

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

SEPT. 29 and OCT. 6, 1933.\*

### I.—GENERAL; PLANT; MACHINERY.

**Thermodynamics of refrigerators.** E. RAUSCH (Woch. Brau., 1933, 50, 161—164).—The use of temp.—entropy, heat content—entropy, and heat content—pressure diagrams in controlling the operation of refrigerators using  $\text{NH}_3$ ,  $\text{SO}_2$ , or  $\text{CO}_2$  is discussed.

A. R. P.

**Centrifugal pumps for special purposes.** K. GRÜN (Chem. Fabr., 1933, 6, 325—328).—In the pumping of a hot or volatile liquid there is a max. lift on the suction side above which vapour is liberated in the pump, stopping pumping. Special cooling devices are indicated. For alkaline liquors cast Fe is the usual material; for strong acids Cr-Ni steels or Si-Fe. The design of glands is discussed. With increasing viscosity the capacity of the pump diminishes, and for such liquids as tar the centrifugal pump is not economical. C. I.

**Filter-press design and operation.** J. A. B. FORSTER (Proc. Austral. Inst. Min. Met., 1932, No. 88, 407—422).—The application of Underwood's equation (Trans. Inst. Chem. Eng., 1926, 4, 19) to certain filtration problems is discussed mathematically.

A. R. P.

**Evaporation and its applications to the concentration of industrial liquids.** E. LAFITTE (Mem. Soc. Ing. Civ. France, 1932, 85, 1258—1290).—The principles of operation of 8 types of evaporator are discussed and their use in the recovery of salts from mineral waters and in the sugar and fermentation industries is described.

A. R. P.

**Condensation and evaporation.** M. JAKOB (Z. Ver. deut. Ing., 1932, 76, 1161—1170; Chem. Zentr., 1933, i, 744).—The direction and velocity of  $\text{H}_2\text{O}$  vapour determine whether condensation occurs as a liquid film or as drops. Small velocity and a direction perpendicular to the cooling surface (I) produce condensation in drops, whilst strong movement parallel to (I) favours film formation. The connexion between superheating at the heating surface and the course of evaporation has been investigated kinematographically.

L. S. T.

**Theory of the depth of the evaporation surface in the drying of flat bodies.** A. V. LUIKOV and A. G. KOLESNIKOV (J. Tech. Phys., Russia, 1932, 2, 708—725).—The inner and outer diffusion processes are considered separately.

CH. ABS.

**Rectification.** W. H. KEESOM (Chem. Weekblad, 1933, 30, 398—401, 550—554; cf. A., 1933, 772).—The graphical determination of the irreversible work of separation and the calculation of the conditions for min. energy consumption are described. Further consideration is given to the question of energy consumption. The separation of  $\text{N}_2$  and  $\text{O}_2$  from air by means of

Linde's apparatus is described, and the application of the graphical treatment previously described is given.

H. F. G.

**Kinematic viscosity and conventional viscosity units.** F. H. GARNER and C. I. KELLY (Petroleum, 1933, 29, No. 28, 1—12; cf. B., 1933, 287).—The advantages of expressing coeffs. of viscosity in c.g.s. units of kinematic viscosity (stokes), and the relation between these units and the conventional units, e.g., Redwood or Saybolt sec., or Engler degrees, are discussed. The relation can be expressed by the formula  $\nu = At - B/t$ , where  $\nu$  is the kinematic viscosity in centistokes,  $t$  the viscosity in any of the above conventional units, and  $A$  and  $B$  are consts., although a single formula of such type does not suffice to cover the whole of the desired range of viscosity. Thus the vals. of the consts. for the standard Redwood No. 1 viscosimeter, determined by measurements with 16 oils of viscosities from 43.9 to 2286 Redwood sec., are  $A = 0.260$ ,  $B = 179$  for the range 34—100 Redwood sec., and  $A = 0.247$ ,  $B = 50$  for the range above 100 Redwood sec. The influence of temp. on the vals. of  $A$  and  $B$  is discussed.

A. B. M.

**Viscosimeter recording c.g.s. units.** L. STEINER (Centr. Zuckerind., 1932, 57, 879—880).—Equal vols. of the liquid under examination are made to flow during a unit of time through tubes of a certain diam., the pressure produced in doing so being a measure of the viscosity at the temp. chosen. An instrument constructed on this principle has been found useful in giving information on the relative viscosity vals. of different molasses in their transport.

J. P. O.

**Preventing fog in distillation.**—See II. **Boiler plates.**—See X. **Viscosity, elasticity, and plastic strength.**—See XIX.

See also A., Aug., 781, **Desiccating agents.** 787, **Rates of dissolution of solid into liquid, and of sublimation.** 800, **Const.-temp. apparatus.**

### PATENTS.

**Kilns and similar apparatus for heat-treatment of materials.** H. E. WORWOOD (B.P. 392,717, 31.3.32).—In a tower ( $A$ ) the material is supported on a no. of shelves extending from opposite walls alternately and passes downwards from shelf to shelf. At an intermediate point a combustion space is formed to which fluid fuel is admitted, the air for combustion being preheated in the lower part of  $A$ . Progression of the material is effected by tilting  $A$ , it being supported by large rings and rollers.

B. M. V.

**Muffle kiln.** L. W. MANION (U.S.P. 1,886,491, 8.11.32. Appl., 7.10.31).—A pair of semicircular muffles

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



are contained in a circular kiln, the exhaust flue being below the diametral space. B. M. V.

**Tunnel ovens.** J. WEDGWOOD & SONS, LTD., and N. WILSON (B.P. 392,839, 15.6.32).—Combustion chambers for a kiln as described in B.P. 390,358 (B., 1933, 447) are formed so that the flame cannot pass directly from the burners into the kiln. The walls of the tunnel in the combustion zone converge towards the chimney so as to equalise the heat in the naturally cooler parts. Automatic regulation of the fuel is provided, but a const. min. is in any event allowed to pass. B. M. V.

**Heating of muffles, retorts, and the like.** F. BREDEL, Assr. to T. D. MILLER (U.S.P. 1,885,583, 1.11.32. Appl., 9.5.30).—The heating walls for tall ovens, retorts, etc. are divided into two levels for primary and secondary combustion respectively. There are twice as many primary flues ( $A_1$ ,  $A_2$ ) as secondary flues ( $B$ );  $A_1$  are provided with too much gas and insufficient air and  $A_2$  vice versa, so that the combined streams give perfect combustion in  $B$ . B. M. V.

**Heat-energy system.** J. J. GREBE, Assr. to Dow CHEM. Co. (U.S.P. 1,889,586, 29.11.32. Appl., 26.1.27).—Steam at a very high temp. (1000°), generated without the use of steam or  $H_2O$  drums, is passed in heat-exchange relation with a large mass of high-boiling liquid, e.g.,  $Ph_2O$ , Hg, or mineral oil, at low pressure which forms a  $\pm$  reserve of heat. The higher-boiling liquid may also be separately heated and serve other useful purposes, e.g., jacketing the steam-using apparatus, reheating the steam between stages of a turbine, etc. B. M. V.

**Mercury vaporising apparatus [boiler].** J. J. GREBE, Assr. to Dow CHEM. Co. (U.S.P. 1,887,807, 15.11.32. Appl., 30.9.29).—Hg is vaporised by the condensation of Na operating in a closed circuit at 28 in. vac., at which pressure the b.p. is 648°. B. M. V.

**Heat-exchange apparatus of the tubular type.** F. HEPWORTH (B.P. 392,748, 9.6.32).—The outer fluid is caused to flow at least twice longitudinally, and the inlet (point of highest pressure) is connected to the outlet by means of a relief valve which opens in the event of the viscosity rising too high. B. M. V.

**Method of transferring heat.** J. F. WAIT, Assr. to SUN OIL Co. (U.S.P. 1,886,436, 8.11.32. Appl., 13.6.30).—Hg, or other metal which is monat. in the gaseous state, is boiled and the vapours are condensed in doing useful heating; the condensate is purified from oxide by skimming, and revaporised. B. M. V.

**[Catalytic] converter.** I. HECHENBLEIKNER and N. TITLSTAD, Assrs. to CHEM. CONSTRUCTION CORP. (U.S.P. 1,889,549, 29.11.32. Appl., 25.2.30).—A Pt-gauze basket is constructed with an imperforate bottom further protected by a  $SiO_2$  tile to prevent premature corrosion of that part. Other constructional details are described. B. M. V.

**Temperature control of exothermic reactions.** H. A. BOND, Assr. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,888,066, 15.11.32. Appl., 2.4.30).—The end only of the catalyst body is cooled, thus causing

the heat to be removed in lines parallel to the gas flow, and not transversely. B. M. V.

**Drying of any granular or like material.** W. L. PAYNTER, and FORTH ENGINE & MOTOR WORKS (NEW-CASTLE-UPON-TYNE), LTD. (B.P. 392,535, 16.11.31).—In an inclined rotary furnace an axial blast causes an indraught of furnace gases through slots in the cylindrical wall near the lower end, the slots being guarded by trailing inclined slats to prevent exit of material. Similar outlet apertures may be provided at the upper end. B. M. V.

**Dryer.** K. E. BELL, Assr. to A. C. LAWRENCE LEATHER Co. (U.S.P. 1,885,505, 1.11.32. Appl., 10.3.26. Renewed 23.8.29).—A tunnel dryer for hides etc., with transverse forced circulation of air, is described. Adjacent circulations are of opposite rotation. B. M. V.

**Manufacture of a drying agent.** W. A. HAMMOND (U.S.P. 1,887,349, 8.11.32. Appl., 30.4.31).— $CaSO_4 \cdot 2H_2O$  is calcined and reheated at 230–250° for 2 hr., sufficient practically to complete dehydration ( $\frac{1}{2}H_2O$ ). B. M. V.

**Thermophoric mixture.** T. P. BELL, Assr. to THERMOPHOR MANUFG. Co., INC. (U.S.P. 1,887,618, 15.11.32. Appl., 20.10.31).—The mixture comprises NaOAc 92, glycerin 3,  $Na_2SO_4 \cdot 10H_2O$  3, and  $Na_2SO_4$  2%. B. M. V.

**Heat application and equipment therefor.** W. A. DARRAH (U.S.P. 1,885,418, 1.11.32. Appl., 20.7.29).—A closed-circuit drying apparatus suitable for wallboard (etc.) is arranged so that circulating gases arrive in the drying chamber through adjustable nozzles in line with the spaces between the sheets. B. M. V.

**Indicating temperatures of moving bodies.** P. B. SCHUSTER, Assr. to MAGNETIC GAUGE Co. (U.S.P. 1,889,385, 29.11.32. Appl., 9.1.29).—Changes of temp. of, e.g., rubber sheet being rolled are indicated by a hollow roller pressed against the sheet, care being taken to avoid friction heat; the roller preferably contains Hg with a thermometer bulb dipping in it. B. M. V.

**Odorant for refrigeration.** A. D. MACALLUM, Assr. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,886,034, 1.11.32. Appl., 4.9.31).—A lachrymatory alkyl bromoacetate is added to inodorous refrigerants as a warning of leakage. B. M. V.

**Crushing mill concave holding means.** H. H. RUMPEL, Assr. to SMITH ENG. WORKS (U.S.P. 1,886,908, 8.11.32. Appl., 9.10.28).—Holding means for a gyratory crusher head are described. B. M. V.

**Crushing and grinding machine.** O. GAISER, Assr. to ALPINE A.-G. MACHINENFABR. & EISENGIESSEREI (U.S.P. 1,885,251, 1.11.32. Appl., 1.10.29).—The claims relate to lubrication and dust protection of bearings in machines of the disintegrator type. B. M. V.

**Apparatus for pulverising materials.** W. DE COURSEY (U.S.P. 1,889,436, 29.11.32. Appl., 23.10.26).—A grinding pan with air separator above is provided with a regulated feed device to maintain const. the depth of the material in the pan. B. M. V.



**Pulverising machine.** P. V. JALI (U.S.P. 1,886,103, 1.11.32. Appl., 1.7.31).—A feed shoot and closure is described. B. M. V.

**Pulveriser.** A. F. WILLIAMS, ASST. to WILLIAMS PATENT CRUSHER & PULVERIZER Co. (U.S.P. 1,887,366, 8.11.32. Appl., 14.4.32).—A disintegrator having ring-shaped hammers is described. B. M. V.

**Colloid mill.** W. OSTERMANN, ASST. to TRAVIS PROCESS CORP. (U.S.P. 1,885,283, 1.11.32. Appl., 6.5.24. Ger., 18.5.23).—The rotor (*A*) is conical and fluted, the stationary cone having corresponding flutes; the feed is to the smaller end of *A*, and a helical rotating blade is provided to assist the feed by centrifugal force. B. M. V.

**Screening and concentrating apparatus.** E. O'TOOLE (U.S.P. 1,888,636, 22.11.32. Appl., 5.2.29).—The material is fed on to a conical spreader and then passes over one or more pairs of inverted conical and upright conical screens, the different sizes being then passed over separate, very obtuse conical air-separating tables, all the above being superposed in one cylindrical casing and oscillated as a whole. The air draught is produced by a suction fan and passes through both tables and screens. B. M. V.

**Apparatus for the wet screening or separation of intermixed materials.** J. A. FLINT and F. T. HAZELTINE, ASSTS. to TRAYLOR VIBRATOR Co. (U.S.P. 1,886,174, 1.11.32. Appl., 10.1.29).—A screen is placed in an inclined shallow box which is kept filled with  $H_2O$  and the whole vibrated. B. M. V.

**Gyro-separator.** E. P. HUMPHREY (U.S.P. 1,888,131, 15.11.32. Appl., 24.2.31).—The material to be classified is fed to the centre of a body of  $H_2O$  whirling spirally upwards, the lighter material being discharged over the edge of the vessel and the heavier working down below the rotating  $H_2O$ -supply jets. B. M. V.

**Filter press.** D. R. SPERRY, ASST. to D. R. SPERRY & Co. (U.S.P. 1,889,225, 29.11.32. Appl., 26.3.31).—A filter press of the circular recessed-plate type is formed of plates and frames, each frame being split into two narrower ones and two are semi-permanently attached to one plate with a pair of cloths between, forming a 5-ply sandwich, the rims of both the frames and the plate being larger than the cloths. In each cake space is a rotatable scraper loosely threaded on a square axial shaft, the frames being shaped to guide the scrapers close to but not touching the cloths during the discharging operation. B. M. V.

**Rotary filter press.** G. R. BRYANT, ASST. to H. VOGT MACHINE Co. (U.S.P. 1,887,798, 15.11.32. Appl., 2.8.30).—An intermittently operated pressure-disc filter is described. B. M. V.

**Continuous filtering apparatus.** H. C. HIRSCH (U.S.P. 1,887,129, 8.11.32. Appl., 13.1.30).—In a vac. filter the leaves are arranged as an overlapping circular series of louvres, sufficient space being left for washing sprays to reach all parts of the surface. B. M. V.

**Still.** H. I. LEA (U.S.P. 1,889,254, 29.11.32. Appl., 14.5.30).—In a still (*A*) heated by combustion, hot liquid descending from the heated upper part of *A* is caused to

evaporate further at the expense of its sensible heat by exposure on surfaces cooled by the air going to the heater, the air being thereby preheated. B. M. V.

**Plant or still for distilling water or other liquids.** E. A. J. KOCH (B.P. 392,851, 14.12.32).—The vapours from the still are cooled in 3 stages: (1) partial condensation by preheating the ingoing liquid to the b.p., (2) complete condensation by a separate cooling fluid, (3) removal of sensible heat from the distillate by initial preheating of the ingoing liquid. The heating medium of the still may be controlled by the temp. of the cooling fluid in (2). B. M. V.

**Distillation of liquids.** O. H. WURSTER (U.S.P. 1,886,256, 1.11.32. Appl., 13.2.28).—In a process of distillation utilising both vac. and the presence of steam to reduce the b.p. of the liquid, the steam is supplied superheated, the steam and vapours are condensed, and part is re-evaporated by exchange of heat with the first vapours; the second vapours are superheated (preferably by boiler steam) and used in the first still. The other part of the first condensate is evaporated by the boiler steam from the superheater. B. M. V.

**Fractional distillation.** W. G. WILLIAMS, ASST. to BYRNES, TOWNSEND, & POTTER (U.S.P. 1,886,142, 1.11.32. Appl., 6.6.29).—A no. of revolvable retorts are partly submerged in separate liquid baths and superposed above a common source of heat so that they attain graded temp. The liquid, *e.g.*, oil, is passed downwardly through them in series, each retort having an inlet and outlets for vapour and residue, respectively, the oil supply preferably being regulated to form a film only. B. M. V.

**Vapour purification process.** E. TRUOG (U.S.P. 1,889,617, 29.11.32. Appl., 17.8.29).—The liquid, *e.g.*, distilled  $H_2O$ , is manufactured by passing the vapours (*e.g.*, steam) in succession through dil. HCl, a jacket embracing charcoal, and through the C. B. M. V.

**Fractionating tower.** E. C. D'YARMETT, ASST. to FRACTIONATOR Co. (U.S.P. 1,888,872, 22.11.32. Appl., 3.9.29).—The tower (*A*) is provided with superposed centrifugal sprayers and with trays acting both as baffles for the upflow and collectors of the condensates, which are removed as they are formed. Any condensate in the vapour-outlet pipe is returned to *A*. B. M. V.

**Dephlegmation [tower].** L. C. HUFF, ASST. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,886,957, 8.11.32. Appl., 10.3.26).—In a tower the trays are fitted with combined up- and down-takes which provide a large irrigated surface over which the vapours (*e.g.*, from oil-cracking processes) are caused to swirl. B. M. V.

(A, B) **Treatment of emulsions, (B) mechanically.** G. H. MEINZER, ASST. to L. BLAKE-SMITH (U.S.P. 1,887,774—5, 15.11.32. Appl., 30.7.30).—Oil- $H_2O$  emulsions are broken, in (A), by passage through a granular solid which is retained in a tower, but may be allowed to agitate slightly; and, in (B), by passage between oppositely disposed surfaces of solid masses which are preferentially wetted by oil and  $H_2O$ , respectively. The distance between the surfaces is  $> 0.1$  in. and they are maintained in relative motion, *e.g.*, by making the slabs circular and rotating half of them. B. M. V.



**Apparatus for causing liquids to react with gases or vapours.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 392,631, 30.11.31. Addn. to B.P. 309,206; B., 1929, 459).—An apparatus on the principle of the prior patent comprises a no. of superposed chambers through which the general flow of the gas is downwards, but the transfer pipes passing through the partitions overlap and form liquid seals causing upward bubbling and preventing reflux of the liquid. Heating may be effected by internal coils or external liquid heaters connected to the reaction chambers by thermosiphon circulation. B. M. V.

**Air filter.** C. W. WILLIAMS (U.S.P. 1,886,927, 8.11.32. Appl., 9.9.27. Renewed 16.1.31).—A filter of the viscous-entrainment type has the irrigated surfaces coated with wood flour or other absorbent divided material bound with liquid bakelite. B. M. V.

**Cleaning of gases.** A. F. NESBIT (U.S.P. 1,888,606, 22.11.32. Appl., 27.4.31).—An electrostatic precipitator has the live electrodes shaped so as to form an electric wind towards the earthed electrodes. B. M. V.

**Gas cleaner.** J. L. MAUTHE (U.S.P. 1,888,603, 22.11.32. Appl., 27.10.31).—The gas is admitted tangentially into a cylindrical casing surrounding a cylindrical wetted screen, the cleaned gas being removed axially. B. M. V.

**Means for treating gases.** G. H. HORNE and M. A. LISSMAN, Assrs. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,886,548, 8.11.32. Appl., 22.4.29).—A cleaner of the type in which the gas is passed in natural helical paths down a no. of parallel vertical tubes is described. B. M. V.

(A) Dehydration or cooling of air or other gases.  
(B) Dehydration of air or the like and gaseous mixtures. (A) R. W. THOMAS, (B) R. W. THOMAS and G. G. OBERFELL, Assrs. to (A, B) PHILLIPS PETROLEUM Co. (U.S.P. 1,889,161—2, 29.11.32. Appl., [A] 29.8.29, [B] 11.5.29).—The  $H_2O$  is condensed out by cooling caused by the evaporation of a hydrocarbon which, in (A), is normally liquid and is mixed with air to form a combustible mixture, and, in (B), is normally gaseous but is liquefied at a v.p. of 25–500 lb./sq. in. at room temp. and is evaporated in surface heat exchange with the air and mixed with it after the  $H_2O$  has been drained away. B. M. V.

**Apparatus for compressing gases.** N. C. CHRISTENSEN (U.S.P. 1,886,164, 1.11.32. Appl., 18.3.30).—The gases are carried down by inverted cups on a vertical elevator chain to a considerable depth in a liquid and released under a hood. The compression is isothermal and the same apparatus may be used as a motor if supplied with compressed gas. B. M. V.

**Gas analyser.** W. KEMP (U.S.P. 1,888,229, 22.11.32. Appl., 4.12.31).—For the apparatus described in U.S.P. 1,727,544 (B., 1929, 965), a method of securing the diffusion plate in a fluid-tight manner and a ring of wax to prevent creeping of the absorption liquid on to the plate are described. B. M. V.

**Packing for the stuffing boxes and covers or lids of apparatus and machines subjected to a high gas pressure.** W. BAENSCH, Assr. to SCHERING-KAHL-

BAUM A.-G. (U.S.P. 1,887,006, 8.11.32. Appl., 23.1.28. Ger., 24.1.27).—The stuffing box of, e.g., an autoclave is extended downwards as a sleeve to below the liquid level so that the packed part of the stirring shaft is in contact with liquid, but not gas. If desired, there may be 3 concentric sleeves forming a treble seal of, in order, the liquid in the autoclave, an inert gas, and an inert liquid in contact with the gland. The shaft may be bored to supply the inert gas and liquid. B. M. V.

**Producing expansion-joint filling material.** J. S. HIPPLE (U.S.P. 1,888,270, 22.11.32. Appl., 17.3.31).—Vulcanisable vegetable oils 16, rosin 4, and "Hydrolene" 1 are mixed and liquefied by heat, then are added S 8 and  $SiO_2$  4 pts., and the final mixture is heated until vulcanised and cooled somewhat before pouring. B. M. V.

**Reagent feeder.** T. A. EVANS (U.S.P. 1,887,347, 8.11.32. Appl., 21.3.31).—A small bucket is caused to run up and down sloping guides dipping into a tank of the reagent. At the top of the run a fixed but adjustable plunger projects into the cup and spills any desired quantity. B. M. V.

[Pneumatic shaking-table for] separation of solid materials of different specific gravities. G. RAW (B.P. 392,578, 18.11.31).

**Dephlegmator.** R. E. NAGEL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,896,245, 7.2.33. Appl., 9.1.28).

**Catalysts.**—See VII. Heat-resistant alloys.—See X. Developing heat by electricity.—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Three coal-cleaning processes new to British practice.** D. T. DAVIES (J. Inst. Fuel, 1933, 6, 291–300).—A description of the Chance sand flotation process, the Elmore vac. flotation process, and the Slater air jig. C. E. M.

**Pulverised coal and colloidal fuel.** H. D. TOLLE-MACHE (Proc. S. Wales Inst. Eng., 1933, 49, 163–198).—An account is given of the use of pulverised coal as fuel. The difficulty of reducing coal to particles of colloidal size can be overcome by peptisation, e.g., by the physical action of a solvent such as  $C_5H_5N$ . Further research is necessary on stabilisation, gel formation, and peptisation of coal before the use of colloidal fuel is likely to become a commercial success. C. E. M.

**Recent developments in the utilisation of coal.** A. DAWE (J. Inst. Fuel, 1933, 6, 314–315).—A résumé of the advances that have been made in the use of coal and coal products as pulverised fuel, a suspension of coal in fuel oil, hydrogenation, coal gas for motor engines, and the products of high- and low-temp. carbonisation. C. E. M.

**Characteristics of pulverised fuels.** H. HEYWOOD (J. Inst. Fuel, 1933, 6, 333–335; cf. B., 1933, 416).—The factor sp. surface of particles is important both for grinding and combustion purposes; coarse particles should be eliminated from coal powder by modification of the separating system rather than by finer grinding. A means of measuring the actual diam. of a particle is described. C. E. M.



**Changes in the constitution and the caking power of coal during heating to the plastic stage.** K. BUNTE, H. BRÜCKNER, and H. G. SIMPSON (Fuel, 1933, 12, 222—232).—From a comparison of the relative amounts of the  $\alpha$ ,  $\beta$ , and  $\gamma$  fractions in a Saar gas coal before and after preheating it at 250°, 300°, 350°, or 400°, in N<sub>2</sub>, for 2 and 20 hr., respectively, it is concluded that the action of heat on the coal leads first to a polymerisation of the  $\beta$  and  $\gamma$  fractions at about 250°, with the formation of large mols. insol. in C<sub>5</sub>H<sub>5</sub>N. At higher temp. a resolution of the  $\alpha$  fraction takes place, yielding increased quantities of C<sub>5</sub>H<sub>5</sub>N-sol. matter, as a result of which the  $\beta$  and  $\gamma$  fraction contents of the coal increase, until these fractions begin to decompose, when the active decomp. stage of the coal sets in. Except for the coal preheated for 2 hr. at 250°, which had a higher caking index than the original coal, the caking index decreased with rising temp. and increased time of preheating; after 20 hr. at 350° or 2 hr. at 400° the caking index was nil. A study of the coking properties of several coals and of mixtures of the  $\alpha$ ,  $\beta$ , and  $\gamma$  fractions of one coal has indicated that although the  $\beta$  and  $\gamma_3$  fractions are responsible to a considerable degree for the ability of a coal to coke, these fractions do not exert their influence on the coking properties unless the  $\alpha$  fraction is present, whilst for the full development of the coking power of the coal it is necessary for all the fractions to be present since reactions between the fractions take place during the coking process.

A. B. M.

**Low-temperature carbonisation of coal in presence of Japanese acid clay.** I, II. K. KOBAYASHI, K. YAMAMOTO, and H. ISHIKAWA (J. Soc. Chem. Ind., Japan, 1933, 36, 427—432B, 432—434B).—I. Addition of Japanese acid clay resulted in a lowering of decomp. temp. (e.g., by 150°), formation of an oil of high light paraffin content and lower *d*, and a considerable increase in % neutral oil on total, though total oil yield decreased as the coal/clay ratio increased.

II. Distillation of the neutral oil gave 62 vol.-% of a light-coloured oil,  $d_4^{20}$  0.8215, b.p. < 200°, and 38% of a dark oil,  $d_4^{20}$  0.9352, b.p. > 200°. These fractions contained, respectively, phenolic oil 10.04, 7.45; basic oil 1.36, 1.6; unsaturated 10.48, 6.54; naphthenes 9.18, 7.48; paraffins 30.94, 9.61%.

V. P. P.

**Free-burning coke for domestic purposes.** J. ROBERTS (J. Inst. Fuel, 1933, 6, 338—339).—A reactive coke can be produced by reducing the time of coking and using a carbonising temp.  $\gtrsim$  800°. The carbonised residue from a highly oxidised non-coking coal is more reactive than that from a coking coal. Mixtures of feebly-coking with normal-coking coals carbonise readily, promote heat penetration, and enable the retort to be discharged before over-coking takes place. The cokes formed from these blends are more readily ignitable and more reactive than normal cokes in domestic grates. A blend of Durham coking and Yorkshire non-coking coal with the time of coking reduced 25% gave an excellent free-burning coke.

C. E. M.

**Production of high-temperature smokeless fuel in horizontal retorts for domestic purposes.** R. E. GIBSON (J. Inst. Fuel, 1933, 6, 339—341).—The

making of a high-temp. coke from a blend of a coking and a non-coking slack is described.

C. E. M.

**Characteristics of a satisfactory domestic coke.** R. A. MOTT (J. Inst. Fuel, 1933, 6, 336—337).—With a design of grate which allows a deep bed to be built up, produces a better draught, and allows easier access of air and discharge of ash than a normal coal grate, the high-temp. coke should be low in ash and moisture, closely sized, and well screened to remove dust and undersize and contain 50—55% of pores.

C. E. M.

**Use of coke for domestic purposes.** H. HOLLINGS (J. Inst. Fuel, 1933, 6, 337—338).—In the central-heating boiler the coke should be of larger size and mechanical strength so as not to be broken down in handling. The greater fuel bed makes the reactivity of the coke of less importance than in a domestic boiler, but to make the boiler efficient the rate of combustion should be controlled by regulating the rate of air supply to the furnace. In this way the formation of clinker is reduced and fuel economised.

C. E. M.

**Coke formation. IV. Temperature range of swelling of single coal particles. V. Assessing the value of laboratory cokes.** L. BURDEKIN and R. A. MOTT (Fuel, 1933, 12, 232—235, 236—239; cf. B., 1933, 610).—IV. The relation between the swelling of 1-in. cubes of coal on preheating to different temp. at a rate of 1°/min. and the amounts of oil distilled during the heating has been studied. Most of the oil obtainable from bright coal was distilled over the range of swelling of the single particles, and the two phenomena are therefore related. The correlation failed, however, in the case of dull hard coals. The expansion of single (1-in. cube) particles of bright and dull coals began at approx. the same temp. as the contraction of a column of fine particles of the same coal subjected to a load of 100 g., and ended (for bright coals) at approx. the temp. of final expansion of the column of particles under load.

V. A laboratory shatter test is described in which 2—10 g. of coke screened over 5-mesh are dropped thrice down a glass tube 3 in. in diam. and 3 ft. long, and the percentages remaining on 5-mesh (hardness index) and passing 60-mesh (abrasion index) are recorded. The variation of these indices with temp. of carbonisation (330—450°) of a coal has been studied. The results of this shatter test agree well with those of the laboratory abrasion test previously described (B., 1928, 916); the former is simpler and therefore to be preferred.

A. B. M.

**Coke formation. VI. Estimation of tar and liquor in the Gray-King assay.** W. BREWIN and R. A. MOTT (Fuel, 1933, 12, 239—242).—The separation of the tar and liquor is effected by washing out the U-tube repeatedly with small quantities of CHCl<sub>3</sub> in order to remove the tar. The residual CHCl<sub>3</sub> is then removed by heating the U-tube to 70° in an air-bath and passing a current of air through it for ½ hr.; the amount of H<sub>2</sub>O thereby vaporised and removed is inappreciable. The wt. of residual liquor is then obtained by direct weighing and the wt. of tar by difference.

A. B. M.



**Rôle of sulphur in destructive hydrogenation.** L. VON SZESZICH and R. HUPE (Brennstoff-Chem., 1933, 14, 221—225).—Experiments on the hydrogenation of a low-temp. tar and of a brown coal in a continuous plant, and of a brown-coal tar oil in a discontinuous plant, using in each case molybdic acid or Mo sulphide as catalyst, have shown that the addition of S (as S, H<sub>2</sub>S, or CS<sub>2</sub>) to the reaction mixture increased the yields of oils produced, particularly those of the lower-boiling oils. The yield of light oil from low-temp. tar passed through a max. and then decreased with increasing addition of S. Other experiments with Mo, W, Fe, Ni, and Co catalysts have shown that the addition of S leads to increased H absorption, increased reduction of tar acids, increased yield and improved quality of light oils, decreased gas and coke production, and increased life of the catalyst. A. B. M.

**Recovery and treatment of by-products of coal carbonisation.** C. BERTHELOT (Chim. et Ind., 1933, 30, 3—38).—A review of present practice in Europe and the United States.

**Preparation of active charcoal from olive press-cake.** L. BERMEJO and L. BLAS (Anal. Fis. Quím., 1933, 31, 65—67).—Olive press-cake distilled at 800°, (a) alone, or after impregnation with (b) CaCl<sub>2</sub> or (c) ZnCl<sub>2</sub>, yields charcoal with activity coeffs. (g. PhOH adsorbed from a 1% solution by 100 g.) of (a) 0.78, (b) 8.7, and (c) 12.3. R. K. C.

**Influence of the reactivity of coke on the behaviour of CO<sub>2</sub>: H<sub>2</sub> mixtures at 600—1200°.** P. DOLCH (Brennstoff-Chem., 1933, 14, 261—263).—The gas mixture (CO<sub>2</sub>: H<sub>2</sub> = 1:2) was passed through a 10-cm. layer of (a) high-temp. coke or (b) beechwood charcoal, of 2—3 mm. particle size, in a vertical, electrically-heated SiO<sub>2</sub> tube, at a rate of 12 litres/hr. Both (a) and (b) increased the rate of the reaction CO<sub>2</sub> + H<sub>2</sub> → CO + H<sub>2</sub>O. Equilibrium was not reached during passage of the gas over (a) even at 970°; above this temp. interaction of (a) with the gases began, but even at 1100° there were still appreciable quantities of steam in the efflux gases. Equilibrium in the gaseous reaction was reached over (b) at > 750°; above 820° the gases reacted with (b) and at about 1000° only CO and H<sub>2</sub> were produced. The results are briefly discussed in relation to gas-producer practice. A. B. M.

**Activity of nickel catalysts for the methane synthesis.** H. BRÜCKNER and G. JACOBUS (Brennstoff-Chem., 1933, 14, 265—268).—The life of a catalyst for the reaction CO + 3H<sub>2</sub> = CH<sub>4</sub> + H<sub>2</sub>O (at 300°) depends to a great extent on the amount of the formation of higher hydrocarbons; thus the addition of SiO<sub>2</sub> gel, which adsorbs these hydrocarbons, to the catalyst prolonged its life. The most effective catalysts were 90:10 mixtures of Ni with Al<sub>2</sub>O<sub>3</sub> or ThO<sub>2</sub>. Ni catalysts containing alkaline-earth oxides, when prepared by heating a mixture of the oxide with Ni(NO<sub>3</sub>)<sub>2</sub>, were also highly active. The activity of the catalysts depended greatly on the method of prep.; thus the Ni, Ni-alkaline-earth oxide, and Ni-ThO<sub>2</sub> catalysts were almost inactive when prepared from the corresponding hydroxides, whereas the Ni-Al<sub>2</sub>O<sub>3</sub> catalyst similarly prepared was highly active. The latter catalyst prepared from

Ni(NO<sub>3</sub>)<sub>2</sub> was inactive, whilst the corresponding Ni-ThO<sub>2</sub> catalyst was highly active. Addition of Co greatly diminished the activity of Ni, and the addition of Cu rendered it completely inactive. A. B. M.

**Determination of acetylene.** A. KRAUSS (Azetylen Wiss. Ind., 1932, 35, 73—75; Chem. Zentr., 1933, i, 819).—An apparatus for determining C<sub>2</sub>H<sub>2</sub> in welding gases is described. L. S. T.

**Determination of the air content of high-percentage acetylene.** M. KONSCHAK (Azetylen Wiss. Ind., 1932, 35, 118—122; Chem. Zentr., 1933, i, 819). L. S. T.

**Application of antioxygenic action to fire extinction. Extinction of flame.** C. DUFRAISSE, R. VIEILLEFOSSE, and J. LE BRAZ (Compt. rend., 1933, 197, 162—164; cf. B., 1932, 719).—The % of 36 gases or vapours, containing halogen, in the air (at 600 litres/hr.) required to extinguish a gas flame burning 30 litres of coal gas per hr. vary greatly, from, e.g., 40% (CO<sub>2</sub> or HCl), 20% (MeCl or HBr) and 16% (s-C<sub>2</sub>H<sub>4</sub>Cl<sub>2</sub> or CCl<sub>4</sub>) to 2.5 (CH<sub>3</sub>I<sub>2</sub>) and 2 (I). I is more effective than Br and still more so than Cl<sub>2</sub>, but there is no apparent relation between effectiveness and amount of halogen or constitution. C. A. S.

**Gas economy [in furnaces] by preheating.** G. NEUMANN (Arch. Eisenhüttenw., 1933—4, 7, 37—39).—Graphs are given to show the economy which can be effected by preheating gas and air required for furnace heating to temp. between 200° and 1000°. The gases considered are blast-furnace gas, producer gas, mixed gas, and coke-oven gas. A. R. P.

**Corrosion phenomena in tar stills.** ANON. (Stahl u. Eisen, 1933, 53, 734—736).—Corrosion of the steam heating tubes and at rivets and along the floor of stills distilling tar is attributed to the presence of NH<sub>4</sub>Cl; it is particularly marked when the temp. in the still is > 240° and may be prevented by mixing a small proportion of CaO with the tar or by treatment with steam and separation of the condensed H<sub>2</sub>O. Addition of the tar obtained in washing crude C<sub>6</sub>H<sub>6</sub> with H<sub>2</sub>SO<sub>4</sub> leads to serious corrosion and the residue in the still may contain 15% SO<sub>3</sub>. A. R. P.

**Prevention of fog entrainment in continuous [oil] distillation.** A. E. BIRCH and H. M. WEIR (Chem. Met. Eng., 1933, 40, 366—368).—A rapid rotation is imparted to the vapours leaving a petroleum still by passing them through a centrifix separator consisting of a pair of fan-like elements with stationary blades. After passing through each set of blades, the entrained matter is thrown on to the walls of the cylindrical conduit, down which it flows into collecting chambers. The apparatus is as effective as are layers of steel wool. D. K. M.

**Occurrence of higher fatty acids in mineral oil distillates.** II. E. HOLZMANN and S. VON PILAT (Brennstoff-Chem., 1933, 14, 263—265; cf. B., 1931, 327).—By methylating the acid mixture obtained as previously described, fractionating the esters in vac., and subsequently further separating the regenerated acids by fractional pptn. of the Mg salts [by addition of Mg(OAc)<sub>2</sub> to a solution of the acids in MeOH], the



presence of myristic, palmitic, stearic, and arachidic acids has been established. A. B. M.

**Acid tars obtained in refining petroleum derivatives [with sulphuric acid].** I. L. BLUM and (MLLE.) I. SCHLESINGER (Bul. Chim. Soc. Române, 1933, 34, 61—70).—Treatment of a Rumanian kerosene with 2% of  $H_2SO_4$  resulted in conversion of 57% of this acid into org. compounds which accumulated chiefly in the 2.46% of tar formed. This tar has been separated into numerous fractions and the S content of these determined to provide a S balance sheet of the process. A. R. P.

**Anti-oxidising constituents of cracking spirit.** E. VELLINGER and G. RADULESCO (Compt. rend., 1933, 197, 417—418).—The oxidisability of refined cracking spirit is considerably diminished by addition of 3—5% of a fraction (I) of the crude spirit distilling  $> 140^\circ$ . The inhibiting effect of (I) increases with its b.p. from  $140^\circ$  to  $170^\circ$ . A. C.

**Determination of paraffin and naphthene contents of gasolines.** C. C. MINTER (Nat. Petrol. News, 1933, 25, No. 8, 25, 27).—The unsaturated and aromatic hydrocarbons are absorbed from the gasoline with  $H_2SO_4$  and the 50% point of the Engler distillation of the residue is determined. Paraffin % =  $[1 - (v/100)][(0.7126 + 0.000244T - d)/(0.0591 + 0.000016T)]100$ ; naphthene % =  $[1 - (v/100)] \times [(1 - 0.7126 + 0.000244T - d)/(0.0591 + 0.000016T)] \times 100$ , where  $v$  is the vol. of unsaturated and aromatic hydrocarbons,  $T$  is the 50% point ( $^\circ F.$ ), and  $d$  the sp. gr. of the distillate obtained after absorption. CH. ABS.

**Rapid determination of mercaptans [in gasoline].** G. R. BOND, JUN. (Ind. Eng. Chem. [Anal.], 1933, 5, 257—260).—The mercaptan content of light petroleum products is determined by titrating with a standard solution of Cu oleate (containing about 4 g. Cu per litre) in kerosene. The end-point is marked by the appearance of a pale green colour. Free S,  $CS_2$ , or org. disulphides have no influence, but  $H_2S$  should be removed. The average abs. error over a range of 0.0007—0.06% S is about 0.0002%. E. S. H.

**Effect of gasoline volatility on the miscibility with ethyl alcohol.** O. C. BRIDGEMAN and D. W. QUERFELD (Bur. Stand. J. Res., 1933, 10, 841—850).—The more volatile petrols were more miscible with EtOH, producing mixtures having lower crit. solution temp. than similar mixtures containing less volatile petrol. Miscibility differences were comparatively small for petrols having the same volatility but different compositions. N. M. B.

**isoOctane index for motor fuels and cetene index for fuel oils.** A. GREBEL (Mém. Soc. Ing. Civ., France, 1933, 86, 41—69).—A survey of the various methods of comparing hydrocarbons with different standards obtained by mixing detonating and non-detonating hydrocarbons in varying proportions. C. E. M.

**Motor benzol.** W. H. COLEMAN (Gas World, 1933, 99, Coking Sect., 90—92).—A review.

**Refining of hydrogenated lubricants.** Y. TANAKA, R. KOBAYASHI, and A. MISONO (J. Soc. Chem. Ind., Japan, 1933, 36, 227—228 B).—Lubricating oils decolor-

ised by hydrogenation often tend to darken on exposure to air. By low-temp. hydrogenation ( $150^\circ$ , 40 atm.  $H_2$  pressure), using a 3:1 Cu—Ni hydroxide catalyst, for 4 hr., and subsequent treatment with 5% of Japanese acid clay, an oil lighter in colour and more stable than a commercial refined high-grade machine oil is obtained. H. F. G.

**Synthesis of benzine from carbon monoxide and hydrogen under ordinary pressure.** XV, XVI. Nickel catalysts. K. FUJIMARA and S. TSUNEOKA (J. Soc. Chem. Ind., Japan, 1933, 36, 413—414 B, 414—416 B).—XV. The accelerating influence of the oxides of Cr, Mo, W, and U on Ni—Mn catalysts increases with the at. wt. of the metal.  $ThO_2$ ,  $Al_2O_3$ , and fuller's earth are good accelerators.

XVI. The optimum amount of Mn in Ni—Mn catalysts is 15%. Yields of benzine are not affected by the presence of Cu up to 0.5% of the Ni, but are lessened by greater amounts. The reduction temp. of the Ni catalyst cannot be usefully lowered in presence of Cu. A catalyst prepared in a voluminous form with starch gave a low yield. A. A. L.

