine-grained  $Ag_2S$  image. Various intensifying formulæ are given, together with details of the results obtainable; in general, only significant portions of the tonal scale are intensified, and the lower fog densities are unaffected. The intensification effected may be very great, without staining the photograph. Other simple insol. Ag deposits, e.g., AgI, Ag<sub>2</sub>Se, can also be intensified. J. L.

Colour photography. KODAK, LTD. From EAST-MAN KODAK Co. (B.P. [A, B] 454,498-9 and [C] 454,622, 29.3.35. [A] Addn. to B.P. 427,518 and [B] to B.P. 427,520; B., 1935, 703).-(A, B) The loading agents used in the parent patents may include substances which either produce an anti-swelling action on the gelatin or increase the  $\eta$  of the treatment bath; the loading agents must be H<sub>2</sub>O-sol. Sp. substances claimed are: sugar and/or NaCl, higher alcohols, cellulose derivatives, ketones, alkyl- or alkoxy-amines which may be acylated, polyhydric alcohols (e.g., glycols, glycerol), colloids (e.g., gelatin, gum arabic), and high concns. of  $K_2SO_4$  or other inert salts. (c) The process of B.P. 427,518 (loc. cit.) is modified in that the developing bath, containing a loading agent, may be a colour-developing bath; this bath, and the fixing or toning baths, may contain the new loading agents J. L. described above.

**Production of multicolour photographic pictures.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. [A] 454,788 and [B] 454,842, 5.1.35).—(A) Multilayer material is prepared from three superimposed emulsions sensitised to blue, green, and red light, respectively, without any filter layers in between. The photographic layers are prepared with fine-grained emulsions rich in Ag, which it is claimed can be predominantly sensitised to the required colours. Yellow dye may, however, be incorporated to damp the blue sensitivity. (B) The above principle is applied to a bipack consisting of a lenticular film, carrying an emulsion sensitised to blue and green, and an adjacent red-sensitive emulsion on a smooth film. J. L.

 $C_5H_5N$  etc. derivatives.—See III. Ag from  $M_2S_2O_3$  solutions.—See VII.

# XXII.-EXPLOSIVES ; MATCHES.

#### PATENTS.

Purification of organic explosive compounds. W. A. SMITH, ASST. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,024,396, 17.12.35. Appl., 29.4.32).—The compound is cryst. from a neutral org. solvent, consisting of a higher aliphatic alcohol [amyl alcohol,  $(CH_2 OH)_2$ , glycerol], or a compound having a keto-linking (COMeEt, COEt<sub>2</sub>, furfuraldehyde), or a base such as an ethanolamine or  $C_5H_5N$ , the solvent in each case being miscible with  $H_2O$ , having b.p. > 100°, and not giving rise to inflammable vapours. W. J. W.

[Preparation of] lead salts of [3:5-]dinitrosalicylic acid [detonators]. W. BRÜN, Assr. to REMINGTON ARMS CO., INC. (U.S.P. 2,021,497, 19.11.35. Appl., 7.11.30). — The salts 2:3:5:1-Pb[ $O \cdot C_6 H_2(NO_2)_2 \cdot CO_2$ ](I) and -Pb[OH· $C_6 H_2(NO_2)_2 \cdot CO_2$ ]<sub>2</sub> [? containing a little (I)] are described. H. A. P.

Manufacture of [explosive] copper ammonium salts of diazoaminotetrazole. W. BRÜN, Assr. to REMINGTON ARMS Co., INC. (U.S.P. 2,021,478, 19.11.35. Appl., 14.12.32. Cf. U.S.P. 2,001,299; B., 1936, 718).— A Cu<sup>II</sup> salt (in  $H_2O$ ) is added (dropwise) to a solution of the Na salt of diazoaminotetrazole (I) containing NH<sub>3</sub> and AcOH; alternatively, a solution of a salt of (I) is added to cuprammonium acetate, or vice versa. H. A. P.

