

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JULY 5 and 12, 1935.\*

### I.—GENERAL; PLANT; MACHINERY.

**Chemical engineering in the stratosphere.** J. and (MME.) J. PICCARD (Ind. Eng. Chem., 1935, 27, 121—127).—In order to maintain the atm. of the gondola used in stratosphere flights at  $> 2\%$   $\text{CO}_2$  bags with KOH or NaOH were used, in addition to bags with  $\text{Mg}(\text{ClO}_4)_2$  to absorb  $\text{H}_2\text{O}$  and of  $\text{SiO}_2$  gel to remove org. compounds present in stale air. Circulation of air was produced by the lower half of the gondola being black and the upper half white, whilst a small fan was also used.  $\text{CO}_2$  determinations were made by the use of a set of tubes (of 50 c.c. capacity) containing 5 c.c. of 0.01N-NaOH and phenolphthalein. The gondola should be of Al in preference to Mg alloy as the softer metal takes the shape of highest resistance without loss of strength. Fittings and struts should be of Mg alloy and riveted.

C. I.

**Mass transfer (absorption) coefficients. Prediction from data on heat transfer and fluid friction.** T. H. CHILTON and A. P. COLBURN (Ind. Eng. Chem., 1934, 26, 1183—1187).—Colburn has predicted a set of curves for estimating heat-transfer coeffs. by means of a heat-transfer factor  $j = (h/cG)(c\mu/k)^{2/3}$ , where  $h$  = film coeff. of heat transfer and  $k$  = thermal conductivity. A mathematical analogy is drawn between heat transfer and mass diffusion, and the curves applied in this way have been compared with various experimental data for falling-film towers, the evaporation of liquid from plane surfaces, and transverse flow across tubes. Fairly close agreement is obtained.

C. I.

**Stable organic compounds in power generation. Diphenyl-diphenyl oxide mixtures in a boiler plant.** D. H. KILLEFFER (Ind. Eng. Chem., 1935, 27, 10—15).—In the steam section of the boiler, which consists of 20 parallel seamless tubes,  $\text{H}_2\text{O}$  is pumped at 1600 lb./sq. in. through an economiser to the different heating sections in such a way that vaporisation is incomplete. The wet steam passes to a superheater where it is brought to a pressure of 1450 lb. by the vapour of a eutectic  $\text{Ph}_2\text{-Ph}_2\text{O}$  mixture (I) at  $374^\circ$ . The latter is generated at 100 lb. pressure. The steam then passes to a back-pressure turbine, the exhaust from which is reheated by (I). The section of the boiler generating (I) is oil-fired and resembles a  $\text{H}_2\text{O}$ -tube steam boiler with forced circulation. The first cost of the plant is  $<$  that of a comparable steam boiler, owing to the low working pressure of (I), or of a Hg boiler, which has also other disadvantages.

C. I.

**Siliceous scales [in boilers] and their prevention.** R. E. SUMMERS (Fuel Econ., 1935, 10, 784—785).—Hardness is not an index of the tendency of a siliceous  $\text{H}_2\text{O}$  to encrust boilers. Conventional softening of  $\text{H}_2\text{O}$ ,

which removes little siliceous material, may tend to promote formation of siliceous scale (S). With high silicate contents of boiler  $\text{H}_2\text{O}$  the maintenance of a high alkalinity, e.g.,  $p_{\text{H}}$  11, is desirable. S has a lower thermal conductivity than have other scales and a relatively thin deposit may have a dangerous heat-insulating effect. Increasing the blow-down rate is not a suitable preventive measure; not only does it increase the amount of scale-forming substance brought into the boiler in raw  $\text{H}_2\text{O}$  make-up, but it may lower the  $p_{\text{H}}$  of the  $\text{H}_2\text{O}$  to  $< 9-10$ .

A. B. M.

**Synthetic mill stones.** R. SIMPSON (Off. Digest, 1935, No. 145, 160—162).—Aloxite gives better results than buhrstone.

D. R. D.

**Design of fractionating columns. II. Number of plates for gas and gasoline fractionators.** G. G. BROWN, M. SOUDERS, JUN., H. V. NYLAND, and W. W. HESLER (Ind. Eng. Chem., 1935, 27, 383—392; cf. B., 1934, 431).—A method of calculating the theoretical no. of plates required for the separation by distillation of complex liquids, based on the absorption-factor concept, gives results in agreement with those obtained by other methods and with experimental results obtained in the distillation of a mixture of  $\text{C}_3\text{H}_8$ ,  $\text{C}_4\text{H}_{10}$ , and  $\text{C}_5\text{H}_{12}$ . The overall plate efficiency of a gasoline fractionator operating at about 300 lb. per sq. in. is approx. 100%.

D. K. M.

**Relative efficiencies of packed fractionating columns.** P. DOCKSEY and C. J. MAY (J. Inst. Petroleum Tech., 1935, 21, 176—199).—Assuming that the rate of interchange of material between vapour and liquid in a column depends on the rate of diffusion through that part of the vapour in which the flow is stream-line, a no. of equations have been derived (for both stream-line and turbulent flow) from which the relative efficiencies of columns packed with material of any given shape can be calc. The effect of altering the pressure and vapour velocity and of introducing a diluent such as steam can also be calc. The formulæ were checked by experiments using columns 8 mm. and 2 in. in diam. The mixtures used included  $\text{C}_6\text{H}_6\text{-PhMe}$  and  $\text{PhNO}_2\text{-NH}_2\text{Ph}$ . Results show good agreement between the theoretical and experimental figures.

C. C.

**Application of the graphical method of Ponchon to distillation and extraction.** E. W. THIELE (Ind. Eng. Chem., 1935, 27, 392—396).—Ponchon's method (Tech. Moderne, 1921, 13, 20—24, 55—58) for the analysis of distillation columns is described and applied to countercurrent extraction with immiscible solvents.

D. K. M.

**Design of cooler condensers for mixtures of vapours with non-condensing gases.** A. P. COLBURN

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



and O. A. HOUGEN (Ind. Eng. Chem., 1934, 26, 1178—1182).—In the passage of a mixture of vapour and non-condensing gas through a condenser the over-all heat-transfer coeff.,  $U$ , varies from point to point and the position can be expressed as  $dA = dq/Udt$ , where  $q$  = heat transferred. It is not possible to express  $U$  and  $dt$  separately as functions of  $q$ , but the product  $Udt$  may be calc. for various vals. of  $q$  and  $A$  thus obtained by graphical integration.  $Udt$  is obtained by trial and error, knowing the temp. at the gas-condensate interface and the corresponding v.p. An actual example of this procedure is given, the length of the tubes of a condenser cooling inert gas saturated with  $H_2O$  vapour at  $95^\circ$  to  $40^\circ$  being calc. Finally the pressure drop is estimated. In this case the true mean temp. difference is  $>$  the log mean temp. difference or  $>$  either of the terminal differences. The use of high flow rates is of great val. in reducing the size of heat-exchange apparatus. C. I.

**Rate of mixing of gases in closed containers.** A. S. SMITH (Ind. Eng. Chem., 1934, 26, 1167—1169).—The rate of mixing of 2 (or more) gases under pressure may be calc., if  $\Delta$  is known, on the assumption that the 2 gases are placed in the upper and lower halves of a cylinder separated by a sliding plate and the plate is then withdrawn. Variation of  $\Delta$  with  $P$  and  $T$  is given by  $\Delta = \Delta_0(T/T_0)^m P_0/P$ , where  $m = 1.75-2.00$ . Results for the gas pairs  $He-CH_4$  and  $C_4H_{10}$ -air for varying cylinder dimensions are given. These represent maxima, as some convection always occurs. C. I.

**Fractionating column for gasoline.**—See II.

#### PATENTS.

**Ovens for baking, roasting, and drying purposes.** J. B. COX (B.P. 426,176, 30.8.34).—The goods are placed in a no. of superposed imperforate trays or baking chambers ( $T$ ), and means are provided to control from outside the oven the passage of gases between  $T$  and the walls, to regulate the amount of heat trapped at different levels. B. M. V.

**[Manufacture of] compositions particularly adapted for heat transmission and distribution.** WHESOE FOUNDRY & ENG. CO., LTD., and O. D. LUCAS (B.P. 427,170, 11.9.33).— $Ph_2$ ,  $Ph_2O$ , or their mixtures with  $NHPh_2$  are heated at  $>$  their b.p. in order to obtain products stable at  $400-500^\circ$  and of m.p.  $< 0^\circ$ . *E.g.*,  $Ph_2O$  is heated at  $520^\circ/450-540$  lb. per sq. in. for 8 hr. and distilled to separate products of low b.p. (15% had b.p.  $< 250^\circ$ ). The product is stable at  $400^\circ$  and has a f.p. of approx.  $-38^\circ$ . Similar results are obtained by heating  $Ph_2O$  (2 pts.) +  $NHPh_2$  (1 pt.) at  $460^\circ/225$  lb. per sq. in., and  $Ph_2$  at  $500-520^\circ$ . Further f.p. depression is achieved by addition of  $C_{10}H_8$  (10%). H. A. P.

**[Gastight rotating joint for] waste-heat recovery apparatus.** A. W. WAERN and L. F. BUNDE, Assrs. to NEKOOSA-EDWARDS PAPER CO. (U.S.P. 1,971,358, 28.8.34. Appl., 8.4.32).—A gastight connexion between a rotary cylindrical furnace and a stationary boiler (or other utilisier of heat) is described. B. M. V.

**Preparation of [thermal] insulation.** W. H. GITZEN, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,972,188, 4.9.34. Appl., 4.1.33).— $Al_2O_3 \cdot H_2O$  is stabilised by heating at  $425-760^\circ$ . B. M. V.

**Manufacture of [thermal] insulating block.** E. R. POWELL, Assr. to JOHNS-MANVILLE CORP. (U.S.P. 1,972,493, 4.9.34. Appl., 29.1.32).—The block is composed of mineral fibre and  $< 5\%$  of stiffening agent, *e.g.*, an aq. suspension of unpeptised starch or the like. The mass is moulded, drained, and heated to peptise it. B. M. V.

**[Thermal] insulating material.** E. A. TOOHEY and E. R. WILLIAMS, Assrs. to JOHNS-MANVILLE CORP. (U.S.P. 1,972,500, 4.9.34. Appl., 26.9.31).—Paper containing asbestos is made up in alternate plain and corrugated sheets held together by a hygroscopic binder, and over all is applied montan wax or other  $H_2O$ -repellent material. B. M. V.

**Filling an absorber-generator [for refrigeration].** E. NOEBEL, Assr. to SIEMENS-SCHUCKERTWERKE A.-G. (U.S.P. 1,972,426, 4.9.34. Appl., 10.12.32. Ger., 14.12.31).—Molten absorbent for a refrigerating medium ( $R$ ) is applied, by dipping, to metallic wool or other good-conducting network, and the mass is then placed in a container ( $C$ ); on admission of  $R$  the mass will swell and entirely fill  $C$ . B. M. V.

**Yielding-jaw crushing machine.** W. W. GUEST, Assr. to M. E. GUEST (U.S.P. 1,972,096, 4.9.34. Appl., 6.12.30).—The fixed jaw is also pivoted and is held in its working position by springs, the beam forming the abutment for which can be adjusted by screwed rods at its ends. B. M. V.

**Separating and extracting apparatus.** H. B. ROSS (U.S.P. 1,972,495, 4.9.34. Appl., 9.1.33).—A conical classifier or Hg trap comprises a truncated, inverted conical vessel having an upright conical or domed bottom, the heavy product being withdrawn from the gutter thus formed through a conical hood by suction. The overflow leaves through an axial conduit and the feed is peripheral. B. M. V.

**Mechanism for separating intermixed divided materials.** K. DAVIS, Assr. to PEALE-DAVIS CO. (U.S.P. 1,971,678, 28.8.34. Appl., 7.2.28. Renewed 11.12.31).—A form of pneumatic shaking table divided into zones having individually regulable supplies of air which is supplied to a common blast chamber beneath all is described. B. M. V.

(A) **Process, (B) apparatus, for centrifugal separation.** J. BERGÉ, Assr. to RAFFINERIE TIRLEMontoise, Soc. ANON. (U.S.P. 1,970,551—2, 21.8.34. Appl., [A] 20.5.30, [B] 10.10.32. Renewed [B] 18.11.33. Ger., [A] 5.3.27. Austr., [B] 9.7.29).—(A) Sugar, *e.g.*, is centrifuged in an atm. which is maintained unchanged, thereby preventing increase in  $\eta$  of the mother-liquor by evaporation, the liquor being withdrawn continuously. (B) In an apparatus for carrying out the above process, the surrounding casing is freely open to atm., as is also the top central inlet of the basket; the latter aperture is provided with a fan which induces an outward axial impulse = the centrifugal effect on the air, thus preventing circulation. B. M. V.

**Spraying of powdered material.** P. P. ALEXANDER, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,971,804, 28.8.34. Appl., 4.12.29).—Hard materials ( $M$ ), *e.g.*, WC and Co, or the like, are heated to a high temp.  $<$  their m.p. and



while in an atm. of inert gas (*G*) are sprayed on to a softer body metal with sufficient force to form a firmly embedded coat. *G* and *M* are supplied under pressure to, and commingled in, a refractory tube which is heated electrically by its inherent resistance.

B. M. V.

**Desiccation of liquids and semi-solids.** W. J. ELSER (U.S.P. 1,970,956, 21.8.34. Appl., 16.5.31).—A method especially applicable to the drying of biological sera comprises subjecting a film thereof, or a froth formed with  $\text{CO}_2$ , to temp. of  $0^\circ$  to  $-2^\circ$  under vac., the vapours being removed to a remote place at  $\geq -70^\circ$ . Reconstitution may be effected by addition of  $\text{H}_2\text{O}$ .