**Desulphuration of shale-tar benzines by hydrogenation in presence of sulphur-containing catalysts.** S. S. NAMETKIN, P. I. SANIN, S. V. MAKOVER, and A. N. TZYBA (J. Appl. Chem. Russ., 1933, 6, 494—507).—The fraction of b.p.  $< 220^\circ$  of Kaschpir shale-tar oil contains up to 10% S, chiefly as thiophen derivatives, which may be completely eliminated as  $H_2S$  by hydrogenation at  $350^\circ/80$ — $90$  atm. in presence of  $MoS_2$  or CoS catalyst. Hydrogenation at  $450^\circ/1$  atm. does not give the desired results. The  $d$  and b.p. of the product are  $<$  those of the original benzine. R. T.

**Kinematic viscosity.**—See I. CO.—See VII. Decolorising C.—See XVIII.

See also A., Aug., 786, Aspects of gaseous explosions. 789, Catalysts for prep. of  $H_2$  by the water-gas reaction. 790,  $CH_4$ — $H_2O$  reaction. Reaction regions [arresting coal-mine explosions]. 791 and 804, Action of the silent discharge on  $C_2H_4$ . 798, CO indicator.

#### PATENTS.

(A) Handling of coal. (B) Coal and coke treated with oil. F. C. BROEMAN (U.S.P. 1,886,632—3, 8.11.32. Appl., [A] 14.5.29, [B] 14.7.30).—Run-of-mine coal at some stage of its progress is sprayed with light mineral oil to prevent dusting, the quantity being, in (A), 1 gal. and in (B)  $\frac{1}{2}$ —1 gal. of oil/ton of coal.

B. M. V.

**Manufacture of fuel briquettes.** F. D. SNELL (B.P. 394,572, 19.11.32).—Anthracite culm (A) or other fine carbonaceous matter is mixed with a binder formed by heating Na silicate (85%) with silicic acid gel (15%), and the mixture is moulded and baked at  $220^\circ$  (max.). If desired, A may first be treated with a waterproofing agent. A. B. M.

**Fuels in paste or like form.** J. S. B. FLEMING, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 394,696, 31.12.31 and 3.3.32).—A nitrocellulose insol. (A) in MeOH or EtOH is incorporated in a solution of sol. nitrocellulose (B) in MeOH or EtOH; suitable proportions are 3 pts. of A and 2 pts. of B in 95 pts. of alcohol. If desired, a



small proportion of an anti-gelating agent, *e.g.*, citric acid, may be added. A. B. M.

**Manufacture of amorphous carbon.** J. V. G. VAN DEN DRIESSCHE (U.S.P. 1,887,792, 15.11.32. Appl., 11.1.32. Belg., 16.1.31).—Water-gas formed by passing a mixture of  $\text{CO}_2$  and steam over heated Fe is mixed with a further quantity of steam and passed over  $\text{CaC}_2$  at  $250^\circ$  ( $3\text{CO} + 3\text{H}_2 + 4\text{CaC}_2 + \text{H}_2\text{O} = 4\text{CaO} + 4\text{H}_2 + 11\text{C}$ ) and the C formed is separated from the  $\text{CaO}$ , *e.g.*, by treatment with HCl. The  $\text{CO}_2$  used may be produced by passing water-gas over a heated mixture of  $\text{Ca}(\text{OH})_2$  and  $\text{Fe}_2\text{O}_3$  and subsequently calcining the carbonates so formed. A. B. M.

**Manufacture of carbon black.** W. B. WIEGAND and L. J. VENUTO (U.S.P. 1,889,429, 29.11.32. Appl., 2.12.27).—C black is thoroughly wetted with  $\text{H}_2\text{O}$  or aq. solution or suspension of subsequently useful chemicals, *e.g.*, rubber accelerators, and then agitated in an immiscible liquid, *e.g.*, petrol, producing pellets which are suitable for transport but will recover the useful properties of the C on crushing. B. M. V.

**Catalytic treatment of carbonaceous materials in gaseous or liquid phase.** W. T. R. BINDLEY (B.P. 394,506, 16.2.32. Cf. B.P. 386,982; B., 1933, 256).—The gases or liquids are passed in intimate contact with a catalytic surface consisting of powdered catalytic material attached to a non-combustible supporting material by means of a Si ester. *E.g.*, such a catalyst prepared from oxides of Co, Mn, Ce, and Cr is very effective in the conversion of S-free water-gas (at  $200$ — $230^\circ$ ) into hydrocarbons. A. B. M.

**Apparatus for gasification of fuels which tend to agglomerate.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 394,747, 8.2.32. Cf. B.P. 214,544; B., 1924, 549).—The fuel, in the form of nuts or smaller particles, is fed on to a thick, glowing layer of small-sized fuel in the producer, through which the gasifying agent is blown at such a rate as to keep the carbonised fuel in movement similar to that of a boiling liquid; the particles of fresh fuel are thereby kept substantially separated from each other until they have lost their agglomerating power. A. B. M.

**Purification of town gas or other fuel gas.** D. Y. BRYANT, and FIRTH, BLAKELEY, SONS & CO., LTD. (B.P. 394,085, 15.3.32).—The hot gas is admitted to a chamber (A) wherein it suffers reduction of velocity and some fall in temp., thereby effecting pptn. of the heavier tarry constituents, and is led thence up into a bubble-washer section (B) surmounting A. B contains a tar and a liquor zone, the former serving as a trap for the  $\text{C}_{10}\text{H}_8$ . The gas is then passed, countercurrent to a descending stream of liquid, through a scrubber superimposed on B. Finally the direction of flow of the gas is reversed and it is passed downwards through vertical pipes traversing the scrubber, B, and inlet chamber, into a gas-collecting and -outlet section. A. B. M.

**Removal of ammonia from fuel gas.** KOPPERS CO. OF DELAWARE, ASSEES. OF F. W. SPERR, JUN. (B.P. 394,666, 28.12.31. U.S. 27.12.30).—The gas is passed successively through a cooler, in which it is brought in contact with  $\text{NH}_3$  liquor, a cyanide scrubber, wherein

it is scrubbed with a suspension of S in  $\text{NH}_3$  liquor, an electrical precipitator for removing tar fog and entrained liquor, a heater, and an "ammoniator" (A). In A the gas is brought in contact with dry superphosphate material supported on horizontal trays and kept in motion by means of rabble arms on a vertical shaft, which convey the material from tray to tray through the apparatus countercurrent to the gas. The ammoniated superphosphate is withdrawn from the apparatus when its  $\text{NH}_3$  content is about 4%, *i.e.*, before any substantial amount is converted into phosphate insol. in aq.  $\text{NH}_4$  citrate. The temp. of the gas entering A is adjusted to  $30$ — $50^\circ$ . If desired,  $(\text{NH}_4)_2\text{SO}_4$  may be added to the superphosphate before it is fed into the apparatus. A. B. M.

**Removal of hydrogen sulphide from combustible gases.** C. J. HANSEN (B.P. 394,917, 13.2.33. Ger., 11.2.32).—The gases are freed from  $\text{NH}_3$  and washed with aq.  $\text{NH}_4\text{CNS}$ , or other sol. thiocyanate, containing  $\text{SO}_2$ . The reaction  $\text{SO}_2 + 2\text{H}_2\text{S} = 3\text{S} + 2\text{H}_2\text{O}$  proceeds without the formation of polythionic acids, and the thiocyanate is recovered unchanged. A. B. M.

**Determination of oxygen or combustible gas constituents by combustion.** O. RODHE (B.P. 394,292, 8.3.33).—The gas to be tested and the reaction gas ( $\text{H}_2$  or  $\text{O}_2$ ) are brought in contact only in the combustion chamber, wherein the gases burn quietly without risk of explosion. In other respects the gas analysis apparatus is of known design. A. B. M.

**Distillation of tar.** E. H. ELLMS, ASSR. to BARRETT CO. (U.S.P. 1,883,642, 18.10.32. Appl., 17.9.28).—The tar is brought in direct contact with hot coal-distillation gases, *e.g.*, by being sprayed into the collector main, the gases and vapours are freed from tar fog, *e.g.*, by being passed through an electrical precipitator operating above the dew point of the condensable oil constituents, and the cleaned gases and vapours are passed through one or more condensers wherein they are cooled by indirect heat exchange with boiling liquids, of approx. const. b.p., so chosen as to condense the desired oil fractions. Thus the first condenser may operate with coal-tar naphtha of b.p.  $160$ — $175^\circ$ , and the second with PhMe at  $110^\circ$ , condensing heavy and light cresote oils, respectively. A. B. M.

**Distillation of tar.** S. P. MILLER, ASSR. to BARRETT CO. (U.S.P. 1,888,235—6, 22.11.32. Appl., [A] 14.3.27, [B] 15.3.27).—(A) Part of the hot gases from coke ovens pass forward to effect cooling etc. in the usual manner, and part are used to distil tar from the same or other ovens, the tar being sprayed in several stages in the same flow of gas and the pitches of different distillation temp. being collected separately; the final gases are treated separately from the first-mentioned part, which treatment comprises, in (B), flooding the gases with tar to effect substantial cooling without much distillation of tar, the thinner tar thus produced going to process (A). A. B. M.

**Manufacture of asphalt.** A. LOEBEL, ASSR. to BATAAFSCHE PETROLEUM MAATS. (U.S.P. 1,881,753, 11.10.32. Appl., 14.2.29. Holl., 9.12.27).—The asphalt, prepared by distilling crude oil, petroleum residues, etc., is improved by adding to the initial material < 20%



of hydrocarbon oils sol. in liquid  $\text{SO}_2$  and having an initial b.p.  $\leq 250^\circ$ . A. B. M.

**Production of asphalt.** H. B. PULLAR (U.S.P. 1,889,697, 29.11.32. Appl., 10.6.29).—Residuum oil is heated and sprayed or flowed in thin films in the presence of air which is also finely divided into streams at about the same pressure as the oil; the air and volatile matter are removed and the oil is recycled, a proportion of the reaction product being continuously removed. B. M. V.

**Manufacture of asphalt emulsion.** L. A. HALL and E. E. MORTELL, Assrs. to J. W. MORTELL Co. (U.S.P. 1,882,834, 18.10.32. Appl., 15.8.29).—Relatively small quantities of a heated aq. suspension of colloidal clay and an asphaltic substance heated above its m.p. are emulsified, and to the emulsion relatively large quantities of the same ingredients are then added with agitation. The agitation is continued, and when the emulsion has cooled to about the m.p. of the asphalt rapid cooling is brought about by the addition of cold  $\text{H}_2\text{O}$ . A. B. M.

**Manufacture of cold asphalt or bitumen dispersions, more particularly for street constructional purposes.** W. R. ROEDERER (B.P. 395,384, 3.12.31. Ger., 3.12.30).—Brown bituminous coal ("Schwelkohle") or oil shale is finely ground (with or without treatment with alkali or petroleum oils), and 10 pts. of this prep. are stirred with, e.g., molten asphalt (40 pts.), tar oil (10 pts.), and  $\text{H}_2\text{O}$  (40 pts.). [Stat. ref.] J. A. S.

**Production of bituminous emulsion.** P. R. SMITH, Assr. to BARBER ASPHALT Co. (U.S.P. 1,888,295, 22.11.32. Appl., 26.7.28).—An emulsion of bitumen or asphalt in  $\text{H}_2\text{O}$  is produced by the aid of an alkaline salt of Turkey-red or other sulphonated oil with, if desired, another emulsifying agent, e.g., soap, the  $\eta_{\text{H}}$  being 11.5—13.2 (11.68—12.80). B. M. V.

**Preparation of creosoting compositions.** S. P. MILLER, Assr. to BARRETT Co. (U.S.P. 1,886,967, 8.11.32. Appl., 18.12.28).—Hot coal-distillation gases are subjected to a regulated partial removal of suspended heavy tar and pitch constituents in an electrical precipitator at a temp., e.g.,  $200^\circ$ , sufficiently high to maintain a large proportion of the oils in the form of vapour. The resulting partly cleaned gases are then cooled to condense the tarry oils, which are in a form suitable for creosoting purposes. A. B. M.

**Treating oils and composition therefor.** E. MORRILL (U.S.P. 1,886,293, 1.11.32. Appl., 8.2.29).—An emulsion-breaking reagent comprises a polynuclear hydrocarbon, a caustic alkali, and metallic soap, e.g.,  $\text{C}_{10}\text{H}_8$  75,  $\text{NaOH}$  18,  $\text{Al}$  stearate 7%. B. M. V.

**Stabilisation of gasoline and the like.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,885,190, 1.11.32. Appl., 4.11.29).—For prevention of deterioration in storage of cracked, anti-knock petrol, anthraquinone (up to 2%) is added. B. M. V.

**Treatment of [heavy] liquid hydrocarbons.** A. S. KNOWLES and C. W. ANDREWS, Assrs. to TAR & PETROLEUM PROCESS Co. (U.S.P. 1,885,920, 1.11.32. Appl., 27.10.28).—The hydrocarbons are completely distilled in a chamber having a floor heated to coking temp., the gases leaving through a bubbling tower

countercurrent to the entering liquid, which is thereby heated nearly to the coking temp. B. M. V.

**[Hydrogenation] treatment of hydrocarbon oils.** H. C. WEBER, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,887,051, 8.11.32. Appl., 3.11.30).— $\text{H}_2$  is generated by electrolysis of an electrolyte mixed with the oil. The anode is so chosen that the oxidised products of it will form catalysts for the hydrogenation. The operation may be carried out at cracking or other elevated temp. B. M. V.

**Removal of wax from hydrocarbon oils.** H. LOWERY, Assr. to STANDARD OIL Co. (U.S.P. 1,886,344, 1.11.32. Appl., 27.10.28).—Slop wax is rendered separable by sedimentation by dilution of the oil with low-boiling hydrocarbons, adding a small quantity of petrolatum wax, and chilling. B. M. V.

**Compound for purifying [used] oils.** H. H. MORETON, Assr. to O. B. ENGLISCH and C. F. CRAIG (U.S.P. 1,885,619, 1.11.32. Appl., 8.10.28).—A filtration medium comprises  $\text{H}_2\text{SO}_4$  1 gal., 300-mesh  $\text{SiO}_2$  100 lb.,  $\text{CaSO}_4$  100 lb., the mixture being set aside for 2 hr. before using. B. M. V.

**Testing combustible fluids.** W. HELMORE (B.P. 394,736, 19.1.32).—The spontaneous ignition temp. of a liquid fuel and the delay time between contact of the fuel with heated air and its subsequent ignition are determined in an apparatus wherein the fuel is injected in a finely-divided state into air at a predetermined temp. and pressure in an electrically-heated vessel, the injection being effected in such a manner that the liquid can ignite in the air without making contact before ignition with any solid surface at a temp.  $\leq$  that of the air. The moment of injection of the fuel and the time of subsequent ignition are automatically recorded. A. B. M.

**Engine fuel.** N. H. GULLANDER, Assr. to C. & S. CLEMENTSON (U.S.P. 1,889,474, 29.11.32. Appl., 26.2.31. Swed., 14.2.31).—A lubricating oil is mixed with hydrogenated  $\text{C}_{10}\text{H}_8$  and hydrogenated  $\text{PhOH}$ . B. M. V.

**Lubricant.** H. T. BENNETT (U.S.P. 1,889,168, 29.11.32. Appl., 25.1.30).—Lubricants suitable for the flanges of railway wheels are composed of mixtures of high-viscosity residuum from atm. distillation and low-viscosity residuum from cracking, in various proportions. B. M. V.

**Lubricating composition containing sulphonates.** A. E. BECKER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,888,974, 29.11.32. Appl., 18.7.27).—The lubricant comprises petroleum lubricating oil  $> 90$ , alkali soap of fatty acid 0.1—0.5%, and an oil-sol. alkali sulphonate derived from mineral oil. B. M. V.

**Dense opaque smoke and irritating fume- and gas-producing chemicals.** B. C. GOSS, Assr. to LAKE ERIE CHEM. Co. (U.S.P. 1,886,394, 8.11.32. Appl., 24.2.27).—A composition claimed is: chloroacetophenone (A) 12.5—30.0,  $\text{C}_2\text{Cl}_6$  27.3—52.5,  $\text{NH}_4\text{ClO}_4$  4.5—12.0,  $\text{Zn}$  dust 22.0—34.0,  $\text{ZnO}$  8.0—21.5%. About 20 alternatives for A are claimed. B. M. V.

**Dip of seal pipes for use in the manufacture of gas and/or in coke-oven plant.** GAS LIGHT & COKE Co., W. J. B. LEECH, S. HAY, H. HOLLINGS, and A. E.



BURTON (B.P. 396,552, 25.7.32. Addn. to B.P. 326,173, 3.12.28).

Recovering condensable vapours from incondensable gas. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,897,618, 14.2.33. Appl., 25.11.29).

Cracking of petroleum oil. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,897,245, 14.2.33. Appl., 11.2.22. Renewed 10.12.28).

Cracking and separating gasoline stock from petroleum oils. J. C. BLACK, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,895,345, 7.2.33. Appl., 20.10.26).

Cracking process [for hydrocarbons]. C. W. WATSON and J. H. GRAHAME, Assrs. to TEXAS Co. (U.S.P. 1,897,348, 14.2.33. Appl., 23.1.29).

Cracking of hydrocarbon oils. J. G. ALTHER, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,895,873, 31.1.33. Appl., 23.8.26).

Fractional distillation. Dephlegmation tower. Treating emulsions.—See I.  $C_2H_4$ . Emulsions.—See III. Filtration of emulsions.—See V.  $H_2$ .—See VII. Bituminous cement etc.—See IX. Petroleum dehydrators.—See XI.

### III.—ORGANIC INTERMEDIATES.

Technical application of azeotropy, particularly for the dehydration of alcohol. R. FRITZWEILER and K. R. DIETRICH (Z. Spiritusind., 1933, 56, 165—167, 171—173, 177—179).—A detailed description of the process already noted (B., 1933, 11, 327).

Furfuraldehyde, an industrial raw material and solvent. A. J. HAMMER (Chem. & Ind., 1933, 608—612).—The manufacture, properties, and uses of furfuraldehyde (I) are described. Indications are given of the approx. solubility of a large no. of org. compounds or mixtures in (I),  $Bu^+$  furoate, tetrahydrofurfuryl alcohol, and furfuryl alcohol, respectively. E. S. H.

Detection of citric acid in its compounds. W. ALTNER (Apoth.-Ztg., 1932, 47, 1387; Chem. Zentr., 1933, i, 821—822).—For Li, Na, K, Mg, and caffeine citrates, the solution is heated with  $HgSO_4$  in presence of  $H_2SO_4$ , and a few drops of 0.5%  $KMnO_4$  are added, when a white ppt. shows the presence of citric acid. With Ca citrate, the  $CaSO_4$  is first filtered off. With Fe quinine citrate, Fe  $NH_4$  citrate, and Fe citrate the base is first removed with NaOH; and with Mn citrate, with  $Na_2CO_3$ . L. S. T.

Fatty acids in mineral oil distillates.—See II. Lacquer paints for  $CCl_4$  production.—See XIII. Abs. EtOH.—See XVIII.

See also A., Aug., 791, Electrolytic oxidation or reduction. 791 and 804, Action of the silent discharge on  $C_2H_4$ . 806, Prep. of BuOH and hexyl alcohol, and of EtOAc. 809, Purification of  $COMe_2$  and  $Et_2O$ . 815, Prep. of styrenes. 821, PhCN. Prep. and pyrolysis of  $(CH_2Ph)_2CO$ . Prep. of diaryl-acetic acids. 830, Prep. of camphor. 834, Synthesis of thiophen. 867,  $COMe_2$ -BuOH by fermentation. 878, Raponticin, an anthraquinone derivative from *Rheum emodi*.

### PATENTS.

Drying of [organic] liquids by dialysis. R. H. VAN SCHAACK, JUN., Assr. to VAN SCHAACK BROS. CHEM. WORKS, INC. (U.S.P. 1,885,393, 1.11.32. Appl., 29.4.29).—An org. liquid, e.g., EtOAc, is freed from  $H_2O$  by dialysis into a hygroscopic org. liquid, e.g., EtOH. Gelatin is suitable for the diaphragm, and the liquids may be flowed in opposite direction on opposite sides of it. B. M. V.

Separation of ethylene from its homologues. H. G. WATTS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 393,317, 2.12.31).— $C_2H_4$  is extracted from gas mixtures rich in  $C_2H_4$  and containing  $C_3H_6$  with an ammoniacal  $Cu^{II}$  salt solution, preferably at 20 atm. If desired, the gas is washed before and/or after with kerosene or other preferential solvent for  $C_3H_6$ . The  $C_2H_4$  is recovered by heating or by reducing the pressure. C. H.

Production of alcohols [from olefines]. N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 393,152, 30.9.32. Holl., 17.10.31).—The alkyl H sulphate, diluted with  $H_2O$  (with or without neutralisation and/or hydrolysis), or the alcohol obtained by hydrolysis, is treated with mineral oil (Edeleanu extract) to remove polymerisation products having obnoxious odour and is then worked up as usual. C. H.

Manufacture of ethyl alcohol [from ethylene]. DISTILLERS Co., LTD., W. P. JOSHUA, H. M. STANLEY, and J. B. DYMCK (B.P. [A] 392,289, 29.1., 10.3., and 22.3.32, [B] 392,685, 19. and 24.2.32).—(A)  $C_2H_4$  is hydrated, e.g., at 200—300°/1—40 atm., in presence of a catalyst compounded of excess (> 95%) of  $H_3PO_4$  and U,  $Fe^{II}$ , or  $Co^{II}$  or oxides thereof.  $FeO + 2.5H_3PO_4$  gives 0.83% conversion. (B)  $C_2H_4$  and steam are passed, e.g., at 200—300°/1—20 atm., over a catalyst comprising Cu or Mn or oxides of these and excess of  $H_3PO_4$ . Conversion is about 1%. C. H.

Production of water-free ethyl alcohol. DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 393,265—6, 22.10.31. Ger., [A] 4.2.31, [B] 19.6.31).—In the process of B.P. 368,793 (B., 1932, 700) there is used as dehydrating agent (A) a mixture of KOAc and NaOAc, e.g., 70 : 30, or (B) a mixture of fatty acid salts (acetates) of alkali or alkaline-earth metals, at least 1 being sol. in EtOH and 1 a dehydrating agent, e.g., KOAc (70), NaOAc (20), and  $Ca(OAc)_2$  (10). C. H.

Manufacture of acetaldehyde [from alcohol]. BRIT. CELANESE, LTD., and W. H. GROOMBRIDGE (B.P. 391,444, 26.10.31).—In the oxidation of EtOH vapour to MeCHO the reactants are introduced into the reaction zone, e.g., through perforated conduits, at several points spaced in or parallel to the line of flow. Preferably air or  $O_2$  is introduced in this way into the EtOH vapour (with or without 10—20% of the total  $O_2$ ) flowing over catalyst layers. C. H.

Manufacture of ketones [from secondary alcohols]. N. V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of M. DE SIMO (B.P. 392,652, 7.1.32. U.S., 17.1.31).—In the dehydrogenation of sec. alcohols ( $CHMe_2 \cdot OH$ , sec.-BuOH, sec.- $C_5H_{11} \cdot OH$ ) a brass



catalyst is used at 350—480°, *e.g.*, 440—460°, in absence of O<sub>2</sub>. C. H.

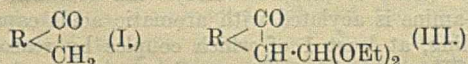
**Manufacture of condensation products from aliphatic organic compounds.** BRIT. INDUSTRIAL SOLVENTS, LTD., W. P. JOSHUA, H. M. STANLEY, and J. B. DYMOCK (B.P. 393,267, 26.10.31).—Oxygenated derivatives of paraffins (alcohols, aldehydes, esters), other than EtOH or EtOH + MeOH, are passed with H<sub>2</sub> (in absence of CO) over a mixed catalyst comprising Al<sub>2</sub>O<sub>3</sub>, U<sub>3</sub>O<sub>8</sub>, and/or ThO<sub>2</sub> with Cu, Ag, Ni, and/or Cr at 150—400°/1—50 atm. MeCHO and H<sub>2</sub> over Al<sub>2</sub>O<sub>3</sub> (100 pts.), CaO (6 pts.), and Cu (1.5 pts.) at 260° give BuOH, PrCO<sub>2</sub>Bu, BuOAc, and higher alcohols. Hexyl alcohol is the main product from a mixture of EtOH, BuOH, and H<sub>2</sub>. C. H.

**Manufacture of aliphatic [acetic] anhydrides.** H. DREYFUS (B.P. 391,802, 3.11.31 and 25.5.32).—Glycol diacetate or other aliphatic esters of glycols or other polyhydric alcohols (except ethylidene diacetate etc.) are subjected to thermal decomp., *e.g.*, at about 400°, in presence of ZnCl<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>, H<sub>3</sub>PO<sub>4</sub>, alkali acetates, filling materials, etc. C. H.

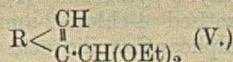
**Manufacture of [aliphatic] diamines.** GOODYEAR TIRE & RUBBER Co. (B.P. 393,093, 21.6.32. U.S., 12.9.31).—Alkylene dihalides < C<sub>9</sub> are treated with NH<sub>3</sub> in presence of a metal oxide or salt capable of forming a complex with the diamine, *e.g.*, ZnCl<sub>2</sub>, ZnO, Cu<sub>2</sub>Cl<sub>2</sub>, or CuSO<sub>4</sub>. C. H.

**Manufacture of addition compounds of formyl halides [with aluminium halides].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 391,600, 15.4.32).—Anhyd. H halide with < 1 mol. of CO is led at 20—250 atm. into a pressure vessel containing anhyd. Al halide, preferably at 50—60°. The compounds may be used for introducing ·CO· into org. mols. C. H.

**Conversion of alicyclic ketones into unsaturated aldehydes.** SOC. ANON. M. NAEF & Co. (B.P. 392,062, 7.10.32. Switz., 30.10.31).—The ketone (I) is converted *via* its OH·CH<sub>2</sub>· derivative (II) into the acetal (III), which is reduced, *e.g.*, with Na and EtOH, to



the *sec.* alcohol (IV) and dehydrated by the xanthate method to the unsaturated acetal (V), from which the aldehyde (VI) is liberated by shaking with dil.



H<sub>2</sub>SO<sub>4</sub>. Products are thus obtained from 2:2:3-trimethylcycloheptanone [b.p. (I) 80—83°/12 mm., (II) 115—118°/12 mm., (III) 140—143°/10 mm., (IV) 150—155°/10 mm., (V) 125—130°/11 mm., (VI) 95—98°/11 mm.] and from the 3:3:5-isomeride (respective b.p. identical). C. H.

**Conversion of carbohydrates into products of higher oxidation [tartaric acid].** W. E. STOKES and A. H. PETER, ASSTS. to ROYAL BAKING POWDER Co. (U.S.P. 1,870,472, 9.8.32. Appl., 21.12.25).—Starch or other carbohydrate is oxidised with HNO<sub>3</sub> in presence of MnCl<sub>2</sub> (> 3%, preferably 20%, of the wt. of carbo-

hydrate). Good yields of tartaric acid are produced with little or no oxalic and saccharic acids. J. H. L.

**Manufacture and use of wetting-out, equalising, peptising, and cleaning agents.** DEUTS. HYDRIERWERKE A.-G. (B.P. 391,610, 4.5.32. Ger., 4.5.31).—Glycols obtained by hydration of the unsaturated alcohols from hydrolysed fish or whale oils are esterified with suitable polybasic acids, *e.g.*, H<sub>2</sub>SO<sub>4</sub>, ClSO<sub>3</sub>H, H<sub>3</sub>PO<sub>4</sub>, or org. sulphocarboxylic acids. The esters may be mixed with soaps or soap substitutes, and alcoholic solvents and/or chlorinated hydrocarbons may be added to the aq. bath for treating textiles. C. H.

**[Manufacture of] cleansing and softening agents.** IMPERIAL CHEM. INDUSTRIES, LTD., and A. J. HAILWOOD (B.P. 391,435, 21.9.31 and 26.2.32).—Unsaturated long-chain alcohols, alone or with saturated long-chain alcohols, are treated with C<sub>5</sub>H<sub>5</sub>N, SO<sub>3</sub> or SO<sub>3</sub> compounds of other *tert.* bases or mixtures capable of producing such compounds, in presence or absence of diluent. The alcohols are those obtained by reducing to ·CH<sub>2</sub>·OH the CO<sub>2</sub>H or ester groups of unsaturated oils or oil acids, or by hydrolysis of sperm oil or arctic sperm oil; an example is oleyl alcohol. The products may be mixed with inorg. salts (NaCl, Na<sub>2</sub>SO<sub>4</sub>) for use in cleansing and softening textiles. C. H.

**Production of soap-like preparations, wetting, dispersing, and penetrating agents, and protective colloids.** H. HUNSDIECKER and E. VOGT (B.P. 392,763, 30.6.32. Ger., 30.6.31).—Heterocyclic *tert.* bases (C<sub>5</sub>H<sub>5</sub>N, quinoline, isoquinoline, homologues and derivatives) are condensed with alcohols > C<sub>5</sub> in presence of conc. HCl or HBr, or with sulphuric esters of such alcohols. Examples are: C<sub>5</sub>H<sub>5</sub>N with cetyl alcohol and conc. HBr at 250°; C<sub>5</sub>H<sub>5</sub>N, SO<sub>3</sub> with lauryl alcohol. C. H.

**Manufacture of emulsions, dispersions, etc.** IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, and W. TODD (B.P. 393,276, 25.11.31).—Quaternary NH<sub>4</sub> salts having an alkyl or alkylene group > C<sub>9</sub> free from alcoholic OH are dispersing agents giving positively-charged dispersions or emulsions of mineral or vegetable oils, fats, tars, etc. in H<sub>2</sub>O. Glue, gelatin, or other gelatinisable substance may be added. Examples are octa- and do-decyl- and stearyl-pyridinium bromide. C. H.

**Manufacture of phenol esters of sulphonated phthalic acids [wetting agents].** SOC. CHEM. IND. IN BASLE (B.P. 391,715, 16.11.32. Switz., 18.12.31. Addn. to B.P. 370,845 and 371,144; B., 1932, 763).—The esterification of phenols with sulphophthalic anhydride is effected in presence of suitable *tert.* bases, *e.g.*, C<sub>5</sub>H<sub>5</sub>N, NMe<sub>3</sub>, NEt<sub>3</sub>, lepidine, collidine, quinoline. C. H.

**Manufacture of diphenylolpropane [pp'-dihydroxy-ββ-diphenylpropane].** IMPERIAL CHEM. INDUSTRIES, LTD., and R. GREENHALGH (B.P. 395,732, 21.1.32).—Equiv. amounts of PhOH and COMe<sub>2</sub> are condensed in presence of < 3 (4—7) times the wt. of PhOH of 65—75% H<sub>2</sub>SO<sub>4</sub> at < 65° (40—50°). H. A. P.

**Dehydration of phenols and mixtures containing phenols.** CHEM. FABR. VON HEYDEN A.-G.



(B.P. 392,878, 18.2.33. Ger., 20.2.32).—An entraining liquid is used, *e.g.*, PhMe or PhCl. C. H.

**Manufacture of new thio-derivatives [from phenols; synthetic mordants and wool reserves].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 393,011, 18.1.32).—A phenol or naphthol is treated with  $S_2Cl_2$  in presence of Sn or Sb or a compound thereof; the Sn or Sb may be introduced after thionation. *E.g.*, a mixture of  $S_2Cl_2$  (5 pts.) and  $SnCl_2$  (2 pts.) is added slowly to molten PhOH (5.4 pts.), and after stirring at 50—100° 35% NaOH (10 pts.) is added. C. H.

**Preparation of alkylbenzylaniline.** L. F. MARTIN and D. E. MACQUEEN, Asssts. to DOW CHEM. CO. (U.S.P. 1,887,772, 15.11.32. Appl., 1.4.29).—A benzyl halide is added gradually, with agitation, to rather > the equiv. of *N*-alkylaniline, at a rate approx. to keep pace with the reaction. The temp. should be kept at 100—200° (140—150°). B. M. V.

**Reduction of nitrogen-containing organic compounds [nitro- to hydrazo-benzene].** H. DREYFUS (B.P. 391,774, 22.10.31).—Finely-divided or pyrophoric Fe is used in the alkaline reduction of  $NO_2$ -, azoxy-, or azo compounds to hydrazo-compounds. C. H.

**Manufacture of organic disulphides.** IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, J. S. H. DAVIES, and A. ECCLES (B.P. 395,726, 21.1.32).—SH compounds are oxidised to disulphides in aq. solution or suspension by  $H_2S_2O_8$  or a  $H_2O$ -sol. salt. Examples of SH compounds are mercaptobenzthiazole,  $NHBT_2$  diethyldithiocarbamate, and Na dimethyldithiocarbamate. H. A. P.

**Electrolytic manufacture of piperidine.** ROBINSON BROS., LTD., and D. W. PARKES (B.P. 395,741, 24.12.31).—A divided cell is used, the catholyte consisting of  $C_5H_5N$  and 2—6 equivs. of aq.  $H_2SO_4$ , and the anolyte of  $H_2SO_4$  suitably adjusted to minimise diffusion (about *d* 1.35); the electrodes are of Pb. The temp. is adjusted according to the amount of acid in the catholyte, *e.g.*, at 70—85° for 2 equivs., and at 20—50° for 6 equivs. The less acid is used the greater is the production of dipiperidyls (I), but the more rapid also is the reduction; (I) are also formed if too little acid is present in the anolyte. The above conditions give > 64% of theory of pure  $C_5H_{11}N$ . H. A. P.

**Manufacture of [hydr]oxydiphenyl ether carboxylic acids.** CHEM. FABR. VON HEYDEN A.-G. (B.P. 393,240, 9.3.33. Ger., 13.5.32).—Hydroxydiphenyl ethers are carboxylated with  $CO_2$  under pressure in aq. alkali. 4:4'-Dihydroxy-3:3'-dicarboxy- (m.p. 255—257°), 2- (m.p. 142°) and 4-hydroxy-3-carboxy- (m.p. 134°) -diphenyl ethers are described. C. H.

**Manufacture of aromatic hydroxyaldehydes [vanillin].** A. HOME-MORTON. From F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 392,399, 26.8.32).—*o*- or *p*-Propenylphenol is oxidised in aq. alkaline solution with aromatic nitro-sulphonic or -carboxylic acids except such as contain halogen *ortho* to  $NO_2$ . Vanillin and 3-aldehydosalicylic acid, m.p. 178—179°, are thus obtained in good yield. C. H.

**Treatment of dinitronaphthalene. [Manufacture of 5-nitro- $\alpha$ -naphthylamine and 1:8-dinitronaphthalene.]** IMPERIAL CHEM. INDUSTRIES, LTD., and H. H. HODGSON (B.P. 392,914, 17.11.31).—Mixed 1:5- and 1:8- $C_{10}H_6(NO_2)_2$  are treated with a limited amount of alkali sulphide, polysulphide, or hydrosulphide, *e.g.*, aq.  $Na_2S$  at 90°, whereby the 1:5-compound is reduced to nitro-amine, which may be separated from unchanged 1:8- $(NO_2)_2$ -compound. C. H.

**Manufacture of 3-[hydr]oxyselenonaphthene and derivatives.** I. G. FARBENIND. A.-G. (B.P. 396,011, 26.4.33. Ger., 27.4.32).—Diazotised anthranilic acid (or a derivative) is caused to interact with Se and an alkali cyanide, the resulting  $\cdot SeH$  compound condensed with  $CH_2Cl \cdot CO_2H$ , cyclised by  $Ac_2O$ , and hydrolysed. H. A. P.

**Manufacture of halogenation products of acenaphthindandione diketimines.** I. G. FARBENIND. A.-G. (B.P. 393,158, 10.10.32. Ger., 10.10.31. Addn. to B.P. 359,201; B., 1932, 174).—The ketimines of the prior patent are halogenated, *e.g.*, with  $KClO_3$  and aq.  $HCl$  at 50—60°, or  $Cl_2$  or  $Br$  in  $H_2O$  at 40°, to give  $C_{10}H_8 \begin{matrix} C(:NX) \\ C(:NX) \end{matrix} CX_2$ . The products are converted by dil. acid into the diketones, and thence by alkalis into  $CO_2H \cdot C_{10}H_8 \cdot CO \cdot CHX_2$ , and by  $NaOCl$  into acenaphthalic acid and naphthalenetetracarboxylic acid. C. H.

**Elimination of sulphonic acid groups from anthraquinone- $\beta$ -sulphonic acids.** SOC. CHEM. IND. IN BASLE (B.P. 392,290, 3.2.32. Switz., 3.2.31).—Anthraquinone-2-sulphonic acids containing in position 1, 3, or 4 OH or NHR (R = H, alkyl, or aryl) are desulphonated *via* the leuco-compounds by reduction, *e.g.*, with  $Na_2S_2O_4$ , in presence of aq. alkali followed by heating. In the examples the substituents are: 4- $NH_2$ -1-OH; 1- $NH_2$ -4-NHPh; 1- $NH_2$ -4-NHPh-5- $SO_3H$  (the 5- $SO_3H$  is retained); 1:5- $(NH_2)_2$ -4:8- $(OH)_2$ -3:7- $(SO_3H)_2$  (the 7- $SO_3H$  may be retained or eliminated). C. H.

**Manufacture of intermediate products and dyes therefrom.** A. G. BLOXAM. From SOC. CHEM. IND. IN BASLE (B.P. 393,271, 17.11.31).—An aromatic di- or poly-amine is acylated with aromatic acids capable of coupling, at least 1 of which couples by reason of a  $\cdot CO \cdot CH_2 \cdot$  grouping. The products are coupled with diazo components or are condensed with aldehydes etc. to give dyes. Examples are: benzidine with 1-*m*-carboxyphenyl-3-methyl-5-pyrazolone (I), coupled on the fibre with diazotised 4-chloro-2-aminodiphenyl ether (yellow) or 5-sulphoanthranilic acid (yellow), or condensed with *p*-dimethylaminobenzaldehyde (orange pigment); dianisidine with 1-*p*-carboxyphenyl-3-methyl-5-pyrazolone,  $\leftarrow$  sulphanilic acid (yellow); benzidine with 1 mol. of (I) and 1 mol. of 2:3-hydroxynaphthoic acid; *p*- $C_6H_4(NH_2)_2$  with (I),  $\leftarrow$  2:5-dichloroaniline on the fibre (yellow). C. H.

**Intermediates.**—See IV. Cyclic ethers.—See V. Wetting agents. Impregnating baths.—See VI.  $CISO_3H$ . Hydrogenation catalysts.—See VII. Distillation of glycerin.—See XII. Vulcanisation accelerators.—See XIV. Lactic acid preps.—See XIX. Terpene alcohols. Iodonaphtholsulphonic acids. cycloPentenylacetic esters.



## IV.—DYESTUFFS.

**Colorimetric determination of sulphur dyes.** G. E. LEVANT and R. I. NEMTZOVA (J. Appl. Chem. Russ., 1933, 6, 546—551).—Sulphur dyes can be determined colorimetrically by comparison with solutions prepared from standard samples, the optimum concns. of which are given for a no. of dyes. The hydrosols used should be stabilised by  $H_2O_2$ . An accuracy of 97—98% is obtained. R. T.

See also A., Aug., 826, **Ketoneimine dyes.** 832, **Dye of acacia wood.** 833, [Dyes from] **dinaphthylene dioxide.** 834, **Blue S dyes.** 840, **Pyridylnitro-pyrazole dye.** 844, **Spectroscopic characterisation of dyes.**

## PATENTS.

**Manufacture of carbotyanine dyes and intermediate products.** KODAK, LTD., Assees. of F. L. WHITE (B.P. 392,410, 21.9.32. U.S., 21.9.31).—2 mols. of a 2-methylselenazoline alkiodide (etc.) are condensed with an ortho-ester, e.g., Et ortho-formate, -acetate, or -propionate. The 2-methylselenazoline, b.p. 71—72°/35 mm., is obtained by heating  $Me \cdot CSe \cdot NH_2$  with  $CH_2Br \cdot CH_2 \cdot NH_2 \cdot HBr$ . Examples are the ethiodide with  $CH(OEt)_3$ , and the methiodide with  $CEt(OMe)_3$ . C. H.

**Manufacture of acid wool dyes [of the anthraquinone series].** W. W. GROVES, Assee. of I. G. FARBENIND. A.-G. (B.P. 391,262, 14.1.32. Ger., 14.1.31. Addn. to B.P. 355,810; B., 1932, 58).—Green-blue wool dyes are obtained by esterification of 5-, 6-, or 7- $CO_2H$ -derivatives of 2-halogeno-1-amino-4-sulphoarylamino- or 1-amino-4-alkyl- or -aryl-amino-2-sulpho-anthraquinones; the 2- $SO_3H$  may be introduced after esterification by replacement of halogen. Examples are Et 2-bromo-1-amino-4-sulphoanilino-, 1-amino-4-anilino- or -methylamino-2-sulpho-anthraquinone-6-carboxylates. C. H.

**Manufacture of acid wool dyes of the anthraquinone series.** I. G. FARBENIND. A.-G. (B.P. 392,056, 26.9.32. Ger., 26.9.31).—Anthraquinones containing replaceable substituents in positions 1 and 4 are condensed with a 4-aminodiphenyl and the product is sulphonated to give green wool dyes. With leucoquinizarin or 1:4-dichloroanthraquinone 4-amino- and 4-amino-3-methyl-diphenyl give intermediates, m.p. 252—254° and 263—264°, respectively; Et 4-amino-diphenyl-4'-carboxylate, m.p. 86—87° (from the 4- $NO_2$ -ester, m.p. 113—115°), may also be used. 5:8-Dichloro-1:2-benzanthraquinone with 4-aminodiphenyl gives after sulphonation a yellow-green dye. C. H.