Smokeless powder. W. DE C. CRATER, ASST. to HERCULES POWDER CO. (U.S.P. 2,024,128, 10.12.35. Appl., 1.6.33).—Nitrated flaked starch (N  $\leq$  10.28%), retaining its original flaked form, is colloided in the cold with a solution of a nitroaromatic compound in nitroglycerin, the mixture being heated at about 57° for 30—40 min. W. J. W.

## XXIII.—SANITATION; WATER PURIFICATION.

Industrial poisoning. W. BRANDT (Chem.-Ztg., 1936, 60, 901-905, 924-925).--A review.

Skin troubles from [contact with] solvents. Köllen (Farben-Ztg., 1936, 41, 1085—1086).—A general dissertation is given on the occurrence of eczema etc. on the skins of workers using paint, solvents, etc.

S. S. W.

Ceramic filter media and high rates of filtration [of sewage]. M. LEVINE, R. LUEBBERS, W. E. GALLI-GAN, and R. VAUGHN (Sewage Works J., 1936, 8, 701-727).-Experimental percolation filters employing granite, ceramic Raschig rings, or a special perforated block as media were used to treat domestic sewage at rates of  $(2-16) \times 10^6$  gals./acre/day. At the highest rate 43-49% reductions of biochemical O2 demand were obtained, and this was increased to 78-89% by sedimentation of the effluents. The proportion removed by post-sedimentation increased with the rate of filtration, probably largely due to the biophysical and biochemical transformation of colloidal and other sewage constituents into readily settleable material which was flushed out of the filter. The biochemical O2 removal per acre-foot of filter by filtration alone rose from 350—420 lb. at the 2  $\times$  10<sup>6</sup>-gal. rate to 2180—2560 lb. at the  $16 \times 10^6$ -gal. rate. C. J.

Disposal of garbage in the sewerage system. L. V. CARPENTER, A. C. ROGEL, and B. GRABOIS (Sewage Works J., 1936, 8, 728-741).—Ground garbage can be disposed of with sewage sludge either in Imhoff tanks or in separate digestion units. The gas produced is similar to that obtained from sewage solids in quantity, composition, and thermal val. In comparing costs with other garbage-disposal systems the increased size of digestion tank and subsequent purification plant must be considered. C. J.

Comparison of sewage purification by compressed air and mechanically aërated activated sludge. I. Purification and sludge-settling characteristics. G. M. RIDENOUR and C. N. HENDERson (Sewage Works J., 1936, 8, 766-779).-The Kessener brush aërator has been compared with compressed air on a practical scale and under otherwise identical conditions. The purification obtained was the same when operating with > 3000 p.p.m. of activated sludge in the aëration chamber. When < 3000 p.p.m. were present the mechanical plant removed less suspended matter than the other, in proportion to the decreasing amounts of sludge, but this difference was not reflected in the biochemical O2 demand removal efficiencies. The settling and compacting ability of the sludge floc was three times as great in the compressed air plant as that of the sludge formed by the brushes.

C. J.

Swimming-pool sanitation control. W. S. JOHNson (Amer. J. Publ. Health, 1936, 26, 928–929).— Results obtained from a large no. of samples covering a period of two years indicate the importance of the immediate dechlorination of samples taken for bacteriological examination and also of maintaining a min. of 0.4-0.7 p.p.m. of residual Cl<sub>2</sub> in the pool-H<sub>2</sub>O during use, depending on whether Cl<sub>2</sub> or NH<sub>3</sub>-Cl<sub>2</sub> treatment is used. C. J.

Removal of iron and manganese from humuscontaining waters in closed rapid filters. A. MARSCHNER (Gas- u. Wasserfach, 1936, 79, 818-820).— Trials have shown that three  $H_2O$  supplies of high humus content and containing Fe and Mn can be satisfactorily treated in a closed aëration and filtration system. The  $H_2O$  with compressed air is forced into a scrubber filled with 3.5-mm. coke, and filtration follows through graded gravel beds. Each  $H_2O$  has its own requirements as to velocity and air used, and those with the least Fe and Mn content, but higher humus, were most difficult to treat. C. I.