B. M. V.

**Vacuum distillation.** S. P. MILLER, Assr. to BARRETT Co. (U.S.P. 1,972,157, 4.9.34. Appl., 16.1.29. Can., 4.12.28).—A normally solid org. material (*M*) having a tendency to sublime, e.g.,  $\text{C}_{10}\text{H}_8$ , is distilled and condensed in vac., the vac. being produced by a wet pump sealed by a circulated liquid in which *M* is sol., or by liquid *M* alone.

B. M. V.

**Distilling apparatus.** J. E. SCHULZE and T. E. MCARDLE, Assrs. to RED RIVER REFINING Co., INC. (U.S.P. 1,971,355, 28.8.34. Appl., 16.5.22. Renewed 19.12.31).—A dome, vapour box, and draw-off pipes for a horizontal cylindrical still are described.

B. M. V.

**Still and evaporation apparatus.** H. I. LEA (U.S.P. 1,971,492, 28.8.34. Appl., 10.10.32).—Within a single casing, vapour is condensed on a coil cooled by the preheating of cold raw liquid, part of which is then evaporated in a series of troughs, superposed on and arranged around, and forming the wall of, a vertical passage in the lower part of which is a fuel burner.

B. M. V.

**Apparatus for crystallising solutions by evaporation or cooling.** R. BONATH, Assr. to WERKSPOR N.V. (U.S.P. 1,970,732, 21.8.34. Appl., 19.2.32. Holl., 8.1.32).—A trough which may be open or vac.-tight, and is provided with heat-exchanging means or not, is fitted with a longitudinal rotating shaft to which are attached sector-shaped plates, each extending over  $> 180^\circ$ , and the centre line of each being at  $180^\circ$  to that of the next. Additional helical blades are described but not claimed.

B. M. V.

**Evaporation and cooling of liquids.** G. A. CONNELL, T. M. CRAMER, and H. B. CALDWELL, Assrs. to PACIFIC COAST BORAX Co. (U.S.P. 1,972,730, 4.9.34. Appl., 18.9.31).—Hot liquid is delivered into a rotating bulk of cold liquid in a vac. chamber.

B. M. V.

**Control of evaporating processes.** M. HECHT and D. S. MCKINNEY, Assrs. to E. G. CAMPBELL (U.S.P. 1,971,816, 28.8.34. Appl., 30.9.30).—The operation of an evaporator or the blow-down of a boiler is controlled by the conductivity of withdrawn vapour alone, before or after condensation, or by that in conjunction with the conductivity of the conc. liquor.

B. M. V.

(A) **Fluid and liquid separation.** (B) **Liquid and gas separation.** J. P. WALKER, Assr. to G. O. MARCHANT and C. G. WELLS (U.S.P. 1,970,783—4, 21.8.34. Appl., [A] 2.2.31, [B] 18.4.32).—(A) The influent (e.g., oil and gas) is admitted tangentially at mid-height to a cylindrical chamber which is unobstructed below the

inlet, but is provided above with 2 storeys of spiral baffles having, underneath each, conical dishes to collect the oil. (B) Successive separation of  $\text{H}_2\text{O}$  and gas from oil is effected in two stages of tangential whirling, the first under the natural pressure of the oil well to separate  $\text{H}_2\text{O}$  while the gas remains in solution, and the second after expansion through a reducing valve to separate gas. Baffles are provided to produce a scrubbing action.

B. M. V.

**Sampling apparatus [for liquids].** W. J. CORTON, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,970,597, 21.8.34. Appl., 22.5.31).—Liquid in a main tank is kept agitated by means that produces some rotation of the bulk; the rotation causes circulation in a by-pass (*B*) which has tangential inlet and outlet for the purpose. *B* is also provided with a pair of isolating valves, and, between them, a drain plug through which a sample may be withdrawn from time to time, or, if the plug is upright, a test-paper may be inserted in the cup thus formed.

B. M. V.

**Viscosity indicating device.** R. J. HAUG (U.S.P. 1,972,736, 4.9.34. Appl., 22.5.29).—The liquid is allowed to emerge from a const.-head tank through a jet having a sufficient amount of wall friction and impinges on a suitably curved vane (having a pointer) restrained by a spring the resistance of which decreases with rise of temp.

B. M. V.

**Apparatus for purification of [mixed] gases.** E. C. KENDALL (U.S.P. 1,970,700, 21.8.34. Appl., 13.12.30).—For the purification of, e.g.,  $\text{N}_2$  from traces of  $\text{H}_2$  and  $\text{O}_2$ , a tube is provided with one or more rolls of Cu gauze heated internally by an electrical resistance wound on a  $\text{SiO}_2$  rod.

B. M. V.

**Condensation of [corrosive] vapours.** G. B. COUBROUGH, Assr. to LUMMUS Co. (U.S.P. 1,970,993, 21.8.34. Appl., 1.12.30).—Corrosive vapours arising from the rectification of mixtures of steam and crude oil are treated in a contact tower, concurrent downwards, with  $\text{NH}_3$  and sufficient cold oil completely to condense the  $\text{H}_2\text{O}$ , which is separated, and part of the oil is then cooled and used again.

B. M. V.

**Expanding high-pressure gas.** R. D. BOOTH, J. R. COFFIN, and A. J. TIGGES, Assrs. to JACKSON & MORELAND and to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,971,518, 28.8.34. Appl., 8.9.32).—After pre-cooling by heat exchange, if desired, but prior to expansion, a substance (*S*), e.g., EtOH, which lowers the f.p. of  $\text{H}_2\text{O}$  is added, the gas (and vapour) is then cooled to not much  $< 0^\circ$ , and the  $\text{H}_2\text{O}$  and *S* are removed as a liquid.

B. M. V.

**Production of air-foam for fire-extinguishing purposes.** J. B. TREICHEL, Assr. to MACANDREWS & FORBES Co. (U.S.P. 1,971,997, 28.8.34. Appl., 22.7.32).—Foam is formed from air,  $\text{H}_2\text{O}$ , liquorice extract, and, as stabiliser, 1—50 (25)% of the oxides or hydroxides of Ca, Ba, or Sr.

B. M. V.

**Manufacture of (A) asbestos board, (B) friction elements.** I. J. NOVAK, Assr. to RAYBESTOS-MANHATTAN, INC. (U.S.P. 1,971,162—3, 21.8.34. Appl., [A] 17.2.32, [B] 2.5.32).—(A) Asbestos board is manufactured in a similar way to paper, using a sizing material (e.g.,



causticised starch, glue, or casein) insol. in  $H_2O$  containing free sol. alkali (NaOH) to  $p_H$  8—10 and adsorbed in the asbestos. (B) A composition of asbestos and a drying oil is treated, after curing, with a solvent capable of removing free fatty acids, and with Mg soaps to render the coeffs. of friction (wet and dry) more alike.

B. M. V.

**Friction facing.** R. J. NORTON, Assr. to BENDIX BRAKE Co. (U.S.P. 1,971,618, 28.8.34. Appl., 11.12.30).—Inorg. salts of low m.p. and high latent heat of fusion are incorporated in a fibrous lining to form a reversible heat absorbent.

B. M. V.

**Salt filter.**—See VII. Measuring colours etc. of fluids.—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**British coals: their analyses and uses.** ANON. (Fuel Econ., 1935, 10, 749—756, 815—818).—Analyses etc. of various types and grades of coal and coke marketed by Horden Collieries, Ltd.; Lothian Coal Co., Ltd.; Lambton, Hetton, and Joicey Collieries, Ltd.; Ocean Coal Co., Ltd.; Ebbw Vale Steel Iron & Coal Co., Ltd.; and John Bowes & Partners, Ltd., are tabulated.

A. B. M.

**Relation of microscopical composition of coal to chemical, coking, and by-product properties.** G. C. SPRUNK and R. THIESSEN (Ind. Eng. Chem., 1935, 27, 446—451).—A chemical analysis of coal-forming constituents is given, and a microscopical study of column samples of various American coals made.

E. S.

**High-temperature carbonisation of coal. Effect of free space above the charge on yields and properties of gases and tars.** J. D. DAVIS and S. AUVIL (Ind. Eng. Chem., 1935, 27, 459—461).—Tests were made on coals (sized 1—2 in.) in a vertical, 18-in. welded-steel cylindrical retort at a const. carbonising temp. of 900° with a free space over the charge varying between 3.9 and 23.1% of the vol. of the retort (3.82 cu. ft.). The yield of gas increased by 3.3 vol.-% and that of light oil from 2.49 to 3.41 gals./ton when the exposure in the free space was increased from 1.3 to 9 sec. Paraffins were practically eliminated and the yield of  $C_6H_6$  increased from 1.4 to 2.7 gals./ton.

E. S.

**Behaviour of sulphur during coal carbonisation.** G. THIESSEN (Ind. Eng. Chem., 1935, 27, 473—478).—Approx. 50% of the S in a coal remains in the coke on carbonisation. Due to the loss of volatile matter, the amount of S in the coke is about 80% of that in the coal from which it was prepared. The almost direct proportionality between coke-S and total coal-S results from the fact that approx. 45% of the org. S and 60% of the pyritic S remain in the coke. The S retention of coke is increased by the presence of Fe compounds, other than  $FeS$ , resulting either from infiltration or oxidation of  $FeS$ .

E. S.

**Carbonisation of low-grade and waste wood.** C. G. SCHWALBE and K. E. NEUMANN (Angew. Chem., 1935, 48, 207—212).—By heating wood at 180° under pressure ( $> 10$  atm.) in contact with aq. solutions of salts which hydrolyse to produce free acid, e.g.,  $MgCl_2$ ,

or of neutral salts to which a little acid has been added, the material undergoes "wet carbonisation" with the production of  $MeOH$  (I) and  $AcOH$  (II). Carbonisation is completed in about 8 hr. The wood of deciduous trees gives smaller yields of (I) but higher yields of (II) by this process than by the normal dry distillation; the wood of conifers gives higher yields of (I) and (II). The charcoal produced has good adsorptive properties, and is particularly suitable for use in suction-gas plants. The mechanism of the process is discussed. Similar results have been obtained by carbonisation at 180—200°/1 atm. of wood previously soaked in the salt solution.

A. B. M.

**Effect of ash on combustion characteristics of carbons.** Y. OSHIMA and Y. FUKUDA (Ind. Eng. Chem., 1935, 27, 212—217).—An electrical method of continuously weighing samples of coke and charcoal during combustion enables ignition temp. and rate of combustion to be determined. Removal of ash from coke with HCl and HF has little effect, the ash merely acting as a diluent. If ash is thus removed from either high- or low-temp. charcoal, however, the ignition temp. is raised. The catalytic behaviour of charcoal ash in promoting ignition may be due to its fine state of dispersion. A similar effect was obtained by salts of K and Na, especially KOH and NaOH, to ash-free wood charcoal. (Cf. B., 1934, 706.)

C. I.

**Analysis of by-product gas of coal liquefaction.** H. OTSUKA (J. Soc. Chem. Ind., Japan, 1935, 38, 135—138 B).—Gas from the hydrogenation of coal and low-temp. tar consists of  $H_2$  (90%) and saturated hydrocarbons. The latter, except  $CH_4$  and some  $C_2H_6$ , were separated by adsorption on active charcoal and analysed in a modified Podbielniak apparatus. The total hydrocarbon gas from coal liquefaction contains, approx.,  $CH_4$  85,  $C_2H_6$  7—11,  $C_3H_8$  1—4, butanes 1—2, and pentanes 1%. The adsorbed hydrocarbon gas from low-temp. tar hydrogenation contains  $C_2H_6$  7,  $C_3H_8$  40, butanes 31, and pentanes 22%.

F. M. E. C.

**Determination of naphthalene in gases.** V. FUNK (Gas- u. Wasserfach, 1935, 78, 263—264).—See Baum and Oppelt's results have been confirmed (cf. B., 1934, 530; 1935, 341). The picric acid method gives accurate results only if the solution is cooled to  $< 7^\circ$ . Preferably it is cooled to  $0^\circ$  since that temp. is more readily maintained than is  $4-7^\circ$  and the results are unaffected by the further cooling. If a measured quantity of the filtrate is titrated a correction must be made for  $H_2O$  condensed from the gas; if preferred, the ppt. can be decomposed and titrated.

A. B. M.

**Coal bitumens. III. Extraction of various types of coal with dioxan at ordinary pressure.** D. J. W. KREULEN and H. P. DEN OTTER. IV. Determination of the bitumen content of various coals and of the micellar phase of the xylene pressure extract. D. J. W. KREULEN (Chem. Weekblad, 1934, 31, 758—761, 761—766; cf. B., 1935, 131).—III. Results are given for the extraction for 24 and 48 hr. of coals containing 15—81% of volatile matter and 0.5—14% of  $H_2O$ . On treatment with  $Et_2O$  the extract yields a bitumen (61—95% of the total) and a



micellary phase containing humic substances; the quantity of extract obtained is not  $\propto$  the bitumen present, but is largely influenced by the hardness of the coal. The humic constituents of soft coals are more readily dispersed than are those of hard coals. The increase of yield with increasing time of extraction is due largely to the dispersion of  $C_5H_{12}$ -insol. protected humic compounds.

IV. Details are given of modified methods of low-temp. distillation, in which the tar and  $H_2O$  are collected separately and the phenols may be determined accurately without distillation with xylene, and of pressure extraction at  $275^\circ$  with xylene followed by separation of the bitumens by means of light petroleum. Results for various grades of coal are recorded. The bitumens (3–57% of the coal) are of approx. uniform properties, and all yield about 80% of tar. The phenols and  $H_2O$  are derived from the humic constituents of the coal. The yield of tar obtained from the non-bituminous constituents confirm the view that these consist of protected humic compounds, of which the properties largely control the behaviour of the coal on coking.

H. F. G.

#### Increasing the yield of benzol from coke ovens.

F. ROSENDAHL (Petroleum, 1935, 31, No. 12, 4–6).—Published methods for increasing the yield of benzol without at the same time increasing that of coke are summarised.