**Manufacture of sulphonic acids of the anthraquinone [anthra-pyridone and -pyrimidone] series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 393,331, 8.12.31).—The halogen in 2-halogeno-1:9-anthra-pyridones or -pyrimidones, carrying  $\cdot NHR$  in position 4 (R = alkyl, aryl, aralkyl, or hydroaryl), is replaced by  $SO_3H$  by reaction with, e.g.,  $Na_2SO_3$  at 130—180°, in a suitable solvent ( $H_2O$  and  $PhOH$ , aq. EtOH). In the examples R = Ph. The products give violet shades on wool. C. H.

[**Manufacture of chrome wool**] **dyes of the anthraquinone series.** IMPERIAL CHEM. INDUSTRIES, LTD.,

F. LODGE, and C. H. LUMSDEN (B.P. 393,316, 2.12.31).—2-Halogeno-4-aminoarylamino-1-alkylaminoanthraquinones are obtained by hydrolysis with  $H_2SO_4$ , in presence of  $H_3BO_3$ , of the corresponding 1-acylalkylamino-compounds, prepared, e.g., by condensing 2:4-dibromo-1-acetmethylamidoanthraquinone with  $p-C_6H_4(NH_2)_2$  (hydrolysed product, m.p. 232—233°; sulphonated to give a chrome black for wool) or *N-p*-aminophenylmorpholine. C. H.

**Manufacture of dyes for dyeing cellulose esters and ethers.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 391,859, 6.11.31).—1:4:5-Tri- or 1:4:5:8-tetra-aminoanthraquinone is treated with alkylating agents so as to leave < 1 unalkylated  $NH_2$  group; e.g., with MeOH and  $H_2SO_4$  at 130—140° for 1.5 hr. C. H.

**Manufacture of water-insoluble dyes [pigments and ice colours].** I. G. FARBENIND. A.-G. (B.P. 392,011—2, [A] 16.7.32, [B] 18.7.32. Ger., 22.7.31).—(A) 3-Amino- and (B) 2-amino-diphenylsulphones are diazotised and coupled in substance or on the fibre with certain 2:3-hydroxynaphthoic arylamides, viz., anilides carrying (A) a 2-alkyl group or their 4-halogen derivatives, (B) 2-alkyl and 4-alkoxyl or 4-Cl, or a 2-alkoxyl with or without 4-Cl. The shades are orange or red-orange, fast to light. C. H.

**Manufacture of azo dyes.** I. G. FARBENIND. A.-G. (B.P. 392,452, 5.11.32. Ger., 30.11.31).—Dyes sol. in oils, fats, waxes, and org. solvents are obtained by coupling a diazotised aminophenylcyclohexane or a tetrazotised di(aminophenyl)cyclohexane with a component carrying a hydroaromatic nucleus,  $H_2O$ -solubilising groups being absent. Examples include: 1:1-di-(6-amino-*m*-tolyl)cyclohexane  $\rightarrow$  2 mols. *p*-hydroxyphenylcyclohexane (yellow) or *ar*-tetrahydro- $\beta$ -naphthol (orange); (6-amino-*m*-tolyl)cyclohexane  $\rightarrow$  *p*-hydroxyphenylcyclohexane (green-yellow). C. H.

**Manufacture of azo dyes.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 395,820, 1.4.32).—Oil-sol. azo dyes of good fastness to light are obtained by coupling tetrazotised  $(NH_2)_2$ -compounds, prepared by condensing aromatic amines (2 equivs.) with aliphatic ketones, with phenols, both dye components being free from  $SO_3H$  or  $CO_2H$ . Examples are 4:4'-diamino- $\beta$ - $\beta$ -diphenylpropane (I)  $\rightarrow$  *p*-cresol, 4:4'-diamino-3:3'-dimethoxy- $\beta$ - $\beta$ -diphenylpropane (from COMe<sub>2</sub> and *o*- $NH_2 \cdot C_6H_4 \cdot OMe$ )  $\rightarrow$  *p*-cresol, (I)  $\rightarrow$  quinol Me ether. H. A. P.

**Manufacture of monoazo dyes.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 395,832, 25.4.32).—Dyes of the metachrome series are prepared by coupling diazotised picramic acid with derivatives of  $m-C_6H_4(NH_2)_2$  in which one or both  $NH_2$ -groups are substituted by hydroxyalkyl groups. Examples of coupling components are mono- (deep brown on mordant) and *as*-di- $\beta$ -hydroxyethyl-*m*-phenylenediamine (bordeaux), mono- and *s*-di- $\beta$ - $\gamma$ -dihydroxypropyl-*m*-phenylenediamine (both violet-brown), 4-chloro-3-amino-1- $\beta$ -hydroxyethylamine (reddish-brown) and -1- $\beta$ - $\gamma$ -dihydroxypropylamine (brown), and 4- $\beta$ - $\gamma$ -dihydroxypropyl-*m*-tolylene-diamine (violet-brown on Cr mordant). H. A. P.

**Manufacture of azo dyes.** DURAND & HUGUENIN A.-G. (B.P. 395,951, 6.1.33. Ger., 14.1.32. Cf. B.P.



220,303; B., 1926, 43).—Dyes for acetate silk are prepared by nitration of derivatives of azobenzene containing both  $\text{SO}_3\text{H}$  and a dialkylamino-group in the same or different nuclei. Examples of starting materials are metanilic acid (I)  $\rightarrow$   $\text{NPhEt}_2$  (reddish-orange), (I)  $\rightarrow$   $m\text{-C}_6\text{H}_4\text{Me}\cdot\text{NEt}_2$  (yellowish-red), (I)  $\rightarrow$   $m\text{-C}_6\text{H}_4\text{Cl}\cdot\text{NEt}_2$  (reddish-orange), (I)  $\rightarrow$   $m\text{-C}_6\text{H}_4(\text{OEt})\cdot\text{NEt}_2$  (orange), (I)  $\rightarrow$   $\text{NPhMeBu}^a$  (bright orange),  $o\text{-NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{SO}_3\text{H}$  (II)  $\rightarrow$   $\text{NPhEt}_2$  (ruby-red), (II)  $\rightarrow$   $m\text{-C}_6\text{H}_4\text{Me}\cdot\text{NMe}_2$  (red), and  $2:5\text{-C}_6\text{H}_3\text{Cl}_2\cdot\text{NH}_2$   $\rightarrow$   $m\text{-C}_6\text{H}_4\cdot\text{NMe}_2\cdot\text{SO}_3\text{H}$  (bluish-red).

H. A. P.

**Manufacture of an azo dye [red ice colour] and intermediate product [base] therefor.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 391,468, 14.9.31).—2-Nitroanisole-4-sulphondiethylamide, m.p. 77–78°, obtained from the sulphonyl chloride, m.p. 66°, and  $\text{NHEt}_2$ , or from 1-chloro-2-nitrobenzene-4-sulphonyl chloride, m.p. 40–41°, is reduced; or the 2- $\text{NH}_2\text{Ac}$ -compound, m.p. 112°, prepared from 2-acetamidoanisole-4-sulphonyl chloride, m.p. 149°, and  $\text{NHEt}_2$ , is hydrolysed. The resulting *o*-anisidine-4-sulphondiethylamide, m.p. 104–105°, when diazotised and coupled with 2:3-hydroxynaphthoic 5-chloro-2:4-dimethoxyanilide on the fibre gives a Turkey-red dyeing fast to light, weather,  $\text{Cl}_2$ , and boiling  $\text{Na}_2\text{CO}_3$  or  $\text{NaOH}$ .

C. H.

**Manufacture of complex metallic compounds of *o*-hydroxyazo dyes.** I. G. FARBENIND. A.-G. (B.P. 395,968, 27.1.33. Ger., 10.5.32. Cf. B.P. 296,819; B., 1928, 849).—*o*-Alkoxyazo dyes are treated with neutral, acidic, or basic Cr sulphates under such conditions that the alkyl group is replaced, *e.g.*, in presence of  $\text{H}_2\text{O}$  at 130–140°. Complex Cr compounds of the corresponding *o*-hydroxyazo dyes having good fastness properties on wool and silk are thus prepared from 5-chloro-2-methoxyaniline (I)  $\rightarrow$   $\alpha$ -naphthol-6:8-disulphonic acid (violet), (I)  $\rightarrow$   $\alpha$ -naphthol-3:8-disulphonic acid (blue), 6-nitro-4-methoxy-*m*-toluidine  $\rightarrow$   $\alpha$ -naphthol-4:8-disulphonic acid (navy-blue).

H. A. P.

**Manufacture of disazo dyes [for cellulose esters and ethers] and their application.** IMPERIAL CHEM. INDUSTRIES, LTD., and A. H. KNIGHT (B.P. 391,862, 6.11.31).—Dyes of the type: arylamine of the  $\text{C}_6\text{H}_5$  or  $\text{C}_{10}\text{H}_8$  series, free from  $\text{NO}_2$ ,  $\text{SO}_3\text{H}$ , and  $\text{CO}_2\text{H}$ ,  $\rightarrow$  2:5-dimethoxyaniline  $\rightarrow$  1:5-aminonaphthol, give violet to blue to black shades fast to light. Suitable 1st components are  $o\text{-C}_6\text{H}_4\text{Cl}\cdot\text{NH}_2$ , cresidine, and  $\alpha\text{-C}_{10}\text{H}_7\cdot\text{NH}_2$ .

C. H.

**Manufacture of primary disazo [acid wool] dyes.** W. W. GROVES. From J. R. GEIGY A.-G. (B.P. 391,626, 2.6.32).—A 1:8-aminonaphtholsulphonic acid is coupled with a diazotised aminoarylsulphonamide (except *o*-aminophenolsulphonamides), and then coupled alkaline with any suitable diazo-compound. Examples include: *o*-chloroaniline-5-sulphonanilide  $\rightarrow$  H-acid  $\leftarrow$  *p*-toluidine-2-sulphonanilide (blue-black) or *p*-aminophenol *O*-*p*-toluenesulphonate (blue-black); *p*-chloroaniline-2-sulphonethylamide (m.p. 119°)  $\rightarrow$  H-acid  $\leftarrow$  2-amino-2'-ethylidiphenyl ether (blue-black).

C. H.

Dyes.—See III.

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

**Scouring of wool fibre.** I, II. Effect of the scouring temperature. H. SOBUE and K. ONO (J.

Soc. Chem. Ind., Japan, 1933, 36, 213–218 B).—Measurements are recorded of the amounts of grease and suint removed by scouring under standard conditions at different temp. for merino (I) and crossbred (II) wools. Removal of suint and grease from (I) increases slowly up to 35° and then rapidly to a max. at 45°. Suint removal from (II) shows a max. at 35°, decrease being slow up to 45°; grease removal shows a max. at 45°, which is therefore the optimum temp. for scouring.

A. A. L.

**Effect of alkalis on the degumming of silk.** V, VI. I. TOYODA (J. Soc. Chem. Ind., Japan, 1933, 36, 368–370 B; cf. B., 1932, 415).—Silk may be degummed without damage by boiling with 0.001N-NaOH or 0.007N- $\text{Na}_2\text{CO}_3$  to which a small amount of soap is preferably added.

A. G.

**Production of hemicellulose from evergreen and leaf-tree waste.** III. O. ROUTALA and J. O. MURTO (Finnish Paper Timber J., 1933, 95–100).—Barked birch waste (loss, 28%) when heated for 4 hr. at 100° with 2.5% NaOH gave 65% of pulp. Unbarked birch, aspen, and alder when heated for 8 hr. with NaOH (10% of wt. of wood) gave pulp yields of 83, 82, and 78%, respectively. Vals. are given for ash, extract, lignin, and pentosan contents of woods and the corresponding hemicelluloses. Spruce gave cellulose yields of 70–77% based on hemicellulose used, or 53–60% based on wt. of barked wood used. Similarly birch gave 64.5–68.5% or 54–56%.

CH. ABS.

**State of the cellulose molecule in solution.** K. ATSUKI and M. ISHIWARA (J. Soc. Chem. Ind., Japan, 1933, 36, 349 B).—Particles of cellulose nitrate obtained by the coagulation of a 1% solution in  $\text{COMe}_2$  are almost spherical and 2–3  $\mu$  in diameter; the particles from 0.01% solution have length/width  $< 5$  and it is estimated that the particles from an infinitely dil. solution have a mass of  $10^{-18}$  g. or 600,000 times the mol. wt.

A. G.

**Action of caustic liquor on native and mercerised cellulose.** I. OKAMURA (Naturwiss., 1933, 21, 393–394).—The heat of reaction of mercerised cellulose with NaOH solution, obtained by subtracting the heat of wetting from the gross heat developed, differs from that of native cellulose. With concns. of NaOH up to 20% the difference is not const. but with higher concns. (25–40%) the difference is const. and apparently corresponds with the heat of mercerisation.

W. O. K.

**Glass spheres for viscosity determination of cuprammonium solutions of cellulose.** L. S. GRANT, JUN. and W. M. BILLING (Ind. Eng. Chem. [Anal.], 1933, 5, 270–271).—Glass spheres having a const. rate of fall through liquids can be selected from materials of different *d* by adjusting their diam. (by grinding) in accordance with a linear relation between diam. and *d*. The spheres are segregated into batches of similar *d* by floating in mixtures of  $\text{C}_2\text{H}_2\text{Br}_4$  (*d* 2.95) and  $\text{C}_2\text{H}_2\text{Cl}_4$  (*d* 1.588).

E. S. H.

**Hydromechanics of viscose and mechanism of coagulation of viscose in the spinning process.** III. Theoretical investigation of viscose spinning viscosimeter. H. SOBUE (J. Soc. Chem. Ind., Japan, 1933, 36, 351–354 B; cf. B., 1933, 619).—Data are



given for computing the diam. of a viscosimeter capillary having a pressure distribution similar to that in a spinning nozzle. A. G.

**Effect of impurities in the mixed acid on the stability and nitrogen content of cellulose nitrates and on the viscosity of cellulose nitrate solutions.** E. BERL and G. RUEFF (Cellulosechem., 1933, 14, 109—115).—Nitration of cellulose with anhyd.  $\text{HNO}_3\text{--H}_2\text{PO}_4$  is complete in  $1\frac{1}{2}$  min., but prolonged treatment does not affect the % N or the viscosity. When mixed acid is used repeatedly it becomes contaminated with oxidation products of cellulose and with nitrosylsulphuric acid (*A*).  $\text{H}_2\text{C}_2\text{O}_4$  has no detrimental effect on nitration, but *A* diminishes the % N, the viscosity, and the stability, and increases the % combined  $\text{H}_2\text{SO}_4$ . Stabilisation is promoted by swelling, and of a no. of solutions tested the best was 50%  $\text{AcOH}$  + 2%  $\text{NaOAc}$ ; the sulphuric ester content is diminished by acid stabilisation. A. G.

**Viscosity of solutions of cellulose esters.** E. BERL and G. RUEFF (Cellulosechem., 1933, 14, 115—119).—Repeated nitration of cellulose with the same mixture causes a greater fall in the viscosity of the nitrate if this is denitrated between each nitration. Use of an anhyd.  $\text{H}_3\text{PO}_4\text{--HNO}_3$  leads to a very high-viscosity nitrate. A. G.

**Viscosity measurements with cellulose acetates.** D. KRÜGER (Cellulosechem., 1933, 14, 120—122).—Cellulose acetates prepared by different methods, which had equal viscosities as 2% solutions in  $\text{HCO}_2\text{H}$  but different viscosities as 10% solutions in  $\text{COMe}_2$ , have different concn.-viscosity relations in  $\text{COMe}_2$ , and when the solutions are more dil. the viscosities are equal. A. G.

**Washing effect.**—See XII. Saccharification of wood.—See XVII.

See also A., Aug., 779, Viscosity of cellulose nitrate and cuprammonium cellulose solutions. 788, Rate of reaction of cellulose with cuprammonium. Kinetics of cellulose reactions. 790, Kinetics of cellulose acetylation. 812, Determination of I val. of cellulose derivatives. Ethylation of cellulose. Benzylcelluloses. Cellulose contents of woods. 849, Fine structure of silk.

#### PATENTS.

**Fibration of emulsions.** E. O. GROSKOPF, Assr. to FLINTKOTE CORP. (U.S.P. 1,889,473, 29.11.32. Appl., 22.4.29).—The fibrous matter, disintegrated in air to such an extent that each fibre is apart from its neighbour, is blown into the emulsion. B. M. V.

**Oiling textile fibres.** I. G. FARBENIND, A.-G. (B.P. 392,027, 4.8.32. Ger., 13.8.31).—Textiles composed of or containing animal fibres are oiled with an aq. solution of alkali or  $\text{NH}_4$  salts of sulphonic acids  $\text{R}\cdot\text{X}\cdot\text{R}'\cdot\text{SO}_3\text{H}$  or alkyl H sulphates,  $\text{R}\cdot\text{O}\cdot\text{SO}_3\text{H}$ , where  $\text{R} = \text{alkyl} > \text{C}_6$ ,  $\text{X} = \cdot\text{CH}_2\cdot\text{O}\cdot, \cdot\text{CO}_2\cdot, \cdot\text{CO}\cdot\text{NH}\cdot$  or  $\cdot\text{CO}\cdot\text{NR}''\cdot$ , and  $\text{R}'$  and  $\text{R}'' = \text{alkyl}$  or aryl. Examples are: Na oleyloxyethanesulphonate, Na oleyl-*N*-methyltaurine, Na dodecyl or cetyl sulphate, K oleyl-*N*-phenyltaurine,  $\text{NH}_4$   $\gamma$ -stearilyoxypropanesulphonate, K *N*-oleylidiphenylaminesulphonate,  $\text{NH}_4$  oleyl-*N*-isopropylxylydinesulphonate, Na sulphophenylpalmitate. C. H.

**Rigidifying composition [hair fixative].** B. N. LOUGOVOY, Assr. to ELLIS-FOSTER CO. (U.S.P. 1,884,015, 25.10.32. Appl., 18.1.28).—Proteins of the gliadin-zein type which are sol. in aq. EtOH are used (a) in the prep. of hair-waving compositions, hair fixatives, etc., (b) as stiffening and sizing agents for fabrics, (c) as adhesives in the manufacture of laminated paper products. D. J. N.

**Waterproof [-sheet] fibrous product.** H. L. LEVIN, Assr. to FLINTKOTE CORP. (U.S.P. 1,884,761, 25.10.32. Appl., 22.11.30).—The product consists of 35% of fibrous material (wood pulp, animal fibre, asbestos, leather, etc.), a waterproof binder having thermoplastic properties (bitumen, rubber, etc.), and a filler (cork, sawdust, rosin powder, etc.). A. J. H.

**Production of soft fabric or the like from vegetable fibrous materials.** F. THIES (U.S.P. 1,885,019, 25.10.32. Appl., 5.5.30. Ger., 16.7.28).—Cotton fabric is shrunk  $\leq 5$  and 8% warpwise and weftwise respectively by treatment with NaOH of mercerising concn., the weaving specification having been adjusted to allow this shrinkage. A. J. H.

**Manufacture of [hydroxyalkyl] ether derivatives of cellulose.** C. F. BURGESS LABORATORIES, INC., Asses. of A. W. SCHORGER (B.P. 392,206, 14.8.31. U.S., 14.8.30).—A wet alkali-cellulose from undegraded cellulose, with  $\geq 24$  hr. ageing, is treated, preferably at  $< 45^\circ$ , with ethylene oxide (up to 20% of wt. of cellulose) or other alkylene oxide, with or without org. solvent, to give an ether containing  $< 1$  hydroxyalkyl per  $\text{C}_6$  cellulose unit. The products are sol. or partly sol. in dil. NaOH, insol. or slightly sol. in aq. KOH, insol. in  $\text{H}_2\text{O}$  and org. solvents, and may be made into filaments, films, etc. C. H.

**Preparation of cellulose solutions, and production of artificial threads, films, bands, and such-like artificial products therefrom.** J. C. DE NOOLJ and D. J. GERRITSEN (B.P. 395,947, 31.12.32. Holl., 5.4.32).—Cellulose is dissolved in  $\text{H}_2\text{SO}_4$  of  $> 60\%$  concn., and diluted with  $\text{H}_2\text{O}$  or other diluent, at reduced temp. and pressure if desired, to an acid concn.  $< 60\%$ ; the metastable solution is spun into a coagulating medium, e.g.,  $\text{H}_2\text{O}$ , EtOH, etc. F. R. E.

**Manufacture or treatment of products or articles made of or containing cellulose derivatives.** H. DREFFUS (B.P. 395,353, 30.11.31).—1:3-Cyclic diethers containing  $\leq 5$  atoms in the ring, e.g., 1:3-cyclotri- or tetra-methylene oxide, are used as solvents for the spinning of cellulose derivatives both of normal and high Ac content (up to 62.5%), also in the coagulation bath, and as agents for swelling or softening of the filaments prior to or simultaneously with stretching, dyeing, printing, delustring, etc. F. R. E.

**Manufacture of artificial threads.** R. E. ELLIS. From Soc. FOUR LA FABR. DE LA SOIE "RHODIASETA" (B.P. 395,440, 17.2.32).—Normal solutions of cellulose acetate, preheated to a viscosity  $\geq 100$  e.g.s. units, are spun downwards in a dil. evaporative atm. while subjected to only a slight stretching (ratio  $\geq 2$ ); the filaments have an elongation at breaking of  $> 30\%$ , and the usual breaking load. F. R. E.



**Manufacture [precipitation] of cellulose derivatives.** BRIT. CELANESE, LTD., J. E. JONES, and D. R. JOHNSTON (B.P. 395,707, 18.1.32).—The esterification solution is treated with a substantial proportion of the precipitant (*A*) just insufficient to effect permanent pptn., and is then mixed with more *A* so as to ppt. the ester in finely-divided form. F. R. E.

**Production of endless bands of gelatin, cellulose derivatives, and the like.** C. W. I. THOMAS. From FOLIEN-U. FLITTERFABR. A.-G. (B.P. 395,816, 16.3.32).—An endless conveyor band passes through a solution of the material and then rises vertically, the thickness of the film layer being adjusted by varying the concn. of the solution or the speed of the band; coating material is removed from the edges of both sides of the band by scrapers. F. R. E.

**Compositions containing cellulose derivatives.** H. DREYFUS (B.P. 395,694, 15.1.32).—Org. derivatives of cellulose are dissolved in one or more org. liquids, e.g.,  $\text{COMe}_2$ , containing  $\lt 7\%$  of  $\text{H}_2\text{O}$  together with  $\gt 5$  (0.5–1.5)% of a basic org. substance (aliphatic amines or hydroxyamines such as  $\text{NMe}_3$  or ethanolamine); the composition has reduced corrosive action on the ferrous materials composing the containers or the spinning apparatus. F. R. E.

**Manufacture or treatment of solutions, plastic masses, films, or the like, containing lacquer bases. Manufacture or treatment of artificial filaments, threads, ribbons, yarns, fabrics, etc. [Manufacture and use of cyclic ethers from trihydric alcohols.]** H. DREYFUS (B.P. 392,186 and 392,160, 5.11.31).—The cyclic ethers obtainable by condensation of a trihydric alcohol (glycerol) with a di- or tri-hydric alcohol or an aliphatic ketone, or ethers or esters of such ethers, are used (A) as solvents, softeners, and plasticisers for cellulose esters and ethers, lacquer bases (especially natural and synthetic resins), and in the manufacture of films, foils, etc. by wet or dry processes, and of dopes, varnishes, plastics, and moulding powders; (B) as solvents in the manufacture of spinning solutions for artificial threads etc., for treatment (softening or re-lustring) of fabrics or yarns of cellulose esters or ethers, and as printing assistants. The preferred ether is diglycerol triether, b.p. 168–176°, obtained from glycerol and acid dehydrating agents. C. H.

**Improving sheets and films of colloids.** I. G. FARBENIND. A.-G. (B.P. 395,243, 1.12.32. Ger., 2.12.31).—The mechanical properties of films etc. prepared from highly polymerised colloids, e.g., cellulose derivatives or albuminoid substances (gelatin), are considerably altered without adversely affecting other properties by removing any solvent left in the film after manufacture, together with the whole or part of the softening agents. This is effected by extraction for, e.g., 24 hr. with a volatile non-aq. liquid which is a non-solvent for the colloid and exerts only a slight swelling action thereon. Suitable liquids are  $\text{Et}_2\text{O}$  for nitrocellulose and  $\text{MeOH}$  for cellulose acetate or gelatin. D. J. N.

**Recovery of relief material [vented from the sulphite-pulp process].** W. H. KENETY, Assr. to CHEMIPULP PROCESS, INC. (U.S.P. 1,885,918, 1.11.32. Appl., 17.1.31).—A system of blowing off a digester into

accumulators in which the heat and chemicals are caught in future digester charges is described, a combination of injection and surface condensation being utilised. B. M. V.

**Manufacture or treatment of paper for use in electrical insulation.** STANDARD TELEPHONES & CABLES, LTD., T. R. SCOTT, and M. C. FIELD (B.P. 395,737, 23.10.31).—Cellulose paper or disintegrated fibres, which have been pretreated with boiling 2% aq.  $\text{NaOH}$  under pressure in absence of air to remove fatty and waxy matter, are partly acetylated with  $\text{Ac}_2\text{O}$ , glacial  $\text{AcOH}$ , and a dehydrating catalyst ( $\text{ZnCl}_2$ ) to an *Ac* content approx. equiv. to that of cellulose monoacetate, then washed free from the reagents, and sheeted; the resulting paper has improved insulating properties with little or no reduction in mechanical strength, owing to its reduced affinity for  $\text{H}_2\text{O}$ . F. R. E.

**Sizing of fibrous material [paper].** F. J. CURTIS, Assr. to MERRIMAC CHEM. CO., INC. (U.S.P. 1,885,185, 1.11.32. Appl., 10.8.29).—In the rosin sizing of paper, the use of  $\text{Na}$  aluminate instead of alum prevents injury due to acidity of the pulp ( $p_{\text{H}} \lt 6$ , instead of about 5) and permits the presence of sufficient colloidal  $\text{Al}(\text{OH})_3$  for proper sizing. B. M. V.

**Centripetal separator [for pulp].**—See I. Wetting etc. agents.—See III. Multi-coloured cellulosic materials.—See VI. Abrasive articles.—See VIII. Acetylated gum [sheets].—See XVII.

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

**Comparison of bleaching of cotton cellulose by means of aqueous calcium and sodium hypochlorite.** D. GALPERIN, D. TUMARKIN, and B. KOZLOVSKI (J. Appl. Chem. Russ., 1933, 6, 536–545).—The oxidising and bleaching properties of solutions of equal active *Cl* content increase in the order alkaline  $\text{NaOCl} \lt$  neutral  $\text{NaOCl} \lt$   $\text{CaOCl}_2$ ; in the concns. used industrially, however, the bleaching effect is practically identical in all cases. At 30° the destructive effect of  $\text{CaOCl}_2$  on the fibres is  $\gt$  that of  $\text{NaOCl}$ , which is for this reason preferable for bleaching at temp.  $\gt$  the ordinary. R. T.

**Application of spectrophotometric colour analysis to dyeing.** O. R. PINEO (Amer. Dyestuffs Rep., 1933, 22, 470–474).—Light-absorption curves obtained by means of an M.I.T. Colour Analyser are given for three depths of dyeings of Fast Wool Red GL and Fast Light Yellow 2G and their mixtures on wool, and Pontamine Yellow CH and Fast Silk Blue 3GL on silk; their significance is discussed briefly. A. J. H.

## PATENTS.

**Cleaning of fibrous materials, textiles, articles of clothing, and the like.** DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 395,517, 21.7.32. Ger., 21.12.31).—The materials are treated in a closed apparatus with perchloroethylene, the residual solvent being subsequently removed by heated air under reduced pressure. F. R. E.

**[Degreasing and wetting] solvent product for the textile industry.** R. MEUNIER (B.P. 394,843, 19.9.32).



—A product suitable for adding to scouring, bleaching, and dyeing liquors, and for impregnating cotton materials immediately before mercerising, is prepared by sulphonating ( $\gt 35^\circ$ ) a mixture of fatty substances (*e.g.*, castor oil, but not fish oils) and solvents of such substances (*e.g.*,  $C_6H_6$ ,  $COMe_2$ , and turpentine), and saponifying the product at  $60-70^\circ$  with a mixture of NaOH and EtOH.  
A. J. H.

**Desizing and bleaching of textile materials with liquids.** H. D. ELKINGTON. From EBERGSCHER STOOMBLEEKERIJ VOORH. G. J. TEN CATE & ZONEN (B.P. 395,306, 20.3.33).—The material is treated successively with solutions containing a starch-liquefying enzyme, active Cl, and  $H_2O_2$  within a kier made of or coated with a resistant Fe alloy containing Cr, Ni, a small amount of C, and  $\lt 2\%$  of Mo (preferably Cr 18, Ni 8, C 0.15, and Mo 34%) and provided with a middle perforated annular zone so that the solution may be supplied from the top and bottom and withdrawn from the middle.  
A. J. H.

**[Oxidation] treatment of vegetable and animal fibres and fabrics.** R. FEIBELMANN (U.S.P. 1,892,548, 27.12.32. Appl., 14.9.31. Ger., 19.4.30).—Suspensions of  $p-C_6H_4Me \cdot SO_2 \cdot NCl_2$  (A) in  $H_2O$  or solutions in dil. alkalis (*e.g.*, NaOH,  $Na_2CO_3$ ,  $Na_2SiO_3$ , etc.) are used for bleaching and desizing textile materials, for chlorinating wool, stripping dyed goods, and oxidising materials dyed with vat and Indigosol dyes. A is insol. in dil. acids but is then most reactive.  
A. J. H.

**Production of fast [vat] dyeings.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 391,252, 14.12.31).—Dyeings fast to light are obtained by using a mixture of at least 10% of a vat dye of the type (I) (B.P. 24,604/08, 337,741, 339,396, 348,671, 358,074; B., 1909, 832; 1931, 239, 338, 835; 1932, 175) and at least one other vat dye.  
C. H.

**Dyeing of cellulose esters and ethers [in green shades].** I. G. FARBENIND. A.-G. (B.P. 391,987, 25.5.32. Ger., 26.5.31).—The material is dyed with a  $H_2O$ -insol. aminoazobenzene,  $NH_2 \cdot Ar(OR) \cdot N_2 \cdot Ar' \cdot NR'R''$ , where  $R'$  and  $R''$  = alkyl, hydroaryl, or aralkyl or  $\cdot NR'R'$  = a cyclic radical; or with a  $H_2O$ -insol. aminoalkoxybenzeneazotetrahydro-naphthylamine or  $N$ -alkylnaphthylamine. The aminoazo compound is diazotised on the fibre and coupled with a 6-alkoxy- or 6-alkyl-2:3-hydroxynaphthoic acid. Examples include:  $p-NH_2 \cdot C_6H_4 \cdot NMe_2 \rightarrow o$ -anisidine  $\rightarrow$  6-OMe-acid; 4-nitro-2:5-dimethoxyaniline  $\rightarrow ar$ -tetrahydro- $\alpha$ -naphthylamine, or phenylmorpholine, reduced  $\rightarrow$  6-OMe-acid.  
C. H.

**Production of multi-coloured artificial cellulosic materials in the form of sheets, bands, or films.** K. S. LOW (B.P. 395,864, 21.6.32).—Variously coloured aq. solutions of cellulose derivatives capable of regeneration are added to a similar colourless or coloured solution immediately before extrusion in such a fashion that regeneration is achieved and the colours are fixed before noticeable diffusion occurs. The solutions may be fed at various points in the nozzle of the extruding device. The solutions may be coloured by sol. or pigment dyes,

and, if necessary, a regeneration process can be applied to dyes decolorised in the process. Opaque effects are produced by the presence of inert fillings.  
H. A. P.

**Dyeing and printing.** I. G. FARBENIND. A.-G. (B.P. 393,408, 9.5.32. Ger., 8.5.31).—Colours of the "Rapid Fast" type are developed on the fibre by an acid bath comprising 1 or more org. acids or  $H_3PO_4$  or acid salts, and the material is thereupon dried by heat. The process is suitable for colour resists under aniline-black, in conjunction with leuco-vat dye sulphuric esters if desired. Prints of such colours together with the sulphuric esters, or with these in separate patterns, may similarly be developed, preferably with addition of  $KClO_3$  and an oxidation accelerator,  $Al_2(SO_4)_3$  being present in the acid padding solution applied to the material before drying. Colour resists on leuco-ester dyeings are obtained by padding with the ester and oxidant ( $NaNO_2$ ,  $K_2CrO_4$ ), printing with a colour of the "Rapid Fast" type and a resist agent, drying the material, and padding it with the acid compounds mentioned [with or without  $Al_2(SO_4)_3$ ], and finally drying by heat. The material may first be printed with a resist paste containing the "Rapid Fast" colour, dried, padded with the leuco-ester,  $KClO_3$ ,  $NH_4VO_3$ , an agent yielding acid, and an  $NH_4$  salt of a weak org. acid, dried on heated cylinders, and steamed to develop the leuco-ester.  
C. H.

**Production of impregnating baths and printing colours containing arylides of 2:3-hydroxynaphthoic acid.** H. T. BÖHME A.-G. (B.P. 395,853, 4.6.32. Ger., 9.7.31).—Sulphuric or phosphoric esters containing  $\lt 9$  C atoms and residual OH groups are used as dispersing agents, stable to hard  $H_2O$ , acids, and alkalis, for the prep. of bottoming baths or printing pastes containing 2:3-hydroxynaphthoic arylides. Examples are the H sulphate of octadecanetriol, obtained by catalytic reduction of ricinoleic acid to the alcohol followed by treatment with  $H_2SO_4$  at  $-5^\circ$  to  $5^\circ$ , a complex ester from mannitol, oleic acid, and  $H_2SO_4$ , and Pesters from  $\alpha\mu$ -octadecanediol, octadecenyl alcohol, and "ricinoleyl alcohol" and  $PCl_3 \cdot AcOH$ ,  $AcCl \cdot H_3PO_4$ , and  $Ac_2O \cdot H_3PO_4$ .  
H. A. P.

**Production of fast prints on cellulose ester fabrics.** DURAND & HUGUENIN (B.P. 395,949, 3.1.33. Ger., 6.1.32. Cf. B.P. 395,951; B., 1933, 417).—Derivatives of azobenzene containing  $SO_3H$ ,  $NO_2$ , and mono- or di-alkylamino-groups, and in addition other substituents if desired, are used in the printing of cellulose esters.  
H. A. P.

**Manufacture of wetting agents for mercerising liquors.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 392,636, 3.12.31).—Oily products (alcohols, ketones, fatty acids, and hydrocarbons of  $C_5-C_7$ ), b.p.  $120-160^\circ$ , obtained from C oxides and  $H_2$ , *e.g.*, according to B.P. 229,714 and 238,319 (B., 1925, 338, 900), are treated with  $ClSO_3H$  or other sulphonating agent at  $80^\circ$ , and preferably at once neutralised with alkali.  
C. H.

**Frosting process [for fabrics etc.].** M. L. MESA (U.S.P. 1,888,969, 22.11.32. Appl., 19.3.30).—The article is dipped twice in a solution of alum and gum



arabic, and after formation of the crystals by drying they are dyed with an  $\text{NH}_2\text{Ph}$  dye and camphor in  $\text{EtOH}$  or other suitable solvent. B. M. V.

**Delustering of artificial silk.** IMPERIAL CHEM. INDUSTRIES, LTD., C. DUNBAR, and L. G. LAWRIE (B.P. 391,214, 22.10.31).—Artificial silks are treated, *e.g.*, for 30 min. at 60–95°, with a solution of tannic acid, a tannin, a tannate, or a tannin substitute, and a solution of a heterocyclic org. base derivative carrying a hydrocarbon chain  $> \text{C}_5$ , *e.g.*, cetyl- or octadecyl-pyridinium bromide or *N*- $\beta$ -hydroxyethyloctadecylmorpholinium bromide, with or without a subsequent treatment with a tannin-fixing agent (tartar emetic). C. H.

**Treatment [reduction of lustre] of artificial filaments, yarns, and the like [of cellulose derivatives].** BRIT. CELANESE, LTD. (B.P. 395,380, 14.1.32. U.S., 23.1.31).—The materials are treated with an aq. suspension or dispersion of a finely-divided, insol. salt of a higher fatty acid (Zn, Al, Mg, Ca, Sr, or Ba stearate, oleate, palmitate, cerotate, elaidate, or carnaubate) in presence of a swelling agent. F. R. E.

**Treatment of fibres or fabrics consisting of or containing cellulose acetate.** BLEACHERS' ASSOC., LTD., and G. D. SUTTON (B.P. 395,722, 21.12.31).—The materials are rendered resistant to delustering by treatment with aq.  $\text{AcOH}$ ,  $\text{HCO}_2\text{H}$ ,  $\text{EtCO}_2\text{H}$ ,  $\text{HCl}$ ,  $\text{HNO}_3$ , or  $\text{CH}_2\text{Cl}\cdot\text{CO}_2\text{H}$  sufficiently dil. to prevent swelling of the fibres, *e.g.*, with about 6% of the acid (calc. as  $\text{H}_2\text{SO}_4$ ) for 2 hr., and are then washed free from acid; after subjecting to crêping, scouring, dyeing, etc., the materials may, if desired, be rendered susceptible to delustering with the dil. acids succinic, lactic, or  $\text{H}_2\text{SO}_4$ , which are subsequently removed by washing with  $\text{H}_2\text{O}$ . F. R. E.

**[Apparatus for] coating or filling textile fabrics.** BRIT. CELANESE, LTD., A. MELIOR, and R. J. MANN (B.P. 394,685, 30.12.31).—A suitably thickened filling material is spread by means of a doctor knife over fabric while being drawn over a stationary rubber resilient bed. A. J. H.

**Treatment of textiles, paper, leather, skins, and other fibrous substances.** ERBA FABR. CHEM. PROD. SPEZIALITÄTEN F. DIE TEXTILIND. (B.P. 394,816, 29.6.32. Ger., 17.12.31).—Treatment is effected in a liquor containing emulsions of org. substances (*e.g.*, vegetable and mineral oils) and electrolytes so that it has an electric charge opposite to that of the material being treated; more complete absorption and better fixation of the org. substances are thereby secured. A. J. H.

**Mordants etc.**—See III. **Application of dyes.**—See IV. **Treating cellulose derivatives.** **Treating filaments etc.**—See V.

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

**Change of water-soluble phosphoric acid content of superphosphate during storage in bulk.** VIII. IX. T. SHOJI and E. SUZUKI (J. Soc. Chem. Ind., Japan, 1933, 36, 380–388 B; cf. B., 1932, 420, 545).—The sol.  $\text{P}_2\text{O}_5$  content of superphosphate on storage is

not affected by Al silicate or by  $\text{Al}_2(\text{SO}_4)_3$  into which any  $\text{AlPO}_4$ ,  $\text{Al}_2\text{O}_3$ , or  $\text{Al}(\text{OH})_3$  present in the original phosphate rock is converted by  $\text{H}_2\text{SO}_4$ . The insol.  $\text{P}_2\text{O}_5$  content is increased by adding  $\text{Al}(\text{OH})_3$  or bauxite to  $\text{CaH}_4(\text{PO}_4)_2$ , the insol.  $\text{P}_2\text{O}_5/\text{Al}_2\text{O}_3$  ratio being nearly 3 : 1. A small increase in insol.  $\text{P}_2\text{O}_5$  on adding  $\text{Al}_2\text{O}_3$  or diasporite is accompanied by formation of  $\text{CaHPO}_4$ ,  $\text{AlH}_6(\text{PO}_4)_3$ , and  $\text{Al}_2\text{H}_3(\text{PO}_4)_3$ . V. P. P.

**Utilisation of carbon monoxide obtained in the electro-distillation of phosphorus.** I. B. N. DOLGOV, B. A. BOLOTOV, and A. N. POPOVA. II. B. N. DOLGOV, B. A. BOLOTOV, and N. P. SILINA (J. Appl. Chem. Russ., 1933, 6, 470–478, 479–493).—I. The gas (I) obtained during the electro-distillation of P, containing 75–85% CO, can be freed of P,  $\text{PH}_3$ ,  $\text{P}_2\text{O}_5$ , and  $\text{O}_2$  by passing a 2 : 1 mixture of (I) and  $\text{H}_2\text{O}$  vapour over siderite ignited at 600–700° and then reduced by  $\text{H}_2$  at 500°; the activity of this catalyst is somewhat augmented by adding CuO, and its durability by adding  $\text{Al}_2\text{O}_3$  or  $\text{Cr}_2\text{O}_3$ .

II. The reaction  $\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{CO}_2 + \text{H}_2$  is catalysed by ignited siderite containing 10%  $\text{Al}_2\text{O}_3$  or  $\text{Cr}_2\text{O}_3$  (optimum reaction temp. 450–475°), and by a mixture containing  $\text{Al}_2\text{O}_3$  60,  $\text{Fe}_2\text{O}_3$  30,  $\text{Cr}_2\text{O}_3$  8, and  $\text{ThO}_2$  2% (reaction temp. 375–400°). R. T.

**Production of hydrogen sulphide and thiosulphates.** G. LYNN, M. ALLEN, B. K. BEECHER, and R. W. DARBYSHIRE (Ind. Eng. Chem., 1933, 25, 916–918).—S, soda ash, and  $\text{H}_2\text{O}$  are boiled under pressure in a steel vessel.  $\text{H}_2\text{O}$  is condensed out selectively from the evolved vapour and returned.  $\text{H}_2\text{S}$  escapes from a reducing valve. It may also be liquefied directly without releasing the pressure. The reaction is  $2\text{NaOH} + 4\text{S} + \text{H}_2\text{O} \rightarrow 2\text{H}_2\text{S} + \text{Na}_2\text{S}_2\text{O}_3$ . 125 lb. of  $\text{H}_2\text{S}$  are produced in a batch in 4 hr. The mother-liquor deposits  $\text{cryst. Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ . H. J. E.