Hydrochemical analysis. II. Determination of oxygen dissolved in water. W. Ohle (Angew. Chem., 1936, 49, 778—784; cf. A., 1936, 577).—Methods of determining  $O_2$  dissolved in natural  $H_2O$  are critically reviewed. In presence of much org. matter, the Cldifference process alone is practicable; with less org. matter, the Br-difference process, Br-salicylic acid process, or Winkler CaOCl<sub>2</sub>-KCNS process may be used. For the fixation of the  $O_2$  in situ the carbonate method is recommended. J. S. A.

Oligodynamic effect of metals. C. P. CALLISTER (Soc. Chem. Ind. Victoria, 1935, 35, 1068–1091).— Using AgNO<sub>3</sub> as a source of Ag', heavy inoculations of *B. coli* in H<sub>2</sub>O containing 0.05 p.p.m. of Ag' are sterilised in 2 hr., but concns. > 10 p.p.m. are insufficient when the medium is milk. It appears, therefore, that Ag' has no preference for combination with bacterial protoplasm, but is strongly adsorbed by extra-cellular colloidal protein, and until the latter is saturated in this respect the Ag is not available for the destruction of bacteria. Fresh orange juice can be sterilised with 0.05 p.p.m. of Ag without resorting to pasteurisation, which impairs the flavour. The colloidal and suspended matters present in this case do not appear to be adsorbent of Ag. C. J.

**Erratum.**—On p. 861, col. 2, line 26 from bottom, for  $[C_2H_3Cl_3]$  read  $[C_2HCl_3]$ .

(CH<sub>2</sub>)<sub>2</sub>O-See III. Dairy wastes for grassland.--See XIX.

See also A., Nov., 1414, Formation of skin blisters in viscose workers. 1417, Poisoning by Be oxyfluoride vapours. Toxicity of dioxan.

## PATENTS.

Electro-filtration of air in rooms. J. T. SHEVLIN. From SIEMENS-LURGI-COTTRELL ELEKTROFILTER GES. F. FORSCHUNG U. PATENTVERWERTUNG (B.P. 453,998, 12.2.36).—Corona-effect pptn. and conditioning are combined in one apparatus. B. M. V.

Cosmetic preparations. W. W. GROVES, Assee. of I. G. FARBENIND. A.-G. (B.P. 454,970, 12.4.35. Ger., 21.4.34. Addn. to B.P. 433,142; B., 1935, 976).— Undecoic acid, or other fatty acid containing an odd no. ( $\lt$  7) of C atoms, is substituted for the Zn or Mg salts used in the prior patent. E. L.

Product for use as germicide, fungicide, or insecticide. J. HYMAN, Assr. to VELSICOL CORP. (U.S.P. 2,020,648, 12.11.35. Appl., 14.3.33).—The lowboiling portion of the polymerides obtained by action of catalysts on unrefined vapour-phase-cracked petroleum gives a peroxide-rich substance when air is blown through at 175° in presence of a siccative catalyst. This oxidised material has germicidal properties (PhOH coeff. 0.15). R. S. C.

Sewage treatment. A. C. DURDIN, JUN., Assr. to CHICAGO PUMP CO. (U.S.P. 2,024,986, 17.12.35. Appl., 5.5.32. Renewed 27.2.34).—A form of mechanically aërated activated-sludge plant is claimed in which the mixed liquor is drawn up a central tube and sprayed out on to a fixed platform by a centrally-placed, revolving impeller. The spray drips back into the body of the liquid and is recirculated. Beneath the platform is a quiescent zone in which the sludge separates and from which purified effluent decants over a peripheral cill. C. J.

Treatment of wet sewage sludge. H. J. STEHLI (U.S.P. 2,026,366, 31.12.35. Appl., 12.7.32).—The wet sludge is dewatered and broken into small pieces which are coated with dry material to prevent adhesion. It is dried by hot gases ( $\gg 107^{\circ}$ ) and heated to  $\ll 150^{\circ}$  to remove combustible products which would cause obnoxious odours. These products are used as a source of heat and the solid residue is incinerated without nuisance. C. J.

Purifying air.—See I. Cosmetic cream.—See XII.