C. C.

Velocity and heat of reaction in cracking [of petroleum oils]. E. L. LEDERER (Petroleum, 1935, 31, No. 12, 1–2).—The velocity of cracking is defined as the reciprocal of the time required to attain a given equilibrium. If  $t$  = time, the velocity coeff. is given by  $k = a/t$ ,  $a$  being a const. depending on the units. The relation between  $k$  and abs. temp. ( $T$ ) is given by  $\log k = -q/RT + C$ , where  $R$  = gas const.,  $q$  = molar heat of reaction,  $C$  = const. From the results of Sachanen,  $q = 67,200$  g.-cal./mol. and  $C = 19,230$ . These figures were confirmed by comparing calc. and experimental results. The heat of reaction can be taken as approx. double the mol. cohesion, or about  $1\frac{1}{2}$  times the heat of sublimation (*i.e.*, the sum of the heats of fusion and vaporisation).

C. C.

Synthesis of benzene from carbon monoxide and hydrogen under ordinary pressure. XXIII. Extraction and washing of alloy catalysts. XXIV. Influence of grain size, hydrogen treatment, and oxidation of alloy catalysts. XXV. General properties and constituents of benzene prepared by alloy contact. S. TSUNEOKA and Y. MURATA (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1935, 27, 13–22, 23–31, 32–38; cf. B., 1935, 132).—XXIII. The results of Fischer and Meyer (A., 1934, 389) are confirmed. Si is best removed from Ni-Co-Si by boiling for 24 hr. with the theoretical amount of NaOH. Traces of NaOH remaining in the Ni-Co catalyst, which increase  $CH_4$  production, can be extracted by EtOH. More benzene is obtained at a lower temp. if the catalyst mass be increased to 25 g.

XXIV. The activity (I) decreases with the particle size. Treatment with  $H_2$  above  $220^\circ$  decreases (I), the hydrogenation (I) diminishing more rapidly than the

polymerisation (I). Oxidation either before or after  $H_2$  treatment inhibits (I).

XXV. Contact benzene contains 90.5% of paraffins and 9.5% of olefines. 75% distils below  $220^\circ$  in fractions  $30-40^\circ$ ,  $60-70^\circ$ ,  $90-100^\circ$ ,  $120-130^\circ$ , and  $140-150^\circ$ . The content of unsaturated hydrocarbons decreases with rising b.p.

R. S.

Discontinuous fractionating column for separation of benzene, toluene, and xylene from the gasoline fraction of Shukoko crude. V. Conclusion. M. MIZUTA (J. Soc. Chem. Ind., Japan, 1935, 38, 160–161 B; cf. B., 1935, 392).—A column with 25 plates is sufficient for the separation of the gasoline into 3 fractions which contain  $C_6H_6$ , PhMe, and xylene, respectively. The concn. of the hydrocarbons in these fractions can be increased by further fractionation with a similar column.

H. G. M.

Naphtha versus gasoline refining and its effect on the finished product. B. C. DODD (Paint, Oil, and Chem. Rev., 1935, 97, No. 9, 30–32).—Various methods for the fractionation and refining of petrol and naphtha are described, with special reference to the properties of the products.

D. R. D.

Solubility of constituents of mineral oil and tar in liquid hydrogen sulphide. E. TERRES and W. VOLLMER (Petroleum, 1935, 31, No. 19, 1–12).—In a study of the suitability of liquid  $H_2S$  (I) as a selective solvent for refining mineral oil etc. the binary systems of  $H_2S$  with  $C_6H_6$ , PhMe, PhOH, *o*-cresol,  $C_{10}H_8$ ,  $2-C_{10}H_7Me$ , anthracene, phenanthrene,  $C_{18}H_{38}$ , cyclohexane, and quinoline have been examined by constructing f.-p. curves for mixtures of varying composition. Results show that at  $0^\circ$  paraffin hydrocarbons, heterogeneous cyclic compounds, and monocyclic hydrocarbons are miscible in all proportions with (I). All the org. substances examined form only one liquid phase with (I). Double compounds are formed by PhMe, PhOH,  $2-C_{10}H_7Me$ , and quinoline, whilst  $C_{18}H_{38}$  and cyclohexane form mixed crystals with (I).

C. C.

Ebullioscopic method for determining the mol. wts. of non-volatile petroleum fractions. B. J. MAIR (J. Res. Nat. Bur. Stand., 1935, 14, 345–357).—A modification of Cottrell's apparatus is described. Accurate mol. wts. are obtained if the val. of the ebullioscopic const. at infinite dilution is first determined by extrapolation from measurements at different concns. Results are given for  $CHPh_3$  in *iso*- $C_8H_{18}$ , *n*- $C_7H_{16}$ , and  $C_6H_6$ , and for  $Ph_2$  in  $C_6H_6$ .

E. S. H.

Formaldehyde formation by preflame reactions in an engine. Spectroscopic study. L. WITHROW and G. M. RASSWEILER (Ind. Eng. Chem., 1934, 26, 1256–1262).—Under knocking conditions the ultra-violet absorption spectra of the non-inflamed gases in an engine always show bands due to  $CH_2O$ , but two fuels which knock equally do not always have the same max.  $[CH_2O]$ .  $CH_2O$  is often found when knocking does not occur, but disappears if the engine conditions are modified so as to reduce sufficiently the tendency to knock, or if the  $C_8H_{18}$  no. of the fuel is raised sufficiently by means other than adding  $PbEt_4$ .  $[CH_2O]$  is reduced when knocking is suppressed by  $NH_2Ph$ , but not by



$\text{PbEt}_4$ ; it is increased when knocking is induced by  $\text{Pr}^{\text{NO}}_2$  or  $\text{Et}_2\text{O}_2$ . A. G.

**Inhibitors in cracked gasoline. III. Storage stability as related to induction period and critical oxidation potential.** C. G. DRYER, J. C. MORRELL, G. EGLOFF, and C. D. LOWRY, JUN. (Ind. Eng. Chem., 1935, 27, 15—20; cf. B., 1933, 691).—During the storage in bottles of a no. of different kinds of gasoline (I), with and without the addition of inhibitors (II), there is a period of slow increase of gum formation and peroxide no. followed by a rapid increase and loss of  $\text{C}_8\text{H}_{18}$  rating. For (I) of the same kind, with and without (II), the storage life can be correlated to the induction period ( $\text{O}_2$  bomb) except in one case. As far as the (II) studied allow, the previous conclusion of the parallelism existing between their inhibiting effectiveness and their crit. oxidation potential (*loc. cit.*) is confirmed.

D. K. M.

**Action of bleaching earths [on benzine].** E. ERDHEIM and O. SCHNEIDER (Petroleum, 1935, 31, No. 16, 1—3).—The action of a no. of bleaching earths (I) in decolorising a 2-year-old sample of Boryslaw benzine has been studied and adsorption isotherms were constructed according to Freundlich's formula. In two cases (fuller's earth and bentonite) the results are not linear. The effect of boiling the spent (I) with  $\text{EtOH}$  varies with different (I), and no definite conclusions could be drawn as to the nature of the decolorising process. C. C.

**Lubrication of internal-combustion engines with olive oil. II. Use of demargarinated olive oil.** A. BASTET (Bull. Soc. d'Encour., 1935, 134, 169—175).—Demargarinated olive oil (I) gave satisfactory performance as lubricant. Dilution has no adverse effect since (I) containing 10% of gasoline (II) functioned normally. Antifriction metal (Sn 87.2, Sb 9.8, Cu 1.2, Al 1.3%) was not attacked by (I) with an acidity of 5%. Neutralised first-quality expressed (I) was satisfactory as an upper-cylinder lubricant, being completely miscible with (II) from  $-10^\circ$  to  $80^\circ$ . Providing the acidity is  $< 5\%$ , (I) can be graphited. A 3:1 blend of mineral oil and (I) gave better results than either constituent alone.

C. C.

**Lubrication as affected by physical properties of lubricants.** R. C. WILLIAMS (Ind. Eng. Chem., 1935, 27, 64—66).—Work done in overcoming friction in the drawing of wire through a die using various lubricants was determined, the work required for deformation being calc. from the tensile strength. Various waxes are most effective as lubricants at temp. a little  $<$  the m.p., but good adhesion of the wax to the wire is important. It is concluded that the lubricant melts between the surfaces during shearing. The coeff. of friction depends on the  $\gamma$  and thickness of film, rather than on the chemical nature of the lubricant or surfaces. C. I.

**Composition of the solid paraffin formed in the Fischer-Tropsch benzene synthesis.** H. KOCH and G. IBING (Brennstoff-Chem., 1935, 16, 141—148; cf. B., 1935, 179).—The solid paraffins produced in this process have been fractionated (a) by extraction with suitable solvents, *e.g.*,  $\text{Et}_2\text{O}$ ,  $\text{C}_2\text{H}_2\text{Cl}_2$ ,  $\text{CHCl}_3$ , and (b) by distillation in vac. The m.p. of successive fractions rose

smoothly, from about  $40^\circ$  to  $117^\circ$ , with increasing mean mol. wt., at first rapidly and then more slowly, indicating that the m.p. finally approaches a const. val. It was impossible to isolate definite single compounds, and it appears that the solid paraffins in question consist of mixtures of all *n*-paraffins from  $\text{C}_{20}\text{H}_{42}$  to  $\text{C}_{150}\text{H}_{302}$ , together possibly with some slightly-branched iso-paraffins. A. B. M.

**Fractionating columns. Rate of mixing of gases.**—See I. Sizes for paper.—See V. Determining CO in gases.—See VII. Road-surfacing materials. Waterproofing masonry. Wood decay and coal formation. Inflammability of construction materials.—See IX. Gas meters.—See X. Coal-tar solvents.—See XIII. Ammoniated peat.—See XVI. Sterilisation [of paraffin].—See XX. Oilfield  $\text{H}_2\text{O}$  analysis.—See XXIII.

PATENTS.

**Retort furnaces and the like for the heat-treatment of solid materials.** GAS CHAMBERS & COKE OVENS, LTD., A. V. KEMP, and A. E. MORRIS (B.P. 425,603, 13.7.33).—In carbonising plant comprising intermittent vertical chambers (C) and recuperators (R) constructed of materials having different coeffs. of expansion, *e.g.*,  $\text{SiO}_2$  and fireclay, respectively, the C and R are built as separate integral units and the gas and/or air and waste-gas connexions between them are provided with self-adjusting joints to compensate for the differential thermal expansion. A. B. M.

**Carbonisation of cellulose-pulp waste lyes.** L. KALB and E. STRUPP (B.P. 425,899, 25.8.33).—The waste lyes are conc. and mixed with a powdered solid residue obtained in the process; the mixture is briquetted and carbonised by direct contact with hot combustion gases (G). After separation of the condensable volatile products ( $\text{CO}_2$ , MeOH, tar, etc.) the gases evolved are burned to produce G. The solid product may be burned under a boiler and the residue then returned to be mixed with a further supply of lye. A. B. M.

**Destructive distillation of mixtures of coal and oil.** E. W. BROCKLEBANK and W. B. MITFORD (B.P. 426,645, 6.10.33).—A mixture of oil and a blend of a coking and a non-coking coal is carbonised by the process described in B.P. 393,601—2 (B., 1933, 738). The blend is obtained by mixing a coal having a composition within the range C 80—91% and O 5.5—14.2% with another having a composition outside that range, the proportions of the two coals being such that the composition of the blend lies within that range. The product forms a coherent coke which does not adhere to the walls of the retort. A. B. M.

**Production of activated carbon from powdered coal, peat, and like carbonaceous materials.** R. W. EASTON (B.P. 425,611, 13.9.33).—The material is mixed with air and steam and injected in an upward direction into the upper part of a vertical retort (R), wherein it undergoes partial combustion. It then passes down through an activating zone formed by the lower part of R, which is of reduced diam. The product is discharged into a  $\text{H}_2\text{O}$ -tank. A. B. M.



**Production of activated carbon.** F. H. CONE and C. B. HOULDER (B.P. 425,891, 24.6.33).—Finely-divided bituminous coal, lignite, peat, etc. is introduced into a zone of incandescent gas in such manner that its temp. is raised suddenly to  $> 1000^\circ$  so that it undergoes an extremely rapid carbonisation. Thus it may be introduced into a combustion chamber (C) in suspension in a mixture of gas and air in suitable proportions. The C is extended downwardly to form a zone at a lower temp., e.g.,  $900^\circ$ , through which the materials fall. The activated material and outgoing gases are led to a scrubber wherein they are washed with (acidified)  $H_2O$ .

A. B. M.

**Production of an oleo-emulsion of graphite.** E. RABETRANO (B.P. 425,884, 13.11.34. Fr., 16.11.33).—Graphite (I) oxides obtained by treating (I) with  $HNO_3 + KClO_3$  or  $H_2SO_4 + K_2Cr_2O_7$  etc. are ground with a liquid (A) of low surface tension, e.g.,  $CCl_4$  or light petroleum, and the paste so formed is mixed or ground with mineral or vegetable oils, A being subsequently removed by distillation. The final mixing may be effected in presence of a sol. emulsifying agent, e.g., camphor and amyl alcohol.

A. B. M.

**Destructive hydrogenation of carbonaceous materials.** R. HOLROYD, C. COCKRAM, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 425,679, 19.7.33).—Coal etc. in the form of a paste or suspension in oil is subjected to destructive hydrogenation in the liquid phase under such conditions of temp., time, catalyst, etc. as to produce a large proportion of volatile heavy oils which issue in vapour form from the reaction vessel or the adjacent hot catch-pot. Such heavy oils are separated from the other volatile products and, together with the unconverted solid material, are subjected to further hydrogenation in a second reaction vessel under conditions producing mainly middle oils in addition to light oils. The middle and/or light oils from both stages are hydrogenated in a third stage to produce mainly benzine. Improved yields of benzine and min. gas losses are obtained by this method of operation.