**Recovery of sulphur dioxide as dilute sulphuric acid.** R. L. COPSON and J. W. PAYNE (Ind. Eng. Chem., 1933, 25, 909–916).— $\text{SO}_2$  and  $\text{O}_2$  are absorbed simultaneously from a gas stream by  $\text{H}_2\text{O}$  containing  $\text{MnSO}_4$  (optimum concn. 0.025% Mn). Johnstone's results are thus confirmed (cf. B., 1931, 613). The max. concn. of  $\text{H}_2\text{SO}_4$  obtainable is 40%. The reaction is strongly exothermic, the rate of absorption increasing with temp. The catalyst is poisoned by approx. 20 p.p.m. of  $\text{Fe}^{+++}$ ,  $\text{Cu}^{++}$ , or  $\text{Hg}^{++}$ ; Sb, As, Cr, Co, Pb, Mg, Mo, Ni, V, and Zn had no effect. Data for batch and continuous runs are summarised and the conditions for optimum absorption are discussed. H. J. E.

**Rectification. Evaporation.**—See I. **Phosphate slags.** Al shavings.—See X. **Burgundy mixture.**—See XVI. **Ferric floc.**—See XXIII.

See also A., Aug., 777, **Prep. of S sols.** 789, **Decomp. of  $\text{H}_2\text{O}_2$  by Fe.** Catalysts for prep. of  $\text{H}_2$  by the water-gas reaction. 790,  $\text{CH}_4$ - $\text{H}_2\text{O}$  reaction. **Catalytic oxidation of  $\text{NH}_3$ .** Catalysts for  $\text{H}_2\text{SO}_4$  production. **Oxidation of  $\text{As}_2\text{S}_3$  by atm.  $\text{O}_2$ .** 791, **Electrolytic oxidation or reduction.** 793, **Decomp. of  $\text{Na}_2\text{SO}_4$  by  $\text{H}_2$ .** **Prep. of chloride of CaO.** 794, **Blue ultramarine.** 795, **Pb oxides.** 797–8, **Determining small quantities of I.**



## PATENTS.

**Manufacture of chlorosulphonic acid and sulphur dioxide.** SOC. CHEM. IND. IN BASLE (B.P. 393,546, 8.2.33. Switz., 17.2.32).—S and  $2\text{Cl}_2$ , or the equiv. S chloride and  $\text{Cl}_2$ , react at  $20^\circ$  with 45% oleum to give  $\text{ClSO}_3\text{H}$  and  $\text{SO}_2$  practically quantitatively.

C. H.

(A) Production of phosphorus and phosphoric acid. (B) Treatment of phosphatic material. (A) W. H. WAGGAMAN and (A, B) S. D. GOOCH, ASSRS. to CORONET PHOSPHATE CO. (U.S.P. 1,888,896 and 1,888,919, 22.11.32. Appl., [A] 21.3.30, [B] 4.3.30).—(A) Sinters of phosphatic and siliceous material and fuel are treated in a blast furnace, the height of the charge being  $> 7\frac{1}{2}$  times the width, and a hot blast at  $760^\circ$  being used. (B) Moist phosphate rock or similar material is dried in a rotary kiln by combustion of P mixed with it in such quantity that  $\text{P}_2\text{O}_5$  is not, but  $\text{H}_2\text{O}$  is, volatilised and part of the rock is converted into the sol. form.

B. M. V.

**Manufacture of hydrocyanic acid gas, particularly for use as a fumigant or the like.** A. STANDEN, T. EWAN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 395,761, 23.1.32).—97–98% NaCN is caused to react with  $\text{NaHSO}_4$ , of which 75% passes an 80-mesh sieve, in presence of a quantity of  $\text{H}_2\text{O}$  equal to about 3.67 times the amount of NaCN.

W. J. W.

**Production of [concentrated] hydrocyanic acid from gases containing the same.** W. WETTSTEIN, ASSR. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,887,608, 15.11.32. Appl., 21.5.31. Switz., 3.6.30).—EtOAc dissolves, at  $-20^\circ$ , 150 g. and PrOAc 105 g. of HCN per litre from a gas containing HCN at 7.6 mm. partial pressure. The use as scrubbing agent of other carboxylic acid esters of b.p.  $> 100^\circ$  is also claimed.

B. M. V.

**Catalytic combustion of ammonia with oxygen or gases containing oxygen.** I. W. CEDERBERG (B.P. 394,406, 12.1.32. Ger., 12.1.31).—The  $\text{NH}_3$ -air mixtures (up to 35%  $\text{NH}_3$ ) are passed through narrow slot-like openings in a non-axial direction into the free space below the usual Pt-10% Rh gauze catalyst (I), at a speed of  $> 100$  m. per sec., so that the speed of the gases through (I) is  $>$  the linear velocity of explosion.

A. R. P.

**Dehydration of Glauber salt.** J. B. PIERCE, JUN., ASSR. to BARIUM REDUCTION CORP. (U.S.P. 1,886,580, 8.11.32. Appl., 15.9.31).—Liquid  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  (m.p.  $32^\circ$ ) is mixed with anhyd.  $\text{Na}_2\text{SO}_4$  in proportions to produce a substance like damp sand, so that neither caking nor melting takes place while raising the temp. to  $600^\circ$ .

B. M. V.

**Manufacture of ammonium sulphate or salts containing it.** S. HORI (U.S.P. 1,888,633, 22.11.32. Appl., 23.10.30).— $(\text{NH}_4)_2\text{SO}_3$  (A) is oxidised under pressure, in the presence of catalysts, and dissolved in a saturated solution of  $(\text{NH}_4)_2\text{SO}_4$  (B) to prevent, on the one hand, too great a concn. of A and, on the other, hydrolytic dissociation which hinders the oxidation, as also does excess  $\text{NH}_3$ . B is crystallised straight out together with other salts (constituents of mixed fertilisers) which may be added to the solution.

B. M. V.

**Production of bleaching powder.** P. PESTALOZZA (B.P. 394,373, 17.12.31. It., 22.12.30).—Finely-divided

$\text{CaO}$  is sucked into a cyclone chamber from which it falls through a second chamber into the reaction chamber which has the shape of a long inverted cone and up which is passed a mixture of  $\text{Cl}_2$  with sufficient air to absorb the heat of the reaction. The velocity of the gas mixture is so adjusted that it keeps the  $\text{CaO}$  in suspension until chlorination is complete.

A. R. P.

**Recovery of manganous compounds.** K. A. KOBE, ASSR. to BRADLEY-FITCH CO. (U.S.P. 1,889,021, 29.11.32. Appl., 29.11.29).—Mechanically and chemically reduced ore is digested in  $(\text{NH}_4)_2\text{CO}_3$ ,  $\text{NH}_3$  being allowed to blow off but being recovered; the solution after separation from the gangue is treated with  $\text{CO}_2$  and, later, with the recovered  $\text{NH}_3$ , whereby  $\text{MgCO}_3$  is pptd. and  $(\text{NH}_4)_2\text{CO}_3$  re-formed.

B. M. V.

**Preparation of lead chlorophosphate.** P. JOLIBOIS and G. CHAUDRON (U.S.P. 1,885,447, 1.11.32. Appl., 19.2.31. Fr., 27.2.30).—Natural  $\text{Ca}_3(\text{PO}_4)_2$  is ground and treated first with HCl, if necessary, to open it out, and then with aq.  $\text{PbCl}_2$ ; the ppt. formed has the composition  $\text{PbCl}_2 \cdot 3(\text{P}_2\text{O}_5 \cdot 3\text{PbO})$ .

B. M. V.

**Refining [of antimony sulphide] and apparatus therefor.** J. E. BURNS and W. H. WOODFORD, ASSRS. to REMINGTON ARMS CO., INC. (U.S.P. 1,887,920, 15.11.32. Appl., 16.11.26).—Fine material is melted with S at below the b.p. and poured in a thin stream through air.

B. M. V.

**Producing oxides of the fourth-group metals.** M. MAYER, ASSR. to KREBS PIGMENT & COLOR CORP. (U.S.P. 1,885,934, 1.11.32. Appl., 14.3.29. Ger. and Czechoslov., 15.3.28).—In the manufacture of  $\text{TiO}_2$  from the fluid chloride, or of other group IV oxides from their halides, the halide is incorporated to a paste with  $\text{Na}_2\text{SO}_4$  or other salt sol. in  $\text{H}_2\text{O}$ , the mass is heated and steamed at  $300$ – $400^\circ$ , and the steamed mass heated to  $800^\circ$ , cooled, and leached with  $\text{H}_2\text{O}$ .

B. M. V.

**Phosphorescent or luminous masses or compounds.** H. M. MINES and W. A. BEECH (B.P. 391,914, 19.1.32).—A mixture of  $\text{SrCO}_3$  (434),  $\text{CaCO}_3$  (108), and  $\text{MgCO}_3$  (22 pts.) is calcined to produce an oxide mixture which is ground and intimately mixed with 4–5% of starch, 12–15% of S, and with  $\text{LiCO}_3$  (11),  $\text{RbCO}_3$  (5), and  $2\text{Al}_2\text{O}_3 \cdot 3\text{SiO}_2$  (5 pts.). A small quantity of a phosphorogen [e.g.,  $\text{Th}(\text{NO}_3)_4$  with  $\text{Bi}(\text{NO}_3)_3$ ,  $\text{Er}(\text{NO}_3)_3$ ,  $\text{RbNO}_3$  or  $\text{AgNO}_3$ , and  $\text{NiSO}_4$ ] and about 26 pts. of a luminophore (I) are added and the mixture is heated first in a non-oxidising atm., then, after regrinding, in  $\text{H}_2$ ; (I) consists of  $\leq 2$  of the following:  $\text{NaCl}$ ,  $\text{KCl}$ ,  $\text{MnCl}_2$ ,  $\text{Na}_2\text{SO}_4$ ,  $\text{K}_2\text{SO}_4$ ,  $\text{BaSO}_4$ ,  $\text{MnSO}_4$ ,  $\text{Rb}_2\text{SO}_4$ ,  $\text{NaF}$ ,  $\text{CaF}_2$ ,  $\text{K}_2\text{Cr}_2\text{O}_7$ ,  $\text{Na}_2\text{S}_2\text{O}_3$ ,  $\text{Na}_2\text{CO}_3$ , according to the colour of the phosphorescent light required.

A. R. P.

**Preparing contact [hydrogenation] catalysts.** GOODYEAR TIRE & RUBBER CO. (B.P. 394,576, 23.11.32. U.S., 8.6.32).— $\text{Ni}(\text{NO}_3)_2$  and kieselguhr are intimately ground together with a little  $\text{H}_2\text{O}$  and the Ni is then pptd. by addition of a slight excess of a sol. carbonate, preferably  $\text{NaHCO}_3$ . The ppt. is washed free of sol. salts and reduced in  $\text{H}_2$  at  $450^\circ$  for 60–80 min. It forms a very active catalyst for the hydrogenation of PhMe,  $\text{COMe}_2$ , benzyl alcohol, and resorcinol.

A. R. P.

**Manufacture of hydrogen.** (A) J. S. BEEKLEY, (B) A. T. LARSEN, ASSRS. to E. I. DU PONT DE NEMOURS &



Co. (U.S.P. 1,889,530 and 1,889,672, 29.11.32. Appl., [A] 8.11.30, [B] 15.5.28).—(A) Hydrocarbons and steam at 650° are caused to react in a no. of catalysers with reheating between stages to about the initial temp., the gases in the reaction vessels being allowed to cool to 525°. The space velocity in any catalyser should be proportional to the reaction velocity. (B)  $2\text{CO} + 3\text{H}_2\text{O}$  are caused to react at  $> 300^\circ$  in the presence of Cu and an irreducible oxide of W, Cr, Mo, Ce, V, Mn, U, or Mg. B. M. V.

**Releasing nascent hydrogen [in an aqueous medium].** O. M. URBAIN, Assr. to C. H. LEWIS (U.S.P. 1,889,714, 29.11.32. Appl., 29.11.29).—A salt of an alkali metal and a positively charged colloid [e.g.,  $\text{Al}(\text{OH})_3$   $10^{-4}$  g./litre] which will absorb the anions are brought together in  $\text{H}_2\text{O}$ , whereby cations of nascent H are released. B. M. V.

**Manufacture of phosphorus.** VICTOR CHEM. WORKS (B.P. 395,865, 22.6.32. U.S., 11.7.31).—The furnace charge of phosphate material and fuel is intermittently checked in its descent and kept stationary while the blast is applied, combustion of the fuel being thus effected. It is also caused to drop intermittently by partial or complete interruption of the blast, and thus becomes compacted. A higher yield, a purer product, and less dust are produced than when the charge is allowed to descend uniformly through the furnace. W. J. W.

**Preparation of phosphorus compounds.** E. C. BRITTON and H. R. SLAGH, Assrs. to Dow CHEM. Co. (U.S.P. 1,888,713, 22.11.32. Appl., 9.7.29).—P and  $\text{Cl}_2$  are caused to react in the presence of  $\text{PCl}_3$  with formation of  $\text{POCl}_3$  (the desired compound) and additional  $\text{PCl}_3$ . The mixture is subjected to hydrolysis and distillation, the  $\text{PCl}_3$  being reduced to its original quantity during the hydrolysis. B. M. V.

**Continuous recovery of bromine from sea-water and the like.** J. J. GREBE and R. H. BOUNDY, Assrs. to Dow CHEM. Co. (U.S.P. 1,885,255, 1.11.32. Appl., 2.7.30).—Br having been liberated by acid and  $\text{Cl}_2$  and blown out of the  $\text{H}_2\text{O}$  by air, the air is passed through active charcoal (which is regenerated by steam) and the vapours are condensed into strata of Br and dil. aq. HBr; the latter is then treated with  $\text{Cl}_2$ , the HCl formed being used to acidify the sea- $\text{H}_2\text{O}$ . B. M. V.

**Manufacture of phosphorus.** VICTOR CHEM. WORKS (B.P. 395,844, 24.5.32. U.S., 8.6.31).—See U.S.P. 1,867,241; B., 1933, 386.

**Thermophoric mixture. Drying agent.**—See I. Removing  $\text{NH}_3$  and  $\text{H}_2\text{S}$  from gases.—See II. Smelting Fe ores high in S. Roasting pyrites. Pptn. of ZnS. Au from solution.—See X. Hydrogenation catalyst.—See XII. Pigments.—See XIII. Fertilisers.—See XVI.

### VIII.—GLASS; CERAMICS.

**Effect of gases on properties of glass. V. Effect of heat treatment on stability in water.** K. NAKANISHI (J. Soc. Chem. Ind., Japan, 1933, 36, 426—427 B).—The stability of glass in  $\text{H}_2\text{O}$  was in the order: heated in  $\text{N}_2 >$  in air  $>$  in  $\text{O}_2$ . Increase of gas pressure when the glass was heated above its softening point

decreased the stability but had no effect below this temp. The solubility of glass in  $\text{H}_2\text{O}$  was increased by saturating it with  $\text{O}_2$  but was unaffected by  $\text{N}_2$ .

V. P. P.

**Tank blocks for glass-melting furnaces.** J. H. PARTRIDGE (J. Soc. Glass Tech., 1933, 17, 169—238).—An extensive review.

**Nature of glass in porcelain body.** S. KONDŌ and K. MOTEKI (J. Soc. Chem. Ind., Japan, 1933, 36, 169 B).—The microstructures of 43 bodies of kaolin, felspar, and quartz or  $\text{Al}_2\text{O}_3$  have been studied. Even thin sections are heterogeneous, those containing quartz exhibiting glass, quartz, mullite, pores, and yellow spots; the  $\text{Al}_2\text{O}_3$  bodies contain in addition cryst.  $\text{Al}_2\text{O}_3$ . The solubility of mullite in the glasses, and the relation between refractive index and composition are discussed. H. F. G.

**X-Ray examination of ceramic materials.** T. NAKAI and Y. TUKAMI (J. Soc. Chem. Ind., Japan, 1933, 36, 349—351 B).—The examination of a no. of finely ground clays showed that quartz and kaolin gave very distinct X-ray photographs whilst felspar was not so sharp. The constituents could be estimated approx. and the quant. vals. coincided reasonably well with those obtained by chemical analysis. C. A. K.

**Substitution of leadless glazes for lead glazes.** V. V. SAGALATOV (Trans. Kiev Div. Ukrain. Sci. Res. Inst. Silicate Ind., 1930, 7, 95 pp.).—For the production of leadless glazes with a good flow, 0.6 mol. of alkali is necessary.  $\text{K}_2\text{O}$  gives viscous glasses. Brilliant glazes are obtained by smearing the sagger with frit rich in alkalis and  $\text{B}_2\text{O}_3$ . CH. ABS.

**Thermal expansion of highly fired clay ware.** T. YOSHIOKA (J. Soc. Chem. Ind., Japan, 1933, 36, 169—170 B).—The mullite (I) content of clays fired at 1300—1400° is 10—20%, according to the temp. X-Ray examination shows the presence of cryst. (I) and high-temp. modifications of  $\text{SiO}_2$ ; the latter are the cause of the irregularity of the coeff. of expansion of the fired clay at 200° and of the high coeff. at higher temp. The (I) increases the coeff. but does not introduce any irregular variation; its coeff. of expansion is  $85 \times 10^{-7}$ . In clays fired at 1500° the (I) content is about 45%, but the high-temp. forms of  $\text{SiO}_2$  are absent; the expansion curve is similar to that of clays fired at temp.  $>$  that of the (I)—cristobalite eutectic (1545°). It appears that the  $\text{K}_2\text{O}$ , CaO, etc. present reduce the eutectic temp. to below 1500°, and thus cause disappearance of the free  $\text{SiO}_2$ . The coeff. of expansion of the eutectic (5.5%  $\text{Al}_2\text{O}_3$ , 94.5%  $\text{SiO}_2$ ) is  $7.5 \times 10^{-7}$ . H. F. G.

**Dimensional changes of clay wares during firing.** T. YOSHIOKA (J. Soc. Chem. Ind., Japan, 1933, 36, 308—309 B).—Precision experiments showed that clays generally display a gradual slight expansion at 20—500°, a definite peak at 500°, and a slight contraction at 500—900°, with marked increase at 900—950°. The shrinkage value then decreases to 1300° with a marked increase in the rate at 1050—1200°. The effects on quartz and felspar are similar though at lower temp.

C. A. K.

**Effect of varying the ratio of soda ash to sodium silicate in alkaline earthenware slips.** J. H. RIGBY



(Trans. Ceram. Soc., 1933, 32, 337—347).—The influence of adding various amounts of  $\text{Na}_2\text{SiO}_3 + \text{Na}_2\text{CO}_3$  (soda ash), and of varying the ratio of  $\text{Na}_2\text{SiO}_3$  to  $\text{Na}_2\text{CO}_3$  in the added alkali, on the viscosity (I) and casting rates (II) of three earthenware slips has been determined. (II) was found closely to follow (I), slips possessing high (I) having in general the quickest (II). (I) and (II) are determined not only by the total amount of, but also by the ratio of  $\text{Na}_2\text{SiO}_3 : \text{Na}_2\text{CO}_3$  in, the added alkali. The effect was compared of adding alkali having the following ratios (by wt.) of  $\text{Na}_2\text{SiO}_3$  to  $\text{Na}_2\text{CO}_3$ : (1) 1.00:0.96, (2) 1.00:5.25; for small amounts of added alkali slips containing (2) cast quicker than those containing (1), but with increasing alkalinity (II) were reversed. Tougher castings, which set more quickly and could be more readily handled, were obtained from slips containing (1) than from those containing (2); the presence of (2) appeared to increase the tendency for soft, slow-setting casts. The best casting slips for general purposes are indicated. A. L. R.

**Permeability of refractory materials to gases. Experiments with fireclay and silica products (I) at ordinary temperatures, (II) at temperatures up to 500°.** F. H. CLEWS and A. T. GREEN (Trans. Ceram. Soc., 1933, 32, 295—318, 319—331; cf. B., 1932, 64).—I. Methods of determining the permeability (I) of refractory products are reviewed, and the results summarised. An apparatus is described in which the (I) to air of a series of representative  $\text{SiO}_2$ , fireclay, and diatomite materials was measured at room temp.; the results are considered in relation to the texture of the products. No definite relation between % porosity and (I) was observed; on the average the (I) of  $\text{SiO}_2$  brick was  $>$  that of firebrick, whilst that of a diatomite insulation product (70% porosity) was very low. The non-uniformity of pore distribution in a single brick is emphasised particularly with relation to lamination, the presence of denser surface layers, or a surface skin, and a rough classification of the average pore size is given.

II. A modified apparatus is described in which the (I) to  $\text{N}_2$  of fireclay,  $\text{SiO}_2$ , and siliceous products was measured at temp. up to 500°. (I) decreased with rise of temp. almost independently of the nature of the material and of its (I) coeff. at room temp., being reduced, for a temp. rise from 10° to 500°, to approx. half its val. for fireclay, and to rather less for  $\text{SiO}_2$  materials. The decrease in (I) is accounted for almost quantitatively by the increase in viscosity of the gas. The expansion accompanying the inversion of low-sp. gr. forms of  $\text{SiO}_2$  at 100—250° is suggested as the cause of the difference of (I) vals. of  $\text{SiO}_2$  and fireclay materials at 500°.

A. L. R.

**Slag-resistant crucibles.**—See XI.

See also A., Aug., 774, Japanese acid clay. 787, Vitrification of  $\text{SiO}_2\text{-Na}_2\text{CO}_3$ . 830, Prep. of Au solution [for ceramics].

## PATENTS.

**Continuous tank furnace [for glass].** E. T. FERNGREN, Assr. to LIBBEY-OWENS-FORD GLASS CO. (U.S.P. 1,888,496, 22.11.32. Appl., 26.3.26).—In a tank furnace supplying two drawing machines, the

streams of outgoing glass are caused to flow more uniformly over their cross-section by sloping the floors of the passages upwards and providing cooled floaters of refractory material lined with  $\text{SiC}$ . B. M. V.

**Electric melting of glass or the like.** HARTFORD-EMPIRE Co., Assees. of H. W. MCINTOSH (B.P. 394,709, 1.1.32. U.S., 5.2.31).—Electrodes for use in glass melting by the bath-resistance method are coated with glass of higher m.p. than that to be made. The furnace is started by preheating with fuel and introducing glass melted elsewhere; during the intermediate time, when the electrodes are near the surface of the glass, they are protected by the coating, which will not melt until, in the normal running stage, the electrodes are fully submerged in the molten glass and protected from oxidation. B. M. V.

(A) **Apparatus for melting glass.** (B) **Rotary glass-melting furnace.** W. O. AMSLER, Assr. to HARTFORD-EMPIRE Co. (U.S.P. 1,889,510—11, 29.11.32. Appl., [A] 19.4.28, [B] 26.7.28).—(A) The glass materials are scraped from an annular rotating shelf down the axis of which a burner projects a melting flame; the passage of the glass into the tank of the furnace is hindered by baffles until it is melted. (B) Glass is melted in a short, rotary, cylindrical kiln in which the fuel is injected at the same end as the exhaust gases and molten glass leave, the glass material alone passing through the nearly closed upper end. B. M. V.

**Fining of glass.** CORNING GLASS WORKS, Assees. of R. H. DALTON (B.P. 396,015, 29.5.33. U.S., 30.7.32).—The fining of glasses, especially those containing C, is accelerated by adding to the batch up to 2% of I or Br or their compounds, e.g., iodides, iodates,  $\text{CHI}_3$ , bromides, or bromates. J. A. S.

**Making laminated glass.** W. O. LITTLE, Assr. to DUPLATE CORP. (U.S.P. 1,885,272, 1.11.32. Appl., 10.10.31).—The final pressing is effected in an autoclave (150 lb./sq. in., 113°) in the presence of conc.  $\text{H}_2\text{SO}_4$ , which dissolves out the cellulosic material around the edges to a depth suitable for sealing. The sheets are then washed in hot  $\text{H}_2\text{O}$  and the edges ground and sealed. B. M. V.

**Production of white opaque enamels.** I. KREIDL (B.P. 396,007, 7.4.33. Austr., 20.4.32 and 28.11.32).—A gas-clouding slip is stable only if the amount of clouding agent (org. dyes, highly polymerised hydrocarbons, etc.) is  $>$  the amount which can be adsorbed by the clay substance present. E.g., to a mixture of enamel (1000 pts.) and clay (80 pts.) may be added malachite-green or methylene-blue (0.25—0.3 pt.), or to a mixture containing 100 pts. of clay, 0.3—0.5 pt. of pitch or gas tar. J. A. S.

**Production of enamelled articles having a multi-coloured appearance.** PORCELAIN ENAMEL & MANUFACTURING Co. OF BALTIMORE (B.P. 395,482, 30.4.32. U.S., 6.5.31).—The article is first provided with a rough surface, which is then sprayed with the various colours from different directions so that, when viewed from any one direction, one particular colour appears to predominate. A final firing produces a smooth surface with multi-colour effects. J. A. S.



**Tunnel kiln.** J. FETZER (U.S.P. 1,885,811, 1.11.32. Appl., 11.1.30).—In a tunnel kiln containing trucks, the latter are provided with hanging baffles (a) to prevent the passage between the wheels acting as a flue, and (b) to serve as scrapers for the floor of the kiln, removing ashes and broken material into pits provided at intervals for the purpose. Besides having combustion chambers at the sides, fuel is placed on top of the goods themselves and burned there. B. M. V.

**Ceramics.** H. L. and R. M. CROWLEY, Assrs. to H. L. CROWLEY & Co., Inc. (U.S.P. 1,885,234, 1.11.32. Appl., 2.3.29).—Corrosion (during heating) between a metal and a ceramic vessel in which it is sealed has been found to be due to presence of alkali. In the construction of, e.g., a wireless valve the ceramic materials are freed from alkali by heating to < their m.p., a binder (of the type which disappears on heating, or, like BeO, which fluxes) is then added, and the article baked in a reducing gas so that occluded air is replaced by CO. A getter may be added. B. M. V.

**Production of refined clay.** G. SIMCOE, Assr. to EDGAR BROS. Co. (U.S.P. 1,888,701, 22.11.32. Appl., 9.11.26. Renewed 8.6.32).—The process claimed comprises partial deflocculation with  $\text{Na}_2\text{SO}_3$  and  $\text{Na}_2\text{SiO}_3$ , removal of impurities by settlement and straining, dissolution of Fe and bleaching of org. matter by  $\text{SO}_2$ , conversion of Fe salts into phosphates, thickening, filter-pressing, and drying. B. M. V.

**Burning of brick.** N. B. KENDRICK (U.S.P. 1,887,206, 8.11.32. Appl., 12.4.32).—A no. of kilns are operated in succession, the gases from a burning kiln being led through at least one other, from the bottom of a hotter kiln to the top of the next. An induced-draught fan draws the final gases from a circular duct put into communication with the correct kiln. B. M. V.

**Making refractory brick.** R. P. HEUER, Assr. to GEN. REFRACTORIES Co. (U.S.P. 1,886,185, 1.11.32. Appl., 2.3.31).—A hydrous  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  material (< 60%  $\text{SiO}_2$ ) is calcined at  $>1400^\circ$  and a mixture of 85% of the calcined material and 15% of the raw is pressed at 1000 lb./sq. in. The particle size is specified. B. M. V.

**Manufacture of waterproof abrasive articles [e.g., sand-paper or emery cloth].** Z. HADNAGY, A. BOUILLARD, and A. AUBY (B.P. 394,442, 8.3.32. Fr., 14.3.31).—The paper or cloth is coated with an agglutinant consisting of materials which are elastic at room temp. but plastic at  $100^\circ$ , e.g., mixtures of (a) balata, linseed oil, and stearin pitch, (b) gutta-percha, vinyl resins, and polymerised oils, or (c) mixtures of any or all of the above with resin, wax, colophony, or  $\text{CH}_2\text{O}$  condensation products. A. R. P.

**[Machine for] manufacture of pottery ware.** W. J. MILLER (B.P. 395,855, 8.6.32).

**Tunnel ovens.**—See I.

## IX.—BUILDING MATERIALS.

I. Reactivity of Rumanian trass in trass-cement mortars. II. Strength of trass-cement mortars. III. Effect of fineness of grinding on the strength of trass-cement mortars. IV. Effect of heating

trass on its reactivity and technical properties. V. Effect of small quantities of sodium carbonate on the binding properties and strength of trass-cement mortars. A. STEOPOE (Bul. Chim. Soc. Române, 1933, 34, 17—22, 23—34, 35—39, 40—49, 50—60).—I. The trass contains  $\text{H}_2\text{O}$  13.8—14.4,  $\text{SiO}_2$  62.5—65.4,  $(\text{Al,Fe})_2\text{O}_3$  13.5—12.5,  $\text{CaO}$  6.5—4.6,  $\text{MgO}$  0.7—0.5, and minor constituents 2.8—2.5%. It takes an active part in the hardening of cement mixtures, since its  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  react readily with the free  $\text{CaO}$  and thereby the hydraulic modulus of the mortar fraction of the mixture is reduced, the silicate modulus increased, and the %  $\text{CaO}$  sol. in 15% sucrose solution reduced the higher is the trass content.

II. Substitution of up to 30% of the usual cement content of cement-sand mortars by trass increases the strength, reduces the  $d$ , and yet results in a less porous product after hardening.

III. Addition of 10% of coarsely crushed or graded trass to cement increases the compressive strength of mortars made from it, whereas fine trass tends to reduce this strength owing to the rapidity with which it reacts with the  $\text{CaO}$ .

IV. Heating of the trass at  $500^\circ$  effects only a temporary improvement in its reactivity, whilst heating at  $1000^\circ$  causes a considerable diminution in its hydraulic properties.

V. Addition of small quantities of  $\text{Na}_2\text{CO}_3$  to trass-cement mortars accelerates remarkably the rate of setting but this effect diminishes with increasing trass content and increasing additions of  $\text{Na}_2\text{CO}_3$ . A. R. P.

**Mercuric chloride impregnation [of wood].** W. KINBERG and K. EISNER (Chem.-Ztg., 1933, 57, 561—562, 582—584).—Determinations of the  $\text{HgCl}_2$  adsorbed from solutions of varying concn. by pine shavings, cellulose, lignin, and animal C are given. Results are concordant with the view that the process is strictly one of adsorption; lignin gave higher adsorptions than cellulose, though this may be due partly to the different state of division of the two materials. The  $\text{HgCl}_2$  adsorbed is gradually reduced to  $\text{HgCl}$  and  $\text{HgO}$ , and wood 5 years after treatment contained only traces of  $\text{HgCl}_2$ . It is to this that the resistance to extraction with  $\text{H}_2\text{O}$  is due. Bacterial tests on pieces of wood are liable to variation owing to uneven impregnation. Tests with *Coniophora cerebella* made on finely-divided samples showed that adsorption of 0.47%  $\text{HgCl}_2$  gave complete protection for 2 months. C. I.

**Seasoning of Australian timbers.** I. C. S. ELLIOT (Comm. Australia, Counc. Sci. Ind. Res., Pamphlet 40, 1933, 53 pp.).—Seasoning characteristics of, and suggested kiln schedules for, 17 species are given, together with notes on drying, drying stresses, and degrade.

**Paints on wood.**—See XIII.

See also A., Aug., 793, Hydrates of  $\text{CaSO}_4$ . Synthesis of Ca silicates.

## PATENTS.

**Manufacture of hydraulic cement.** P. T. LINDHARD, Assr. to F. L. SMITH & Co. (U.S.P. 1,885,845, 1.11.32. Appl., 20.6.30).—In a rotary kiln the calcining zone is shortened, as regards floor space occupied, by



insertion of a transverse baffle in the kiln, which causes the material to enter external tubes travelling down them and then up an equal no. of tubes back to the main kiln at the lower side of the baffle, the upward motion being effected by helical lifters, and the gases passing countercurrent to the material as in the main kiln.

B. M. V.

**Plasticising hydraulic cement.** C. E. KRAUS, Assr. to SIALCO, INC. (U.S.P. 1,885,731, 1.11.32. Appl., 23.2.23. Renewed 19.12.30).—Bentonite pptd. as a flocculent mass from CaO-H<sub>2</sub>O or other alkaline solution is added to CaO, before or after hydration, or to dry Portland or CaSO<sub>4</sub> cement.

B. M. V.

**Manufacture of a bituminous cement mortar, cement concrete, and the like.** A. J. DE LANGE, Assr. to FLINTKOTE CORP. (U.S.P. 1,889,437, 29.11.32. Appl., 15.11.29. Holl., 17.11.28).—The mortar is mixed first with H<sub>2</sub>O (containing any desired addenda, e.g., CaCl<sub>2</sub>, Na<sub>2</sub>SiO<sub>3</sub>) in quantity insufficient to produce proper setting, and then with an aq. emulsion of bituminous material.

B. M. V.

**Production of concrete.** P. ASKENASY (U.S.P. 1,886,933, 8.11.32. Appl., 20.11.30. Ger., 28.11.28).—A proportion of ash from pulverised fuel is added to the mix: e.g., cement 70, ash 30, sand 300 pts. The concrete is stated to resist sea-H<sub>2</sub>O.

B. M. V.

**Composition for use as plaster and manufacture thereof for building and other purposes.** N. P. DEVELOPMENT SYND., LTD., and H. L. BERESFORD (B.P. 395,426, 27.1.32).—Pumice (preferably of trachytic or syenitic nature) or artificial vesicular material is crushed to pass 20-mesh (75–80% passing 150- and > 5% passing 200-mesh) and mixed with Portland cement, with or without the addition of accelerators and pigments.

J. A. S.

**Cleaning of [gypsum] mould parts.** J. M. PORTER and G. W. HANDY, Assrs. to STRUCTURAL GYPSUM CORP. (U.S.P. 1,885,287, 1.11.32. Appl., 16.9.29).—An alkali, e.g., CaO, is added to gypsum cement prior to casting in Al moulds, in order to set up a slight reaction with the metal and prevent sticking.

B. M. V.

**Treatment of fibrous material [boards] with sulphur.** F. V. D. CRUSER, Assr. to DIAMOND MATCH CO. (U.S.P. 1,886,170, 1.11.32. Appl., 19.12.29).—The articles, e.g., match-box boards, are dipped in S and tumbled in a steam-heated screen until the S has been absorbed.

B. M. V.

**Wood-filler composition.** E. T. STILLE (U.S.P. 1,885,780, 1.11.32. Appl., 18.5.29).—Rapid-drying compositions of boiled oil and dryers, asphaltum varnish, pyroxylin lacquers, and pigments are described.

B. M. V.

**Printing or graining [of wood].** L. A. MIZENER, Assr. to CHICAGO MILL & LUMBER CORP. (U.S.P. 1,888,864, 22.11.32. Appl., 30.11.31).—A composition for printing directly upon the surface of wet (> 25% H<sub>2</sub>O) wood comprises starch, colouring matter, glycerin, and AcOH, with, if desired, blood-albumin, aq. NH<sub>3</sub>, and paraformaldehyde.

B. M. V.

**Heat application [for wallboards etc.].**—See I. Asphalt emulsions. Bitumen dispersions.—See II.

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

**Metallurgical processes in open-hearth steel manufacture.** P. BARDENHEUER (Stahl u. Eisen, 1933, 53, 488–496).—The rate of removal of C during the refining stage is a linear function of the FeO content of the slag and of the temp.; the O content of the metal remains const. until the C is reduced to < 0.25%, after which it rapidly rises while the Mn content falls rapidly at first, then remains almost const., and finally rises slightly towards the end of the "boiling." The continuous addition of ore to burn out the C evenly is preferable to adding the ore rapidly in the early stages since the latter procedure gives rise to a vigorous boiling operation and to a high oxide content of the bath at the end of the refining. The mechanism of the deoxidation process with ferromanganese is discussed with reference to recent work and some examples are given of the changes which take place in the composition of the bath and slag under varying operating conditions. A. R. P.

**Direct reduction tests with iron ore and sinter in the blast furnace.** W. FELDMANN, J. STOECKER, and W. EILENDER (Stahl u. Eisen, 1933, 53, 289–296).—The results obtained in 500 tests on the reduction of 4 types of Fe ore and 2 types of sinter in various parts of the blast furnace are summarised in a series of graphs. The rapidity of reduction depends on the nature of the ore and the size of the feed; porous and small-sized ores are more rapidly reduced than dense, lumpy ores. In the upper zones of the furnace reduction by gas is relatively small, only about 13% of the O content of magnetite and 37% of that of N. African hæmatite being removed. Owing to the glassy surface of the sinters they are reduced only with difficulty in spite of their greater effective surface.

A. R. P.

**Desulphurisation of steel with alkalis in the coreless induction furnace.** H. SIEGEL (Stahl u. Eisen, 1933, 53, 646–652).—The S content of steel can be reduced to < 0.02% by treating the molten metal in a high-frequency furnace with a mixture of Na<sub>2</sub>CO<sub>3</sub> and ferro-silicon or -manganese added portionwise to a slag of Na<sub>2</sub>O, SiO<sub>2</sub> above the metal; the slag must be a solvent not only for the desulphurising alkali but also for the Na<sub>2</sub>S formed. CaO slags are unsuitable owing to their higher viscosity. The extent of desulphurisation depends on the quantities of alkali, reducing agent, and slag used.

A. R. P.

**Removal of phosphorus, sulphur, and oxygen in the production of high-quality steel in the open-hearth furnace.** F. BEYTER (Stahl u. Eisen, 1933, 53, 369–375, 398–404).—In the normal basic open-hearth steel process the P content is reduced to an average val. of 0.014% when only one slag is used, but to about 0.008% when the first slag is removed and the bath treated with a second dephosphorising slag during the boiling operation. High Mn and C content of the steel favours a high P content, but the S content decreases with increasing Mn. The duplex process also tends to reduce the S in the finished steel; the best desulphuriser appears to be strontianite, of which 45–50 kg. per ton should be added to the bath after removing thoroughly all the first slag. Desulphurising



is favoured by a low O content of the steel and by the use of gas with a low S content. The O content of steel made by the acid process averages 0.008% and that of basic steel about 0.024%. The finishing procedure for open-hearth practice is discussed and recommendations are made for the various processes based on the type of steel being made and the rate of combustion of the C during the refining period. A. R. P.

**Double nitrogenising [case-hardening of steel].** O. HENGSTENBERG and F. NAUMANN (Arch. Eisenhüttenw., 1933—4, 7, 61—66).—A double nitrogenisation of Al steels in  $\text{NH}_3$ , first (I) at a relatively low temp. ( $480^\circ$ ) then (II) at a higher temp. ( $520^\circ$ ), results in an increased thickness of the nitride layer without affecting the max. hardness of the outer surface (A). If the temp. of the two stages is reversed no beneficial effects result; too high a temp. in (II) produces coagulation of the nitride and decreased hardness. Provided that (I) is prolonged sufficiently to obtain (A), the hardness obtainable in the inner nitride layers in (II) is greater the lower is the N content produced in (I). A theoretical explanation of these effects is given based on micrographic examination of inclined sections cut through the hardened layers. A. R. P.

**Embrittlement of steel at high steam temperatures.** H. A. DICKIE (Engineering, 1933, 136, 108—109).—In consideration of the possible physical changes producing brittleness, the sp. vol. and hardness must be relied on to show the change. Heat treatment of two Ni-Cr steels showed a double change in hardness associated with the period of onset of brittleness. The mechanism of embrittlement probably involves the expulsion of carbide to the grain boundary and this view is corroborated by visual observation. The addition of Mo to steel to prevent temper-brittleness does not prevent the change, but only reduces it. C. A. K.

**Rate of transformations in solid steel.** E. C. BAIN (Arch. Eisenhüttenw., 1933—4, 7, 41—47).—The rate of transformation of austenite (I) in various Mn and Ni steels has been determined by micrographic examination of specimens annealed for various periods at  $315$ — $870^\circ$ . Mn retards (I) much more than does Ni. In steels with 0.5% C and 3.5% Ni the stable form of the C pptd. from the austenite (A) is graphite, but its rate of formation is so small compared with that of cementite (C) that under normal annealing conditions only C separates. In steel with 1.2% C and 6.2% Mn annealing at  $665^\circ$  gradually produces an equilibrium between A, ferrite (F), and C; this occurs in about 1 week starting from the A state and in about 3 weeks starting from the F + C state, and occurs when about 35% of the metal is A. Since in Mn steels white martensite is converted into a coarse-grained F-C mixture at about 1000 times the rate at which A of the same composition is converted it is concluded that pearlite can be formed directly from A. Most of the transformations in steel, after a more or less lengthy period of induction, proceed at a rate similar to that of chemical reactions of the first order, but there are also reactions which proceed with a very high initial rate and then slow down, e.g., the dissolution of C in A at high temp., and the coagulation of C during tempering. In both these cases diffusion of C begins with a rapid

decrease in concn. (II) and a short path of movement (III) but (III) rapidly becomes longer and (II) smaller. A. R. P.

**X-Ray testing of iron and steel.** F. WEYER (Stahl u. Eisen, 1933, 53, 497—505).—A review of modern technique of X-ray testing of metals and its application to the examination of welds and the determination of internal stress. A. R. P.

**X-Ray detection of internal corrosion of twisted wire ropes.** R. GLOCKER, P. WIEST, and R. WOERNLE (Stahl u. Eisen, 1933, 53, 758—761).—The presence of rust in the gaps between the strands of twisted Fe wire ropes is revealed by an irregular weakening of the X-ray pattern; the phenomenon is best seen by taking stereoscopic photographs. The outer wires also frequently appear to be slightly lifted away from the inner strands. A. R. P.

**[Micrographic] examination of the structure [of steel] at temperatures up to  $1100^\circ$ .** H. ESSER and H. CORNELIUS (Stahl u. Eisen, 1933, 53, 532—535).—An apparatus is described by which a polished metal specimen can be studied micrographically while it is being heated in vac. by a wire-wound (W or Ni-Cr) resistance furnace. Pure Fe with 0.03% C, polished but unetched, begins to show a polygonal network of  $\alpha$ -ferrite just below  $\text{Ac}_3$ , and just above this point a new network due to  $\gamma$ -Fe begins to supplant the former; on cooling, a new  $\alpha$ -network develops so that eventually traces of all three structures can be distinguished. The dissolution and re-separation of pearlite in eutectoidal and hypoeutectoidal steels, the decomp. of austenite in quenched Mn steels on tempering, and the formation of temper-C in white Fe have also been followed micrographically. Tests with electrolytic Fe have shown that FeO is insol. in Fe up to  $1100^\circ$ , no change taking place in the shape or position of FeO inclusions in Fe containing 0.1% O after prolonged heating at  $1100^\circ$ , and that Fe and FeO form no eutectic. A. R. P.

**Evaluation of boiler plates by the notched bending test.** A. THUM and H. HOLDT (Stahl u. Eisen, 1933, 53, 505—508).—The nature of the fracture of boiler plates in the notched bending test is not a satisfactory criterion of the val. of the plate. A fine-grained fracture, however, is a sure indication of a faulty plate, as is the appearance of non-metallic surfaces in fibrous or leafy fractures; step-like fractures indicate unsound places, whilst tough fractures with a long fibrous structure are generally characteristic of good-quality plates. A. R. P.

**Prevention of corrosion-fatigue [of steel].** H. BUCHHOLTZ and K. KREKELER (Stahl u. Eisen, 1933, 53, 671—674).—The corrosion-fatigue of steel parts operating in  $\text{H}_2\text{O}$  can be prevented by nitrogenising or Zn-plating the surface, by the addition of 0.02% of  $\text{Na}_2\text{Cr}_2\text{O}_7$  to the  $\text{H}_2\text{O}$ , or by coating the surface with artificial resin, and considerably reduced by galvanising, sherardising, or Cd-plating the metal, or by addition of 0.5—1% of an emulsifiable oil to the  $\text{H}_2\text{O}$ . A. R. P.

**Determination of the coefficient of radial expansion of "Dumet" wire from the chemical analysis.** J. A. M. VAN LIEMPT (Rec. trav. chim., 1933, 52, 399—402).—The analysis is effected by dissolving



the outer sheath of Cu or Cu-rich alloy in cold conc.  $\text{HNO}_3$ , which does not attack the Fe-Ni alloy core, and proceeding in the usual manner. An equation is derived for calculating the coeff. of radial expansion of the wire from the coeff. of expansion of the two component alloys; for alloys of the compositions used in practice the results agree to within about 3% with the vals. determined experimentally. H. F. G.

**Properties of the alloys of nickel with tantalum.** E. THERKELSEN (*Met. & Alloys*, 1933, 4, 105—108).—Ni-Ta alloys with  $> 36\%$  Ta consist of a homogeneous solid solution, the liquidus falling smoothly from  $1450^\circ$  to  $1350^\circ$ ; a max. occurs at  $1545^\circ$  corresponding with the formation of  $\text{Ni}_3\text{Ta}$  and a min. at  $1400^\circ$  with  $61\%$  Ta. Alloys with  $50$ — $78\%$  Ta undergo a transformation in the solid state at  $1350^\circ$  and their structure is highly complex. Alloys with  $36$ — $37\%$  Ta are amenable to pptn. hardening. The Brinell hardness of the solid solution alloys increases almost linearly with the Ta content to  $250^\circ$  at  $35\%$  Ta, and the tensile strength of the cold-worked alloys similarly increases from 40 tons per sq. in. for pure Ni to 116 tons per sq. in. for the  $35\%$  Ta alloy. The cold-drawn  $9\%$  Ta alloy has a resistance of  $0.262(1 + 2 \times 10^{-3}t + 1.6 \times 10^{-6}t^2)$  ohms per m. per  $\text{mm}^2$ . The couple Ni | Ni-(9—15)% Ta has an e.m.f. of about 2 mv. per  $100^\circ$ . The resistance to corrosion is very good at about  $12.5$  at.-% Ta; all the alloys oxidise readily in air at  $350^\circ$ . A. R. P.

**Treatment of aluminium shavings with salt slags.** Systems KCl-NaF, KCl-NaCl-NaF, KCl-CaF<sub>2</sub>, and NaCl-CaF<sub>2</sub>. W. LEITGEBEL (*Metallwirts.*, 1932, 11, 699—700; *Chem. Zentr.*, 1933, i, 842).—Of salt mixtures used as protective slags in the melting of Al, binary mixtures containing cryolite,  $\text{BaCl}_2$ , NaCl, KCl, and NaF give the best results. The systems KCl-NaF and KCl-NaCl-NaF have been investigated in this connexion. L. S. T.

**Microchemical determination of gold in gold alloys.** J. DONAU (*Mikrochem.*, 1933, 13, 165—178).—A 10-mg. sample of the alloy is melted with 3 pts. of Ag, treated with  $\text{HNO}_3$ , washed, and ignited in a quartz tube without transfer. Ag in the sample may be determined by employing a known amount of Ag and determining the total Ag dissolved. J. S. A.

**Tinning and galvanising.** T. AIZAWA, G. WACHI, and T. EBIHARA (*Res. Electrotech. Lab.*, Tokyo, 1932, No. 333, 19 pp.).—The structure of the Sn coating on Cu, and the effect of the addition of a small % of Cd, Mg, Al, Zn, Pb, Sb, or Bi on its properties, are discussed. Alloy layers formed by diffusion of Cu into Sn withstand the action of S fairly well. A small quantity of Cd increases the hardness and resistance to S but makes the tinning operation more difficult. The composition and properties of the alloy layer in galvanised Fe, and the effect of the presence of Al, Ni, Sn, Cu, Sb, and Cd on the composition and properties are discussed. CH. ABS.

**Nature of structural changes during deformation of metal at higher temperatures.** E. F. BACHMETEV, M. D. VOZDVISHENSKI, S. I. GUBKIN, G. F. KOSOLAPOV, and B. M. ROVINSKI (*Mitt. Forschunginst. Luftfahrtmaterialsprüf.*, U.R.S.S., 1933, No. 1, 5—131).

—X-Ray studies indicate that the velocity of deformation,  $v_1$ , of pressed duralumin (I) may exceed that of recrystallisation,  $v_2$ ;  $v_2$  increases with temp. Under foundry conditions,  $v_1$  is often  $> v_2$ . The anisotropy of (I) has been determined at  $20$ — $500^\circ$ , for various velocities and degrees of deformation (up to  $70\%$ ). Deformed (I) gives diffuse X-ray diffraction figures. (I), deformed rapidly at  $450^\circ$ , undergoes only partial recrystallisation at this temp., and complete recrystallisation at  $500^\circ$ ; alloys similarly cold-treated recrystallise at  $450^\circ$ . A sample of (I) with a plane crystal structure did not exhibit transition at  $535^\circ$ . The temp. of (I) on cold-pressing with  $89\%$  deformation attained  $317^\circ$ . Compensation of internal stress in cold-pressed (I) occurs at  $300^\circ$  in  $< 30$  sec., at  $200^\circ$  in 15 min., and at  $100^\circ$  in  $> 3$  hr. A no. of other Al alloys behaved similarly to (I) in the above respects. R. T.

**Determination of heterogeneity and resistance to corrosion of metals.** PROT and (MLLE.) N. GOLDOVSKY (*Compt. rend.*, 1933, 197, 136—137).—The metal is placed in a jelly containing an electrolyte and suitable  $p_H$  indicator. Points in the metal of different composition are indicated by the electrolysis and consequent changes in  $p_H$  and colour caused by current passing between them. To test for pinhole corrosion, sheets 0.1 mm. thick are used; these show such corrosion in a few days, most easily by being placed on photographic paper and illuminated. C. A. S.

**Study of corrosion of metals, and corrosion of magnesium alloys.** A. PORTEVIN, P. BASTIEN, and M. BONNOT (*Compt. rend.*, 1933, 196, 1999—2002).—A standard method of determining rate of corrosion depending on rate of emission of gas is described. Applied to Mg-rich Mg-Al-Cu alloys in dil. aq. HCl or 0.5N-MgCl<sub>2</sub>, alloys near the limit of solid solution show max. corrosion. This is at first diminished and then increased by increase of Al, but in aq. citric acid corrosion decreases continuously with increase of Al. In Mg-Si alloys corrosion increases with amount of Si, though Mg<sub>2</sub>Si gradually checks the rate. C. A. S.

**Corrosion of metals by [vegetable] tan liquors.** IV. **Corrosion of submerged metals in movement.** M. P. BALFE and H. PHILLIPS (*J. Soc. Leather Trades' Chem.*, 1933, 17, 448—459; cf. B., 1932, 801).—Cr-Fe and Ni-Fe-Cr alloys are not corroded and Fe-Si alloy is slightly corroded by vegetable tan liquors (I) and bleaching extracts (II) respectively. Phosphor-bronze, Ni, monel metal, and Cu-Al-Ni alloy are equally resistant, but Al-brass is more resistant than are these to (I) and (II). Cu was equally resistant to chestnut extract but less resistant than these to (II). An Al-Mg-Mn alloy is corroded but the (I) are not contaminated. Brass is not superior to Cu alloys, Ni, or monel metal. Metals are not markedly corroded by algarobilla liquors. D. W.

**Solubility of certain metals and technical alloys in aqueous zinc chloride.** W. KUCZYŃSKI and M. H. WEISS (*Przemysł Chem.*, 1933, 17, 175—178).—The solubility of metals in  $24\%$   $\text{ZnCl}_2$  at  $85$ — $90^\circ$  diminishes in the order  $\text{Pb} > \text{Cu} > \text{non-corrosive steels}$ . Cu is less sol. in  $60\%$  than in  $24\%$   $\text{ZnCl}_2$ , whilst Pb and certain steels are more sol. The solubility of Pb is raised by



addition of  $\text{KNO}_3$  or  $\text{KClO}_3$ , and slightly lowered by starch or gelatin. R. T.

**Failure of elasticity in metals: damping of torsion oscillations.** L. FILLIATRE and P. VERNOTTE (Compt. rend., 1933, 196, 1374—1376). C. A. S.

**[Ore]-concentrating practice of the Broken Hill South, Ltd.** T. A. READ (Proc. Austral. Inst. Min. Met., 1932, No. 88, 247—339).—A detailed account is given of the plant and operating practice for the treatment of Pb-Zn ores at this mine, with flow sheets and costs. About 60% of the Pb in the ore is recovered by jigs and tables in a coarse concentrate assaying 77% Pb and only 3.3% Zn; the remaining Pb is recovered by selective flotation. Zn is recovered by flotation in an alkaline circuit of the tailings from the Pb section after grinding to give 3% of +40-mesh; the product contains 52% Zn and 1—1.5% Pb. A. R. P.

**Physical chemistry of [ore] flotation. II. Adsorption of the soluble collector.** A. B. COX and I. W. WARK. **III. Relationship between contact angle and constitution of collector.** E. E. WARK and I. W. WARK. **IV. Criticism of Ostwald's theory of flotation.** I. W. WARK and A. B. COX (J. Physical Chem., 1933, 37, 797—803, 805—814, 815—819; cf. B., 1932, 347).—II. A crit. discussion. The balance of evidence is against a purely chemical theory. The ability of a mineral to adsorb a S-containing flotation collector (I) is closely related to the solubility of the salt formed from (I) and the metal of the mineral. Full response to a sol. (I) occurs generally in < 10 min.

**III. Angles of contact due to different types of sol. S-containing (I) have been measured. The angle at the line air-solid-solution is completely determined by the non-polar group of the adsorbed (I).**

**IV. Arguments are given in favour of a complete unimol. layer of (I) as opposed to the "adlineation" postulated by Ostwald.** F. L. U.

**Elutriation and microscopical examination of finely-ground mineral grains.** H. K. SHIRREFS and A. F. EVANS (Proc. Austral. Inst. Min. Met., 1932, No. 88, 423—476).—The use of the Andrews kinetic elutriator for the examination and grading of fine ore is illustrated by results obtained on Rosebery Zn-Pb ore. The various fractions obtained are examined under the microscope after briquetting them with 15—20% of bakelite resin at 150° and polishing a section of the briquette. The val. of this type of work in dealing with mineral-separation problems in complex ores is discussed. A. R. P.

**Anode department of the Noranda smelter.** W. B. BOGGS, J. N. ANDERSON, and R. J. WESTWOOD (Trans. Canad. Inst. Min. Met., 1933, 35, 127—161).—A detailed account is given, with diagrams of the furnace and casting plant, of the present practice in casting anodes from converter Cu at the Noranda smelter. The refining reverberatory furnace (I), which is fired with coal dust, has a monolithic bottom made of "magnifrit" (a dolomitic refractory) bonded with iron-scale, and tamped on to a thick fireclay bed. The Cu from the converters (II) is tapped directly into (I) and blown until the O rises to 0.9%, the slags from this operation (2 tons for a 130-ton charge) being returned to (II); after

covering the metal with coke it is poled until the O is reduced to 0.05% and then cast by means of a Walker wheel into sand moulds to make 700-lb. ingots. Fuel consumption in (I) is about 100 lb. per ton of anodes cast. A. R. P.

**Colorimetric determination of antimony in copper alloys.** A. A. VASILIEV and M. E. SCHUB (J. Appl. Chem. Russ., 1933, 6, 560—562).—Clarke's method (A., 1928, 983) gives good results for solutions containing 0.001—0.004% Sb. The colorimetric solution should be cooled to 10° in order to prevent decomp. of the coloured complex (I); when the coloration is feeble, greater accuracy is obtained by extracting (I) with  $\text{C}_5\text{H}_{11}\cdot\text{OH}$ . R. T.

**Determination of phosphorus in copper and copper-zinc alloys.** A. A. VASILIEV and Z. V. TSHURILINA (J. Appl. Chem. Russ., 1933, 6, 563—566).—Complete pptn. of small quantities of  $\text{PO}_4'''$  as phosphomolybdate by Lorenz' method takes place in 1 hr., and not in 12 hr., as stated by Lorenz. The ppt. is best washed with EtOH, followed by  $\text{Et}_2\text{O}$ , and not with  $\text{COEt}_2$ , as in the original method. The Lorenz-Finkener method can be applied in the presence of Cu and of small quantities of Sn. Both methods are applicable to the analysis of alloys containing > 0.1% P. R. T.

**Determination of silver and copper in alloys without preliminary separation.** F. SIERRA and E. BURRIEL (Anal. Fís. Quím., 1933, 31, 129—133).—The sample (1.5 g.) is dissolved in  $\text{HNO}_3$ , the excess of acid is removed, and the solution is neutralised (Me-orange) with NaOH and diluted to 250 c.c. The Cu is titrated by adding the solution from a burette to a mixture of 20 c.c. of 0.05N- $\text{Na}_2\text{S}_2\text{O}_3$ , 30 c.c. of 0.2N-KI, starch, and 10 drops of 0.1N- $\text{AgNO}_3$ , and the Ag is determined by titration of 50 c.c. of the Ag-Cu solution with 0.05N-KI, benzidine sulphate being used as indicator unless the Ag concn. is < 0.01N, when benzidine acetate is preferable. H. F. G.

**Co-ordinated heat conservation at Normanby Park Steel Works, Scunthorpe.** W. J. BROOKE (Iron & Steel Inst., Sept., 1933, Advance copy, 38 pp.).

**Use of measuring instruments for fuel control in the iron and steel industry.** M. KOOPMAN and N. TURNER (J. Inst. Fuel, 1933, 6, 388—398).

**Gas economy. Corrosion in tar stills.**—See II.

See also A., Aug., 767, **Melting of metals. Electro-crystallisation of Ag.** 771, **Systems Na-K, Cu-Be, Sb-Mn, Fe-Ni. Brass. Ga alloys. Diffusion of Th in W. Binary mixed crystals of Mg. Solubility of Cd in Zn, of Al in Ag and in Cu, and, reciprocally, of fused Pb or Bi and fused Zn. V.p. and activities of binary alloys. System Ag-Cu-Ni.** 772, **System Cu-Si-Mg. Carbides in C-containing W and Mo alloys. Fe-Co-W alloys.** 775, **Metallurgical disperse systems.** 777, **Action of aq. KCN on colloidal Au.** 799, **Determining Cu, Fe, Zn, and Al in mixtures.**

#### PATENTS.

**Smelting of iron ores having a high sulphur content.** W. E. EVANS. FROM DEMAG A.-G. (B.P. 395,367, 10.10.31).—In the reduction of Fe compounds



of high S content, e.g., pyrites, the additional air required to convert FeS into FeO + SO<sub>2</sub> is admitted to the blast furnace. Higher in the furnace the SO<sub>2</sub> is reduced to S, which is recovered from the gases by usual methods.

C. A. K.

**Treatment of metallic ores. [Blast furnace for zinc ores.]** E. W. BUSKETT (U.S.P. 1,885,412, 1.11.32. Appl., 20.6.28).—From a blast furnace containing Zn ore and a reducing agent the vapours and gases are drawn off at a comparatively low level and immediately condensed by downdraught among granules of C in a tower, steam being added to generate H<sub>2</sub> and CO; the gases leave by an updraught tower adjacent, both towers having a common metal bath below.

B. M. V.

**Roasting pyrites fines.** R. F. BACON, Assec. of W. JUDSON (B.P. 394,539, 13.10.32. U.S., 21.11.31).—Fine pyritic ore (A) is allowed to fall downwards through a vertical kiln (I) in which it meets a current of hot SO<sub>2</sub> and N<sub>2</sub> from the roasting kiln (II), whereby the FeS<sub>2</sub> is converted into FeS and S, the latter being recovered from the gases after they have been used to preheat further quantities of A. The FeS from the bottom of (I) is transported by a screw conveyor to the top of (II) down which it falls against a rising stream of hot air which is so adjusted that the FeS is converted into Fe<sub>2</sub>O<sub>3</sub> and all the O<sub>2</sub> in the air is consumed; the hot gases from this stage are used in (I).

A. R. P.

**Production of cast iron of high strength in a cupola furnace.** K. EMMEL (U.S.P. 1,887,453, 8.11.32. Appl., 6.3.28. Ger., 15.3.27).—50% of the Fe charge is steel or wrought Fe, and the coke is > 13%, the air blast being 400—800 mm. H<sub>2</sub>O. The metal produced at the tuyère zone has a low C content, which can be increased by trickling the metal through a bed of coke below the tuyères, control being effected by varying the thickness of the bed.

B. M. V.

**Manufacture of magnetic electrolytic iron.** INTERNAT. GEN. ELECTRIC CO., LTD., ASSEES. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 395,505, 22.6.32. Ger., 22.6.31).—During electrolysis, equiv. quantities of C and O<sub>2</sub> are introduced into the Fe and are removed subsequently by annealing *in vacuo*. The C content may be regulated by using anodes containing C, or by means of org. compounds in the electrolyte, and the O content is adjusted by controlling the acidity of the solution.

C. A. K.

**Electrical steel.** C. J. STROBLE, ASS. to ALLEGHENY STEEL CO. (U.S.P. 1,887,339, 8.11.32. Appl., 9.10.29).—Transformer steel is heated and simultaneously stretched by passing it through two pairs of rolls, the delivering pair running at the higher speed.

B. M. V.

**Production of nickel layers on iron or steel.** F. VON WURSTEMBERGER (B.P. 395,979, 14.2.33. Ger., 4.3.32).—A malleable adherent coating of Ni is produced by an anodic pretreatment of Fe in H<sub>2</sub>SO<sub>4</sub> followed by a plating process in a suitable bath, e.g., NiSO<sub>4</sub> 280, Na<sub>2</sub>SO<sub>4</sub> 45, H<sub>3</sub>BO<sub>3</sub> 15, NaCl 17.5, K Na tartrate 10 g., H<sub>2</sub>O 1 litre.

C. A. K.

**Steel alloy.** W. H. WOODHALL, ASS. to HARRISON STEEL CASTINGS CO. (U.S.P. 1,885,667, 1.11.32. Appl., 4.12.30).—A high-strength steel suitable for castings

comprises C 0.20—0.60 (0.43), Mo 0.05—0.50 (0.25), Mn (optional) 1.00—1.50 (1.25), Cu (optional) 0.90—1.50 (1.10), Zr 0.15—0.30 (0.15)%, the balance being Fe.

B. M. V.

**Cutting-tool alloy.** G. R. COULS (U.S.P. 1,886,469, 8.11.32. Appl., 27.2.31).—Fe 26, W 21, Cr 31.5, Co 14, Cu 2.0—2.5, V 1.0—5.0 (as Fe-V), and Ti 1% (optional) are melted and mixed, in that order.

B. M. V.

**Iron alloy particularly for tools working in heat.** R. BELLAK (B.P. 394,346, 16.12.31. Austr., 22.12.30).—Alloys suitable for swages, die-casting apparatus, turbine blades, etc. consist of Fe with W 0.5—15 (2—6), Co 0.3—5 (0.5—2), Ni 1—5 (1—3), C 0.1—0.5, and Mo, V, Ta, Ti, or Zr 0.5%.

A. R. P.

**Extraction of copper from its ores.** A. AMENABAR (B.P. 394,316, 7.12.31).—The ore is leached with H<sub>2</sub>SO<sub>4</sub>, after roasting if necessary, and the solution treated with SO<sub>2</sub> and I or a sol. iodide to ppt. CuI and regenerate the H<sub>2</sub>SO<sub>4</sub>. The CuI is roasted either alone or with S to regenerate I, or treated with Zn or an alkali carbonate or hydroxide to produce a sol. iodide for further use. Alternatively, it may be heated with H<sub>2</sub>SO<sub>4</sub> (d 1.26) and SO<sub>2</sub> at 180°/160 lb. per sq. in. to obtain Cu and I, or introduced into a bath of molten Cu containing dissolved O<sub>2</sub>. [Stat. ref.]

A. R. P.

**Welding rod etc.** D. K. CRAMPTON and H. L. BURGHOFF, ASS. to CHASE COMPANIES, INC. (U.S.P. 1,887,500, 15.11.32. Appl., 26.4.32).—A Cu-Zn alloy is used comprising Cu 50—65, Cu + Zn < 80%; 0.002—0.200% of Be is added to prevent fume.

B. M. V.

(A) **Treatment of [zinc] sulphide ores.** (B) **Precipitation of zinc sulphide and manufacture of zinc sulphide pigments.** N. C. CHRISTENSEN (U.S.P. 1,886,162 and 1,886,165, 1.11.32. Appl., [A] 2.11.25, [B] 5.11.30).—(A, B) Sulphides of Pb, Cu, Ag, and Zn are dissolved out from the comminuted ore by means of hot, conc. CaCl<sub>2</sub> solution (>50CaCl<sub>2</sub>:100H<sub>2</sub>O) containing only a small excess of HCl. Ag is pptd. by Pb, the PbCl<sub>2</sub> by cooling, remaining Pb by H<sub>2</sub>S, Fe by CaCO<sub>3</sub>, and the Zn from the neutral solution by H<sub>2</sub>S. The H<sub>2</sub>S may be derived from the dissolution of the ore, and the HCl from waste aq. CaCl<sub>2</sub> and H<sub>2</sub>SO<sub>4</sub> (cf. U.S.P. 1,539,711—4 and 1,549,063; B., 1925, 598, 812). (B) An equimol. mixture of CaCO<sub>3</sub> and Ca(SH)<sub>2</sub> is an alternative precipitant for white ZnS.

B. M. V.

(A) **Furnace construction [for lead working].** (B) **Reclamation of lead.** (A, B) R. C. BRETT and (B) J. E. DOYLE, ASS. to EAGLE-PICHER LEAD CO. (U.S.P. 1,886,937—8, 8.11.32. Appl., [A] 20.12.30, [B] 26.1.31).—In (A), a two-hearth reverberatory furnace suitable for the process in (B) is described. In (B), the constituents of antimonial Pb metals and oxides (e.g., battery scrap) are separated by charging the material on to a bath of Pb, promoting further oxidation, and skimming off the Pb antimonate slag. The comparatively pure Pb is drawn off continuously under an arch, over a sill, into a second adjacent furnace at a slightly lower level, and the oxidation etc. are repeated.

B. M. V.

**Treatment of oxidised [lead-zinc] ores.** N. C. CHRISTENSEN (U.S.P. 1,886,163, 1.11.32. Appl., 19.10.27).—The ore is leached in NaCl and FeCl<sub>2</sub> brine with heat,



agitation, and aëration, whereby Zn and Pb are dissolved and  $\text{Fe}(\text{OH})_3$  is pptd.;  $\text{PbCl}_2$  is pptd. on cooling and the remaining Pb by Zn. The solution is electrolysed with an Fe anode to re-form  $\text{FeCl}_2$  and to obtain Zn. The  $\text{PbCl}_2$  is dissolved in the same electrolyte, which is again electrolysed to obtain Pb. B. M. V.

**Production of [aluminium-]plated zinc sheet.** F. JORDAN (B.P. 395,585, 13.12.32).—Sheets of Zn (containing 1–2% Al) and Al are heated at 180–450° and rolled together, followed by annealing to increase the surface-alloying. The Zn may be plated on one or both surfaces and then rolled out to form foil. C. A. K.

**Production of coated metal article. [Rust-proof coating for iron, zinc, and their alloys.]** R. R. TANNER and V. M. DARSEY, Assrs. to PARKER RUST PROOF CO. (U.S.P. 1,888,189, 15.11.32. Appl., 29.3.32. New Zealand, 18.12.29).—A balanced phosphate solution is dosed with Cu, Ag, or W inversely as the free acidity and proportionately to the rate of formation of the phosphate coating, the acidity to phenolphthalein being < 5 times the acidity to Me-orange. B. M. V.

**Wrought zinc product.** W. M. PEIRCE, Assr. to NEW JERSEY ZINC CO. (U.S.P. 1,888,567, 22.11.32. Appl., 14.3.29).—A Zn-base alloy containing Cu or Cd (0.05–5.0%) or Ni 0.05–1.0%, Mn 0.01–2.0%, and Mg or Li 0.005–0.5% is worked in the final stages at 175–200°, to avoid work-hardening but preserve the absence of cold-flow due to the alloy. B. M. V.

**(A) Zinc-aluminium-mercury, (B) zinc-aluminium anode for electrodeposition of zinc.** (A) G. B. HOGABOOM, (B) A. K. GRAHAM, Assrs. to HANSON-VAN WINKLE-MUNNING CO. (U.S.P. 1,887,841 and 1,888,202, 15.11.32. Appl., 20.2.31).—(A) An anode suitable for both acid and cyanide baths comprises Zn alloyed with Al 0.5 and Hg 0.2%. Other proportions of the metals (within the range < 5% Al and < 1% Hg) are claimed. (B) The alloy consists of Zn alloyed with < 5 (preferably 0.25–1.0)% of Al. B. M. V.

**Extraction of cadmium from its oxy-compounds.** A. H. STEVENS. From AMER. SMELTING & REFINING CO. (B.P. 393,764, 29.6.32).—Roasted ores or flue dusts containing Cd in association with As compounds are boiled with neutral aq.  $\text{Fe}_2(\text{SO}_4)_3$ , whereby the Cd is dissolved and the As pptd. with  $\text{Fe}(\text{OH})_3$ . The solution is treated with CdS to remove heavy metals and electrolysed for the recovery of Cd. A. R. P.

**Production of [age-hardening] alloys containing nickel.** MOND NICKEL CO., LTD. From INTERNAT. NICKEL CO., INC. (B.P. 394,378, 23.12.31).—Alloys of Ni having a face-centred cubic lattice, e.g., Ni-Cu alloys with 15–95% Ni and 0.5–80% Cu, Ni-Cr alloys with 3–30% Cr, and high Ni-Cr steels, are alloyed with 1–10% Ti and, after working, annealed at 600–800° until hardening is complete, then quenched. A. R. P.

**[Heat]-treating nickel and its alloys.** W. S. SMITH, H. J. GARNETT, and W. F. RANDALL (B.P. 395,371, 13.1.32).—The heat-treatment in all stages from ingot to wire or sheet is carried out in a gastight muffle lined with a Cr-Ni alloy to prevent contact of the metal with gases containing S, which produce a deterioration in the mechanical properties. A. R. P.

**Prevention of tarnishing of the surface of silver and its alloys.** VER. F. DIE PROBIER- U. FORSCHUNGS-ANST. F. EDELMETALLE (B.P. 395,491, 23.5.32. Ger., 23.5.31).—The articles are dipped for 1 min. in  $\text{H}_2\text{O}$  containing 0.5 g. of  $\text{CrO}_3$  per litre with or without a small amount of  $\text{K}_2\text{S}_2\text{O}_8$  and/or  $(\text{NH}_4)_2\text{CuCl}_4$ . A. R. P.

**Stainless silver alloy.** T. TANABE (U.S.P. 1,888,188, 15.11.32. Appl., 12.2.32. Jap., 17.2.31).—The alloy comprises Zn 0.5–20.0, Sn 10.0–40.0, Ag 50.0–89.5%. B. M. V.

**Extraction of gold from solutions.** H. C. PARKER (U.S.P. 1,889,489, 29.11.32. Appl., 5.1.32).—The Au-bearing solution (e.g., sea- $\text{H}_2\text{O}$ ) is agitated with FeS (120–200-mesh, 5lb. per ton of solution) and the auriferous FeS is separated. B. M. V.

**Manufacture of tungsten carbide.** O. F. MARVIN, Assr. to MILLS ALLOYS, INC. (U.S.P. 1,887,729, 15.11.32. Appl., 3.2.30).—Cu or other high-conductivity metal, in considerable mass, is used for moulds for casting WC. B. M. V.

**[Carbide] material for tool or tool parts.** W. W. TRIGGS. From NAAML. VENN. MOLYBDENUM CO. (B.P. 395,721, 16.11.31).—A material for tools contains 21–50% of carbides of the elements of group VI, < 22% of metals of groups VI and/or VIII, with carbides of group IV as remainder, e.g., carbides of Ti or Zr (31–74%) with Cr, Ni, or Co as the auxiliary metal. C. A. K.

**Magnesium alloys.** P. BRISKE and V. PROHL (BRISKE & PROHL), and A. LUSCHENOWSKY (B.P. 393,668, 15.12.31. Ger., 17.12.30).—Strong, corrosion-resistant Mg alloys contain < 80% Mg, > 15% Zn, > 2% Fe, > 3% Mn, and about 1.5–2% Ag or Be. A. R. P.

**Magnesium-base alloys.** DOW CHEM. CO., Assees. of J. A. GANN and F. L. REYNOLDS (B.P. 394,551, 26.10.32. U.S., 1.2.32).—Claims are made for Mg alloys containing 0.5–12% each of Cd and Sn, especially (a) 2% Cd and 4–8% Sn, and (b) 4–8% Cd and 8–10% Sn, (a) being suitable for hammer-forging and (b) for rolling and extrusion. A. R. P.

**Magnesium-manganese-zinc alloys.** J. A. GANN Assr. to DOW CHEM. CO. (U.S.P. 1,886,251, 1.11.32. Appl., 20.11.30).—The alloys comprise Mn 0.1–6.0, Zn (always > Mn) 0.5–16.0, Mg the remainder (always > 80%). B. M. V.

**Protecting magnesium and its alloys at elevated temperatures.** DOW CHEM. CO., Assees. of E. O. BARSTOW (B.P. 395,633, 8.2.33. U.S., 21.3.32).—The surface of heated Mg is protected by contact with a mixture of  $\text{CS}_2$  (1–10%) and one (or more) of the gases  $\text{N}_2$ , CO,  $\text{CO}_2$ , and  $\text{SO}_2$ . C. A. K.

**Aluminium-base alloys.** A. H. STEVENS. From ALUMINIUM, LTD. (B.P. 395,586, 14.12.32).—Claim is made for Al alloys containing Mn 0.75–3 (1–1.5) and Mg 0.2–0.5 (0.25)%. The Mg tends to restrain the coarse grain growth of Mn-Al alloys. A. R. P.

**[Zinc]-magnesium alloys.** C. ARNOLD. From DOW CHEM. CO. (B.P. 393,837, 21.10.32).—Claim is made for alloys containing Mg 81–99.4, Mn 0.1–3, and Zn 0.5–16%. The alloy with 1.4% Mn and 3% Zn



has yield point 5000 lb. per sq. in., tensile strength 26,000 lb. per sq. in., elongation 11%, impact val. 12 ft.-lb., and Brinell hardness 37. A. R. P.

**Electrolytic treatment of aluminium or its alloys.** C. H. R. GOWER and E. WINDSOR-BOWEN (B.P. 395,390, 6.1.32).—The Al articles are treated anodically at 15–20° with 1.5–3 amp. per sq. ft. at 100 volts in a bath made by mixing 1 vol. of 20% aq.  $Al_2(SO_4)_3$  with 8 vols. of a 5:4 vol.-mixture of conc.  $H_2SO_4$  and  $H_2O$  and adding 1% of a colloid, *e.g.*, dextrin, Iceland moss, or a vegetable gum. A. R. P.

**Production of galvanic deposits on aluminium and its alloys.** SIEMENS & HALSKE A.-G. (B.P. 394,637, 13.4.33. Ger., 30.4.32. Addn. to B.P. 385,067; B., 1933, 196).—The articles are treated with a.c. at 40 volts for 5 min. in a solution containing 40 g. of  $Na_2CO_3$  per litre, then plated thinly with brass in a cyanide bath which also acts as a degreaser, and finally plated with any suitable metal. A. R. P.

**Casting freely oxidisable metal.** J. A. GANN and M. E. BROOKS, Assrs. to DOW CHEM. CO. (U.S.P. 1,886,252, 1.11.32. Appl., 30.7.31).—A mould is composed of pulverised C, a plasticiser, an oxidation inhibitor, *e.g.*,  $H_3BO_3$ , S, or a hygroscopic halogen salt, and a little  $H_2O$ . B. M. V.

**Mould for high-temperature castings.** LE R. P. BENSING, Assr. to LEBANON STEEL FOUNDRY (U.S.P. 1,886,249, 1.11.32. Appl., 4.10.30).—The mould is faced with zircon (in rounded grains) and a bentonite or org. binder. B. M. V.

**Mould part.** G. H. WEIDEMANN, Assr. to DOW CHEM. CO. (U.S.P. 1,888,441, 22.11.32. Appl., 30.8.30).—Moulding sand, especially for light alloys, has admixed  $\frac{1}{2}$ –5 (1–3)% of an  $NH_4$  salt. B. M. V.

**Manufacture and use of light-metal alloy.** VEREIN. GLÜHLAMPEN U. ELECTRICITÄTS A.-G. (B.P. 395,989, 24.2.33. Ger., 29.2.32).—An alloy containing Ba 80–90 (85)% and Al 20–10 (15)% is used for the chemical fixation of gases and activation of cathodes in discharge tubes. C. A. K.

**Production of (A) electroplating [baths for tungsten], (B) tungsten plating, (C) substantially pure tungsten plating.** (A–C) C. G. FINK and (B) F. L. JONES (Assr.) (U.S.P. 1,885,700–2, 1.11.32. Appl., [A] 12.4.27, [B] 13.5.29, [C] 27.9.29. Renewed [A] 7.12.31).—W-plating baths are described; similar methods may be applied to the plating of Th, Ti, Zr, or Al. In (A),  $WO_3$  (30 g./litre) is dissolved in an alkali salt or hydroxide (75 g./litre approx.) at  $p_H$  12 and 55°. An excess of sol. carbohydrate and a minute quantity of cathode depolarising agent, *e.g.*, Ni, may also be added. In (B),  $WO_3$  (125 g./litre) is dissolved in  $Na_2CO_3$  (330 g./litre), at 60–100° and  $p_H$  10–13.  $H_2$  should be evolved freely in both cases. In (C), the use of Ni (> 1% of the  $WO_3$ ) is claimed and electrolyte is preferably withdrawn continuously, passed through  $NiCO_3$ , and returned to a remote point of the tank. B. M. V.

**Hg boiler. Catalytic converter.**—See I. Enamelled articles.—See VIII. Finishing gun barrels.—See XXII.

## XI.—ELECTROTECHNICS.

**Preparation of slag-resistant crucibles for the coreless induction furnace.** H. SIEGEL (Arch. Eisenhüttenw., 1933–4, 7, 21–23).—For the melting of Fe in high-frequency induction furnaces crucibles made of magnesite (I) behave better than those made of siemensite (II). In large furnaces the crucibles are tamped into a thin refractory container just inside the coil; for the best results careful grading of the particles is necessary to obtain max. refractoriness. The best binder for (I) is a mixture of 3% CaO and 0.5% clay; a higher % of the latter renders the crucible more liable to attack by basic slags. Owing to the rapid circulation of the Fe caused by the current severe undercutting of the crucible walls occurs just below the slag line; this may be prevented by building into the walls a ring of (II) made of small fused bricks having a slight projection into the crucible below the slag line. A. R. P.

**Smelter anodes.**—See X.

See also A., Aug., 767, **Electro-crystallisation of Ag.** 772, **Fe-Co-W alloys.** 791, **Electrolytic oxidation or reduction.** 791 and 804, **Action of the silent discharge on  $C_2H_4$ .** 792, **Effect of electrophoresis on photographic emulsions.** 800, **Const.-temp. apparatus.** 801,  **$p_H$ -meter.**

## PATENTS.

**Electric furnace.** C. R. JOHNSON, Assr. to AMERSIL CO., INC. (U.S.P. 1,889,665, 29.11.32. Appl., 9.9.31).—The apparatus comprises a D-shaped chamber wound with electric resistances; a ground cap with transparent window and inlet and outlet pipes are provided. B. M. V.

**Apparatus for developing heat by electricity.** A. S. WILLIAMS (U.S.P. 1,887,533, 15.11.32. Appl., 8.8.30).—Any  $H_2$  and  $O_2$  generated in an electrical  $H_2O$  heater or steam generator are caused to pass through an annular space between two catalysts and are reconverted into  $H_2O$ , the catalyst rod and cylinder forming part of the electric circuit. B. M. V.

**Thermionic tube.** H. W. PARKER, Assr. to ROGERS RADIO TUBES, LTD. (U.S.P. 1,889,105, 29.11.32. Appl., 13.2.30).—An anode is composed of Ni (or other metal of m.p. < 1427°) coated with Cu (or other metal of m.p. 1027–1427°), the coating being oxidised and capable of absorbing  $H_2O$  vapour. B. M. V.

**Vacuum tube.** L. L. PECK, Assr. to CLAUDE NEON LIGHTS, INC. (U.S.P. 1,888,421, 22.11.32. Appl., 19.12.27).—In a Ne lamp blued by Hg, striation of the latter by freezing is prevented by the presence of A and He. B. M. V.

**Incandescence electric lamp.** J. FORCE, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,889,598, 29.11.32. Appl., 4.6.28).—A getter for W lamps comprises a powdered metal of the W class adjacent to or in contact with the junction of the lead-in and filament. B. M. V.

**Composition for battery boxes.** R. N. CHAMBERLAIN, Assr. to AMER. CONTAINER CORP. (U.S.P. 1,886,269, 1.11.32. Appl., 6.12.27).—The moulding mixture is composed of diatomaceous earth, vegetable fibre, blown asphalt, and gilsonite. B. M. V.



(A) [Electrical] resistance element and (B) method of manufacture. (A, B) S. RUBEN, (A) Assr. to RUBEN PATENTS Co. (U.S.P. 1,859,344 and 1,889,379, [A] 24.5.32, [B] 29.11.32. Appl., [A] 6.10.30, [B] 1.4.31).—The base material is a strip of fused or vitrified  $\text{Cu}_2\text{O}$  having very high resistivity. In (A) the resistance is reduced, where desired, by a layer of a compound of Cu and S or other element of the same group (excluding  $\text{O}_2$ ), the thickness being preferably inversely as that of the base material, which may be improved by the addition of  $< 1\%$  of another metallic oxide. In (B) various schemes of manufacture of the CuS film are described, involving the use of  $\text{H}_3\text{PO}_4$  and S dissolved in  $\text{CS}_2$ .

B. M. V.

Producing an electrical [resistance] element. W. C. GEER, Assr. to B. F. GOODRICH Co. (U.S.P. 1,888,762, 22.11.32. Appl., 7.4.23).—The resistor comprises a vulcanised intimate mixture of hard rubber compound and about 20% of graphite, according to the conductivity required.

B. M. V.

(A) Excited electrolytic condenser. (B) Electrolytic rectifier. R. D. MERSHON (U.S.P. 1,889,415 and 1,889,417, 29.11.32. Appl., [A] 6.11.28, [B] 4.12.31).—(A) A combined rectifier and condenser is constructed with one electrolyte by the use of electrodes of different metals for the two functions. Al or an alloy of Al and Mg, Cu, Ta, or Ti is suitable for the rectifier electrode, acquiring the non-durable film, and borax- $\text{H}_3\text{BO}_3$  free from  $\text{H}_3\text{PO}_4$  for the electrolyte. (B) A filming electrode suitable for use in an electrolyte containing free  $\text{H}_3\text{BO}_3$  or  $\text{H}_3\text{PO}_4$  is composed of Al alloyed with Cd and preferably also with Ni or Cu, e.g., Cd 8.7, Ni 3.6%.

B. M. V.

Insulator bushing for electrical petroleum dehydrators. J. M. CAGE, Assr. to ELECTRIC SEPARATION Co., Ltd. (U.S.P. 1,887,010, 8.11.32. Appl., 11.2.30).—Concentric sleeves of a bushing are joined only by narrow rings of cement, the rest of the spaces being filled with insulating oil fed from a compartment surrounding the insulator outside the treatment tank.

B. M. V.

Cleaning gases.—See I. Distillation of tar. Creosoting compositions. Hydrogenation of oils. Testing combustible fluids.—See II. Piperidine.—See III. Paper for insulation.—See V. Melting glass. Ceramics [in valve making].—See VIII. Electrolytic Fe. Electrical steel. Ni layers on Fe. Reclaimed Pb [from battery scrap]. Treating Pb-Zn ores. Zn electrodeposition. Extracting Cd. Treating Al and its alloys. Uses of light-metal alloys. W-plate.—See X. Treating alimentary products.—See XIX. Modifying photographic images.—See XXI. Finishing gun barrels.—See XXII.