A. B. M.

**Destructive hydrogenation of solid carbonaceous materials.** INTERNAT. HYDROGENATION PATENTS Co., LTD., Assees. of I. G. FARBENIND. A.-G. (B.P. 427,275, 11.9.34. Ger., 29.9.33).—Coal etc. prior to being subjected to destructive hydrogenation is impregnated with a solution of a catalyst, e.g., a compound of Mo or Sn, in an org. solvent, e.g., MeOH,  $COMe_2$ , AcOH, etc.

A. B. M.

**Gas-making with emulsified fuel.** M. W. DITTO, Assr. to GAS FUEL CORP. (U.S.P. 1,970,996, 21.8.34. Appl., 6.3.30).—"Furnace" or other heavy hydrocarbon oil is homogenised with  $H_2O$  and 10–25 vol.-% of air and the emulsion is sprayed into a retort at  $870$ – $980^\circ$ , producing a gas of high calorific val.

B. M. V.

**Purification and separation of gaseous mixtures.** M. SHOELD, Assr. to KOPPERS Co. of DELAWARE (U.S.P. 1,971,798, 28.8.34. Appl., 30.1.32).— $H_2S$  or the like is removed from a gas by a regeneratable absorbent liquid in two stages (I, II); both the saturated liquid from (I) and the unsaturated from (II) are regenerated together by a countercurrent of steam, and a portion of the liquor from an intermediate point of the regenerator is returned

to (I) and the remainder, fully steamed, is returned to (II).

B. M. V.

**Decomposition of sulphur compounds in coal gas, water-gas, producer gas, waste furnace gas, or other combustible power gas, or in by-products obtained in the manufacture thereof.** H. BAKER (B.P. 426,153, 26.1.34).—The gases etc. are treated with  $H_2$ , produced, e.g., by the action of acids on metals or by electrolysis, by being passed into or through the solution wherein the  $H_2$  is being formed.

A. B. M.

**Purification of hydrocarbon compounds.** T. T. GRAY, Assr. to GRAY PROCESSES CORP. (U.S.P. 1,952,855, 27.3.34. Appl., 19.5.30).—Cracked petroleum is freed from unsaturated constituents (I) by passing the vapour under pressure through a tower filled with kieselguhr (K) and maintained at such a temp. that part of the oil condenses and washes out from the K the gummy residue formed by polymerisation of (I).

A. R. P.

**Production of motor fuel.** H. E. POTTS, From INTERNAT. HYDROGENATION PATENTS Co., LTD. (B.P. 427,905, 11.12.33).—A safety motor fuel, b.p.  $130$ – $240^\circ$  ( $150$ – $220^\circ$ ), is obtained by fractionating a hydrocarbon mixture, e.g., crude benzol, and hydrogenating the  $150$ – $240^\circ$  fraction at  $200$ – $390^\circ$  ( $300$ – $390^\circ$ )  $> 20$  ( $> 50$ ) atm. in presence of catalysts, e.g., compounds of heavy metals of group VI.

C. C.

**Production of lubricating oils by heat-treatment of solid carbonaceous materials in presence of hydrogenating gases.** INTERNAT. HYDROGENATION PATENTS Co., LTD., Assees. of I. G. FARBENIND. A.-G. (B.P. 425,814, 13.7.34. Ger., 15.8.33).—Coal, in particular brown coal, is treated with a catalyst, e.g.,  $NH_4$  molybdate or Sn oxalate, and is made into a paste with a high-boiling oil, preferably one produced in the process; this is then mixed with  $H_2$  under pressure ( $100$ – $300$  atm.), preheated to  $350$ – $410^\circ$ , and passed through a reaction vessel giving a sufficient time of contact to bring about a  $> 90\%$  conversion of coal into oil, and a liquid product containing  $< 2\%$  of asphalt. The heavy oil is separated from the product, e.g., by distillation in vac., and is further hydrogenated under pressure, and in presence of catalysts, at  $300$ – $450^\circ$ .

A. B. M.

**Separating liquids and gases. Condensing corrosive vapours.**—See I. Chlorohydrins. Separating *m*-cresol from phenolic mixtures.—See III.

### III.—ORGANIC INTERMEDIATES.

**Zinc oxide–chromium oxide catalysts for methyl alcohol synthesis.** M. C. MOLSTAD and B. F. DODGE (Ind. Eng. Chem., 1935, 27, 134–140).—Catalysts were prepared by pptg. ZnO and/or  $Cr_2O_3$  from their nitrates with aq.  $NH_3$  or  $Na_2CO_3$  at  $85^\circ$ . Conversion tests were made with a space velocity of 25,000 at  $300$ – $450^\circ$ ; results at any temp. were reproducible to  $< 10\%$ . Although in a short test the most efficient catalyst is  $6ZnO, Cr_2O_3$ , such high-Zn catalysts are adversely affected by higher temp.; on the other hand, mixtures such as  $2ZnO, Cr_2O_3$  are improved by high temp. and prolonged use. This is in spite of an error introduced by shrinkage of the catalyst, and resulting in higher space velocities, in the last type. Catalysts prepared from



$\text{CrO}_3$  are much less active. Conversion yields as compared with those found by other investigators are discussed in detail. The liquid product in almost all cases was MeOH of  $> 90\%$  purity, the balance being  $\text{H}_2\text{O}$ .

C. I.

**Catalytic oxidation of ethyl alcohol.** J. A. PATTERSON, JUN., and A. R. DAY (Ind. Eng. Chem., 1934, 26, 1276—1279).—Increasing the pressure in the catalytic oxidation of EtOH over a Ag catalyst diminishes the yield; more  $\text{H}_2$  and hydrocarbons are formed. At atm. pressure the max. process yield is obtained at  $415^\circ$  and at a theoretical ratio of  $\text{O}_2$ ; the max. yield efficiency is obtained at  $370^\circ$  and a lower ratio, but too low a ratio results in temporary poisoning of the catalyst. The addition of  $0.2\%$  of  $\text{Sm}_2\text{O}_3$  to the Ag increases the process yield by 3—4%.

A. G.

**Vapour-liquid equilibrium data for commercially important systems of organic solvents. Binary systems *n*-butyl alcohol-*n*-butyl acetate and *n*-butyl alcohol-acetone.** A. S. BRUNJES and C. C. FURNAS (Ind. Eng. Chem., 1935, 27, 396—400).— $d^{25}$  for  $\text{Bu}^\circ\text{OH}-\text{Bu}^\circ\text{OAc}$  mixtures (I),  $d^{25}$  and  $n$  for  $\text{Bu}^\circ\text{OH}-\text{COMe}_2$  mixtures (II), and vapour-liquid equilibrium data for (I) and (II) have been determined. D. K. M.

**Organic acids giving oil-soluble metallic salts. Relationship between structure and solubility.** H. A. BRUSON and O. STEIN (Ind. Eng. Chem., 1934, 26, 1268—1271).—The Pb, Co, and Mn salts of polycarboxylic acids are insol. in petroleum naphtha (I), but dissolve in hot drying oils (II) as a result of double decomp. Those of *n*-fatty acids (III) are poorly sol. in linseed oil and exhibit a tendency to gel formation, whereas those of ketonic acids,  $\text{R}\cdot\text{CO}\cdot[\text{CH}_2]_n\cdot\text{CO}_2\text{H}$ , increase in solubility in oil as R increases in mol. wt.; when R has 25 C atoms and  $n = 3$ , the product is an oil-sol. resin. The Pb, Co, and Mn salts of the aryloxyacetic acids are sol. in (I) if the aromatic nucleus contains a 4-C chain in the *p*-position (lower homologues are insol.), or is reduced, or if aryl =  $\text{CH}_3\text{Ph}$ . The series  $\text{RO}\cdot[\text{CH}_2]_n\cdot\text{CO}_2\text{H}$  gives easily sol. salts when R has at least 4 C and  $n = 1-3$ . The solutions have a lower  $\eta$  than those of (III).  $o\text{-C}_6\text{H}_4(\text{CO})_2\text{O}$  with many hydrocarbon derivatives gives  $\text{R}\cdot\text{CO}\cdot\text{C}_6\text{H}_4\cdot\text{CO}_2\text{H}$  (R is aliphatic or aromatic), the Pb, Co, and Mn salts being sol. in aromatic hydrocarbons and hot (II). Alkyl groups increase the solubility, which is also dependent on the mol. configuration of R rather than on its size. Unsaturated acids do not afford pure salts. The Pb, Co, and Mn alkylated benzoates are more oil-sol. than those of the arylated ones. When alkyl = amyl, optimum solubility is obtained, but *sec*-alkyl groups give solutions of lower  $\eta$ ; the higher homologues give soaps. When Bz is *ortho* to  $\text{CO}_2\text{H}$ , more marked siccativ properties are observed. Co 2-hydroxy-5-isopropylbenzoate dries linseed oil as fast as Co resinate in spite of the oxidation-inhibiting properties of OH.

J. L. D.

**Determination of traces of nitro-compound in aniline or *o*-toluidine.** S. UENO and T. SUZUKI (J. Soc. Chem. Ind., Japan, 1935, 38, 140—142 B).—The  $\text{NH}_2\text{Ph}$  (I) containing traces of  $\text{PhNO}_2$  (II) [or  $o\text{-C}_6\text{H}_4\text{Me}\cdot\text{NH}_2$  (III) with traces of  $o\text{-C}_6\text{H}_4\text{Me}\cdot\text{NO}_2$  (IV)] is distilled in vac. and then compared colorimetrically

with  $\text{K}_2\text{Cr}_2\text{O}_7$  solutions of various concns. The colour of the (I)-(II) mixture  $\propto$  [(II)]. The % of (II) is given in terms of the concn. of the  $\text{K}_2\text{Cr}_2\text{O}_7$  solution with which it matches. In a second method, dil. HCl is added to (I) containing a trace of (II) and half of the liquid is distilled over. More HCl is then added to the distillate, which is again distilled. The resulting distillate contains (II) free from (I). The (II) is then determined by reduction ( $\text{Zn}-\text{HCl}$ ) to (I), which is titrated at  $0-5^\circ$  with standard  $\text{NaNO}_2$  in presence of HCl and KBr (starch-iodide paper as indicator). Traces of (IV) in (III) can be similarly determined. H. G. M.

**Determination of dyestuff intermediates by coupling. I. Selection of diazo solutions for coupling.** S. UENO and H. SEKIGUCHI (J. Soc. Chem. Ind., Japan, 1935, 38, 142—144 B).—*m*-Diamines (I),  $\beta\text{-C}_{10}\text{H}_7\cdot\text{OH}$ , and naphtholsulphonic acids are best titrated with a diazo solution of  $p\text{-C}_6\text{H}_4\text{Cl}\cdot\text{NH}_2$  (II), whilst for aminonaphtholsulphonic acids a diazo solution of  $p\text{-C}_6\text{H}_4\text{Me}\cdot\text{NH}_2$  (III) should be used. 25 c.c. of the diazo solution (IV) at  $0^\circ$  are run by means of a previously cooled pipette into the coupling solution, which should be equiv. to 25—26 c.c. of (IV). The end-point is then reached by adding (IV) from a 1-c.c. pipette graduated in 0.01 c.c., and is detected by a method involving the use of H-acid. [A modified method is described for the titration of (I).] A 0.1*N*-diazo solution of (III) decomposes at  $0^\circ$  at the rate of 0.017% per hr. for 7 hr. after prep., whilst that of (II) remains almost unchanged at  $0^\circ$  for 6 hr. H. G. M.

**Determination of resorcinol by nitrosation.** S. UENO and T. SUZUKI (J. Soc. Chem. Ind., Japan, 1935, 38, 139—140 B).—Resorcinol, to which HCl has been added, may be titrated by means of a standard  $\text{NaNO}_2$  solution at  $0-5^\circ$ , starch-iodide paper being used as indicator:  $\text{C}_6\text{H}_4(\text{OH})_2 + 2\text{HNO}_2 = \text{C}_6\text{H}_2(\text{OH})_2(\text{NO})_2 + 2\text{H}_2\text{O}$ . About 95% of the theoretically required amount of the  $\text{NaNO}_2$  solution must be added within the first 4—5 min., and the remainder dropwise until the end-point is reached. The titration (time occupied about 20 min.) is unaffected by the presence of a small quantity of PhOH or pyrocatechol. H. G. M.

**Org. compounds in power generation.—See I. Determining  $\text{C}_{10}\text{H}_8$  in gas, and mol. wts. of non-volatile petroleum fractions.—See II. Analysis of process solvents.—See V. Solvents.—See XIII.  $\text{Pr}^\circ\text{OH}$  and  $\text{BuOH}$  by fermentation.—See XVIII.**

## PATENTS.

**Manufacture of vinyl ethers.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 427,036, 16.10.33. Addn. to B.P. 369,297; B., 1932, 670).—The process of the prior patent is carried out in the vapour phase, using strongly alkaline, non-volatile catalysts. *E.g.*,  $\text{Bu}^\circ\text{OH}$  and  $\text{C}_2\text{H}_2$  (2 : 1 by vol.) is passed over soda-lime at  $265^\circ$  to give  $\text{Bu}^\circ$  vinyl ether. H. A. P.

**Manufacture of chlorohydrins.** N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 426,519, 21.2.34. U.S., 27.2.33).—Apparatus is claimed in which an excess of aliphatic olefine (I) (*e.g.*, from cracking of petroleum) is passed (at room temp.) in countercurrent to a spray of (or bubbled through)  $\text{HOCl} + \text{HCl}$  [a continuously



circulated aq. solution of  $\text{Cl}_2$  + chlorohydrin (II)]. The aq. (II) formed is dehydrated by distilling with a paraffin [having the same no. of C atoms as (I)].

H. A. P.

**Hydrogenation of  $[\alpha]$ -ethyl- $[\beta]$ -propyl-acrolein [ $\alpha$ -acraldehyde].** CARBIDE & CARBON CHEMICALS CORP., and W. J. TOUSSAINT (B.P. 427,368, 8.5.34).— $\alpha$ -Ethyl- $\beta$ -propylacraldehyde is reduced with  $\text{H}_2$  and Ni (+1 to 5% of Cr) in the liquid or vapour phase at 100–230°/0–900 lb. per sq. in. (190–200°/20 lb. per sq. in.). Under the preferred conditions the main product is  $\beta$ -ethylhexyl alcohol (73% of theory); other products are  $\text{C}_7\text{H}_{16}$  and  $\beta$ -ethylhexaldehyde.

H. A. P.

**Manufacture of substances having wetting, washing, emulsifying, and like properties.** E. A. MAUERSBERGER (B.P. 427,641, 27.10.33. Ger., 2.11.32).—Alcohols having  $\leq \text{C}_8$  are converted into their borates, which are treated with a  $\text{H}_2\text{O}$ -free sulphonating agent ( $\text{ClSO}_3\text{H}$ , oleum). Sulphonation occurs in the case of saturated alcohols (and with unsaturated alcohols also after full addition to the double linking) at the  $\alpha$ -C atom, to give true sulphonic acids. Thus sperm oil alcohols are heated with 10%  $\text{H}_3\text{BO}_3$ , finally to 220°/2 mm., and the product sulphonated with 10% oleum at 45–60°.

H. A. P.

**Separation of *m*-cresol from liquid phenolic mixtures.** MONSANTO CHEM. CO., ASSEES. OF F. COMTE (B.P. 427,246, 17.8.34. U.S., 25.11.33).—Anhyd.  $\text{NaOAc}$  is added to a mixture of cresols in an org. solvent [petroleum, b.p. 90–100° (I)] at 45°, the mixture is allowed to cool, and the cryst.  $\text{NaOAc}$ -*m*-cresol compound filtered off, washed with (I), and heated at the b.p. with (I) to regenerate *m*-cresol.

H. A. P.

**Manufacture of hydroxydiphenylene compounds and derivatives thereof.** I. G. FARBENIND. A.-G. (B.P. 426,403, 3.10.33. Ger., 5.10.32).—The corresponding  $\text{NH}_2$ -compound is diazotised and the diazonium salt heated with  $\text{H}_3\text{PO}_4$  (and Cu). Carboxylation of the OH-compound and conversion into an arylamide by known methods is claimed. *E.g.*, 3-aminodiphenylene oxide, m.p. 128°, is diazotised in technical (? 70%)  $\text{H}_3\text{PO}_4$  and the product added to  $\text{H}_3\text{PO}_4$  at 200–210° in a stream of superheated steam; the 3-OH-compound, m.p. 136°, distils. This is converted into its K salt, which is heated with  $\text{K}_2\text{CO}_3$  and  $\text{CO}_2$  under pressure at 210–220° to give 3-hydroxy-2-carboxydiphenylene oxide, m.p. 293° (4-chloro-*o*-anisidide, m.p. 255°). 2-Hydroxydiphenylene oxide [-3-carboxylic acid, m.p. 245° (4-chloro-*o*-anisidide, m.p. 215°)] and -sulphoxide, m.p. 147°, 3-hydroxydiphenylene sulphide, m.p. 156° (-2-carboxylic acid, m.p. 283°), -sulphoxide (-2-carboxylic acid, m.p. 258°), and -sulphone (-2-carboxylic acid, m.p. 284°), are similarly prepared.

H. A. P.

**Manufacture of mono[hydr]oxychrysene.** SOC. CHEM. IND. IN BASLE (B.P. 427,236, 28.6.34. Switz., 30.6.33).—Chrysene is nitrated ( $\text{HNO}_3$ -AcOH at 100°), the  $\text{NO}_2$ -compound, m.p. 209°, is reduced ( $\text{Sn-HCl-AcOH}$ ), and the  $\text{NH}_2$ -compound, m.p. 199–201°, is heated with an acid (*e.g.*, 10% aq.  $\text{H}_2\text{SO}_4$  or AcOH at 220–225°), or a salt of it is heated with  $\text{H}_2\text{O}$ . The OH-compound has m.p. 240°.

H. A. P.

**Manufacture of intermediates for dyes.** IMPERIAL CHEM. INDUSTRIES, LTD., and E. G. BECKETT (B.P. 427,257, 18.8.33).—4-Chloro-5-nitrophthalic anhydride is condensed with a hydrocarbon or halogenated hydrocarbon of the  $\text{C}_6\text{H}_6$  or  $\text{C}_{10}\text{H}_8$  series ( $\text{AlCl}_3$ ), and the product reduced [ $\text{Fe}(\text{OH})_2 + \text{NH}_3$ ]. The prep. of 4(or 5)-chloro-5(or 4)-nitro-2-benzoylbenzoic acid (I), the corresponding  $\text{NH}_2$ -derivative (cyclised by  $\text{H}_2\text{SO}_4$  to 3-chloro-2-aminoanthraquinone), and Cl-derivatives of (I) (from  $\text{PhCl}$  and *o*- $\text{C}_6\text{H}_4\text{Cl}_2$ ) is described.

H. A. P.

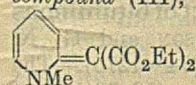
**Manufacture of 1-azanthraquinone and its derivatives.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 427,485, 23.10.33).—Derivatives of  $\beta$ - $\text{C}_{10}\text{H}_7\text{-NH}_2$  unsubstituted in the 3-position and substituted by Cl or a direct C-C linking in the 1-position are condensed (in  $\text{H}_2\text{SO}_4$ ) with glycerol (I) or its Cl-derivatives in presence of a mild oxidising agent ( $\text{PhNO}_2$ ,  $\text{H}_3\text{AsO}_4$ ), and the resulting azanthracene is oxidised (apparently with elimination of the substituent in position 1). *E.g.*, 2:2'-diamino-1:1'-dinaphthyl is heated with (I) and *m*- $\text{NO}_2\text{-C}_6\text{H}_4\text{-SO}_3\text{H}$  in 66%  $\text{H}_2\text{SO}_4$  at the b.p., and the 9:9'-diazanthracyl, m.p. > 350°, oxidised by  $\text{CrO}_3$ -AcOH (III) to azanthraquinone (II). Use as starting material of 2:1:6- $\text{NH}_2\text{-C}_{10}\text{H}_5\text{ClBr}$  gives 6-bromoazanthraquinone, m.p. 268°, but *ms*-chloroazanthracene, obtainable from 2:1- $\text{NH}_2\text{-C}_{10}\text{H}_6\text{Cl}$ , is oxidised by (III),  $\text{MnO}_2\text{-H}_2\text{SO}_4$ , or  $\text{PbO}_2$  to (II).

H. A. P.

**Manufacture of 1:2:2':1'-anthraquinono-anthraquinones.** I. G. FARBENIND. A.-G. (B.P. 427,327, 19.10.33. Ger., 20.10.32).—A 1:5-diaroyl-2:6-dimethylnaphthalene is fully chlorinated in the Me groups and the  $\text{Cl}_6$ -compound heated with conc.  $\text{H}_2\text{SO}_4$ . *E.g.*,  $\text{Cl}_2$  is passed into 2:6:1:5- $\text{C}_{10}\text{H}_4\text{Me}_2\text{Bz}_2$  in  $\text{C}_6\text{H}_3\text{Cl}_3$  in ultra-violet light at 200° and the  $\text{Cl}_6$ -compound heated with conc.  $\text{H}_2\text{SO}_4$  at 150–180° until HCl ceases to be evolved. Similarly, from 1:5-bis-2'-chlorobenzoyl-2:6-dimethylnaphthalene 8:8'-dichloro-, and from 1:5-bis-2':5'-dichlorobenzoyl-2:6-dimethylnaphthalene 5:8:5':8'-tetrachloro-1:2:2':1'-anthraquinonoanthraquinone, m.p. 417°, are obtained.

H. A. P.

**Manufacture of pyridine compounds.** CHEM. FABR. VON HEYDEN A.-G. (B.P. 427,498, 26.10.33. Ger., 28.10.32).—A pyridinium salt having in the 2- or 4-position a halogen or a group capable of replacing halogen (*e.g.*, OH, OAlk, SH,  $\text{SO}_3\text{H}$ ) as substituent is condensed with a compound containing a reactive CH,  $\text{CH}_2$ , or Me group under the influence of basic catalysts (alkali or alkaline-earth alkoxides, hydroxides, carbonates, or metals). *E.g.*, 2-chloropyridine methosulphate (I) and  $\text{CH}_2(\text{CO}_2\text{Et})_2$  (II) with NaOEt in EtOH give the compound (III), m.p. 80–83° (*picrate*, m.p. 95–96°),



(III)

also obtained by use of the methosulphates of 2-hydroxy-, 2-ethoxy-, and 2-sulpho-pyridine, and of *N*-methyl- $\alpha$ -pyridone and  $\alpha$ -thio-pyridone. A similar compound, m.p. 114–115° (*picrate*, m.p. 114–115°), is obtained using  $\text{CH}_2\text{Ac-CO}_2\text{Et}$  in place of (II). Other examples are the condensation of 2-ethoxypyridine methosulphate with fluorene to give a 9-fluorenylpyridinium metho-salt (*picrate*, m.p. 179–180°) and with  $\text{COPhMe}$  to give a base (*picrate*, m.p. 125–126°), and of *N*-methyl- $\alpha$ -quinolone and 4-bromo-



pyridine methosulphates with (II) to give *compounds*, m.p. 118—120° and 164—165°, respectively.

H. A. P.

**Compositions for heat transmission etc.**—See I. **Disinfectants.**—See XXIII.

#### IV.—DYESTUFFS.

**Adsorption and dyeing processes.** XII. **Chromatographic adsorption analysis as applied to aqueous solutions of synthetic organic dyes.** P. RUGGLI and P. JENSEN (Helv. Chim. Acta, 1935, 18, 624—643).—Chromatographic analysis can be applied to the examination of the purity or homogeneity of individual dyes. Most technical dyes and those which have not been subjected to special purification yield several zones or filtrates the no. of which usually increases with the no. of operations involved in the manufacture of the dye. The product from any particular zone is pure after a further chromatographic adsorption (*A*). The separation of mixtures of dyes which exhibit differences in adsorptive behaviour can be effected analytically. For both purposes the chromatographic method is much superior to the customary methods, the degree of separation being more satisfactory owing to the displacement of one dye by another from its *A* zone. In the case of azo dyes the no. of azo groups, and in that of polymethine dyes the no. of vinyl groups, is the deciding factor; in both cases in the analogous series *A* is parallel to the deepening of colour. In the  $\text{CHPh}_3$  group *A* is somewhat related to the size of the mol. In the fluorescein series, introduction of halogen increases *A* ( $\text{I} > \text{Br} > \text{Cl}$ ). In many cases an approx. parallelism exists between *A* and substantivity towards cotton wool, but exceptions are encountered. Comparison of *A* by  $\text{Al}_2\text{O}_3$  with rate of diffusion through gelatin shows in individual cases that slowly-diffusing dyes are, in general, more readily adsorbed, but a pronounced parallelism does not exist.

H. W.

**Flavinduline and its derivatives.** K. YAMADA and T. NOGUCHI (J. Soc. Chem. Ind., Japan, 1935, 38, 134—135 B).—Flavinduline hydrochloride ( $\text{C}_{26}\text{H}_{17}\text{N}_2\text{Cl}_2 \cdot \frac{1}{2}\text{ZnCl}_2$ ), *hydrobromide* ( $\text{C}_{26}\text{H}_{17}\text{N}_2\text{Br}$ ), and *hydriodide* are described (cf. B.P. 18,374 of 1893; B., 1894, 942). Their m.p. are  $> 285^\circ$ . The colours of the solutions of these substances in conc.  $\text{H}_2\text{SO}_4$  and that of the ppt. obtained from an aq. solution and NaOH are given. These dyes have a good affinity mainly for silk and wool; their fastness to various agents is recorded.

H. G. M.

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

**Moisture absorption of jute.** Effect of bituminous treating mixtures. J. H. INGMANSON and G. N. VACCA (Ind. Eng. Chem., 1934, 26, 1274—1275).—The addition to jute of asphalt-wax mixture (52.7%) or of anthracene oil-coal-tar pitch mixture (40.8%) does not affect the equilibrium  $\text{H}_2\text{O}$  absorption of the jute, or the formation of hysteresis loops, but greatly diminishes the rate of absorption.

A. G.

**Effect of temperature and humidity on absorption of moisture by fibrous materials.** W. WELTZIEN and W. RHEIN (Monatsh. Seide Kunstseide, 1934,

39, 343—349, 390—394; Chem. Zentr., 1934, ii, 3332).—Data are recorded for the  $\text{H}_2\text{O}$  lost or absorbed when samples of the materials were kept over aq.  $\text{H}_2\text{SO}_4$  of varying concn. The effect of temp. was small for acetate silk and greater for viscose and Cu-silks.

H. J. E.

**Injury of cotton fibres by salts.** W. NEUMANN (Melliands Textilber., 1934, 15, 305—310; Chem. Zentr., 1934, ii, 3067).—The action of solutions of  $\text{NaCl}$ ,  $\text{Na}_2\text{SO}_4$ ,  $\text{MgSO}_4$ ,  $\text{MgCl}_2$ , and  $\text{CaCl}_2$  has been investigated. The injurious effect  $\propto$  the  $p_{\text{H}}$  of the salt solution; with conc. solutions other factors become evident.

H. N. R.

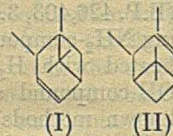
**Analysis of process solvents in leathercloth manufacture.** C. I. COX (J. Soc. Chem. Ind. Victoria, 1934, 34, 909—913).—In a  $\text{C}_6\text{H}_6$ - $\text{COMe}_2$ - $\text{EtOH}$  mixture,  $\text{C}_6\text{H}_6$  is determined by washing with  $\text{H}_2\text{O}$ ,  $\text{COMe}_2$  in the  $\text{H}_2\text{O}$  layer (I) and in a kerosene solution of the  $\text{C}_6\text{H}_6$  by Messenger's method, and  $\text{EtOH}$  either by treatment of (I) with  $\text{K}_2\text{Cr}_2\text{O}_7$  under standard conditions or by fractionation of (I).  $\text{EtOH}$  is determined in  $\text{EtOH}$ -wet nitrocotton by distillation with 10% NaOH.