## XII.—FATS; OILS; WAXES.

Antioxygens of fatty oils. X, XI. Inversion of oxidative catalyst due to the species of fatty oils. M. NAKAMURA (J. Soc. Chem. Ind., Japan, 1933, 36, 335—337 B; cf. B., 1933, 637).—In general compounds (e.g., pyrocatechol, quinol) which act either as positive or negative oxidation catalysts during the insolation of linseed oil act in the same sense with regard to other

oils (Tsubaki, olive, rape, cottonseed, soya, and perilla oils). Whether the effect of (a)  $\beta$ -naphthol, (b) *p*-nitro-aniline, (c) vanillin, (d) yara-yara, is positive or negative, however, appears to depend on the I val. of the particular oil. These catalysts shorten the induction period of oxidation in the early stages of insolation, but decrease oxidation at later stages; the inversion, in the case of (b), (c), and (d), occurs sooner with oils of low I val., whilst with (a) inversion occurs earlier in the insolation of oils of high I val.

E. L.

Antioxygens of fatty oils. XII. Action of  $\beta$ -naphthol on the oxidation of soya-bean oil. M. NAKAMURA (J. Soc. Chem. Ind., Japan, 1933, 36, 408—410 B; cf. B., 1933, 637).—The addition of 0.05—0.2% of  $\beta$ - $\text{C}_{10}\text{H}_7\text{OH}$  (I) to soya-bean oil accelerates oxidation during insolation; at concns. of 0.6—1% (I) acts as a pro-oxidant in the early stages of insolation, but later retards oxidation. The inversion of the catalytic effect occurs the sooner, the greater is the concn. of (I) and the higher the velocity of the oxidation (e.g., in thinner layers).

E. L.

Antioxidants [for oils]. T. YAMADA (J. Soc. Chem. Ind., Japan, 1933, 36, 176—177 B).— $\alpha$ - and  $\beta$ - $\text{C}_{10}\text{H}_7\text{OH}$ ,  $\alpha$ - $\text{C}_{10}\text{H}_7\text{NH}_2$ , and *p*- $\text{NH}_2\text{C}_6\text{H}_4\text{OH}$  absorb  $\text{O}_2$  with an induction period in a similar manner to oils. Quinol is exceptional. Addition of oil to  $\alpha$ - and  $\beta$ - $\text{C}_{10}\text{H}_7\text{OH}$  accelerates absorption. Antioxidant activity is nearly destroyed by immersion of a strip of Cu in an oil, if the concn. of the antioxidant is  $<$  a certain val., and is still low above this val. Oils containing a large proportion of peroxides when treated with  $\alpha$ - $\text{C}_{10}\text{H}_7\text{OH}$  absorb more quickly after a short induction period. Antioxidants, with the exception of quinol, reduce peroxides slowly.

A. A. L.

Determination of unsaturated acids in commercial linseed oil by bromometric and thiocyanometric methods. E. YAMAGUCHI (J. Soc. Chem. Ind., Japan, 1933, 36, 180—181 B).—The CNS val. method is to be preferred.

A. A. L.

Solidification point of [China] wood oil. E. ROSSMANN (Farbe u. Lack, 1933, 341—342; cf. B., 1933, 315).—Samples of oil from *Aleurites fordii*, Florida (I), and *A. cordata*, Russia (II), carefully cold-pressed and protected from light so as to minimise formation of  $\beta$ -elaeostearin (III), did not solidify after several hr. at  $-20^\circ$ . A considerable rise in  $\eta$  takes place, however, which is attributed to the presence of  $\alpha$ -elaeostearin (IV), although a solution of the oil in  $\text{Et}_2\text{O}$  did not ppt. crystals at  $-50^\circ$ . Some (III) which had been added to (I) did not increase in quantity after 3 years' storage, although (II) had deposited crystals of (III) after 2 years and became a buttery mass at  $0^\circ$ ; this difference in behaviour is apparently due to the presence in (II) of (III) and also of mucilage which converts (IV) into (III). The sap. val., acid val.,  $d$ ,  $n$ ,  $\eta$ , Br val., rate of polymerisation, and Lovibond colour nos. of (I) and (II) are tabulated; (I) resembles a Chinese and (II) a Japanese product. The presence of oxidised or polymerised compounds is shown by the comparatively shallow zone of fluorescence in filtered ultra-violet light.

S. M.



**Heat test for the specification of Chinese wood oil.** M. OGURA (J. Soc. Chem. Ind., Japan, 1933, 36, 178—180 B).—The effects of temp. of heating, proportion of fatty acids, and various adulterants on the setting time of some Chinese wood oils are recorded.

A. A. L.

**Seeds and oil of *Staphylea pinnata*, L.** G. PAVLOV (Masloboino-Zhir. Delo, 1932, No. 4—5, 93—95).—The seeds contained H<sub>2</sub>O 10.41, oil 13.06, crude protein 12.92, crude fibre 55.05, ash 1.60, N-free extract 6.96%. The (edible) oil had  $\bar{d}$  0.9238,  $n_D^{20}$  1.4710, acid val. 2.78, sap. val. 193.65, I val. 117.46, CNS val. 82.77, Hehner val. 95.55, mean mol. wt. 289.7. The fatty acids (95.5%) consist of oleic 53.4, linoleic 38.3, and palmitic acid 3.8%.

CH. ABS.

**Application of fish oils as soap-making material.**

**I. Preparation and properties of soap-making material [polymerised oils] from fish oils.** II (1) Components of treated oils and (2) the properties of soaps. S. UENO, Z. OKAMURA, and N. KUZUI (J. Soc. Chem. Ind., Japan, 1933, 36, 313—316 B, 316—322 B).—I. As refined sardine oil was polymerised (A) (in a slow stream of H<sub>2</sub>) at 270—275° the  $\bar{d}$ ,  $n$ , and relative  $\eta$  increased steadily with continued heating; the I val. fell from 173 to 115 in the first hr., and to 98.8 in 5½ hr. When polymerisation was conducted (B) in presence of spent Ni-kieselguhr catalyst (inactive for ordinary hydrogenation) the  $\bar{d}$ ,  $n$ , and relative  $\eta$  showed pronounced increases in the first ½ hr. of reaction, and but little change afterwards; the I val. fell to 95.7 and 75.5 in ½ hr. and 5½ hr. respectively. All the polymerised oils gave a deep green colour in the Tortelli-Jaffe reaction.

II. The fatty acids separated from the polymerised oils (A) and (B) include solid unsaturated acids of the series C<sub>n</sub>H<sub>2n-2</sub>O<sub>2</sub> and C<sub>n</sub>H<sub>2n-4</sub>O<sub>2</sub> ( $n > 18$ ). The lathering power, drop no., surface tension, and relative  $\eta$  of 0.25% solutions of Na soaps made from the polymerised oils and from tallow, coconut, and cottonseed oils (alone and various mixtures) have been compared (figures detailed). The polymerised-oil soaps yield excellent results and improve the viscosity of the mixed soaps; soaps from the (A) oils developed an unpleasant odour in 6 months, which was not observed, however, in the case of soaps from the (B) oils.

E. L.

**Fish oils as soap-making material. III. Influence of various substances. IV. Influence of pressures.** S. UENO, Z. OKAMURA, and N. KUZUI (J. Soc. Chem. Ind., Japan, 1933, 36, 392—395 B, 395—396 B; cf. preceding abstract).—III. Figures showing the effect of the addition of 1% of CaO, Mg, MgO, ZnO, ZnCl<sub>2</sub>, Sn, Pb, etc. on the polymerisation of sardine oil at 270—280° (1—3 hr.) are detailed.

IV. Refined sardine oil was polymerised at 270—280° in H<sub>2</sub> at 50 and 100 atm. pressure. The I val. (original 173.6) rose slightly after an initial fall (to about 110), indicating slight dehydrogenation during polymerisation; the products from 4½ hr. treatments still yielded Et<sub>2</sub>O-insol. bromides and gave a green colour in the Tortelli-Jaffe reaction (A). Polymerisation in presence of 0.25% of spent Ni catalyst caused considerable fat splitting (acid val. after 4½ hr., e.g., 140); the

I val. fell lower (to < 10 after 1½ hr.) but slight dehydrogenation followed and the products no longer yielded insol. polybromides or a positive (A).

E. L.

**Shaving-soap manufacture.** G. KNIGGE (Allgem. Oel- u. Fett-Ztg., 1933, 30, 299—301).—Practical notes on commercial methods.

E. L.

**Quality of Japanese fish oils from point of view of the hardened oil industry. IV. Quality of sardine oils. 2.** S. UENO, G. INAGAKI, and K. KOIZUMI (J. Soc. Chem. Ind., Japan, 1933, 36, 396—400 B; cf. B., 1933, 754).—Commercial Japanese sardine oils obtained between spring, 1931, and autumn, 1932, from Hokkaido and Chosen had, respectively: acid val. 0.5—7, 0.5—10.9; sap. val., 187—193, 187—203; I val. 170—182, 170—183. These figures are detailed for >140 samples, and indicate the more uniform and improved quality of the modern industrial oils.

E. L.

**Capillarity of marine-animal oils.** H. MARCELET (Compt. rend., 1933, 197, 130—131; cf. B., 1927, 304).—The extent to, and velocity with, which various marine-animal oils (30 samples from 22 animals, of mol. wt. 399—1494 were examined) ascended a vertical strip of filter-paper dipping into the oil at one end, or spread horizontally from a drop, decreases as the mol. wt. increases; it is also decreased by oxidation of the oil, but is independent of density, viscosity, and chemical constitution.

C. A. S.

**Japanese beeswax. II. Composition of free and combined fatty acids.** H. IKUTA (J. Soc. Chem. Ind., Japan, 1933, 36, 377—379 B; cf. B., 1931, 125, 307).—A sample of Japanese beeswax (secreted by *Apis indica*, Fab., var. Japonica, "Rads") had  $\bar{d}_4^{20}$  0.8210, m.p. 65—65.5°, acid val. 7.5, sap. val. 87.8, I val. (Wijs) 12.6, unsaponifiable matter 56.3% (m.p. 75—75.5°). The combined fatty acids included palmitic acid (about 60%), oleic acid (trace), acids C<sub>26</sub>H<sub>52</sub>O<sub>2</sub> (cerotic) and C<sub>31</sub>H<sub>62</sub>O<sub>2</sub> and a OH-fatty acid; the C<sub>26</sub>, C<sub>31</sub>, and OH-acids were also isolated from the free fatty acids.

E. L.

**Washing effect.** E. GOTTE [with W. KLING] (Kolloid-Z., 1933, 64, 222—227).—Washing efficiency is determined by placing the detergent solution in a large glass jar, which is rotated at 60 r.p.m. in a const.-temp. bath. Agitation can be increased by adding rubber balls. Using Na stearate a max. washing effect is obtained at  $p_H$  10.7.

E. S. H.

**C from olive press-cake.**—See II. Rancidity.—See XIX.

See also A., Aug., 807, Polymerisation of the elæostearic acids. 846, Liver oil of *Etmopterus spinax*. 848, Milk-fats of Indian goats and sheep. 870—1, Vitamins in oils. 876, Waxy substances of *Ceroplastes rubens*. Nigaki oil. Alteration of castor oil by heat.

PATENTS.

**Stabilising fats and oils.** R. C. NEWTON and W. D. RICHARDSON, Assrs. to SWIFT & Co. (U.S.P. 1,890,585, 13.12.32. Appl., 28.3.30).—The fat (e.g., lard) is mixed with a carotenoid pigment (conveniently in the form of 5—10% of palm oil), deacidified with NaOH, hydrogenated if desired, and heated at, e.g., 182—215° in a steam (vac.) deodoriser.

E. L.



**Hydrogenation process [for fatty oils] and catalyst therefor.** O. G. BENNETT, ASSR. to CATALYST RESEARCH CORP. (U.S.P. 1,893,155, 3.1.33. Appl., 18.6.32).—The Ni-Cr oxide catalyst is prepared by heating pptd. Ni NH<sub>4</sub> chromate [from a sol. Ni salt and (NH<sub>4</sub>)<sub>2</sub>CrO<sub>4</sub>] at about 350°, in order to convert it into Ni chromite, which is reduced with H<sub>2</sub> at about 500°. E. L.

**Recovering retained oils and fats from filter-press cake and reactivation of the spent cake.** W. KELLEY, ASSR. to W. S. BAYLIS (U.S.P. 1,891,715, 20.12.32. Appl., 12.11.30).—The oily cake is digested with steam at 176–205°/150 lb., whereby the fats are hydrolysed; the H<sub>2</sub>O is allowed to evaporate and the fatty acids, together with impurities, are extracted with EtOH, recovered, and distilled. The purified cake is ready for re-use after washing with EtOH and steaming. E. L.

**Distillation of glycerin.** O. H. WURSTER (U.S.P. 1,885,166, 1.11.32. Appl., 13.2.28).—A system in which superheated steam at a temp. above that of the glycerin-H<sub>2</sub>O vapours and at least as high as that of the boiling liquid is injected into the still, and in which the evaporators for the sweet waters are connected in multiple effect, is described. B. M. V.

**Manufacture of soaps having a disinfecting action.** DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM ROESSLER ([A] B.P. 395,570, 26.11.32. Ger., 28.11.31, and [B] Addn. B.P. 395,572, 28.11.32. Ger., 28.7.32).—(A) The soap contains Ag, in the form of powder or flakes (I), which may have been activated previously by treatment with oxygenating compounds (II) (Na<sub>2</sub>O<sub>2</sub>, NaBO<sub>3</sub>, etc.), or (I) and (II) may both be added to the soap, H<sub>2</sub>O being excluded as far as possible. (B) A difficultly sol. Ag compound, e.g., a Ag subhalide or AgCNS, is incorporated in the soap, (I) and (II) being also added if desired. E. L.

**Manufacture of [titanium] metallic soaps.** TITANIUM PIGMENT CO., INC. (B.P. 395,406, 15.1.32. U.S., 16.1.32).—A preheated solution of alkali hydroxide and fatty acid(s) or resin acid are mixed at 40–70°, and a salt of a quadrivalent metal of group IV(a) (especially Ti) is added with agitation. Mixed soaps, e.g., a Ti-Co tungstate-linoleate may be prepared. E. L.

**Soap product.** J. H. JAMES, ASSR. to C. P. BYRNES (U.S.P. 1,894,096, 10.1.33. Appl., 7.5.29).—The fractions of higher mol. wt., obtained by fractional distillation and solvent treatment of the products of the oxidation of hydrocarbons (cf. B., 1929, 425), are saponified. E. L.

**Bleaching of soap stock.** A. M. LAGASSE, ASSR. to S. G. TURNBULL (U.S.P. 1,890,121, 6.12.32. Appl., 31.5.30).—Soap stock (grease etc. preferably filtered through suitable earths or C), which has been bleached by means of a non-alkaline or acid solution of H<sub>2</sub>O<sub>2</sub> (≧ 1% H<sub>2</sub>O<sub>2</sub> on wt. of stock) at 45–55° until all H<sub>2</sub>O<sub>2</sub> is decomposed, yields a non-porous soap. E. L.

**Emulsion [for shaving].** E. L. MACK and C. W. BENNETT, ASSR. to BENNETT-MACK CORP. (U.S.P. 1,888,601, 22.11.32. Appl., 7.3.30).—A shaving cream composed chiefly of stearic acid, coconut oil, and paraffin

wax in glycerin and dil. aq. NH<sub>3</sub>, with traces of other addenda, is claimed. B. M. V.

**Floor polishing compositions, shoe creams, etc.** HENKEL & Co., G.M.B.H. (B.P. 395,538, 13.9.32. Ger., 6.10.31).—Sulphuric esters of alcohols (e.g., derived by catalytic hydrogenation of coconut oil) or their (Na) salts are used to stabilise wax-water-oil emulsions. E. L.

**Purifying used oils.**—See II. Emulsions. Soap-like preps.—See III. Preventing formation of lumps.—See XVII. Stabilising lard.—See XIX. Disinfectant detergents.—See XXIII.

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

**Alcohol fraction of turpentine oil from *Pinus sylvestris*.** V. KRESTINSKI and F. SOLODKI (J. Appl. Chem. Russ., 1933, 6, 508–518).—A sample of turpentine oil contained 72% of alcohols, which after separation formed two layers, of which one contained equal vols. of saturated and unsaturated alcohols, amongst which phellandrene hydrate, terpinen-4-ol, *p*-menthanol, a dicyclic alcohol, C<sub>10</sub>H<sub>18</sub>O (probably a carene derivative), and traces of *d*-camphor were identified, whilst the other consisted of 20% of borneol and 80% of an unseparated mixture of sylveoterpinols. R. T.

**Determination of hexabromide number of linseed oil.** F. FRITZ (Chem.-Ztg., 1933, 57, 596).—Modifications of the author's method (B., 1930, 620) are given. The inlet tube for the air stream which is to remove excess Br vapour should terminate just above the surface of the solution, whilst the short tube, opening just below the cork, is connected to the pump. E. L.

**Microscopical detection of mineral oil in linseed oil.** N. FUCHS (Farben-Ztg., 1933, 38, 1217).—Whilst small amounts of mineral oil mix completely with raw linseed oil, owing to the presence of hydrophilic OH and CO<sub>2</sub>H groups, separation takes place on oxidation and gelation of the oil, and drops of mineral oil may be seen under low magnification. The effect is enhanced by moistening the dried film, as little as 2% of light (spindle) oil and 1% of heavy (machine) oil being discernible in this way. S. S. W.

**"Extenders."** H. WOLFF, G. ZEIDLER, and B. ROSEN (Farben-Ztg., 1933, 38, 1188–1189, 1214–1215).—The functions of extenders as part of the paint system as a whole are detailed, and their indiscriminate banning is criticised. A series of weathering and H<sub>2</sub>O-absorption experiments shows that whilst blanc fixe is inferior to barytes as extender, addition of the latter to lithopone, red oxide, etc. appreciably diminishes swelling and improves durability. To an extent the action of BaSO<sub>4</sub> is bound up with lowering of "crit." oil content of paint. S. S. W.

**Testing lacquer paints under the conditions of carbon tetrachloride production.** M. V. BORODULIN and R. G. BELOZERSKAYA (Trans. State Inst. Appl. Chem., 1932, No. 15, 37–39).—Bituminous compositions are least resistant; bakelite and cellulose nitrate lacquers can possibly be used in coating storage and transportation equipment requiring no heating.



**Effectiveness of paints in retarding moisture absorption by wood.** F. L. BROWNE (Ind. Eng. Chem., 1933, 25, 835—842; cf. B., 1927, 821).—Specimens cut from southern yellow pine, Douglas fir, northern white pine, and redwood were brought to equilibrium at 60% R.H. and 27° and coated with paints the moisture-resistance of which was measured, both before and after exposure, by comparing the wt. increase after storing for 1 week at 97% R.H. with the increase of an unpainted specimen. Data are tabulated for many paints made from white, transparent, and coloured pigments, Al [bronze] powder, and mixtures of Al powder and granular pigments in linseed oil, as well as for various vehicles, variable pigment concns., and brushed and sprayed paints. In nearly all cases the greater portion of moisture-resistance is obtained by application of the second coat, the primer alone being inefficient. Al paint alone is a less effective primer than one containing a granular pigment, but when covered with one or more coats of white-Pb the Al primer becomes the more efficient. Al is suitable for all coats only when finely-divided and dispersed in a special varnish medium. A theory of wood priming is advanced. S. M.

**Permeability of paint films to moisture.** R. I. WRAY and A. R. VAN VORST (Ind. Eng. Chem., 1933, 25, 842—846).—The permeability of 12 clear varnishes and enamels was determined by (a) Browne's method (see above), and by measuring the increase in wt. of activated  $Al_2O_3$  contained in (b) a Petri dish covered with the dried film and (c) a bottle sealed with discs of wood painted on both sides. The results were not in good agreement and show that each method is empirical. Method (a) gave particularly wide discrepancies for the first coat. With (c) an equilibrium was soon reached, after which the efficiencies were const. Addition of Al-bronze powder in flake form to a clear varnish reduced the permeability considerably, the finer grades, within limits, being most effective. Lithopone and white-Pb were less effective in consequence of their granular structure. S. M.

**Interpretation of moisture permeability measurements [on paints].** J. D. EDWARDS (Ind. Eng. Chem., 1933, 25, 846—847).—Discrepancies in the results obtained by various methods (see preceding abstract) are discussed. S. M.

**Oil absorption of brushable paints.** E. KLUMPP (Farben-Chem., 1933, 4, 285—287).—A table is given to show the  $d$ , the particle size (I), the oil absorption to form a paste (II), and the oil absorption to form a brushable paint (III) for 13 pigments. (III) varies from 1 to 4.7 times the vol. of pigment, is 1.3—3.5 times (II), and bears no obvious relation to (I) when small. When (I) is comparatively large, as in barytes, (III) is low and is approx. only 1.5 times (II). With mixtures of barytes and a fine pigment in varying proportions (II) passes through a min., but (III) shows a more or less gradual increase with increase in the quantity of fine pigment. It is concluded that the quantity of oil required for brushable paints can be reduced to a min. by the use of coarse pigments or of mixtures of coarse and fine pigments containing a large excess of coarse particles. S. M.

**Analysis of oil varnishes.** J. SCHEIBER (Farbe u. Lack, 1933, 353—354, 365—367).—A general discussion is given of the identification and determination of natural and synthetic resins and of tung oil in varnishes. Resins may dissolve in the drying oil or react with it, with or without formation of free fatty acids; the reaction product is best removed by distillation in  $CO_2$  up to 150—180°. The determination of resin acids and oil acids by extraction with MeOH is inaccurate when oxidation has taken place since oxidised fatty acids are less easily esterified by MeOH and therefore behave like the resins; a gravimetric process (details given) gave much lower resin contents, but in the case of Manila copal varnishes both methods were unsatisfactory. S. M.

**Linoleum and oilcloth manufacture.** J. ALLAN (J. Oil & Col. Chem. Assoc., 1933, 16, 257—282).—A lecture.

**Brazilian balsams and their most frequent adulterants.** F. W. FRAISE (Ann. Falsif., 1933, 26, 324—331).—Physical and chemical characteristics of a no. of pure balsams, and the probable adulterants of copaiba, pao, sapote, sorva, and tamararé balsams, are given. E. C. S.

**Sealing wax and lead seals with a base of bitumen composition.** E. I. BARG (Plast. Massi, 1932, Nos. 2—4, 29—34).—Mixtures of coumarone resins and benzyl-cellulose with chalk give a good sealing wax. Certain Russian asphalts are good bases for sealing wax and lead seals. CH. ABS.

See also A., Aug., 829, **Kawa and manila-elemi resins.** 831, **Amber.** 833, **Benzopyrillium pigments.** 847, **Lyochromes.** **Lactoflavin.**

## PATENTS.

**Manufacture of base for paints, varnishes, and lacquers.** E. C. HOLTON, ASST. to SHERWIN-WILLIAMS Co. (U.S.P. 1,888,595, 22.11.32. Appl., 24.2.31).—A mixture of castor oil and a vegetable drying oil having I val.  $> 160$  is destructively distilled down to 70—90% of its original wt. and then heated with phthalic anhydride (or other polybasic acid) and/or glycerin (or other polyhydric alcohol) until the melt becomes homogeneous, clear, and tranquil. B. M. V.

**Manufacture of pigments.** E. M. LOFLAND (U.S.P. 1,888,464, 22.11.32. Appl., 27.9.28).—Comminuted Fe is wetted and allowed to oxidise to  $FeO_{aq}$ , which is removed from the parent metal, further oxidised at room temp., dried, and heated. The colour may be darkened by addition of alkali before or after the second oxidation. B. M. V.

**Manufacture of sunfast and inert lithopone.** K. S. MOWLDS and W. SEGUINE, JUN., ASSTS. to KREBS PIGMENT & COLOR CORP. (U.S.P. 1,885,940, 1.11.32. Appl., 21.9.29).—ZnO is removed from heat-treated lithopone by means of  $(NH_4)_2CO_3$ , preferably formed *in situ*, by injection of  $CO_2$  into a suspension of the pigment in aq.  $NH_3$ . The  $(NH_4)_2CO_3$  remaining on the lithopone is removed by calcination. B. M. V.

**Production of titanium pigments.** C. DE ROHDEN, ASST. to KREBS PIGMENT & COLOR CORP. (U.S.P. 1,885,187, 1.11.32. Appl., 17.1.28).— $TiO_2$  hydrolytically pptd. in



dil. HCl is digested with a reducing agent, *e.g.*, H<sub>2</sub>S or a thiosulphate, to enhance the whiteness. B. M. V.

**Reducing the oil absorption of titanium dioxide pigment.** G. KUBIN, ASSR. to KREBS PIGMENT & COLOR CORP. (U.S.P. 1,885,921, 1.11.32. Appl., 16.2.29).—Hydrolytically pptd. TiO<sub>2</sub> is ground wet, dried, and then ground dry. B. M. V.

**Manufacture of titanium white.** S. J. LUBOWSKY, ASSR. to METAL & THERMIT CORP. (U.S.P. 1,888,993, 29.11.32. Appl., 10.7.28).—In the process described in U.S.P. 1,640,952 (B., 1927, 815), the Ti—Mg salt solution is heated at 100° until the Ti is completely pptd., and digestion in the mother-liquor is prolonged under pressure at 135–140° to improve the whiteness. B. M. V.

**[Washable] coating for wallpapers.** E. I. DU PONT DE NEMOURS & Co. (B.P. 394,974, 31.12.31. U.S., 2.1.31).—A solution of nitro- or benzyl-cellulose is applied to which sufficient wax and Al or Zn stearate have been added to produce a matt effect. S. M.

**Transferring of designs and transfer composition.** W. S. LAWRENCE, ASSR. to KAUMAGRAPH Co. (U.S.P. 1,893,626, 10.1.33. Appl., 20.6.28).—A base, *e.g.*, paper, carrying a design consisting of a cellulose ester film is immersed in a transfer liquid comprising an active solvent for the cellulose ester of relatively high b.p., *e.g.*, Et lactate, and a non-solvent of relatively low b.p., *e.g.*, benzine, the proportion of these being such that the design does not dissolve or run. The transfer is removed from the liquid, the non-solvent is allowed to evaporate leaving the design moist and sticky, and the film is applied by pressure to the surface to be marked. S. S. W.

**Manufacture of [tung oil] varnishes.** ELEKTRO-CHEM. WERKE MÜNCHEN A.-G. (B.P. 395,299, 9.3.33. Ger., 18.4.32).—Aq. emulsions of heat-thickened tung oil prepared in a rapid disperser with addition of K resinates or other emulsifying agent and turpentine oil can be brushed or sprayed to give satisfactory films, 0.01–0.02 mm. thick, which dry in 2–3 hr. with a Co drier. S. M.

**Composition for coating, decorating, and printing wood, metal, paper, cloth, and other surfaces.** T. F. BRADLEY (U.S.P. 1,884,553, 25.10.32. Appl., 11.4.27).—The ink comprises nitrocellulose dissolved in a composite volatile solvent, *e.g.*, EtOH and benzol, a resin, a plasticiser, a pigment, and sufficient wax, *e.g.*, ceresin ( $\frac{1}{2}$ –2%), to reduce evaporation at room temp. but insufficient to spoil the hard smooth finish. B. M. V.

**Protective coating.** A. W. SEYMOUR (U.S.P. 1,893,599, 10.1.33. Appl., 23.5.30).—A colloidal, aq., bituminous emulsion is applied to structural surfaces as undercoat, and followed by a top coat, wholly bituminous and non-colloidal, which is normally non-fluid and is heated before application. A permeable fabric layer may be interposed between the coats if desired. S. S. W.

**Manufacture of oil-soluble synthetic resins.** I. G. FARBENIND. A.-G. (B.P. 392,382, 20.7.32. Ger., 20.7.31).—An aromatic halide is condensed with a recent natural resin and the product is esterified with

a mono- or poly-hydric alcohol; the esterification may precede the condensation. Examples are: American colophony with CH<sub>2</sub>PhCl and ZnCl<sub>2</sub> at 100–210°, esterified with glycerol in xylene at 100–250°; copal with 2:4-dimethylbenzyl chloride, esterified with CH<sub>2</sub>Ph·OH; colophony esterified with glycerol, condensed with chlorinated retene; abietic acid with chlorinated abietic acid, esterified with glycerol. C. H.

**Pigments.**—See III. **IV. Plastics etc.**—See V. **Oxides.**—See VII. **Abrasive articles.**—See VIII. **Wood fillers.**—See IX. **ZnS pigments.**—See X. **Ti soaps.**—See XII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Coagulation of [rubber] latex and latex mixings for industrial purposes.** R. G. JAMES (Trans. Inst. Rubber Ind., 1933, 9, 42–58).—A review is given of the coagulation process as applied to natural or preserved latex, or operated in various manners, *e.g.*, by spreading, spraying, dipping, filtration, or electrodeposition. Consideration is also given to coagulation by salts, and production of continuous coagula, *e.g.*, with heat-sensitised latex mixtures or by using coagulants with delayed action, and of discontinuous or crumb-like coagula. D. F. T.

**Interfacial relationships between rubber and fillers.** G. D. LEFCADITIS (Trans. Inst. Rubber Ind., 1933, 9, 67–93).—The influence of compounding ingredients on the resistance of rubber to tearing is independent of their reinforcing qualities; C black, therm-atomic black, and ZnO increase the tear-resistance (I), but Mg carbonate and china clay reduce it. Surface treatment of clay, *e.g.*, by coating the particles with various materials or by the action of HF, gives no outstanding alteration in its properties, although a slight improvement in its effect on (I) is obtained by passing the powder through a flame. The physical properties of vulcanised rubber prepared from compounded latex (with and without clay) without mechanical working are superior to those of similar mixtures prepared in the ordinary manner, especially with respect to (I); the mastication during ordinary compounding has a remarkably adverse effect on (I), whilst other mechanical properties are unaffected. The interfacial affinity of rubber and fillers is the factor probably responsible for resistance to tearing and abrasion of vulcanised rubber, whereas tensile strength is probably dependent on friction and film-formation. The mechanical properties are also functions of the rate and temp. of vulcanisation. The best physical properties with any particular compound were obtained by producing it direct from latex or by milling in N<sub>2</sub> and by using a powerful accelerator of vulcanisation. D. F. T.

**Effect of reclaim on the manipulation of rubber.** G. MARTIN and H. C. BAKER (Trans. Inst. Rubber Ind., 1933, 9, 14–41).—Contrary to the common view, mixtures of "reclaim" (I) and rubber are not generally more easy to masticate than mixtures without (I) and the amount of power needed in mastication is not definitely reduced. Mixtures containing also C black, ZnO, and oil are not caused to mould or extrude more





readily by the presence of (I). The only advantageous effect observed was that (I) reduced the extent to which the unvulcanised compounded rubber mixings recovered after deformation. D. F. T.

**Stress-strain relationships of vulcanised india-rubber.** J. R. SCOTT and C. W. SHACKLOCK (Trans. Inst. Rubber Ind., 1933, 9, 94—96; cf. B., 1933, 640).—An error in the mathematical treatment is corrected. Although the network structure suggested for vulcanised rubber, gelatin, and vulcanised oils offers some explanation for the inflexion point in the stress-strain curve, this explanation is insufficient to account for the pronounced inflexion observed. D. F. T.

**Volumetric determination of free sulphur [in rubber].** II. W. D. GUPPY (Trans. Inst. Rubber Ind., 1933, 9, 59—66; cf. B., 1932, 156).—The reduction method for determination of free S when applied to vulcanised rubber mixtures containing an aldehyde-amine antioxidant or an accelerator, e.g., diphenylguanidine or tetramethylthiuram disulphide, gave results below those of methods involving oxidation of the COMe<sub>2</sub> extract; the latter methods presumably include some of the combined S. On account of the inability of the S compounds in brown substitute or in ebonite to resist nascent H, the method cannot be applied to these materials directly, but it can be used to determine the free S in the COMe<sub>2</sub> extract from ebonite or from vulcanised rubber containing dark substitute. The combined S in white substitute is stable towards the reducing agents used. D. F. T.

PATENTS.

**Manufacture of rubber.** P. SCHIDROWITZ, and PIRELLI, LTD. (B.P. 395,066, 12.2.32).—Conc. rubber latex, compounded and vulcanised or otherwise, may be stabilised against thickening or its viscosity may be reduced without material dilution, by the addition of a small proportion of a strong org. base, e.g., piperidine or NHEt<sub>2</sub>. D. F. T.

**Manufacture of rubber compositions.** DUNLOP RUBBER CO., LTD., D. F. TWISS, and F. A. JONES (B.P. 395,109, 2.4.32).—In order to effect the uniform incorporation of org. acids containing one or more OH or > 1 CO<sub>2</sub>H, which are introduced for temporary retardation of vulcanisation, the acids are previously rendered easily fusible by combination with or dissolution in easily fusible substances such as glycerol, N(C<sub>2</sub>H<sub>4</sub>-OH)<sub>2</sub>, NH<sub>2</sub>Ph, C<sub>10</sub>H<sub>7</sub>-NH<sub>2</sub>, the proportion of any basic substance being desirably such as to satisfy only part of the acidity of the acid. D. F. T.

**Composition [from rubber].** H. A. WINKELMANN, Assr. to B. F. GOODRICH CO. (U.S.P. 1,890,924, 13.12.32. Appl., 25.10.27. Cf. U.S.P. 1,605,180; B., 1927, 85).—Rubber is converted into a thermoplastic product by heating in intimate admixture with an org. sulphinate of a heavy metal, especially a multivalent metal, e.g., with Fe<sup>III</sup> toluenesulphonate (20%) for 6 hr. at 140°, or phenolsulphonate. D. F. T.

**Rubber composition and method of preserving rubber.** W. L. SEMON, Assr. to B. F. GOODRICH CO. (U.S.P. 1,890,916—7, 13.12.32. Appl., 16.5.32).—

Rubber is treated with a product obtained by condensing a diarylamine, e.g., NHPh<sub>2</sub>, or an alkoxydiarylamine and (A) CH<sub>2</sub>O or (B) an aliphatic aldehyde > C<sub>2</sub>, e.g., α-ethyl-β-propylacetaldehyde or PrCHO. Acid favours the condensation. D. F. T.

**Rubber product.** F. L. SHEW (U.S.P. 1,893,966, 10.1.33. Appl., 16.9.29).—A rubber product of substantially pure white colour is obtained by incorporating a pigment such as TiO<sub>2</sub>, and, after vulcanisation, bleaching, e.g., with HOCl. Vulcanite may be polished before bleaching. D. F. T.

**Vulcanisable compounds [containing rubber] and vulcanised products derived therefrom.** J. C. PATRICK and N. M. MNOOKIN (U.S.P. 1,890,231, 6.12.32. Appl., 28.5.28).—The "olefine polysulphide" plastics, such as the products from ethylene dichloride and Na polysulphide, are mixed with unvulcanised rubber (e.g., 1:1) and S; other compounding ingredients also may be added. Although the mixture may desirably contain considerably more excess S than is necessary for vulcanisation, the vulcanised products do not bloom and show remarkable resistance to oxidation and sun-cracking. D. F. T.

**Manufacture of vulcanised rubber.** O. BEHREND, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,891,743, 20.12.32. Appl., 12.2.30).—Vulcanisation is accelerated by the reaction product derived from an ω-Cl-substituted PhMe, e.g., CH<sub>2</sub>PhCl, and an aliphatic aldehyde-NH<sub>2</sub> compound such as heptaldehyde-ammonia. D. F. T.

**Vulcanisation of rubber.** H. A. MORTON (U.S.P. 1,893,868—9, 10.1.33. Appl., [A] 30.9.29, [B] 7.1.31).—(A) The vulcanisation characteristics of rubber are rendered more uniform and the tensile qualities of low-grade rubber are improved by the presence of a univalent salt of AcOH, e.g., NaOAc, preferably hydrated, and with concurrent presence of an org. accelerator such as α-ethyl-β-propylacetaldehyde-aniline (I). (B) Vulcanisation in presence of an org. accelerator, e.g., (I), is further expedited by an alkali salt of an aliphatic monobasic acid > C<sub>2</sub>, e.g., Na ethylhexoate or laurate. D. F. T.

**Vulcanisation of rubber.** W. SCOTT, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. [A] 1,890,165, 6.12.32; [B] 1,893,846, 10.1.33. Appl., [A] 15.3.28, [B] 16.5.28).—Vulcanisation is accelerated (A) by the product obtained by successively treating butylidene-aniline (e.g., 1 mol.) with PrCHO (e.g., 2 mols.) and sulphurising, e.g., with S or an alkali polysulphide, or (B) by a mixture of a benzthiazyl disulphide and an accelerator containing a substituted NH<sub>2</sub> group, e.g., 2:4-diaminodiphenylamine, or a disubstituted guanidine, e.g., NH:C(NHPh)<sub>2</sub>. D. F. T.

**Vulcanisation of rubber latex or other rubber dispersions.** F. C. VAN HEURN, Assr. to FLINTKOTE CORP. (U.S.P. 1,893,477, 3.1.33. Appl., 31.12.30).—Latex which has been rendered acid-stable, e.g., by addition of AlCl<sub>3</sub>, is treated successively with a thio-sulphate, e.g., Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, and an acid, e.g., HCl. The liberated S, with the aid of an accelerator if desired, effects vulcanisation of the dispersion, e.g., on heating. D. F. T.



[Manufacture of] accelerators for the vulcanisation of rubber. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 391,120, 14.10.31).—A thioxanthate derived from an amine  $\text{NHRR}'$ , in which R and R' are alkyl, aralkyl, or hydroaryl and one may be aryl, or  $\text{RR}' = \text{a polymethylene chain which may be interrupted by hetero-atoms, is condensed with Ar}\cdot\text{S}\cdot\text{X}$ , in which Ar = aryl (preferably  $p\text{-NO}_2\cdot\text{C}_6\text{H}_4\cdot$ ) and X = halogen or SCN. Examples are products from:  $p\text{-NO}_2\cdot\text{C}_6\text{H}_4\cdot\text{SCl}$  and  $\text{NMe}_2\cdot\text{CS}_2\text{K}$  (m.p. 149–150°);  $o\text{-NO}_2\cdot\text{C}_6\text{H}_4\cdot\text{SCl}$  and  $\text{NMe}_2\cdot\text{CS}_2\text{Na}$  (m.p. 160–161°),  $\text{C}_5\text{H}_{10}\text{N}\cdot\text{CS}_2\text{Na}$  (m.p. 145°) or  $\text{C}_6\text{H}_{11}\cdot\text{NEt}\cdot\text{CS}_2\text{Na}$  (m.p. 125°). C. H.

Manufacture and application of accelerators for vulcanisation. I. G. FARBENIND. A.-G. (B.P. 392,046–7, 16.9.32. Ger., [A] 17.9.31, [B] 19.9.31).—The SH group of compounds containing the grouping  $\cdot\text{NH}\cdot\text{C}(\text{SH})\cdot\text{S}$  or  $\cdot\text{N}\cdot\text{CS}_2\text{H}$  is etherified (A) with an aromatic *m*-nitro-ketone, -aldehyde, -amide, or -carboxylic alkyl ester, or (B) with an aromatic *m*-nitro-sulphone, -sulphonamide, or -sulphonic alkyl ester. Suitable S compounds are: 2-thiolbenzthiazole,  $\text{NMe}_2\cdot\text{CS}_2\text{Na}$ ,  $\text{C}_5\text{H}_{10}\text{N}\cdot\text{CS}_2\text{Na}$ , Na dicyclohexyldithiocarbamate. These are etherified by reaction with: (A) 4-chloro-3-nitrobenzaldehyde, 2-chloro-5-nitrobenzoic butylamide, Me 4-chloro-3-nitrobenzoate; (B) 4-chloro-3-nitrobenzenesulphonmethylamide, 4-chloro-3-nitro-diphenylsulphone or -benzenesulphonanilide. The accelerators are used alone or in admixture with others, e.g., with  $\text{NH}\cdot\text{C}(\text{NHPh})_2$ . C. H.

Antioxidant for vulcanised rubber products. J. R. INGRAM, ASSR. to RUBBER SERVICE LABS. CO. (U.S.P. 1,890,154, 6.12.32. Appl., 13.11.30).—Rubber is vulcanised in presence of the reaction product of an  $\text{NH}_2$ -substituted  $\text{CHPh}_2$ , e.g., the *pp*- $(\text{NH}_2)_2$ -derivative (1 mol.), and a OH-derivative of an aromatic hydrocarbon, e.g.,  $\beta\text{-C}_{10}\text{H}_7\cdot\text{OH}$ . The antioxidant products have the constitution  $\text{CHPh}(\text{C}_6\text{H}_4\cdot\text{NHR})_2$ . D. F. T.

(A) Age resister, (B) antioxidant. A. M. CLIFFORD, ASSR. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,894,230–1, 10.1.33. Appl., 14.9.31).—Rubber is vulcanised in presence of (A) a OH-derivative of fluorene or of diphenylene oxide, e.g., 2-hydroxyfluorene or 2:7-dihydroxydiphenylene oxide, or of (B) a primary  $\text{NH}_2$ -derivative of phenanthrene, e.g., 3-aminophenanthrene. D. F. T.

Temp. of moving bodies [rubber sheet].—See I. C black.—See II. Resistance element.—See XI. Preventing formation of lumps.—See XVII.

#### XV.—LEATHER; GLUE.

[Vegetable] tanning materials. Report of a joint Committee of the Society of Leather Trades' Chemists and the I.V.L.I.C. A. GANSSER and W. VOGEL (J. Soc. Leather Trades' Chem., 1933, 17, 462–464).—The determination of the  $p_{\text{H}}$  val. should be included in tannin analysis. Methods should be established for the determination of the liability of tannin extracts to sediment and of the colour of tanned leather. In the analysis of sulphite-cellulose waste liquors, the report should give "materials absorbed by hide powder."

15–17 g. of liquid sulphite-cellulose extract and 5 g. of the solid should be used in preparing 1 litre of infusion for analysis. D. W.

Flavone compounds of woods, their fluorescence and relations with tannins. F. VIGNOLO-LUTATI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1933, 11, 436–448).—A review.