E. H. S.

**Nitric acid process for recovery of cellulose from bagasse.** J. H. PAYNE (Ind. Eng. Chem., 1934, 26, 1339—1341).—A 32—33% yield of cellulose pulp (93—96% of  $\alpha$ -cellulose) is obtained from bagasse at a cost of  $< \$10.00$  per ton of pulp by digesting for 1 hr. at 90—100° with 2% aq.  $\text{HNO}_3$ , boiling for 45 min. with 2% aq. NaOH, bleaching, and digesting for 30 min. with 5% aq. NaOH.

A. G.

**Resin acids of spruce and their fate in the manufacture of sulphite cellulose.** EMDE (Cellulose-chem., 1935, 16, 13—22).—Sulphite-liquor lactone may be obtained from  $\text{Et}_2\text{O}$  or  $\text{C}_6\text{H}_6$  extracts of the liquor, and Erdtman's formula is accepted. It is identical with tsugaresinol from Japanese hemlock spruce, and the common name tsugalactone is proposed for the two substances. For *l*-pimaric acid the skeletons (I) or (II) are suggested; these account for the val. of the optical rotation and for the isomerisation to abietic acid.



A. G.

**Composition of hemicellulose in the waste soda of viscose factories, and its fermentation.** K. TANEMURA (J. Cellulose Inst., Tokyo, 1935, 11, 105—109).—The waste soda contains 2.19% of hemicellulose (I), composed of 7% of degraded cellulose, 30% of mannan, and 32% of pentosan.  $\text{EtOH}$  ppts. 93% of the (I), Fehling's solution 75%, and  $\text{H}_2\text{SO}_4$  55%; the compositions of these ppts. are recorded.  $\text{CH}_4$ -fermentation bacteria induce  $\text{H}_2$  fermentation in the neutralised soda.

A. G.

**Composition of viscose spinning bath.** K. TANEMURA (J. Cellulose Inst., Tokyo, 1935, 11, 100—105).—The  $p_{\text{H}}$  of the spinning bath may be adjusted by changing the  $[\text{H}_2\text{SO}_4]$  or  $[\text{Na}_2\text{SO}_4]$ ; the best val. is 0.6. When the  $p_{\text{H}}$  is too low the action is too rapid, and the evolution of gas causes poor lustre, irregular structure, rough surface, and mechanical weakness. Too high a  $p_{\text{H}}$  results in a poor affinity for direct dyes.

A. G.



**Stability of sodium cellulose xanthate solutions.**

**I. Action of cations of different valency.** L. MIRLAS (Cellulosechem., 1935, 16, 37—40).—When heavy-metal (M) salts are added to Na cellulose xanthate (CX) solutions MCX is generally pptd., and the ppt. is free from Na. In sufficiently dil. (0.017*N*) solutions coagulation occurs, and is probably due to the displacement of a few Na atoms by M. The swelling val. of  $Mg^{++}$  is of the same order as that of  $Na^+$ . In coagulating power  $Li < Na < K, Mg < Ca < Sr < Ba, Pb < Co[NH_3]_6$ , and in pptg. power  $Na < Mg < Ba < Zn < Th < Co[NH_3]_6, Pb$ . Na is quantitatively displaced by an equiv. amount of Pb, but by Zn only when a slight excess is added.

A. G.

**Viscose. LVI. Change in viscosity and degree of xanthation during ripening. LVII. Viscosity measurements on dilute viscose solutions.** G. KITA, M. SHÔJINO, and I. SAKURADA (J. Soc. Chem. Ind., Japan, 1935, 38, 14—15 B, 15—16 B).—LVI. During the ripening of viscose  $\eta$  falls to a min. and then rises, whereas the degree of xanthation (*D*) rises to a max. and then falls. The min.  $\eta$  and max. *D* occur at approx. the same time. The fall of  $\eta$  is thus not due to loss of xanthate groups, but is caused by increasing dispersion.

LVII. The apparent sp. vol. of the colloidal particles in viscose, calc. from the  $\eta$  of dil. solutions, falls with increasing duration of ripening; it rises to a max. at about 10% NaOH and is increased 2—3-fold by 0.1*N*-NaCl, -KCl, -NaBr, or - $K_2SO_4$ .

A. G.

**Ripeness determination of viscose solutions.**

J. D'ANS and A. JÄGER (Cellulosechem., 1935, 16, 22—36).—When ripeness is determined by the film method the solution should contain 0.02—0.5% excess AcOH, and I is then consumed only in oxidising xanthate to dioxanthate; with less acid other oxidations also occur. In Jentgen's flask method the excess of AcOH should be 0.02—0.05%. Under these conditions concordant vals. for xanthate-S are given by these two I methods and by the gravimetric determination of xanthate-S. Between the I index no. (*N*) and the salt point (*S*) there exists the approx. relation  $N = (30 \text{ to } 35) - 24S/(1.2 + S)$ . The  $NH_4Cl$  ripeness is much more dependent on the composition of the viscose than is *S*, and there is no single curve relating the two quantities.

A. G.

**Two nomograms for calculating the fluidity of cellulose solutions.** J. R. WOMERSLEY (J. Text. Inst., 1935, 26, T 165—170).—Nomograms are described and illustrated for calculating the fluidity from the time of flow in a capillary viscosimeter of the type used at the Shirley Institute, and for converting the fluidity of a 0.5% solution into  $\eta$  of a 2% solution.

A. G.

**Combined manufacture of cellulose pulp from wood and straw.** G. DUPONT and J. DE FAYARD (Bull. Inst. Pin, 1935, 56—59).—The black liquor from either the soda- or sulphate-digestion of wood (pine) is used for the digestion of straw and gives a satisfactory straw pulp. The liquor from this operation may be used further with straw to give a kraft pulp. The significance of the process in the treatment of French pine and straws is discussed.

E. H. S.

**Distribution of sulphur during the kraft pulping operation.**

O. KRESS and J. W. MCINTYRE (Paper Trade J., 1935, 100; T.A.P.P.I. Sect., 225—228).—From a no. of semi-scale experimental cookings of wood by the kraft process, it is concluded that (a) very little S escapes from the digester as non-condensable gases during blowing (conc.  $HNO_3$  is the best absorbent for mercaptans); (b) the amount of S retained by the chips varies with the concn. of the liquor and the temp. at which it is drained off, but is only about 4% (readily removed by washing); (c) practically all the S is present in the black liquor (*L*), the amount being about half of that present in the original white liquor. Methods for determining the S content of *L* are examined. The most accurate is to evolve the sulphide with  $NH_4Cl$ , using  $CdSO_4$  as absorbent. Titration of *L* with ammoniacal  $Zn(NO_3)_2$ , after treatment with  $BaCl_2$ , using ammoniacal  $Ni(NO_3)_2$  as indicator, is satisfactory only when the sample is fresh.

H. A. H.

**Cooking process. VIII. Volatile organic acids [produced] by saponification of aspen wood.**

S. I. ARONOVSKY and R. A. GORTNER (Ind. Eng. Chem., 1935, 27, 451—454; cf. B., 1934, 315).—Aspen sawdust was cooked with  $Na_2CO_3$  for 2 hr. at  $170^\circ$  at various concns. Distillation of the black liquor with  $H_3PO_4$  yielded 4.6—7.0% of volatile org. acids (*A*) (calc. on bone-dry wood), isolated as their Ba salts. Decrease in  $[Na_2CO_3]$  gave less *A*, but change in wood/ $H_2O$  ratio had negligible effects. Production of *A* is attributed to the effects of saponification. The distribution coeff. of these acids between  $H_2O$  and  $Et_2O$  showed that only  $HCO_2H$  and  $AcOH$  were present, in the ratio 1/5.9 to 1/9.4.

D. A. C.

**Principles of Bennett size-making process [for paper].**

O. F. NETZKE (Paper Trade J., 1935, 100; T.A.P.P.I. Sect., 219—211).—The process consists in the continuous saponification and dispersion of thermoplastic materials suitable for sizing paper, including rosins, paraffin, amorphous, and synthetic waxes, montan and carnauba waxes, stearic and oleic acids, asphalts, and certain types of oils. The plant and a range of products are described.

H. A. H.

**Testing of non-fibrous raw materials [for paper-making].**

C. E. PETERSON (Paper Trade J., 1935, 100; T.A.P.P.I. Sect., 249—250).—Results of a questionnaire regarding current American practice in the testing of chemicals, fuel, etc. purchased for paper-making are summarised.

C. I.

**Report for 1934 of the Strength Testing Committee [of the Verein der Zellstoff- u. Papier-Chemiker u. -Ingenieure].**

E. UNGER (Papier-Fabr., 1935, 33, 153—158, 161—165).—The results of comparative tests with the German Standard equipment, viz., the Jokro mill and the Rapid-Köthen (*R*) sheet-making equipment, are discussed. Further, the Jokro mill showed less differentiation between pulps than the ball mill or the Rieth hollander. Experiment indicates that Swedish, British, and German sheets would probably be equally strong if all were dried in the *R* apparatus. Jokro sheets, being unpressed, were too weak for comparison with practice.

D. A. C.



**Contributions to paper microscopy from the Government Material Testing Office, Dahlem, Berlin.** B. SCHULZE (Papier-Fabr., 1935, 33, 165—167).

—(i) The detection of starch formation in genuine parchment paper, (ii) the failure of the Shaffer rapid brazilin method for distinguishing bleached sulphate from bleached sulphite fibres, (iii) the fluorescent differentiation between linters and cotton, with rhodamine, and (iv) the application of the author's method for examining mixtures of groundwood and chemical pulp are described. D. A. C.

**S burner gases for sulphite pulping.**—See VII. **Inflammability of fibrous materials.**—See IX. **Sterilisation [of straw].**—See XX.

#### PATENTS.

**Manufacture of cellulose.** H. DREYFUS (B.P. 427,626, 23.9.33).—Chemical wood pulp (I) is treated successively with dil. alkali solution ( $\frac{1}{2}$ —3%) at 100—200°/ > 1 atm., and then with more conc. alkali (10—20%) at > 35° (10—25°). After washing with H<sub>2</sub>O, (I) is finally treated with an org. acid (AcOH) before use in the manufacture of org. cellulose derivatives. F. R. E.

**Manufacture and use of cellulose derivatives.** H. DREYFUS (B.P. 427,559, 26.10.33).—An org. ester of cellulose (I), after partial ripening in an acid medium in the usual manner, is pptd., and subjected to further ripening by means of an org. base, e.g., NH<sub>2</sub>Me, C<sub>2</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub>, while suspended in a medium (H<sub>2</sub>O, EtOH) which may, if desired, have a swelling action on (I). F. R. E.

**Manufacture of artificial filaments, yarns, horse-hair, films, and like products.** BRIT. CELANESE, LTD. (B.P. 426,912, 1.8.34. U.S., 21.8.33).—A solution of a suitable base (B), e.g., cellulose acetate, containing a solid substance, e.g., a white pigment (TiO<sub>2</sub>) for reducing lustre, a coloured pigment (C black), or a weighting material (compounds of Sn, Pb, or Zn), which has been prepared in a finely-divided form by working it in a plastic mass containing a thermoplastic cellulose derivative similar to or different from B and a softener or solvent, is spun in the usual manner. F. R. E.

**Manufacture of dulled filaments, films, and like artificial products of viscose.** FELDMÜHLE A.-G. (B.P. 426,751, 4.6.34. Ger., 6.6.33).—A paste mixture of TiO<sub>2</sub> with liquid paraffin, pinene, petroleum, benzene, C<sub>6</sub>H<sub>6</sub>, tetralin or decalin, and sulphonated mineral oil is emulsified with viscose, homogenised, and spun in the usual manner. F. R. E.

**[Manufacture of] threads, ribbons, tubes, and the like from polyvinyl compounds.** CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 427,106, 16.10.33. Ger., 17.10.32).—The articles are made, under sterilising conditions at > 100° if desired, from polyvinyl compounds which are free from OH radicals or do not contain them exclusively, with the addition of other materials for special medical or surgical purposes, e.g., KCNS or sugar to increase their strength, gelatin, H<sub>2</sub>O-sol. gums, org. or inorg. electrolytes for promoting resorption, or bactericidal or therapeutically active substances. F. R. E.

**Manufacture of impervious paper.** ETABL. COT-TEREAU SOC. ANON. (B.P. 427,267, 12.2.34. Fr., 19.12.33).—Viscose (V) is applied to both sides of the paper web (W) by distributors exerting no pressure on it. W travels freely for natural capillary impregnation, and is then pressed between rollers to remove excess V. After travelling freely again to assume original thickness and to distribute absorbed V uniformly, W is immersed in V-coagulating baths, washed, and dried. D. A. C.

**Asbestos board. Insulating material.**—See I. **Carbonising pulp lyes.**—See II.

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

**Dyeing cotton. Substantive dyes and salt-sensitivity.** S. LENHER and J. E. SMITH (Ind. Eng. Chem., 1935, 27, 20—25).—A micro-method for measuring diffusion phenomena is described and applied to solutions of 12 representative commercial dyes of the stilbene, benzidine, urea, and arylamide types. Diffusion consts. and particle size (I) of the dyestuffs are correlated with the dyeing properties. (I), which is an optimum for dyeing at < 17 × 10<sup>-8</sup> cm. at 25°, and salt-sensitivity (II), which is closely related to substantivity (S), are two important physico-chemical characteristics of substantive dyeing. Fastness (III) to soap is closely related to (I). The dyestuffs tested fall into 3 groups with regard to (III), viz., (A) with micelle radii (IV) < 15 × 10<sup>-8</sup> cm. in 0.6% NaCl solution at 25°, having very poor (III); (B) with (IV) < 17 × 10<sup>-8</sup> cm. in the commercial dyestuff, but > in NaCl solution, having improved (III) especially when dyed at the boil; and (C) with (IV) < 20 × 10<sup>-8</sup> cm. in either condition, having poor (III) when dyed at 25°, but excellent when dyed at 100°. It is more reliable to use (II) as a measure of S than to depend on the presence of so-called substantive groups in the dyestuff mol. S. C. A.

**Cotton dyeing and substantivity.** A. WAHL (Rev. Gén. Mat. Col., 1935, 39, 161—166).—A review of published information. H. A. P.

**Printing of non-chlorinated wool.** E. JUSTIN-MUELLER (Textilber., 1935, 16, 322).—Wool acquires an increased affinity for dyes when suitably treated with NaHSO<sub>3</sub>, and this treatment (I) may replace the usual chlorination in preparing wool fabric for printing. (I) gives satisfactory results only when the wool is sufficiently "NaHSO<sub>3</sub>-active" that a test-piece sinks within 30—50 sec. when placed on the surface of a liquor consisting of 15 c.c. of NaHSO<sub>3</sub> (d 1.32) (II) and 85 c.c. of H<sub>2</sub>O. Wool may be made "NaHSO<sub>3</sub>-active" by boiling it with 4—6% aq. H<sub>2</sub>SO<sub>4</sub>. (I) consists in printing the wool fabric with a colour paste thickened with gum tragacanth and British gum and containing 125—135 g. of (II) per kg., followed by drying, steaming for 1 hr. in dry steam without pressure, and washing. Suitable dyes are described. A. J. H.

**Manufacture of sizing compounds and sizing of viscose and acetate yarns.** E. S. GENSTEIN and V. S. NEWMAN (Rayon and Melliand Text. Month., 1935, 16, 268).—The importance of exact technical control in the prep. and application of sizes is stressed. A. G.



**Silk weighting.** M. CHINN and E. L. PHELPS (Ind. Eng. Chem., 1935, 27, 209—211).—Samples of degummed silk (*S*) were immersed in weighting baths (*B*) containing aq.  $\text{SnCl}_4$  (*d* range 1.0755—1.3499) and the amount of retained weighting (*W*) was obtained by determining (I) the gain in wt. of the samples, (II)  $\text{SnO}_2$  present in the ash, and (III) residual  $[\text{SnCl}_4]$  in *B*. (I) showed a max. of 10.57% and (II) 8.35%, both in *B* of *d* 1.275, although at *d* 1.3499 both (I) and (II) fell to the same level as at *d* 1.0775. (III) shows a progressive decrease up to *d* 1.275, where it tends to be greater after than before immersion of *S*. This is explained by a preferential adsorption of  $\text{H}_2\text{O}$  by *S*. It is deduced that the retention of *W* by *S* from aq.  $\text{SnCl}_4$  of high concn. is a negative, and from that of lower concn. a positive, adsorption reaction. The amount of  $\text{H}_2\text{O}$  absorbed under ordinary atm. conditions by weighted silk is independent of *W*. D. A. C.

**Adsorption and dyeing processes.**—See IV. **Moisture absorption of jute.**—See V. **Textile starch.**—See XVII.

## PATENTS.

**Treatment of cellulose fibre to improve dyeing properties thereof.** DEUTS. HYDRIWERKE A.-G. (B.P. 426,482, 28.9.33. Ger., 28.9. and 12.11.32).—Cellulose is treated with (a) a CO-compound (I) ( $\text{CH}_2\text{O}$ , MeCHO, furfuraldehyde, PhCHO) and a salt of a  $\text{C}_5\text{H}_5\text{N}$  base (II), (b) an acid derivative of (I) and (II), (c) alkyl (aryl, etc.) chloromethyl ethers (III) and (II), or (d) the quaternary salts from (III) and (II). *E.g.*, cotton is treated with  $(\text{CH}_2\text{O})_n$ ,  $\text{C}_5\text{H}_5\text{N}$ , and  $\text{C}_5\text{H}_5\text{N}\cdot\text{HCl}$  (IV) in  $\text{CHCl}_3$  at the b.p., with  $\text{CHMe}(\text{OAc})_2$ , (II), and (IV) at  $120^\circ$ , or with the quaternary salt from  $\text{CH}_2\text{Cl}\cdot\text{OBu}$  and  $\text{C}_5\text{H}_5\text{N}$  (V); as an alternative, cotton or viscose rayon is suspended in (V) containing 10 wt.-% of  $\text{C}_{16}\text{H}_{33}\cdot\text{SO}_3\text{H}$  and gaseous  $\text{CH}_2\text{O}$  is passed in. The fabric is generally softer and shows considerably greater affinity for acid dyes, and the dyed material is faster to washing. H. A. P.

**Dyeing of textiles.** DEUTS. HYDRIWERKE A.-G. (B.P. 427,089, 12.7.33. Ger., 12.7.32 and 28.6.33).—Two components are used so as to form on the fibre a  $\text{H}_2\text{O}$ -insol. salt of a substantive diquaternary  $\text{NH}_4$  salt and a substantive or non-substantive org. acid of  $\text{C}_{10}$  (I), at least one component being a dye. *E.g.*, cotton (II) dyed with Congo-red is after-treated in  $\text{H}_2\text{O}$  with the yellow compound  $(\text{C}_6\text{H}_4\cdot\text{N}_2\cdot\text{C}_6\text{H}_4\cdot\text{NMe}_2\cdot\text{SO}_4\text{Me})_2$  [prepared from benzidine (III)  $\rightleftharpoons$   $\text{NPhMe}_2$  and  $\text{Me}_2\text{SO}_4$ ], or (II) is pretreated with a 1% aq. solution of the dicinnamoyl derivative of 1 : 5 : 3 : 7- $\text{C}_{10}\text{H}_4(\text{NH}_2)_2(\text{SO}_3\text{H})_2$  and dyed with (III)  $\rightleftharpoons$  2 : 7-OH- $\text{C}_{10}\text{H}_6\cdot\text{NMe}_2\text{Cl}$ . Other examples of (I) are synthetic tanning agents, sulphurised phenols, higher fatty acids, and sulphonated oils. H. A. P.

**Production of dyeings on the fibre [ice colours].** SOC. CHEM. IND. IN BASLE (B.P. 427,208, 12.1.34. Switz., 16.1.33).—A diazotised ester of an  $\alpha$ -aminoarylcarboxylic acid is coupled with a diamide of a  $\beta$ -ketomonocarboxylic acid with a diamine of the  $\text{Ph}_2$  series. Examples are: Me or Et anthranilate, 2 : 5 : 1- or 2 : 4 : 1- $\text{NH}_2\cdot\text{C}_6\text{H}_3\text{Cl}\cdot\text{CO}_2\text{Me}$   $\rightarrow$  diacetoacet-*o*-tolidide. Yellow

to orange dyeings fast to light, washing, and boiling aq.  $\text{Na}_2\text{CO}_3$  are obtained. H. A. P.

**Treatment of textile materials.** H. HUNSDIECKER and E. VOGT (B.P. 426,508, 14.12.33. Ger., 14.12.32).—The addition to textile-treatment baths (as wetting or scouring agents, dyeing assistants) of higher *O*-alkylisoureas or their salts is claimed. *Dodecylisourea hydrochloride* is prepared by heating *n*- $\text{C}_{12}\text{H}_{25}\cdot\text{OH}$  (2 mols.) with  $\text{NH}_2\cdot\text{CN}$  (1 mol.) and  $\text{NH}_2\cdot\text{CN}\cdot\text{HCl}$  (1 mol.) at  $60$ – $80^\circ$ . H. A. P.

**Wetting etc. agents.**—See III.

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

**Contact sulphuric acid manufacture.** IV. **Platinum catalyst on magnesium sulphate.** M. MATSUI and K. ODA (J. Soc. Chem. Ind., Japan, 1935, 38, 148—151 B; cf. B., 1935, 492).—The prep. of the catalyst is described and data for conversion rates covering the range  $375$ – $600^\circ$  are recorded. J. A. S.

**Alumina as a catalyst of hydrocyanic acid formation [from carbon monoxide and ammonia].** W. FUCHS and H. VERBEEK (Ind. Eng. Chem., 1935, 27, 410—413).—The best method of preparing an  $\text{Al}_2\text{O}_3$  catalyst for synthesis of HCN is to add aq.  $\text{NH}_3$  (*d* 0.882) to a solution of  $\text{Al}(\text{NO}_3)_3$  at room temp., stir for 30 min., filter, and dry at  $100^\circ$ . The vitreous  $\text{Al}(\text{OH})_3$  shatters when moistened, and if this is carried out in Zr solution an activated granular catalyst of very low gas-resistance is obtained. With an  $\text{NH}_3$  : CO ratio of 1 : 9.4 and at  $570^\circ$  it gives HCN yields of about 50% (on vol. of  $\text{NH}_3$ ) with little loss of  $\text{NH}_3$ . The reaction rate remained const. for 250 hr. and was unaffected by C deposits on the catalyst. C. I.

**Effects of addition of common salt during calcination of limestone.** II, III. T. NODA (J. Soc. Chem. Ind., Japan, 1935, 38, 138—139 B; cf. B., 1934, 716).—Ferruginous impurities were removed from  $\text{CaCO}_3$  by heating (for 5 hr. at  $1000^\circ$ ) in presence of C + NaCl vapour. The reaction  $\text{Fe}_2\text{O}_3 + 6\text{NaCl} \rightleftharpoons \text{Fe}_2\text{Cl}_6 + 3\text{Na}_2\text{O}$  was studied quantitatively and found to be accelerated in presence of CO. J. A. S.

**Presence of manganese in commercial lime and bleaching powder.** J. E. DRIVER and H. A. TURNER (Quart. J. Pharm., 1935, 8, 113—115).—The periodate method (A., 1918, ii, 84) gave contents of 77—137 p.p.m. With bleaching powder- $\text{NaHCO}_3$  preps., the pink colour which slowly develops owing to oxidation of Mn to  $\text{MnO}_4^-$  does not occur when the proportion of  $\text{NaHCO}_3$  is < approx. 30%; the max. colour produced corresponds with only partial oxidation. F. O. H.

**Potassium sulphate from polyhalite.** E. P. SCHOCH (Ind. Eng. Chem., 1935, 27, 467—473).—Earlier processes for the recovery of  $\text{K}_2\text{SO}_4$  from polyhalite (I) by CaO treatment are reviewed. The principal difficulty is to avoid the formation of  $\text{K}_2\text{SO}_4\cdot 5\text{CaSO}_4\cdot \text{H}_2\text{O}$  (II), which is not easily decomposed. This may be avoided by using  $\text{CaCO}_3$ , but a better method is to heat (I) and CaO slurry separately at  $220^\circ$  in an autoclave with 2 compartments, mix at this temp., and cool rapidly to  $100^\circ$ . Sufficient  $\text{H}_2\text{O}$  is added to give a solution containing



13.7 g. of  $K_2SO_4$  per 100 g. of  $H_2O$ . (II) is not formed at  $> 210^\circ$ . It is proposed to work a continuous process with several autoclaves, employing the steam to evaporate  $K_2SO_4$  solution. The reaction product is filtered at  $100^\circ$ , the filter cake mixed with cold  $H_2O$ , and treated with  $CO_2$  (limekiln gas) to form light  $MgCO_3$ . About 10% of the K is in the form of KOH and serves to prevent the formation of Ca scale in the evaporators. The  $CaSO_4$  will be used as plaster.

C. I.

**Action of urea on calcium orthophosphates.** C. W. WHITTAKER, F. O. LUNDSTROM, and J. H. SHIMP (Ind. Eng. Chem., 1934, 26, 1307—1311).—In aq. or EtOH solutions or as mixed powders  $CaH_2PO_4$  and urea react to yield  $Ca_2HPO_4$  (I),  $H_3PO_4$ ,  $CO(NH_2)_2$ , and  $H_2O$ . After storage of the mixed powders for 3 months the reaction had proceeded nearly to completion at 47—65% R.H. and considerable reaction occurred in a dry atm. The reaction results in caking and stickiness, but no stickiness occurs in absence of excess urea. Urea does not react with (I) or with  $Ca_3PO_4$ .

A. G.

**Gas analysis. I. Determination of carbon monoxide with various cuprous chloride solutions.** H. BRÜCKNER and W. GRÖBNER (Gas- u. Wasserfach, 1935, 78, 269—273).—The rate of absorption (*A*) of CO by acid solutions of CuCl (I) increases, but the total absorptive capacity decreases, with increasing [HCl]; the further addition of  $NH_4Cl$  (II) has little effect on *A*. The time-*A* curve of CO by acidified  $Cu_2SO_4$  containing  $\beta\text{-}C_{10}H_7\cdot OH$  (cf. B., 1924, 772) is similar to that for (I) in conc. HCl. The rate of *A* of CO by ammoniacal solutions of (I) is small, and is little affected by the further addition of (II). The best absorptive properties, in respect both of rate of *A* and total absorptive capacity, are exhibited by a neutral solution of (I) containing (II). A solution containing 125 g. of (I) and 265 g. of (II) per litre is recommended for analytical purposes. The reactions underlying the process are briefly discussed.

A. B. M.

**Catalysts for oxidation of ammonia to oxides of nitrogen.** S. L. HANDFORTH and J. N. TILLEY (Ind. Eng. Chem., 1934, 26, 1287—1292).—The addition of Rh (e.g., 10%) to Pt increases the conversion efficiency and diminishes the loss of metal; many other metals were tested, but these were not advantageous. If the catalyst is deposited on a refractory base it is soon lost, and when used as a coating on a refractory base metal it is soon poisoned by the base metal.