Hides and skins. Report of a Committee of the Society of Leather Trades' Chemists. A. GANSSER (J. Soc. Leather Trades' Chem., 1933, 17, 460–462).—Warble fly damage, the use of unsuitable salt, dirty hides, insect damages, and bad flaying are reported. D. W.

Adsorption and swelling. IV. Influence of temperature on swelling of hide powder. V. KUBELKA and V. SAMEC (Kolloid-Z., 1933, 64, 84–86).—The swelling of hide powder in dil. HCl ( $p_{\text{H}} 5\cdot23\text{--}0\cdot01$ ) at 0–30° has a positive temp. coeff. Swelling at 40° is < at 30°. At all temp. the swelling is greatest at  $p_{\text{H}} 1\cdot85$ . E. S. H.

Physical properties of leather. Report of a joint Committee of the Society of Leather Trades' Chemists and I.V.L.I.C. L. JABLONSKI and C. H. SPIERS (J. Soc. Leather Trades' Chem., 1933, 17, 464–468). D. W.

Corrosion by tan liquors.—See X. Soya-bean protein.—See XIX.

See also A., Aug., 796, Cr tanning.

#### PATENTS.

Dryer [for hides etc.].—See I. Endless bands of gelatin.—See V. Treating leather.—See VI.

#### XVI.—AGRICULTURE.

Volumetric determination of adsorption capacity [of soil]. S. N. ALESHIN and T. A. MALOMAKHOVA (Khim. Sotzial. Zemled., 1932, No. 7, 17–26).—Aleshin's method is modified. The soil is not washed with  $\text{H}_2\text{O}$  after treatment with  $\text{BaCl}_2$ , but is dried for 3 hr. at 105°, treated with 300 c.c. of 0·05N- $\text{H}_2\text{SO}_4$ , shaken for 5 min., filtered, the first portion discarded, and an aliquot part titrated using bromocresol-purple as indicator. CH. ABS.

Thermal analysis applied to soils. V. AGAFONOV and S. PAVLOVITCH (Compt. rend., 1933, 197, 166–168).—A temp.-time curve from about 1000° is determined, using the Le Chatelier-Saladin differential galvanometer, for the powdered soil freed from  $\text{CaCO}_3$  by 10% aq. HCl, and the arrests etc. are compared with similar curves for various minerals likely to occur in the soil and giving characteristic curves, e.g., kaolin, diaspore, hydrargillite, and various forms of hydrated  $\text{Fe}_2\text{O}_3$ . C. A. S.

Determination of the agricultural value of limes and limestones. M. LENGLEN (Ann. Falsif., 1933, 26, 359–361).—Agricultural limes are evaluated by determining free CaO and  $\text{CaCO}_3$ , limestones by determining  $\text{CaCO}_3$  and the grain size. E. C. S.

Manurial action of some potash salts used alone and in admixture. W. V. BEHRENS (Z. Pflanz. Düng., 1933, 12, B, 347–350).—No evidence was obtained



that mixtures of K salts were more effective than the individual constituents. A. G. P.

**Action of various phosphates on moor soils.** W. ZIELSTORFF and K. NEHRING (*Z. Pflanz. Düng.*, 1933, 12, B, 334—347).—In both pot-culture and field trials with acid highmoor soils ( $p_H$  4.6, 5.3) basic slag, superphosphate, and phosphate rock produced similar yields. In less acid lowmoor soils ( $p_H$  5.8) rock phosphate was less effective. The effect of soil reaction on  $PO_4'''$  utilisation was shown by differences in P content of crops even where significant differences in yield were not apparent. In the less acid soils the P intake from basic slag was < that from superphosphate. A. G. P.

**Influence of chemical composition of organic matter on rate of decomposition [in soil].** T. L. MARTIN (*J. Amer. Soc. Agron.*, 1933, 25, 341—346).—Rates of decomp. of ground lucerne (I) and sweet clover (II) in soil are recorded. In (II) crude protein disappears much more rapidly than does cellulose.  $H_2O$ -sol. constituents of roots and tops break down at approx. the same relative rates for material of the same source, but the rates for (I) were > for (II). During decomp. lignin accumulates more rapidly in roots than in tops. A. G. P.

**Relation of elevation to nitrogen content of grassland and forest soils in the Rocky mountains of Colorado.** R. D. HOCKENSMITH and E. TUCKER (*Soil Sci.*, 1933, 36, 41—45).—The total N content of the surface 3 in. of forest soil was > that of similar soil under grass. The vals. for 3—6 and 9—12 in. were, however, greater in grassland. In general the N content of soils rises with elevation, but at > 10,000 ft., fluctuations are considerable. Differences result from variation in vegetation rather than in rates of decomp. of plant material. A. G. P.

**Potash content of east Prussian soils.** Goy (*Ernähr. Pflanze*, 1933, 29, 221—225).—Neubauer tests of a large no. of soils are recorded. K deficiency was more general on small than on large farms and more prevalent on light than on medium or heavy soil types. A. G. P.

**Soluble aluminium [in soils]. III. Relationship of nitrification and sulphur oxidation to the aluminium- and hydrogen-ion concentration of some very acid soils.** G. G. POHLMAN (*Soil Sci.*, 1933, 36, 47—55; cf. B., 1932, 952).—In soils of which the displaced soil solutions had  $p_H$  3.9—4.9, nitrification and S oxidation increased the proportion of sol. Al. These biological processes were not inhibited by  $[Al^{+++}]$  of 27—190 p.p.m. in soil solutions nor by  $p_H$  3.5—3.6. The Ca and  $NO_3'$  contents of the soil solutions were closely correlated, and the process of nitrification is largely controlled by the amount of available Ca present in soil. A. G. P.

**Adsorption capacity of soils.** E. N. IVANOVA (*Trans. Dokuchaiev Soil Inst.*, 1933, 8, No. 8, 23—41).—The results obtained in the determination of the adsorption capacity depend on the dilution of the replacing ion, the abs. amount of the ion being const. The presence of  $Ca^{++}$  depressed the amount of Na adsorbed from mixtures of  $CaCl_2$  and NaCl, the depression effect increasing with increasing  $[Ca^{++}]$ . With

Na/Ca const. the ratio of Na adsorbed from the mixture to that adsorbed from a pure NaCl solution of the same  $[Na^+]$  is a const. for all concns.  $CaSO_4$  acts similarly to  $CaCl_2$ , whereas  $CaCO_3$  hinders but little the adsorption of  $Na^+$ . A. M.

**Reversal of soil charge by acids and adsorption of anions.** I. N. ANTIPOV-KARATAIEV and A. I. RABINERSON (*Proc. Leningrad Dept. Inst. Fert.*, 1933, 17, 101—133).—The ease with which the electrical charge was reversed by acids ( $HCl$ ,  $HNO_3$ ,  $H_2SO_4$ ,  $H_3PO_4$ ,  $AcOH$ ,  $H_2C_2O_4$ ) was, in descending order, Chakva krasnozern, podsol, yellow podsol (Sochi), red clay (Crimea). Neither solonetz nor chernozem had their charges reversed. Methylene-blue reversed the charges in all cases. There was no strong correlation between  $SiO_2/R_2O_3$  ratio of the clay (< 1  $\mu$ ) and the ease of charge reversal, but there is between the latter and the content of mobile  $R_2O_3$  (Tamm's oxalate method and 5% KOH method of Gedroiz). The reversal of charge depends on the adsorption of anions, and the amount adsorbed in an acid medium depends on the amount of mobile  $R_2O_3$  and the solubility of the salt-like compounds formed with the anions. A. M.

**Colorimetric determination of hydrogen-ion concentration of soils.** H. W. KERR and N. J. KING (*Proc. IV Cong. Int. Soc. Sugar Cane Tech.*, 1932, Bull. No. 110).—The  $p_H$  of a suspension in  $N-KCl$  is of greater use than that of an aq. suspension. Vals. for Queensland sugar soils are 3.8—5.5. At  $p_H$  < 3.8 liming is not economical. CH. ABS.

**Oxygen absorption in soils.** F. B. SMITH and P. E. BROWN (*Iowa State Coll. J. Sci.*, 1933, 7, 153—162).—Preliminary aeration decreased the initial rate of absorption of  $O_2$  by Carrington and Tama silt loams. The presence of glucose greatly increased it. An unidentified gas, not  $CO_2$ , appeared to be evolved from Tama silt loam. CH. ABS.

**Action of lime, superphosphate, and silicic acid on soil reaction in pot experiments.** E. RAUTERBERG (*Z. Pflanz. Düng.*, 1933, 30, A, 324—331).—Increases in the  $p_H$  of soils following treatment with superphosphate (I) in pot trials are attributed to the preferential intake of  $PO_4'''$  by the plants. The acidifying action of  $SiO_2$  applied in conjunction with (I) is due to fixation of the  $Ca^{++}$ . Crop increases thus obtained result from the greater solubility of  $PO_4'''$ . A. G. P.

**Importance of time of sowing in pot cultures of oats, especially in the determination of  $b$  [Mitscherlich].** K. OPITZ and K. RATHSACK (*Z. Pflanz. Düng.*, 1933, 30, A, 249—268).—In Mitscherlich cultures the yield of oats in manured and unmanured soils varied with the time of sowing, and calc. vals. of  $b$  vary accordingly. A. G. P.

**Determination of the nutrient condition of soils by means of the solubility of nutrient compounds in boiling water.** G. ROHDE (*Z. Pflanz. Düng.*, 1933, 30, A, 331—344).—The soil (25 g.) is heated for 30 min. with 2 litres of  $H_2O$  under a reflux condenser. A Chamberland filter is immersed in the boiling mixture and sufficient filtered extract is removed by means of a filter-pump. The  $PO_4'''$  and K content of the extract



is directly proportional (12.5%) to the Neubauer vals. for the soil. The utilisation of vals. so obtained for determining the fertiliser requirement of soils is discussed.

A. G. P.

**Adsorption of potassium by plants in relation to replaceable, non-replaceable, and soil-solution potassium.** D. R. HOAGLAND and J. C. MARTIN (Soil Sci., 1933, 36, 1—32).—In soils containing much replaceable K (I), all or nearly all of the K removed by crops is accounted for by loss of (I). Under such conditions "luxury" consumption by the crop is favoured. With continued cropping plants utilise increasing proportions of non-(I) until no further removal of (I) occurs and the solubility of the non-(I) determines the entire supply to plants. In certain soils this is adequate for normal growth. The [K] in the soil solution usually represents only a small proportion of the total K intake by the crop, but is of similar magnitude to that of normal culture solutions. Among soils of different type no relationship exists between the [K] of the soil solution and the intake by the crop, although in any one soil a consistent proportion was observed. The supplying power of a soil for K is reflected in the composition of the crop. Growth is not limited by sp. ratios of bases. Some soils rapidly fix added K of fertilisers in a non-(I) but in others added K was completely recoverable by leaching with  $\text{NH}_4\text{OAc}$ .

A. G. P.

**Relations of *Bacillus mycoides* with ammonification, nitrification, and soil fertility.** M. TYAGNY-RYADNO (J. Agric. Sci., 1933, 23, 335—358).—In soil inoculated with *B. mycoides* (I), there is rapid decomp. of org. N. materials with abundant formation of  $\text{NH}_3$  and increased nitrification (II). Initially ammonification (III) and (II) are similarly accelerated, and whereas (III) declines at a later stage (II) continues for a considerable period. In chernozem soils the activity of (I) controls the rate of (II). Farmyard manure is an important source of (I). Rock phosphate stimulates (II) and (III) organisms in soils, and in turn soil phosphate is rendered more available by the acids thus produced. Joint applications of rock phosphate and dung are recommended for Russian soils.

A. G. P.

**Composition of soya-bean plants at various growth stages as related to their rate of decomposition and use as a green manure.** L. M. TURK (Missouri Agric. Exp. Sta. Res. Bull., 1932, No. 173, 40 pp.).—With increasing age of the plants there was a steady decline in the % of total N (< 1.7% N in roots after early stages) and of  $\text{H}_2\text{O}$ -sol. constituents in both roots and tops. The alkali-sol. matter (slowly decomposable) increased rapidly toward maturity, and the N content of this fraction increased, especially that of the alkali-sol. lignin. Carbohydrate material including reducing sugars, cellulose, and pentosans (all decomposable by soil organisms in presence of sol. N) also increased towards maturity, and was accompanied by a widening of the C:N ratio. The % of total lignin increased enormously during the season's growth. The narrow pentosan:lignin ratio indicates slow decomp. especially of the roots. In general the rate of decomp. of the various plant parts decreased with advancing age, and was usually in the order tops > complete plants >

roots. The cold- $\text{H}_2\text{O}$ -sol. fraction was chiefly responsible for the immediate and rapid evolution of  $\text{CO}_2$  during decomp. in soil. During a 30-day decomp. period there was a marked narrowing of the C:N ratio of the material. In soil incorporated with soya-bean tops there was a greater proportion of alkali-sol. humus than where roots had been used. Accumulation of  $\text{NH}_3$  and  $\text{NO}_3'$  in soil after admixture with the plant material decreased with advancing age of the plants. With entire plants there was a positive accumulation of  $\text{NO}_3'$ , but with roots there was a decrease in  $\text{NO}_3'$  formation.

A. G. P.

**Increase of yield by chemical treatment of sugar-beet seed.** P. A. VLASSUK (Nauk. Zapiski, 1932, 25, 1—25).—Beet seed was soaked for 24 hr. in solutions of various concn. of various salts, germinated in pure sand, and planted out in a slightly alkaline black soil. In general it was found that solutions of uni- and ter-valent metal salts retarded the germination in comparison with the same seed soaked only in distilled  $\text{H}_2\text{O}$ . On the other hand, treatment with bivalent metal salts distinctly improved germination. In the field experiments, the most marked influences were shown by Mn, Mg, and Cu.

J. P. O.

**Productivity of sugar beets as influenced by spacing and inbreeding.** C. BONNE (Z. Ver. deut. Zucker-Ind., 1933, 83, 357—375).—In field trials from 1923 to 1928, spacings ranging from 1312 to 2000 sq. cm. per plant yielded roots of average wt. ranging from 529 to 715 g. and average sugar content ranging from 19.9 to 19.4%, representing wts. of beets and sugar per acre ranging from 16.1 to 14.2 and from 3.2 to 2.75 tons respectively. The closest spacing therefore gave the highest yields of roots and sugar per acre. Continued propagation of plants from the same stock for many years, whether with free fertilisation or in isolation, revealed no change in the variability of individual roots in wt. or sugar content. In isolated propagation the average wt. per root increased in course of years, and in some cases a no. of roots with red skin were produced.

J. H. L.

**[Sugar] filter-press cake on Louisiana plantations.** R. D. RANDS (Sugar Bull., 1933, 11, No. 10, 2—5).—Application of filter-press cake has been found from investigations over a no. of years to assist the growth of cane retarded in its early development by root troubles. Often the press cake contains as much as 40 lb. of N per ton, equiv. to 200 lb. of  $(\text{NH}_4)_2\text{SO}_4$ , as well as considerable amounts of  $\text{PO}_4'''$ . It should not be applied at a greater rate than about 2000 lb. per acre. As 1 ton of cane usually produces about 40—50 lb. of cake where the yield is 20 tons per acre, there will be enough to give normal treatment to a large proportion of the stubble lands.

J. P. O.

**Grasslands of Australia and some of their problems.** W. DAVIES (Counc. Sci. Ind. Res., Australia, 1933, Pamphlet No. 39, 36 pp.).—A report of a survey including the management and utilisation of pastures and of the principal botanical species concerned.

A. G. P.

**Nutritive value of lucerne. I. Yield, composition, and nutrient value (season 1932).** H. E.



WOODMAN, R. E. EVANS, and D. B. NORMAN (J. Agric. Sci., 1933, 23, 419—458).—With advancing age the protein (I) content of lucerne declined and the proportion of fibre increased. At the flowering stage, plants were as fibrous as grass at the haying stage and the (I) content was < that of grass herbage cut at 5-week intervals. In all stages of growth the proportion of "amides" in the crude (I) of lucerne was > and the Et<sub>2</sub>O extract and N-free extractives were < that of the pasture cuts. Lucerne also contained a higher proportion of SiO<sub>2</sub>-free ash especially rich in Ca. The % of the latter increased with age up to the flowering stage; that of P declined from budding to flowering. The K content decreased steadily with growth. The % of org. S and of cystine was similar to that of pasture cuts, but the latter had higher proportions of SO<sub>4</sub>". The digestibility and nutrient val. of lucerne in bud or flower were notably < in grass cut at 1—5-week intervals, the difference being specially marked in the case of fibre. Growth of lucerne in soils of varied type indicated that the mineral but not the org. composition may be influenced to a considerable extent by soil conditions and manurial treatment. A. G. P.

**Evaluation of applications of lime-sulphur for the control of apple scab.** W. O. GLOYER (New York [Geneva] Agric. Exp. Sta. Bull., 1933, No. 624, 39 pp.).—Data showing the effect of varied timing of applications of CaO-S on its efficiency are considered from the viewpoint of the dual purpose, eradivative and protective, of the fungicide. The bearing of this on modifications of spray programmes to meet varied environmental conditions is examined. A. G. P.

**Potato scab-gnat (*Pnyxia scabiei*, Hopkins).** H. L. GUI (Ohio Agric. Exp. Sta. Bull., 1933, No. 524, 21 pp.).—Seed treatment with hot aq. CH<sub>2</sub>O destroys all insects. Damage to potato crops is less in soils of high org. matter content or where straw mulching has been practised. The insect is less active in soils having p<sub>H</sub> < 5.0. A. G. P.

**Control of *Alternaria* blight of ginseng with Bordeaux mixture and injuries accompanying its use.** H. A. RUNNELS and J. D. WILSON (Ohio Agric. Exp. Sta. Bull., 1933, No. 522, 15 pp.).—Best results were obtained by the use of a 3:4½:50 Bordeaux mixture containing 1 lb. of K fish-oil soap and 1 lb. of Ca arsenate per 50 gals. Leaf injury occurred in frosty or drought conditions. A. G. P.

**Zones of stability of different copper compounds in Burgundy mixture as function of p<sub>H</sub>.** L. MAUME and A. BOUAT (Compt. rend., 1933, 196, 2024—2026).—Varying amounts of aq. Na<sub>2</sub>CO<sub>3</sub> were added to aq. CuSO<sub>4</sub> and the composition of the ppts. was determined. Between p<sub>H</sub> 4.7 and 5.1 CuSO<sub>4</sub>.nCuO (n = 2—9) is pptd. This is accompanied by the hydrated basic carbonate CuCO<sub>3</sub>.CuO.2H<sub>2</sub>O (I) above p<sub>H</sub> 5.1. At p<sub>H</sub> 6 the pptd. Cu reaches an approx. const. level of 94% of total Cu, but with a large excess of Na<sub>2</sub>CO<sub>3</sub>, Cu is completely pptd. as Cu(OH)<sub>2</sub> + (I). A. C.

**Superphosphate.**—See VII. **Phosphate slags.**—See X. **Sugar-cane juice.**—See XVII.

See also A., Aug., 774, Absorption of AsO<sub>3</sub>" by soils. 803, Soil profiles. 873, "Maturity" of

sugar cane. 874, Nutrient intake of submerged plants. Response of plants to H<sub>2</sub>O deprivation. Ash of the Wagener apple. P compounds of plants. Effect of p<sub>H</sub> on K and P absorption by wheat plants. 875, Determining As in plant materials, and of K in plant tissues. Determining uronic acids and OMe in plants etc. 878, N metabolism of alkaloidal plants.

## PATENTS.

**Fertiliser.** R. GRIESSBACH and H. WEISS, ASSTS. to OHIO SANITARY ENG. CORP. (U.S.P. 1,889,654, 29.11.32. Appl., 20.12.28. Ger., 14.1.28).—Solid SiO<sub>2</sub> is heated with 1½—8 times its wt. of 60% H<sub>3</sub>PO<sub>4</sub> to a temp. > 250°, and, after cooling, the mixture is treated with NH<sub>3</sub> gas. The fertiliser will slowly decompose in the soil into sol. H<sub>3</sub>PO<sub>4</sub> and active SiO<sub>2</sub>. B. M. V.

**Fertiliser.** J. T. TRAVERS and O. M. URBAIN, ASSTS. to OHIO SANITARY ENG. CORP. (U.S.P. 1,889,712, 29.11.32. Appl., 25.8.28).—Phosphate rock, SiO<sub>2</sub>, and NaCO<sub>3</sub> are fused and leached with HCl in order to form colloidal SiO<sub>2</sub>.P<sub>2</sub>O<sub>5</sub>, to which are added N and K compounds. B. M. V.

**Manufacture of fertilisers.** H. L. HARTENSTEIN (U.S.P. 1,888,921—2, 22.11.32. Appl., [A] 18.10.26, [B] 4.4.27).—(A) Muck and peat in their natural state are digested with finely-divided Fe and S oxide gases; after digestion calcareous matter may be added and air blown through. In (B) the digestive reagents are NaNO<sub>3</sub>, CaSO<sub>4</sub>, phosphate rock, Al(NO<sub>3</sub>)<sub>3</sub>, and S oxide gases; a potash fertiliser may be added and the cooked material aged for < 10 days. B. M. V.

**Manufacture of mixed fertilisers containing ammonium nitrate.** DIRECTIE VAN DE STAATSMIJNEN IN LIMBURG (B.P. 394,447, 18.3.32. Holl., 1.4.31).—HNO<sub>3</sub> mixed with < 1 other acid is atomised into a reaction chamber (I) into which NH<sub>3</sub> is passed in excess of that required to neutralise the acids. The H<sub>2</sub>O content of the acid mixture is so regulated that the salts formed collect as a slurry with 5—10% H<sub>2</sub>O, heat being supplied or withdrawn as required to obtain such a product as will crystallise on cooling after withdrawal from (I). A. R. P.

**Insecticide.** F. B. McLANE (U.S.P. 1,888,997, 29.11.32. Appl., 15.3.30).—The conc. spray may consist of nicotine sulphate 7, sugar or other carbohydrate retarding agent 3, and H<sub>2</sub>O 4 pts. B. M. V.

(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.—See VII.

## XVII.—SUGARS; STARCHES; GUMS.

**Influence of basic lead acetate on the marc volume in [the determination of sugar in beets by hot aqueous] digestion.** O. SPENGLER and W. PAAR (Z. Ver. deut. Zucker-Ind., 1933, 83, 343—356).—Marc from 26 g. of beets, washed free from sugar, was digested with solutions of 2.6 or 5.2 g. of sucrose at 75—80° for 30 min. in absence and in presence of basic Pb acetate (I) and, after cooling, the liquids were made up to 100 c.c., filtered, and polarised. It is calc. that the effective vol. of the marc was 1.5—2.6 c.c. in absence and 0.7—1.1 c.c. in presence of (I). In the former case part of the marc vol. is attributed to colloid H<sub>2</sub>O



which does not dissolve any sugar; in presence of (I) most of this  $H_2O$  is withdrawn and the marc shrinks. From these and similar experiments with beet juice the average val. calc. for the vol. of the marc from 26 g. of beets, under the usual conditions for analysis of beets by hot aq. digestion, was 0.83 c.c., agreeing well with the vals. found by Pellet and Saillard. J. H. L.

**Colorimetric determination of pectin substances.** P. M. SILIN and Z. A. SILINA (Z. Ver. deut. Zucker-Ind., 1933, 83, 390—397).—A micro-method suitable for determining pectins in beet juices etc. 5 or 10 c.c. of the liquid, containing 5—10 mg. of pentosans or < 15 mg. of pectin, is treated with half its vol. of conc. HCl and sufficient HCl of  $d$  1.06 to make up to 40 c.c., and distilled from a 100-c.c. flask at such a rate that 30 c.c. of distillate are obtained in 3 hr. The distillate is neutralised with 10% aq. NaOH and made up to 100 c.c. Its furfuraldehyde (I) content is determined by mixing 5 c.c. with 4 c.c. of glacial AcOH free from (I) and 0.5 c.c. of freshly distilled  $NH_3$ , setting aside for 10 min. in the dark, and then matching the red colour in a Duboscq colorimeter against that of 5 c.c. of a freshly prepared standard (I) solution similarly treated. A 0.1% solution of freshly distilled (I) will remain unchanged for 2 months and can be diluted as required. One part of (I) in the distillate corresponds to 1.894 pts. of araban, 1.732 pts. of xylose, 1.813 pts. of pentosan, 3.8 pts. of the pectins of raw beet juice, or 5.5 pts. of the pectins of purified juice or molasses. A correction must be made for sucrose, 1 g. of which yields 1.25 mg. of (I) under the conditions stated. J. H. L.

**Determination of sucrose and raffinose in beet cossettes by the enzyme method.** H. S. PAINE and R. T. BALCH (Int. Sugar J., 1933, 35, 238—239).—The method consists in hydrolysing with the enzymes contained in top and bottom yeasts, using a de-leaded solution of the product under examination, the sucrose and raffinose being calc. from the formulæ  $S = (P - 2.202A + 1.202B)/131.26$ , and  $R = 1.354(A - B)$ , in which  $A$  and  $B$  are the corr. polarisations after hydrolysis by top and bottom yeast respectively. The rotation of the non-sugars is variable, being dextro in Idaho and negative in Utah, Colorado, and Nebraska. When  $P - S$  is > normal (0.2 to 0.3), then the unaccountable loss of sugar in manufacture is also greater. J. P. O.

**Darkening of [beet] juice due to presence of small amounts of first saturation scums at the second saturation, owing to defective filtration.** O. SPENGLER and S. BÖTTGER (Z. Ver. deut. Zucker-Ind., 1933, 83, 331—341).—The presence of first carbonatation ppt. at the second carbonatation causes a reddish-brown discoloration of the final juice, similar to that which results if the first carbonatation is carried too far. As even 0.01% of this ppt. produces measurable darkening, refiltration of first carbonatation juice is recommended as a regular practice. J. H. L.

**Pre-defecation experiments.** F. SOLDNER (Deut. Zuckerind., 1933, 58, 164—166).—In liming raw beet juice, the best results as regards rate of filtration and colour of juice were obtained by the optimum pre-defecation method of the Institute of Sugar Industry,

Berlin, in which the cold juice was first limed with 0.3%, rapidly heated to 80°, and again limed to make a total of 2% of the roots. Another useful variant was pre-defecating with 0.3% CaO and following with 25% of cold unfiltered juice and 20% of hot unfiltered juice, with a final addition of enough CaO to make 2% CaO on the roots. J. P. O.

**Differentiation between beet-refinery and beet-factory molasses.** J. ČERMÁK (Z. Zuckerind. Czechoslov., 1933, 57, 341—347).—100 c.c. of a 5% solution of the molasses are treated with 0.2 g. of "Carboraffin," some kieselguhr is added, the solution filtered, and the colour before and after the operation read by the Stammer colorimeter, the proportion of the latter to the former being the "adsorption coeff." This for factory molasses was found to be 0.531—0.862 and for refinery molasses, 0.482—0.582. J. P. O.

**Nitrogen in condensate and molasses.** E. SAILLARD (Suppl. Circ. hebdom., 1933, Nos. 2293 and 2294).—Samples of condensate from the evaporators of a large no. of French sugar factories contained an average of 100 mg. N per litre, corresponding to 15 g. per 100 kg. of roots. As a rule the N content increases from the first to the last effect of the evaporator, but in some factories this order appears to be reversed. In French beet molasses the average N content was 1.8% for 1932 with a max. of about 2.23%. J. P. O.

**Conductometric determination of ash in molasses.** E. SAILLARD and R. SAUNIER (Compt. rend., 1933, 196, 2026—2028).—The product of the electrical resistance and the ash content (I) of dil. solutions of molasses is approx. const. if (I) is between 0.05 and 0.1%. This has been applied to the conductometric determination of sulphated ash in a large no. of samples of molasses, suitably diluted, with a max. error of 0.25%. A. C.

**Effect of fertiliser on composition of [cane] juice.** L. A. HURST and M. McKAIG (Sugar Bull. 1933, 11, 6—7).—Application of N as fertiliser increased the N content of the cane juice to an appreciable degree and also decreased the ash content of the juice, compared with juice from unfertilised cane. K or P fertilisers increase the ash, but the data failed to establish that application of P fertiliser increases the  $P_2O_5$  content of the juice. J. P. O.

**Relation between surface tension, colloid content, colour, and alkalinity of sugar-factory products.** L. E. DAWSON, J. C. KEANE, and H. S. PAINE (Int. Sugar J., 1933, 35, 236—237).—Surface tension was found to be highest in white sugar, next in diffusion juice, and lowest in carbonatation juice; high green has the lowest val. There was good agreement between the colloidal matter by the dye test and the non-sugars based on apparent purities, the correlation becoming closer as the products approach raw massecuite in the factory process, indicating actual production of colloidal matter in white and raw massecuites. Colour and dye vals. correlated well, excepting in the case of thick-juice. Titrated alkalinity and  $p_H$ , as well as CaO by the soap test, correlated with some of the other properties in the case of certain individual products. J. P. O.



**First saturation, using hydrochloric acid.** J. DĚNEK and J. KMNÍČEK (Z. Zuckerind. Czechoslov., 1933, 57, 337—339).—In laboratory experiments on the neutralisation of limed beet juice with HCl the amount of colour remaining was about the same as after normal carbonation; it was concluded that in the clarification operation the colouring matters were mainly removed by the action of the CaO (*i.e.*, the defecating effect) rather than by the CaCO<sub>3</sub> ppt. J. P. O.

**Determination of saturation temperature of sucrose solutions.** R. W. HARMAN (Int. Sugar J., 1933, 35, 261—263).—At the saturation temp. (*ts*), *i.e.*, the temp. at which a syrup is just saturated, the sucrose per 100 of H<sub>2</sub>O in the syrup is the solubility in pure H<sub>2</sub>O at this temp. (*H<sub>ts</sub>*) multiplied by the saturation coeff. (*h*) at this temp. (*h<sub>ts</sub>*); or  $100S/W = h_{ts}H_{ts}$ . Hence the saturation coeff. =  $h_{ts}H_{ts}/h_tH_t$ ; and on the assumption that *h* does not vary with temp., this expression simplifies to  $H_{ts}/H_t$ . Apparatus is described for the determination of *ts*, depending on the heating of a supersaturated solution containing sucrose crystals, which dissolve as soon as *ts* is passed; this can be seen by observing the crystals under a microscope. J. P. O.

**Inversion of sucrose by active carbons.** V. NETUKA (Z. Zuckerind. Czechoslov., 1933, 57, 349—351).—50 c.c. of a 60% solution of sucrose are heated with 0.5 g. of the C in an autoclave for 2 hr. in a boiling H<sub>2</sub>O-bath, and the amount of invert sugar formed is compared with that found in a control test in absence of C. Neutral carbons do not cause appreciable inversion in this way, *i.e.*, < 100 mg. per 100 g. of sucrose; others (as the "Carboraffin" of former years) may do so, and will require neutralisation. J. P. O.

**Automatic second carbonation.** E. SCHMIDT (Deut. Zuckerind., 1933, 58, 285—286).—Juice from the last filter-presses is heated to 104°, and allowed to flow boiling hot into the second carbonators, steam being automatically injected into the juice line from the heaters whenever the temp. falls below 103°, and the CO<sub>2</sub> warmed by a continual injection of steam into the gas line. If the alkalinity of the finished juice at 100° tends to become too low, a little Na<sub>2</sub>CO<sub>3</sub> is injected; otherwise this second carbonation operation requires no control. J. P. O.

**Coloration of sugar solutions on heating and during evaporation.** O. REISS (Z. Zuckerind. Czechoslov., 1933, 57, 361—363).—Sugar solutions of different concn. and containing various non-sugar substances (*e.g.*, invert sugar and aspartic acid) were (*a*) heated in an autoclave for 1 hr at 105°, and (*b*) concn. from 20° to 60° Brix in vac., so as to imitate as closely as possible the conditions of these operations obtaining in practice. The amount of coloration taking place was found to be about the same in both operations. J. P. O.

**Denaturation of raw sugar for fodder.** K. ŠANDERA (Z. Zuckerind. Czechoslov., 1933, 57, 363—364).—Exhausted active C or finely ground bone black is recommended for use as a denaturing agent for raw sugar. J. P. O.

**Crystallisation of sugar.** K. ŠANDERA (Z. Zuckerind. Czechoslov., 1933, 57, 333—337).—Concn. solutions of sucrose and a no. of non-sugar substances were poured

on glass plates at 20° (in air of R.H. 60%). Most of the mixtures in the proportions examined crystallised, certain of them requiring some months at suitable R.H. to do so. On allowing pure sugar solutions of 86, 80, and 78 Bg. to crystallise between glass plates, the transition from conglomerates and meal to well-developed crystals with the full no. of faces could be observed; by adjusting the effects of the supersaturation and rate of cooling the sucrose could be made to crystallise in acicular form. J. P. O.

**Nozzle tube for covering granulated sugar crystals with steam in the centrifugals.** H. KUPSKÝ (Z. Zuckerind. Czechoslov., 1933, 57, 332). J. P. O.

**Saccharification of wood by means of dilute sulphuric acid.** L. V. KOTOVSKI (J. Appl. Chem. Russ., 1933, 6, 519—528).—Up to 24% yields of sugars are obtained by hydrolysis with 2—2.5% H<sub>2</sub>SO<sub>4</sub> at 2 atm. pressure during the first 2 hr.; increase in concn. of acid or duration of hydrolysis does not give higher yields. With 1—3% acid the yield increases with the pressure up to 4 atm., above which it falls rapidly. These results are ascribed to complete hydrolysis during the first 2 hr. of an unstable constituent of wood, to yield sugars unstable to acids, whilst further saccharification is due to hydrolysis of a more stable constituent, with the production of acid-stable sugars. 30—35% yields are obtained by mild hydrolysis of the untreated wood, followed by more intense hydrolysis of the residue. About 5% of the sugars formed undergo decomp. during neutralisation of the acid solution by means of CaCO<sub>3</sub>, and considerably more when NaOH or Na<sub>2</sub>CO<sub>3</sub> is used. The residue after hydrolysis is capable of adsorbing up to 16% of the sugars formed. R. T.

**Hydration capacity of starch.** R. A. GORTNER (Cereal Chem., 1933, 10, 298—312).—The KUNITZ formula (A., 1926, 1005) has been applied to the viscosity of starch sols. Starches from various plants differ greatly in hydration capacity (I). Starches from different wheat varieties differ in (I) but not so much as their relative viscosity vals. would indicate. (I) for wheat starch (vol. occupied by 1 g. of heat-gelatinised starch) is the same at 90° as at 25°. The continued heating of gelatinised starch pastes produces a rapid decrease in the hydration; this change is much more rapid in potato- than in wheat-starch sols. The vols. occupied by cold-gelatinised and heat-gelatinised wheat starch are respectively 28—35 c.c. and 12.5—17.5 c.c. The cold-gelatinisation of starch appears to involve identical reactions irrespective of the chemical (II) (*e.g.*, NaOH, NaCNS, KCNS, Na salicylate, or urea) used, but max. (I) occurs at different concns. of (II). At least 3 reactions are involved in the cold-gelatinisation of starch: (1) at low concn. no hydration takes place; (2) as the concn. of (II) is increased a rapid swelling of the intact granules occurs, resulting in a rupture of the granules; (3) the individual micelles released from the starch granules continue to increase in vol. (peptisation) through an increasing concn. of the gelatinising (II); (4) finally, as more (II) is added, the concn. of (II) in the dispersion medium becomes so great that a slow osmotic dehydration of the hydrated nucleus takes place.

E. A. F.



**Evaporation. Viscosimeter.**—See I. Beet seed. Sugar beets. Filter-press cake.—See XVI.

See also A., Aug., 787, Velocity of inversion of sucrose. 801, Pore viscosimeter and stalagmometer. 844, Determination of sugars. 873, "Maturity" of sugar cane. 875, Raffinose in cereals. Sol. carbohydrates of unripe pumpkin.

## PATENTS.

**Recovery of sucrose from cane molasses.** A. L. HOLVEN (U.S.P. 1,878,144—5, 20.9.32. Appl., [A] 6.2.28, [B] 21.8.29).—(A) The invert sugar (*A*) present is decomposed by treating the diluted molasses, at or near its b.p., with  $\text{Ba}(\text{OH})_2$  or  $\text{BaO}$ , added during 20—40 min. to keep the alkalinity too low for pptn. of  $\text{Ba}$  saccharate. After removing  $\text{Ba}$  salts of org. acids formed from *A*, the liquid is conc. to syrup and added hot to a hot conc. solution or suspension of  $\text{Ba}(\text{OH})_2$  or  $\text{BaO}$  to ppt. the sucrose (*B*). The ppt. is decomposed by  $\text{CO}_2$  and after filtration the filtrate is preferably treated with  $\text{Al}_2(\text{SO}_4)_3$  to ppt. final traces of  $\text{Ba}$ . (B) *A* is decomposed as in (A), between the limits  $p_{\text{H}}$  10—11. Without filtration the liquid is then treated at  $85^\circ$  with further aq.  $\text{Ba}(\text{OH})_2$  to ppt. *B*. The filtered and washed ppt. is decomposed by carbonation to  $p_{\text{H}}$  9.5—10.5, and after filtration the filtrate is sulphited to  $p_{\text{H}}$  6.5—7, again filtered, and worked up for *B*. Suitable mother-liquors are used to prepare the  $\text{Ba}(\text{OH})_2$  solutions and to thin the saccharate paste before filtration. J. H. L.

**Manufacture of dextrose.** C. EBERT, W. B. NEWKIRK, and M. MOSKOWITZ, Assrs. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,876,883, 13.9.32. Appl., 1.4.27).—In the acid conversion of starch (*A*), formation of condensation products from the glucose (*B*) during the later stages may be lessened by interrupting the conversion at an intermediate stage and removing impurities which catalyse the condensation. *E.g.*, when about 40% of *B* has been formed from *A* the liquid is partly neutralised, to  $p_{\text{H}}$  4.5—4.8, filtered from pptd. matter and preferably through bone char (*C*), then further neutralised to  $p_{\text{H}}$  5.5—6.5, conc. to *d* 1.2—1.26, and again filtered through *C*. After suitable dilution and re-acidification to the original acidity, the conversion is completed. The *B* in the final liquor may amount to 96—98% of the dry substance. J. H. L.

**Preparation of dry non-hygroscopic crude lactose.** R. M. WASHBURN, Assr. to DEE-HY PRODUCTS CO. (U.S.P. 1,870,270, 9.8.32. Appl., 12.1.29).—Whey from which the casein has been separated is neutralised to an acidity of 0.05—0.1%, heated to boiling, separated from the pptd. albumin, and spray-dried. The product cakes on exposure to air, but when it absorbs no more moisture it is re-powdered and will remain powdery. J. H. L.

**Sugar refining.** C. A. SPRECKELS (U.S.P. 1,862,719, 14.6.32. Appl., 26.3.28).—Raw cane sugar is mixed with suitable syrup, and warm air is forced through the mixture to keep it well agitated and evaporate part of its moisture, so that the crystals grow (cf. U.S.P. 1,844,020; B., 1932, 1101). Further syrup is added as required, and when the crystals have attained the desired size the magma is centrifuged without addition

of any or much  $\text{H}_2\text{O}$  in the centrifugals, yielding an improved, large-grained, affined sugar (*A*) and practically exhausted molasses. The *A* is re-melted, and after char filtration the resulting syrup may be conc. and crystallised by the same aëration process, suitable apparatus for which is described. The low temp. employed minimises decomp. of sugar and caramelisation, so that the mother-syrups from the refined sugar can be re-conc. repeatedly without much discoloration. J. H. L.

**Manufacture of thin-boiling starch.** C. BERGQUIST, Assr. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,871,027, 9.8.32. Appl., 6.8.28).—Dried starch containing 1.5—2% of  $\text{H}_2\text{O}$  is treated with 0.02—0.06% of dry  $\text{Cl}_2$  in an oil- or steam-jacketed mixing vessel at  $105$ — $120^\circ$  until the desired modification is attained, usually in 4—5 hr. J. H. L.

**Recovery of chicle and similar gum from filter-press residue.** M. A. GORDON, Assr. to AMER. CHICLE CO. (U.S.P. 1,877,299, 13.9.32. Appl., 16.10.31).—To recover gum chicle retained by the filtering media, *e.g.*, sawdust, used in its purification, the material is extracted, in apparatus described, with melted carnauba wax, stearin, or similar materials which, being themselves suitable ingredients of chewing gums, do not need to be separated from the extracted chicle. J. H. L.

**Manufacture of acetylated carbohydrate gum.** A. B. DAVIS, Assr. to A. B. DICK CO. (U.S.P. 1,861,209, 31.5.32. Appl., 21.12.26).—By acetylation of powdered gum tragacanth, *e.g.*, with  $\text{Ac}_2\text{O}$  and  $\text{AcOH}$  in presence of  $\text{H}_2\text{SO}_4$ , a horny product is obtained suitable for the manufacture of stencil sheets. J. H. L.

**Preventing the formation of lumps during the swelling of dissolution of organic powders.** HENKEL & CO., G.M.B.H. (B.P. 392,342, 7.4.32. Ger., 26.8.31).—The powdery material is agglomerated into coarser, porous particles of a suitable size, *e.g.*, by moistening and subsequent drying, by admixture of a hygroscopic substance or by introducing the powder into a hot org. liquid in which it is only slightly sol. The moistening liquid may be a pure solvent or contain adhesive or wetting agents or substances which reduce the rate of dissolution of the powder. The process is applicable to powdered gums, soaps, starch products, rubber, etc. J. H. L.

Carbohydrates.—See III.

## XVIII.—FERMENTATION INDUSTRIES.

**Determination of foam [in beer].** E. HELM (Woch. Brau., 1933, 50, 241—243).—The foam is produced by free falling of the beer in a standard apparatus, and the following measurements are made: the beer drawn off from the foam after 2 min. (*a*), the beer produced from the foam after a further 8 min. (*b*), and the residual foam, condensed by addition of a little  $\text{EtOH}$ , as beer (*c*). The foaming ability of the beer is given by  $100(b+c)/(a+b+c)$  and the foam stability by  $100c/(a+b+c)$  as % of the total beer. R. H. H.

**Decolorising charcoal with selective adsorbent qualities for wines.** J. MILBAUER (Chem.-Ztg., 1933, 57, 581—582, 602—604).— $\text{CaCl}_2$  proposed as activator



in place of  $ZnCl_2$  or mixed therewith is of no special val. and acts merely as a diluent. Experiments on the decolorisation of red wine by activated C prepared with  $ZnCl_2$  showed that many wines contained so little yellow colouring matter that the final colour was unsatisfactory and an acid taste was produced. Tests with a suitable wine are made quant. by determining the wt. of C required per 100 c.c. of wine to give the desired colour. Tests with mixtures of dyes showed that prolonged heating of the C improves its selectiveness. A too active C is not desirable. A photographic method of comparing the decolorising power of active carbons is described. C. I.

**Anomalies of the Halphen ratio in white wines of Huelva province.** I. P. PALACIOS and F. GONZÁLEZ NÚÑEZ (Anal. Fís. Quím., 1933, 31, 139—143).—The relation [% non-volatile acid (as  $H_2SO_4$ ) + 0.7] ÷ [% EtOH by vol.] in white Andalusian wines (82 analyses) may differ by as much as  $\pm 0.075$  from the normal val. (Possete and Issoglio, Giorn. Farm. Chim., 1914, 63, 149, 190, 241) or even more in a wet year. The results of Blarez (Ann. Falsif., 1912, 6, 67, 228) are thus confirmed. R. K. C.

**Wines of the Gard and the Ardèche.** AUBOUY and MERCOYROL (Ann. Falsif., 1933, 26, 348—359).—Analytical data of 100 samples of wine are given. E. C. S.

**Manufacture of sweet and fermented cider by the closed cuvée method.** M. B. DAVIS (Fruit Products J., 1933, 12, 294—298, 315).—The relative advantages of the two methods are discussed and the equipment and process for manufacture in the closed cuvée are described. E. B. H.

**Production of absolute alcohol by the Hiag process.** ANON. (Int. Sugar J., 1933, 35, 266—268).—Plant for the operation of the Gorhan process, according to which the dehydration of the EtOH and the regeneration of the salt mixture proceed automatically, is described. No production of dil. alcoholic fractions occurs, the whole of the spirit being obtained as abs. EtOH in a single operation, in consequence of which lower steam and  $H_2O$  consumptions are claimed than in other processes. Applied directly to the mash (containing about 10% EtOH), 100 Imperial gals. can be produced daily at 6s. in a plant having a capacity of 13,000 gals.; using neutral spirit at 94% the corresponding cost would be nearly 1s. 6d. J. P. O.

**Composition of the high-boiling components of fusel oil.** P. SCHORIGIN, V. ISSAGULIANZ, V. BELOV, and S. ALEXANDROVA (Ber., 1933, 66, [B], 1087—1093).—The residues are hydrolysed with KOH-EtOH and subsequently separated into fatty acids (13% whereas only about 1.4% is originally present as free acid), alcohols (about 36%), org. bases (about 4.5%), and neutral, unsaponifiable matter (about 9.6%). The deficit is accounted for by losses and the presence (or formation) of substances sol. in  $H_2O$  in the residues. Fractional distillation of the Et esters of the acids establishes the presence of hexoic, octoic, decoic, lauric, myristic, and palmitic acid. Acids with higher mol. wt. or an odd no. of C atoms are not present. The alcohols are contaminated with a malodorous impurity characteristic of fusel oil residues, from which they are

best purified by treatment with boric or phthalic acid. The presence of hexyl, heptyl, octyl, and nonyl alcohol is established. Trimethyl-, tetramethyl-, 2:5-diethyl-, and (?) methyltriethyl- (I), b.p. 232°, -pyrazine are identified. (I) gives a picrate, m.p. 101—102°, and a chloroaurate,  $C_{11}H_{18}N_2 \cdot HCl \cdot AuCl_3 \cdot H_2O$ . H. W.

**Evaporation.**—See I. Abs. EtOH.—See III. **Sterilised food substances.** **Enzymatised stock feed.**—See XIX.

See also A., Aug., 862, **Determination of phenolase.** **Purification of plant-amyloses.** 863, **Phosphatase.** 864, **Prep. of pure uricase solutions.** 865, **Action of metals on enzymes.** 867, **COME<sub>2</sub>-BuOH fermentation.**

## XIX.—FOODS.

**Wheat evaluation on a commercial basis.** E. GALTER (Mühlenlab., 1933, 3, 131—136).—A general discussion is given of the economic evaluation of wheat on the basis of yield and of milling and baking qualities from the point of view of the wheat breeder. E. A. F.

**Chemical study of rancidity. III. Recent developments in the study of oxidative rancidity of special interest in the cereal industry.** H. O. TRIBOLD, R. E. WEBB, and W. J. RUDY (Cereal Chem., 1933, 10, 263—276; cf. B., 1932, 269, 474, 612).—Experimental crackers, baked from 10 samples of lard shortenings and the same flour under standard, approx. commercial, conditions, were autoxidised at 95° without stirring. A relationship was found, respectively, between the keeping qualities (I) of the lard samples at room temp. and at 40°, the (I) of the lards at room temp. and the length of their induction periods (II), the length of (II) and active O val. (III) of the lards, and between (III) and fat-aldehyde val. There was no definite correlation between the free acidities of the lards and their (I) on storage or their (II). (I) of cracker is not always correlated with the (II) of the lard from which it is produced; hence crackers of good (I) may be obtained from shortenings of poor (I). That this may be due to a destruction of the deteriorative compounds during the baking process is confirmed by the fact that limited oxidation of lard considerably increases (III) and decreases or inhibits (II). Crackers from lards with high (III) show a reduced (III) for their extracted fat. The (I) of a shortening as measured by storage tests may not be a true index of the (I) of the crackers baked from it. 0.2—0.02% of maleic acid did not appreciably increase (II) of lards autoxidised at 90° without stirring. E. A. F.

**Diastatic activity and its relation to the baking quality of flour.** N. P. KOSMIN (Mühlenlab., 1933, 3, 113—118).—The amount of  $CO_2$  developed in the fermentation of 4 flours differing greatly in diastatic activity (I) and, therefore, in sugar content with 1.5% of yeast was very uniform at all stages, but with 6% of yeast the differences in the flours were shown by the  $CO_2$  production. The min. desirable (I) of flour, using the straight dough method and 1% of yeast, is 150—200 Rumsey units. The stickiness and moistness of the crumb of a loaf baked from sprouted wheat flour is due, not to insufficient baking, but to excessive dextrinisation of the starch which leads to loss in  $H_2O$ -absorptive power.



The composition of the aq. extract (*A*) in loaves from sprouted (*B*) and unsprouted (*C*) grain is practically the same but the actual amount of *A* is approx. twice as great in *B* as in *C*. The starch degradation can be inhibited during the baking process by acidifying the doughs ( $p_H$  4.4); *A* does not then increase during baking.

E. A. F.

**Diastatic activity in relation to baking quality and methods for its determination.** B. CZYZEWSKY (Mühlenlab., 1933, 3, 119—130).—The autolytic are the correct methods of determining the diastatic activity of flours. A crit. account, with experimental details, is given of the standard methods of determining the diastatic activity and maltose figures of flours in America, England, and Germany (Ritter, Bertrand, Rumsey, Kent-Jones, Elhardt, Bruhns, and Reischauer methods).

E. A. F.

**Apparatus for accurate delivery of solutions used in experimental baking tests.** P. TALBOTT and R. WEAVER (Cereal Chem., 1933, 10, 367—369).—An apparatus is described and figured by means of which the yeast, sugar and salt, and bromate solutions necessary in experimental baking tests can be delivered into the mixing bowl. It is claimed that this equipment increases baking capacity and accuracy, is inexpensive, and can easily be kept clean.

E. A. F.

**Vitamin-B and -G content of wheat germ, rice polishings, cottonseed flour, and the residue from fermented rye grains.** H. E. MUNSELL and G. M. DEVANEY (Cereal Chem., 1933, 10, 287—297).—The vitamin-B vals. of various substances in terms, respectively, of Sherman units (yeast 11) and on the arbitrary basis of yeast as 100 were found to be: wheat germ 7, 60; wheat 1.5, 13; rice polishings 6, 53; cottonseed flour 5, 43; residue from fermented rye grain 0.4, 4. The corresponding vitamin-G vals. were: yeast 17, 100; wheat germ 3, 15; wheat 1, 5; rice polishings 1, 4.5; cottonseed flour 2, 10. Whole (soft winter) wheat is therefore a relatively poor source of vitamins-B and -G.

E. A. F.

**Influence of composition of yellow corn [maize] on effectiveness of a rachitogenic ration.** A. D. HOLMES and F. TRIPP (Cereal Chem., 1933, 10, 313—329).—Analyses of samples of rachitogenic rations from different laboratories and consisting of yellow maize 76, wheat gluten 20,  $CaCO_3$  3, and NaCl 1%, showed variation in ash, Ca, and P contents and in the Ca/P ratio. Samples of ground maize from representative milling concerns showed considerable variation in fineness, ash, Ca, and P contents, and in Ca/P ratio. It is known that the mineral content of yellow maize is affected by soil conditions, fertilisers, and rainfall, and that the vitamin and protein contents are very variable. Hence in the prep. of a rachitogenic ration, attention should be paid to the composition of the maize sample used.

E. A. F.

**Spectrographic determination of carotenoid pigment content of wheat flour.** C. G. FERRARI (Cereal Chem., 1933, 10, 277—286; cf. B., 1929, 1029).—Spectrographic equipment for the determination of carotenoid pigments of wheat flour is described. The flour extract is prepared by agitating with 93%

of light cleaner's naphtha (b.p. 93.3—160°) and 7% of abs. EtOH at intervals, keeping overnight, and centrifuging at high speed for 20 min. Readings are made with a Koenig-Martens spectrophotometer at wave-length 435.8 m $\mu$ , first with the pigmented extract in one beam, and then in the other, using the first quadrant of the circular scale on the photometer. The larger angular reading is  $\theta_1$  and the smaller  $\theta_2$ . Transmittancy is given by  $T = \cot \theta_1 \tan \theta_2$  and can be calc. with a special slide-rule. Transmittancy is converted into carotene concn. (cf. B., 1929, 657), expressed as p.p.m. of flour.

E. A. F.

**Yoghurt and fermented milks.** M. FOUASSIER (Ann. Falsif., 1933, 26, 331—345).—Yoghurt is characterised by its consistency, bacterial flora, dry wt., and its fat, total N, sugar, and ash contents.

E. C. S.

**Determination of fat in cheese.** H. HALSTENSEN (Tids. Kjemi, 1933, 13, 100—103). The cheese is boiled with HCl and the fat filtered off; the filter is dried and extracted with  $Et_2O$ . Results agree with those of the usual extraction method, and the method is specially suitable for whey cheeses.

R. P. B.

**Increased acidity [of prunes] inhibits corrosion.** E. F. KOHMAN and N. H. SANBORN (Ind. Eng. Chem., 1933, 25, 920—922).—Dehydration of prunes increases their tendency to attack the can, when canned in syrup. Addition of citric acid (as lemon juice) inhibits corrosion and improves the flavour.

H. J. E.

**Canning tomato juice without vitamin-C loss.** E. F. KOHMAN, W. H. EDDY, and C. Z. GURIN (Fruit Products J., 1933, 12, 299—301).—A process for protecting the vitamin-C of tomato juice by removal of dissolved  $O_2$  is described. The juice is allowed to fall upon a steam-heated pipe suspended over a trough. The efficacy of the process is proved by biological experiments.

E. B. H.

**Erratum.**—On p. 731, col. 1, line 22 from the bottom, for "6 hr." read "< 6 hr."

**Composition of potted meat and fish.** A. R. TANKARD (Med. Off., 1933, 50, 45).—It is recommended that potted meat and fish should contain no foreign ingredient (starch) and  $\geq 70\%$   $H_2O$ .

E. C. S.

**Chemical composition of Spanish foodstuffs.** I. Foodstuffs of vegetable origin. II. Fish of the north coast of Spain. III. Mountain meats. IV. Milk and cheese. J. PUYAL and I. TORRES (Anal. Fis. Quím., 1933, 31, 74—80, 81—86, 87—90, 91—96).—Carbohydrate, protein, and, in most cases, fat,  $H_2O$ , ash, Ca, and K have been determined in 20 fruits and vegetables, 25 fish, 12 meats, cows' milk throughout a year, and 10 cheeses, as consumed in Santander. With the exception of the vegetable foodstuffs, the results show significant differences from the results of other workers with foods from other sources. The foodstuffs examined are tabulated in order of protein and fat content and the figures are discussed in relation to diet. A modification of Lawrence's dietary scheme is proposed.

R. K. C.

**Analysis and calorific values of some Indian foodstuffs.** A. D. STEWART, T. C. BOYD, and D. C. DE (Indian J. Med. Res., 1931, 19, 675—689).—Vals. for many common foods are recorded.

CH. ABS.



**Replacement of potato flakes by sugar-beet products in pig fattening.** O. NOLTE, H. MÜNZZBERG, F. STOCKKLAUSNER, F. DAUM-GRUB, H. BÜNGER, A. WERNER, and J. SCHULTZ (Bied. Zentr. [Tierernähr.], 1933, B, 5, 116—139).—Potato flakes may be replaced to the extent of 50% by dried or ground beet pulp without effect on the live-wt. increase of the pigs or on the % utilisation of the total ration. A. G. P.

**Mannitic fermentation of beet fodders.** J. VON-DRÁK (Z. Zuckerind. Czechoslov., 1933, 57, 317—321).—Fresh sugar beets, whole or sliced, stored in closed trenches, yielded fodders which in some cases were purgative in large doses owing to high acidities, e.g., 2% of non-volatile acid calc. as lactic acid in the moist fodder, besides some volatile acid. Most of the sugar disappeared owing to alcoholic and mannitic fermentations. A fodder from sliced beets contained 11.4% of total solids, including 2.3% of fixed acid, 0.7% of sugar, 0.36% of alcohol, and 2.9% of mannitol the greater part of which could be easily obtained in cryst. form. J. H. L.

**Fattening experiments with ducklings.** H. BÜNGER, A. WERNER, and H. BRANDENBURG (Bied. Zentr. [Tierernähr.], 1933, B, 5, 140—161).—The smaller growth of ducklings receiving rations in which fish and meat meals were replaced by milk curd is associated with the absence of cystine from the proteins of the latter. A. G. P.

**Denaturing sugar. Pectin substances. Hydration of starch.**—See XVII.

See also A., Aug., 814, **Prep. of *d*-glutamic acid hydrochloride from wheat gluten.** 820, **Purification of vitamin-A preps.** 843, **Casein.** 844, **Determination of reducing sugars [in milk].** 847, **Pigment of milk. Dialysis of milk. Growth of lactic streptococci in milk.** 848, **Proteins of carabao milk. Milk-fats of Indian goats and sheep.** 857, **Nutritive vals. of fats.** 870—3, **Vitamins (various).** 878, **Nitrogenous constituents of Florida Valencia orange-juice.**

## PATENTS

**Processing of cheese.** R. PASTERNAK and R. W. BURNHAM, Assrs. to C. PFIZER & Co. (U.S.P. 1,890,948, 13.12.32. Appl., 13.6.30).—Na, NH<sub>4</sub>, K, or Ca gluconate (0.1—5.0%) may be used as the emulsifying agent in the manufacture of processed cheese, thereby preventing the subsequent appearance of gritty Ca salts often found therein. E. B. H.

**Drying of casein.** F. L. CHAPPELL (U.S.P. 1,892,233, 27.12.32. Appl., 14.7.30).—Apparatus is described whereby pressed casein, broken into particles the shape and size of bird shot (or alternatively as flakes), may be blown and dehydrated in a current of hot air (49—177°). The humidity of the air is controlled by injecting steam, thus ensuring a gradual drying of the particles and preventing a case-hardening effect. E. B. H.

**Production of solid preparations of lactic acid.** F. A. V. KLOPPER (B.P. 395,990, 27.2.33).—An 80% aq. solution of lactic acid is kneaded with an equal wt. of a dry vegetable mucilage or mucilaginous substance, e.g., carob bean, carrageen, etc. E. B. H.

**Stabilising shortening [e.g., lard].** SWIFT & Co., Asses. of D. P. GRETTE (B.P. 395,971, 30.1.33. U.S., 18.8.32).—Addition to the lard of < 10% of sesamé oil hydrogenated to the consistence of lard stabilises the fat against rancidity as shown by lower active O val. of the fat kept at 70°. E. B. H.

**Vitamin fruit composition.** E. R. ALEXANDER, Assr. to VITAMIN Co. OF AMERICA (U.S.P. 1,886,931, 8.11.32. Appl., 1.8.30).—Any fruit rich in acid or alkaline-earth salts of the fruit acid, invert sugar, essential oils, and pectin is either cryst. in sugar and dehydrated, or merely dosed with preservative and reduced to fine pulp. The fruit is then mixed with dried vitamin concentrate from, e.g., cod-liver oil, yeast, or wheat embryo. B. M. V.

**Treatment of alimentary products.** N. K. CHANEY, Assr. to NAT. CARBON Co., INC. (U.S.P. 1,894,158, 10.1.33. Appl., 13.3.28).—Foodstuffs are irradiated over a moving belt by means of a Hg-arc lamp (or a C-arc lamp with specially prepared carbons) to increase their vitamin-D contents. The undesirable flavours usually obtained by such treatment, due to production of O<sub>3</sub>, are eliminated by allowing light only of certain wave-lengths (by the use of screens) to impinge on the material and by treating the foodstuffs with an inert atm. (CO<sub>2</sub>). E. B. H.

**Apparatus for sterilising food substances.** H. W. ROHDE, Assr. to J. SCHLITZ BEVERAGE Co. (U.S.P. 1,888,472, 22.11.32. Appl., 1.10.31).—An apparatus for subjecting viscous fluid (e.g., malt extract) to the action of light is described. B. M. V.

**Treating chocolate liquor containing proteins, starches, and fats.** F. P. DENGLER, Assr. to L. M. BROWN (U.S.P. 1,892,449, 27.12.32. Appl., 5.6.29).—Ground chocolate liquor is suspended in H<sub>2</sub>O acidulated with HCl and digested at 79.5—82° with a mixture of papain and pepsin, whereby the fat is liberated and may be removed by skimming (cf. B.P. 361,461, B., 1932, 116). E. L.

**Making enzymatised stock feed.** E. M. BROWN (U.S.P. 1,885,411, 1.11.32. Appl., 30.3.29).—Cereal grains are caused to sprout in a solution of mineral salts (which are absorbed) in presence of CaCO<sub>3</sub> to neutralise acidity. When the max. enzyme content is reached the growth is stopped by drying and grinding and the powder mixed with cut "roughage," moistened, and maintained at > 54°. B. M. V.

**Machine for making cream-like compositions from butter or substitute.** F. J. and S. G. CLARKE (B.P. 396,044, 25.1.32).

[Machine for] **manufacture of [tempered] chocolate.** O. SCHRAMM, O. and W. LAUENSTEIN, and W. H. EYERMANN (B.P. 395,834, 27.4.32. Ger., 6.11.31. Addn. to B.P. 392,350). E. B. H.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Determination of morphine in admixture with other opium alkaloids.** H. BAGGESGAARD-RASMUSSEN and F. REIMERS (Parm. Acta Helv., 1932, 7, 249—258; Chem. Zentr., 1933, i, 822).—A modification of Mannich and Schwede's method for the determination of morphine



and the associated alkaloids in "tetrapon" (a mixture of the hydrochlorides of morphine, narcotine, codeine, and papaverine) is described. L. S. T.

**Reactions of the barbituric acids.** PAGET and DESODR (Bull. Sci. pharmacol., 1932, 39, 532—534; Chem. Zentr., 1933, i, 822).—The following reactions are obtained with Millon's reagent: in EtOH gardenal (I) gives a white ppt., sol. in HCl; dial (II) a white ppt. in the cold, grey in hot solution, sol. in hot HCl; veronal (III) a white ppt., sol. in hot HCl; in COMe<sub>2</sub> (I) transient white ppt.; (II) and (III) white ppts. at the b.p. (I) grey ppt. after 10 sec.; (II) grey ppt.; (III) a white ppt. which dissolves and then gives a grey ppt. Barbituric acid derivatives give a rose to red colour in EtOH solution (1—2 c.c.) with Co(NO<sub>3</sub>)<sub>2</sub> (1 drop), KCN (1 drop of 0.1N solution), and NH<sub>3</sub> (1 drop). L. S. T.

**Decomposition of sodium phenylethylbarbiturate in aqueous solution.** L. NIELSEN (Dansk Tidsskr. Farm., 1933, 8, 137—152).—The salt decomposes to CHPhEt·CO·NH·CO·NH<sub>2</sub> and CO<sub>2</sub> in aq. solution. Data are given for velocity at 1°, 20°, and 39° for a 10% solution. The rate has an abnormally high temp. coeff., and increases with increasing *p*<sub>H</sub> in the range 8.9—9.9. A 1% solution is more stable. R. P. B.

**Rivanol [6:9-diamino-2-ethoxyacridine].** I. M. KORENMANN (Pharm. Zentr., 1933, 74, 485—488).—The diazotisation test for rivanol (I) is most delicate if equimol. quantities are taken; the resulting solution gives sol. dyes with phenols (yellow to brownish-red in alkaline, and yellow in acid, solution). (I) (>0.2 × 10<sup>-6</sup> g.) and C<sub>5</sub>H<sub>5</sub>N-saturated aq. KI-dil. HNO<sub>3</sub> (1:1:2) give a yellow, cryst. ppt. R. S. C.

**Citric acid.**—See III. **Turpentine oil.**—See XIII.

See also A., Aug., 808,  $\alpha\beta$ -Ethylenic straight-chain aldehydes. 811, Cardiac glucosides. 817, Alkyl derivatives of halogenophenols as bactericides. 820, Purification of vitamin-A preps. 834, Synthesis of  $\alpha$ -substituted *N*-methylpyrrolidines. 836, Prep. of "atophans." 837, Derivatives of antipyrine and of 3-phenyl-1-methylbarbituric acid. 840—1, Alkaloids (various). 841, Cryst. atisine. 842, Salts of quaternary NH<sub>4</sub> bases. Alcoholic arsenic acids. Alkoxy-pyridine-arsenic acids. 844, Reaction of caffeine. Identification of org. compounds. 858, New alkalines in the tetrahydro-naphthalene series. 859, Harmine and harmaline derivatives. Test for morphine. Detection of tropine alkaloids. 860, Pharmacognosy of *Tanacetum vulgare*. 863, Substance from mesenteric extracts. 870, Oestrogenic substances in bitumen. Prep. of cryst. thylokinin. Detection of the luteo-hormone in human tissue and body fluids. Assay of testicular hormone. 870—873, Vitamins (various). 876, Ginseng root. 878, O-free alkaloid in *Gelsemium sempervirens*. Alkaloids of *Mahonia philippinensis*.

#### PATENTS.

**Production of a medicinal [iron-copper-protein] preparation.** A. L. MOND. From MCKESSEN & ROBBINS, INC. (B.P. 393,319, 24.5.32).—H<sub>2</sub>O-sol. fruit

acid salts (Na citrate) are added as stabilisers in prep. of Fe or Cu nucleinate or caseinate solutions for treatment of anæmia.

**Production of medicinal oil solutions.** W. W. TRIGGS. From ABBOTT LABORATORIES (B.P. 393,102, 4.7.32).—Acidic organo-metal (Hg) compounds, e.g., 4-nitroanhydrohydroxymercuri-*o*-cresol, insol. in oil, are converted into amine salts, which are incorporated in mineral oil with addition of an amine soap, e.g., ephedrine laurate. C. H.

**Manufacture of compounds from terpene alcohols and hydroaromatic alcohols.** HOWARDS & SONS, LTD., J. W. BLAGDEN, and W. E. HUGGETT (B.P. 392,571, 15.10.31. Addn. to B.P. 374,893; B., 1932, 960).—Monohydric hydroaromatic alcohols other than menthol give cryst. compounds with H<sub>3</sub>PO<sub>4</sub>, e.g., cyclohexanol (m.p. 35°), fenchyl alcohol (m.p. 63°), borneol (m.p. 130°), terpineol of m.p. 34° (m.p. 45°), and liquid terpineol (m.p. 39°). C. H.

**Manufacture of idonaphtholsulphonic acids [therapeutic agents].** CHEM. FABR. VORM. SANDOZ (B.P. 393,238, 27.2.33. Switz., 29.2.32).—An aminonaphtholsulphonic acid is diazotised and the diazo-group replaced by I, e.g., by treating with HI and Cu, or boiling with HI. C. H.

**[Manufacture of] esters of cyclopentenylacetic acids.** COMP. DE BETHUNE (B.P. 391,579, 3.3.32. Fr., 6.3.31).— $\Delta^2$ -cyclopentenylacetic acid (I) and its  $\alpha$ -Me (II),  $\alpha$ -Et (III),  $\alpha$ -allyl (IV), or  $\alpha$ - $\Delta^2$ -cyclopentenyl (V) derivatives are esterified with alcohols or phenols to give products useful for perfumery and hygienic purposes. The following (b.p. in parentheses) are described: isoamyl (116—117°/14 mm.), Pr<sup>n</sup> (99°/17 mm.), Pr<sup>s</sup> (70—73°/5 mm.), Bu<sup>s</sup> (107°/11 mm.), sec.-Bu (96—101°/11 mm.), C<sub>2</sub>H<sub>4</sub>Ph (171°/11 mm.), cyclohexyl (132—133°/10 mm.), CHPh:CH·CH<sub>2</sub> (201—202°/14 mm.), CH<sub>2</sub>Ph (162—163°/13 mm.), terpinyl (165—170°/6 mm.), tert.-Bu (117°/12 mm.), geranyl (160—165°/5 mm.), Ph (112—113°/4 mm.), *p*-tolyl (147°/4 mm.), *m*-tolyl (141°/4 mm.), guaiacyl (174—176°/11 mm.), thymyl (159—160°/6 mm.), eugenyl (187—190°/7 mm.), isoegenyl (208°/10 mm.), carvacryl (165—168°/5 mm.), and menthyl (170°/12 mm.) esters of (I) (acid chloride, b.p. 62—63°/12 mm.); Et (68°/3 mm.), Bu<sup>s</sup> (74—78°/2 mm.) and CH<sub>2</sub>Ph (135—136°/2 mm.) esters of (II) (acid chloride, b.p. 83—84°/19 mm.); Me (93—95°/19 mm.), Et (101°/16 mm.), Bu<sup>s</sup> (114—115°/9 mm.), isoamyl (122°/10 mm.), and C<sub>2</sub>H<sub>4</sub>Ph (174—175°/9 mm.) esters of (III) (acid chloride, b.p. 94°/18 mm.); Me (97°/12 mm.), Et (113—114°/17 mm.), Pr<sup>n</sup> (124—125°/14 mm.), Pr<sup>s</sup> (116°/16 mm.), Bu<sup>s</sup> (127—128°/12 mm.), isoamyl (123—125°/5 mm.), allyl (124—125°/15 mm.), CH<sub>2</sub>Ph (162—164°/5 mm.), and CHPh:CH·CH<sub>2</sub> (207—208°/9 mm.) esters of (IV) (acid chloride, b.p. 96—98°/11 mm.); Me (112—114°/4 mm.), Et (142—143°/15 mm.), and Bu<sup>s</sup> (157—158°/13 mm.) esters of (V) (acid chloride, b.p. 135—137°/13 mm.). C. H.

**Manufacture of *N*-allyl- and *NN'*-diallyl-*CC*-disubstituted barbituric acids.** F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 391,741, 20.1.33. Ger., 29.2.32).—Alkali salts of 5:5-disubstituted barbituric acids or



their *N*-monosubstituted derivatives are treated with an allyl halide in  $H_2O$  in presence of small quantities of Cu. The following barbituric acids are described: 5:5:*N*-triallyl, m.p. 68—69°; 5-*isopropyl*-5:*N*:*N'*-triallyl, b.p. 171—174°/14 mm.; 5-phenyl-5-ethyl-*N*-allyl, m.p. 68—69°, b.p. 225—230°/14 mm.; 5-benzyl-5-ethyl-*N*-allyl, m.p. 77—78°, b.p. 225—229°/14 mm.; *N'*-phenyl-5:5-diethyl-*N*-allyl, m.p. 72—73°, b.p. 208—210°/14 mm. C. H.

**Manufacture of purified germ-gland preparations.** SCHERING-KAHLBAUM A.-G. (B.P. 391,686, 5.10.32. Ger., 17.10.31).—A crude hormone prep. is treated with a salt-forming compound containing a group serving to condense with the hormone, the salt of the condensation product is isolated, and the hormone liberated by hydrolysis. By means of *o*- $C_6H_4(CO)_2O$  or salicyl chloride, the follicular hormone,  $C_{18}H_{22}O_2$ , m.p. 240°, is isolated from crude hormone oil of  $2 \times 10^5$  mouse units/g. Other suitable reagents are  $NH_2 \cdot NH \cdot C_6H_4 \cdot SO_3H$ ,  $ClSO_3H$ , etc. C. H.

**Pocket optical instrument for testing organic liquids [especially urine].** E. ANSEMI (B.P. 393,524, 11.1.33. Italy, 11.1.32).—A metal instrument suitable for observing pptns. in urine, and for investigations of blood or serum, is described. C. H.

**Tissue filler.** E. H. HAABESTAD, Assr. to HYDROL CHEM. CO., INC. (U.S.P. 1,889,655, 29.11.32. Appl., 24.4.31).—A solution suitable for hypodermic injection into the visible parts of corpses comprises a solution of nitrocellulose (or other compound insol. in  $H_2O$ ) in MeOH or other solvent readily miscible with  $H_2O$ . B. M. V.

**[Moisture-retaining] treatment of tobacco leaves and composition therefor.** S. JORDAN, Assr. to APPLIED SUGAR LABS., INC. (U.S.P. 1,888,173, 15.11.32. Appl., 7.3.32).—A moisture-retaining spray for the leaves comprises invert sugar,  $Ca(NO_3)_2$ , and  $H_2O$  of approx. the same *d* as 95% glycerin. B. M. V.

OH-aldehydes.—See III.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Photographic plates for use in spectroscopy and astronomy.** III. C. E. K. MEES (J. Opt. Soc. Amer., 1933, 23, 229—233; cf. B., 1932, 162, 657).—The properties of a new type of plate of low sensitivity, high resolving power, and very fine grain, together with new and improved sensitising (no details given) for special spectral regions, are recorded. N. M. B.

**Pharmaceutical-technical parallels in photographic plates with special reference to the constitution and significance of single substances included among developers.** F. HAGELSTEIN (Pharm. Zentr., 1933, 74, 409—415, 454—460). J. L. D.

**Physical development of plates, films, and lantern slides.** A. F. ODELL (Ind. Eng. Chem., 1933, 25, 877—879).—The developer is a solution of  $Na_2S_2O_3$ ,  $Na_2SO_3$ ,  $AgNO_3$ , and amidol. With 5 times normal exposure negatives of normal density are developed in 1.5—2 hr., giving very fine-grained deposits of Ag.

For intensification by physical development a more vigorous pptn. of Ag (e.g., by metol or glycine) is necessary. Working details are given. H. J. E.

See also A., Aug., 791, **Colour-sensitivity of emulsions. Latent image for physical development. 792, Increase of sensitivity of emulsions by electrophoresis. Behaviour of Ag halide emulsions with new desensitisers and developers.**

### PATENTS.

**Production of photosensitive emulsions.** R. GRIESSBACH and T. KOLLMANN, Assrs. to AGFA ANSCO CORP. (U.S.P. 1,892,725, 3.1.33. Appl., 6.8.29. Ger., 14.8.28).—The Ca salts of the polymerides of aldehyde-carboxylic acids (e.g., glycuronic or galacturonic acid) such as can be prepared from the alkaline extracts of algæ are used as the binding agent of photographic emulsions instead of gelatin. They have a high adhesive power on glass etc., and are very resistant to high temp. and warm  $H_2O$ . J. L.

**Photographic anti-halation layer.** I. G. FARBERIND. A.-G. (B.P. 395,889, 1.9.32. Ger., 22.9.31).—Anti-halation layers which are easily detachable in the dry or moistened state are made by coating a solution of colloid (gelatin or a cellulose derivative), mixed with suitable pigment (e.g., soot or graphite), on to the back of the film, which has received only limited treatment or no treatment, with the customary solution (gelatin and AcOH) used to aid adherence of emulsions. The anti-halation mixture may contain EtOH, which has a feeble swelling action on the support. The thickness of the layer should be 5—10  $\mu$ . [Stat. ref.] J. L.

**Making a printing plate.** T. E. RICHARDS (U.S.P. 1,892,682, 3.1.33. Appl., 4.6.29. Renewed 21.5.32. N. Zealand, 22.11.26).—A developed negative image (from dichromate emulsion) on a printing plate is coated with a mixture prepared from turpentine with asphaltum, transfer ink with asphaltum, and a prep. of oleic acid and essential oil ("Elbagreen oil"). The plate is heated to 44° to harden the coating, the undeveloped colloid (glue) dissolved in aq.  $H_3PO_4$ , and the remaining positive matrix heated to about 125°. J. L.

**Colour-sensitised collodion photographic plates.** SILLIB & BRÜCKMANN U. GEBR. FREUNDORFER G.M.B.H. (B.P. 395,818, 23.3.32. Ger., 28.3.31).—A collodion-Ag halide emulsion plate is treated with a solution comprising a softening agent for collodion (glycerin or sugar) and a Ag salt of one or more dyes of the phthalein or quinoline groups, or Et-violet. Alternatively, the solution may contain  $AgNO_3$  and the dye, or alkaline  $AgNO_3$  alone, the dye having been incorporated in the emulsion previously. J. L.

**Production of coloured photographic images and kinematograph films.** B. GASPAR (B.P. 395,718, 16.11.31).—Diffusely dyed emulsions containing metallic images (free from metal halide) are decolorised at the points free from metal by means of oxidising agents, e.g., dil. solutions of  $Ca(OCl)_2$  with AcOH, or  $KBrO_3$  and  $NaBrO_3$  with HCl. A catalytically active salt, e.g., of U, V, or Mo, may also be present. Mixtures of dyes which bleach at different rates, or dyes which change colour on oxidation, may be employed. J. L.



**Modifying photographic images.** A. W. CORNELL, Assr. to FORBES LITHOGRAPH MANUFACTURING CO. (U.S.P. 1,892,099, 27.12.32. Appl., 3.3.31).—Retouching is performed by treating the hardened emulsion with conc. aq.  $K_4Fe(CN)_6$ , connecting the gelatin film through a suitable frame etc. to the negative pole of a battery (e.g., 6-volt), and applying the other electrode in pencil-point form to the areas to be reduced. The process is specially applicable to screen diapositives. J. L.

**Manufacture of photographic pictures.** KALLE & Co. A.-G. (B.P. 391,677, 21.9.32. Ger., 21.9.31).—The paper or other base is sensitised by means of diazo-compounds which on decomp. by light give reducing products, and Ag salts (which need not be light-sensitive and are preferably  $H_2O$ -sol.). The coloured positive is converted by alkaline agents ( $Na_2CO_3$ ) into a black or brown print by reduction of the Ag salt, and fixed with  $Na_2S_2O_3$ . Examples of diazo-compounds are: chlorobenzenesulphonates of diazotised 5-diethylaminoanthranilic acid,  $p$ - $C_6H_4(NH_2)_2$ ,  $p$ - $OH \cdot C_6H_4 \cdot NH_2$ ; sulphates of diazotised 4- $NH_2 \cdot C_6H_4 \cdot NHPH$ ,  $o$ - $NH_2 \cdot C_6H_4 \cdot NMe_3$ . C. H.

**Three-colour photography.** I. G. FARBENIND. A.-G. (B.P. 395,124, 28.4.32. Ger., 2.5.31).—A bi-pack is used comprising a smooth film placed behind a lenticular film, the latter recording 2 of the 3 colours selectively. J. L.

## XXII.—EXPLOSIVES; MATCHES.

See A., Aug., 779, Sp. viscosity of cellulose nitrate. 788, Detonation of explosives by collision.

### PATENTS.

**Priming mixture.** W. BRÜN, Assr. to REMINGTON ARMS CO., INC. (U.S.P. 1,887,919, 15.11.32. Appl., 3.6.31).—Primers containing a Pb 3:5 dinitrobenzoate (A) are claimed; e.g., a mixture of Hg fulminate 40.8,  $Ba(NO_3)_2$  24.5, monobasic A 8.2, glass 25.5, and gum 1.0% is suitable. B. M. V.

**Blasting cap.** W. DE C. CRATER, Assr. to HERCULES POWDER CO. (U.S.P. 1,887,290, 8.11.32. Appl., 30.9.30).—The base charge (A) is cryst. nitromaltose and the primer (B) fulminate-chlorate, diazodinitrophenol, or  $PbN_6$ . Part of B may be mixed with A. B. M. V.

**[Apparatus for] manufacture of blasting explosives and the like.** E. I. DU PONT DE NEMOURS & Co. (B.P. 395,759, 23.1.32. U.S., 24.1.31).—A jacketed bowl, constructed of, or lined with, a Cu alloy (Cu 94.8—96, Si 3—4, Mn 1—1.5%), is provided with a pair of vertical, non-metallic rollers (A), adjustable with respect to the bed of the bowl and either under- or over-driven. Two ploughs of non-metallic material (e.g., hard rubber), the distance of which from the sides of the bowl can be varied, rotate in front of A, and there are two non-metallic raking devices, B, which can be raised or lowered. The apparatus is capable of dealing with both gelatinous and non-gelatinous types of explosives. Pregelatinisation of nitroglycerin and nitrocotton with temp. control is effected with the aid of B, these being raised from the bowl before the remaining ingredients are added. W. J. W.

**Blasting method and apparatus.** C. E. KARSTROM, Assr. to SAFETY MINING CO. (U.S.P. 1,886,960, 8.11.32.

Appl., 7.10.29).—With a blasting cartridge are incorporated a deliquescent material to allay the dust after the explosion, and a colouring substance to identify the rock broken down. B. M. V.

**Finishing of gun barrels.** J. M. OLIN and A. G. SCHURIGHT, Assrs. to WESTERN CARTRIDGE CO. (U.S.P. 1,886,218, 1.11.32. Appl., 29.6.27).—The bore is Cr-plated in sufficient thickness to restore the calibre (after re-boring if necessary). B. M. V.

**Treating [match-box] boards.**—See IX. Fumigants.—See XXIII.

## XXIII.—SANITATION; WATER PURIFICATION.

**Formation of floc by ferric coagulants.** E. BARTOW, A. P. BLACK, and W. E. SANSBURY (Ind. Eng. Chem., 1933, 25, 898—903; cf. B., 1933, 734).—The effect of  $FeSO_4$ ,  $Fe_2(SO_4)_3$ , and chlorinated aq.  $FeSO_4$  in  $H_2O$  treatment was examined. The time of floc formation at varying  $pH$  due to  $NaOH$  and  $NaHCO_3$  was measured. A min. time was found at  $pH$  6.1—6.4. On the acid side  $SO_4^{--}$  has a much greater effect than  $Cl^-$  on the rate of floc formation. Between  $pH$  6.5 and 8.5 there is a zone at which  $Fe^{III}$  floc forms slowly or not at all; in and beyond this zone  $Na^+$  and  $Ca^{++}$  are most effective. Change of sign of the floc from positive to negative explains this zone. The residual Fe in solution at varying  $pH$  was approx. proportional to the time of floc formation. H. J. B.

**Poisoning of fish by waste waters from chemical factories and the "fish test."** V. DEMYANENKO (Igi Epidem., Russia, 1931, 10, No. 6—7, 13).— $H_2O$  is injurious to fish if the alkalinity is  $> 8$  or the acidity  $> 1.6$  c.c.  $N$ -solution per litre.  $C_{10}H_8$  is lethal to fish at  $1:10^5$ ,  $p$ - $C_6H_4Cl_2$  at  $1:2 \times 10^4$ ,  $CaCl_2$  at 12.5 g. per litre,  $PhOH$  and cresol at  $1:5$ — $6 \times 10^4$ ,  $NH_4SCN$  at 1:5000, and  $NH_2Ph$  at 1:4000.  $C_5H_5N$  has a feeble effect at 1:1000. CH. ABS.

See also A., Aug., 798, CO indicator. 817, Bactericides.

### PATENTS.

**[Manufacture of] disinfectants and disinfectant detergents.** HENKEL & Co. G.M.B.H. (B.P. 392,814, 6.10.32. Ger., 29.10.31).—Alkali hypochlorites are mixed with alkali salts of arylsulphonchloroamides, e.g.,  $NaOCl$  and  $p$ - $C_6H_4Me \cdot SO_2 \cdot NHNa$ , with or without other alkaline substances ( $Na_2SiO_3$ ,  $Na_2CO_3$ ,  $Na_2HPO_4$ ). C. H.

**Fumigant.** J. G. WRIGHT, G. A. GRAHAM, and A. K. LIGHT (B.P. 396,004, 28.3.33. Can., 18.5.32).—A composition, which may be applied to match-heads, contains gum benzoin 2, balsam of tolu 2, gum olibanum 2, powdered sandalwood 2.5,  $KNO_3$  2.25, gum tragacanth 1.3, and  $H_2O$  16 pts. C black may be added as colouring matter. W. J. W.

**Treatment of sludge, sewage, and the like.** K. IMHOFF (U.S.P. 1,887,490, 15.11.32. Appl., 14.3.32).—In a sewage digester, heating coils are attached to the rotating sludge-raking mechanism, the rakes being under a disc and the coils above it. B. M. V.

**Still for  $H_2O$ . [ $H_2O$ ]-vapour purification.**—See I. Fumigants.—See VII. Disinfectant soaps.—See XII.