A. G.

**Factors influencing the formation and dissociation of sulphur trioxide in sulphur burner gases for sulphite pulping.** B. L. BROWNING and O. KRESS (Paper Trade J., 1935, 100; T.A.P.P.I. Sect., 237—249).—Artificial burner-gas mixtures were made up from  $SO_2$ ,  $O_2$ , and  $N_2$  and passed through heated tubes of various materials and under various conditions as to temp. and  $[SO_2]$ . It is concluded that: increasing  $[SO_2]$  decreases  $SO_3$  formation; rise of temp. from  $650^\circ$  to  $1100^\circ$  reduces  $SO_3$  formation;  $SiO_2$  does not catalyse  $SO_3$  formation, whereas Fe does at lower temp.; at  $1000^\circ$  Fe reacts rapidly with both  $SO_2$  and  $SO_3$ . Special alloy steels have no advantage over ordinary steel for burner construction. A refractory brick lining is desirable. Dew

points for burner gases of various composition are calc.

C. I.

**Technical extraction of protoactinium.** A. V. GROSSE and M. S. AGRUSS (Ind. Eng. Chem., 1935, 27, 422—426; cf. A., 1934, 1319).—Every natural W mineral contains 8 pts. of Pa per 10 pts. of Ra. The residue from Ra extraction by Curie's method contains 300 mg. of  $Pa_2O_5$  per ton. It is treated with hot 25% HCl, the residue (chiefly  $SiO_2$ ) fused with NaOH, the melt leached with  $H_2O$ , and the extract filtered. The residue is dissolved in acid, most of the remaining  $SiO_2$  being pptd., coagulated with steam, and filtered off. The Pa in the acid filtrate is pptd. by addition of Zr salts and  $H_3PO_4$ . The last  $SiO_2$  ppt. contains 70% of the Pa and is redissolved and the Pa recovered as above. The final product is  $ZrP_2O_7$  + graphite + Pa in a concn. of 1 : 5000. This is worked up in the laboratory and, to date, 100 mg. of pure  $Pa_2O_5$  have been prepared.

C. I.

**Catalysts for MeOH synthesis.**—See III. **Steels for  $HNO_3$  [plant].** Attack of metal by S.—See X. **Colour-permanency of iron oxides.**—See XIII. **Fertilisers. Ammoniated peat.**—See XVI. **Sterilisation of  $CaCl_2$ .**—See XX.

## PATENTS.

**Treatment [hydration] of lime.** W. J. KUNTZ (U.S.P. 1,954,211, 10.4.34. Appl., 2.5.28).—Claim is made for an apparatus in which CaO is hydrated with steam or hot  $H_2O$  formed by the heat of reaction and supplied in regulated amounts, the  $Ca(OH)_2$  formed being separated by levigation from the heavier particles of unburnt or unhydrated CaO, which are then ground and returned to the hydrator.

A. R. P.

**Salt filter.** W. A. NEILL, Assr. to DORR CO., INC. (U.S.P. 1,971,792, 28.8.34. Appl., 24.11.30).—In an internal rotary drum filter, provision is made for: (1) quick and easy renewal of filter panels, (2) an internal atm. of warm air, large quantities of which are drawn through the cake, (3) additional heat radiated to the cake from a steam chest, and (4) cleansing of the filter cloth after the blow-off by a backwash with steam.

B. M. V.

**[Black-]ash smelter.** A. W. WAERN (U.S.P. 1,972,116, 4.9.34. Appl., 12.11.32).—Black ash or the like is burned inside a boiler of which the heating surface is composed almost entirely of approx. vertical tubes enclosing the combustion chamber. Tuyères project through the tube wall downwards into a crucible forming the hearth.

B. M. V.

**Production of pure hydrogen.** C. J. WARNEKE, Assr. to ADAMS & WESTLAKE CO. (U.S.P. 1,955,780, 24.4.34. Appl., 27.4.31).—The gas is passed through a chamber in which Na is caused to volatilise by means of a no. of small electric arcs, and then through a filtering medium to remove Na compounds etc.

A. R. P.

**Recovery of sulphur.** R. F. BACON (Assee.) and I. BENCOWITZ (U.S.P. 1,954,279, 10.4.34. Appl., 31.12.30).— $SO_2$  is passed through a mixture of FeS and  $MgSO_4$  at  $600\text{--}800^\circ$ , the residue of MgO and  $Fe_2O_3$  is separated magnetically, and the MgO is made into a slurry with  $H_2O$  and used for absorption of the  $SO_2$  in the gases



remaining from the condensation of the S, the resulting  $MgSO_3$  being used again. A. R. P.

**Separation of sulphur from sulphur dioxide.** H. AHLQVIST (U.S.P. 1,955,722, 24.4.34. Appl., 7.10.31).— $SO_2$  is allowed to react with  $H_2S$  to produce S, part of which is then treated with a hot hydrocarbon oil to produce the  $H_2S$  for the reaction. A. R. P.

(A) **Raising the sulphur dioxide content of a gas.**  
 (B) **Production of sulphuric acid.** W. H. LEVERETT, Assr. to NAT. ZINC Co., INC. (U.S.P. 1,954, 879—80, 17.4.34. Appl., 24.2.31).—(A) Part of the roaster gases (G) from roasting pyrites is passed through a S burner without additional air so as to raise its  $SO_2$  content without increasing its vol., and is then remixed with the remainder of G and passed to the contact plant. (B) In the manufacture of  $H_2SO_4$  by the 3-chamber process, dil.  $H_2SO_4$  instead of  $H_2O$  is supplied to the 2nd and 3rd chambers. A. R. P.

**Purifying mixed gases.**—See I. Electrolytic  $[Cl_2]$  baths.—See XI.

### VIII.—GLASS; CERAMICS.

**Use of red selenium glasses as ceramic colours.** A. GRANGER (Céram., Verre, Email., 1934, 2, 293—294; Chem. Zentr., 1934, ii, 3160).—In place of colouring materials containing Cd, Se, and S the single components can be employed. Glazes are used with a composition similar to that of glasses coloured with Cd and Se, *i.e.*, Pb-free and rich in Zn. H. J. E.

**Action of soft X-rays on glass.** J. CHLOUPEK (Skláf. Rozh., 1933, 10, 123; Chem. Zentr., 1934, ii, 3296).—A gradual coloration occurs which is accelerated at  $100^\circ$ . H. J. E.

**Radiochemical surface determination for glass.** M. HECKTER (Glastechn. Ber., 1934, 12, 156—172; Chem. Zentr., 1934, ii, 3027).—The method depends on measurements of the activity (A) of glass treated with radioactive material. Formulæ are given for calculating the surface from A. H. J. E.

**Microscopical examination of matt electric-lamp bulbs.** A. SALMONY (Skláf. Rozh., 1933, 10, 121—122; Chem. Zentr., 1934, ii, 3027).—Etching of the inside of glass bulbs by HF, fluorides (I), and baths containing (I) has been studied microscopically. Protective films were observed which retarded further etching. H. J. E.

**Determination of ferrous iron in glass.** S. SHINKAI and K. TAKAHASHI (J. Soc. Chem. Ind., Japan, 1935, 38, 133 B).—The source of error affecting the  $KMnO_4$  titration in such determinations is considered to be  $As^{3+}$  resulting from the hydrolysis of  $AsF_3$  (I), which is derived from As in the glass. Good results are obtained if (I) is removed immediately it is formed, *e.g.*, by heating on an air-bath at  $120$ — $130^\circ$  instead of on a water-bath. A. L. R.

**Replacement of borax [in enamels] by sodium phosphate.** VIELHABER (Emailwaren-Ind., 1934, 11, 290—291; Chem. Zentr., 1934, ii, 3027).—The expansion of the enamel is increased. H. J. E.

**Effect of milling admixtures on the m.p. of enamel.** VIELHABER (Emailwaren-Ind., 1934, 11,

289—290; Chem. Zentr., 1934, ii, 3027).—The m.p. is unchanged by additions of  $< 6\%$  of clay, but is increased by larger amounts. 2—4% of  $SnO$  has little effect; larger proportions lower the m.p. Simultaneous addition of clay and  $SnO$  may leave the m.p. unchanged. H. J. E.

**Substitution of enamelled iron vessels for glass beakers.** P. S. KLUNNE (Chem. Weekblad, 1934, 31, 366—367).—Enamelled Fe vessels have given satisfactory service for  $1\frac{1}{2}$  years when used at temp. between  $-10^\circ$  and  $300^\circ$ . Flaking of the enamel does not occur if the film is sufficiently thin. H. F. G.

**Resistance [of ceramic materials] to softening under load at high temperatures.** S. KONDO and S. SUZUKI (J. Soc. Chem. Ind., Japan, 1935, 38, 129 B).—By applying the Clausius-Clapeyron law to explain the lowering of the softening point under load, the following expression is derived:  $l = Ce^{-kp}$ , where  $l$  = softening point,  $C$  = refractoriness,  $p$  = load, and  $k$  is a const. A. L. R.

**Moulding of clay after heating at various temperatures.** R. MORETTI (Corr. d. Ceram., 1934, 15, 221—223; Chem. Zentr., 1934, ii, 3296).—Heating at  $100$ — $500^\circ$  before moulding increases the apparent porosity of the product fired at  $900^\circ$ , and decreases the resistance to breaking of a specimen dried at  $120^\circ$ . H. J. E.

### PATENT.

**Production of fibres and threads from glass, slag, and like meltable materials.** N. V. MAATS, TOT BEHEER EN EXPLOIT. VAN OCTROOIEEN (B.P. 428,433, 17.12.34. Ger., 18.12.33. Addn. to B.P. 373,932).

### IX.—BUILDING MATERIALS.

**Heat and material balances for a rotary cement kiln.** W. N. LACEY and H. WOODS (Ind. Eng. Chem., 1935, 27, 379—382).—Material and heat balances are given together with the methods used for obtaining data and calculating the results. D. K. M.

**Effect of the burning process on the clinker concrete lining of the rotary cement furnace.** K. KRASSOWSKY (Tonind.-Ztg., 1934, 58, 746—747; Chem. Zentr., 1934, ii, 3028).—The resistance is decreased by  $SO_2$  in the furnace gases, and by chemical processes in the mortar (*e.g.*, separation of  $CaO$ ). H. J. E.

**Low-heat cement.** R. F. BLANKS (Cement, 1935, 8, 128—129).—The low-heat cement (I) used for Boulder Dam is nearly identical in physical properties with standard Portland cement (II) except that the fineness of (I) is  $>$  that of (II). (I) has high contents of  $2CaO, SiO_2$  and of  $4CaO, Al_2O_3, Fe_2O_3$ , and low  $3CaO, SiO_2$  and  $3CaO, Al_2O_3$  compared with (II). For standard and mass-curing, the compressive strengths of (I) and (II), at 90 days, are equal, but the temp. rise (A) and heat of hydration (B) of (I) are  $<$  those of (II). A arising from B varies with the sp. heat and thermal diffusivity of the concrete. T. W. P.

**Boulder Dam.** G. R. ROBERTSON (Ind. Eng. Chem., 1935, 27, 242—249).—Constructional details of Boulder Dam are outlined, together with the requirements of the cements used, for heat of hydration and fineness. T. W. P.



**Bleeding of cements.** L. S. BROWN (Ind. Eng. Chem., 1935, 27, 97—102).—Bleeding (*B*) (separation of  $H_2O$  during setting, either at the surface or under pieces of aggregate in concrete, where it causes channeling) decreases with increasing fineness of cement and is also minimised by the use of low  $H_2O$ -cement ratios. It is also controlled by addition to the solid cement of an org. dispersing agent such as "T.D.A." (a mixture containing salts of polymerides of condensed naphthalene-sulphonic acids). *B* tests in which  $H_2O$  separated was measured, and microscopical examination of sections of concrete confirm the view that *B* and " $H_2O$ -gain" under aggregate are the same phenomenon. Photomicrographs show the elimination of the latter as above described. C. I.

**Calcium hydroxide process for preparation of Portland cement.** HESS (Zement, 1934, 23, 445—448; Chem. Zentr., 1934, ii, 3028).—Replacement of  $CaCO_3$  by  $Ca(OH)_2$  produces a crude sludge (*I*) of very high  $H_2O$  content. The process is uneconomical, even after filtering off the (*I*). H. N. R.

**Trass and free calcium hydroxide.** W. WITTEKINDT (Tonind.-Ztg., 1935, 59, 139—142).—The removal of free  $Ca(OH)_2$  in set Portland cement by trass is small at the outset, but increases markedly with age. In mixes with  $Ca(OH)_2$ , the combination in the case of trass is  $>$  in that of blast-furnace slag. T. W. P.

**Relation between composition and properties of Portland cement.** F. M. LEA (Chem. & Ind., 1935, 522—527).—A review.

**Adhesion, capillary force, and hardening [of cement].** K. WÜRZNER (Zement, 1934, 23, 572—575; Chem. Zentr., 1934, ii, 3296).—A discussion. H. J. E.

**Impact-resistance of concrete.** A. GUTTMANN and F. WENZEL (Zement, 1934, 23, 528—534, 545—551; Chem. Zentr., 1934, ii, 3296—3297).—A discussion of the determination of resistance and of factors which influence it. H. J. E.

**Deterioration of concrete structures in alkaline and sea-water.** W. WATSON and Q. L. CRADDOCK (Cement, 1935, 8, 130—140).—A review of the literature. T. W. P.

**Asphalt-concrete surfacing for automobile roads.** HERRMANN (Bitumen, 1934, 4, 172—174; Chem. Zentr., 1934, ii, 3074).—Data on the composition and properties of the road surfaces are recorded. H. J. E.

**Exterior waterproofing materials for masonry.** D. W. KESSLER (J. Res. Nat. Bur. Stand., 1935, 14, 317—343).—The waterproofing effectiveness and durability (over periods up to 13 years) of 32 commercial products and 10 non-proprietary materials have been studied. Wax types were the most durable, but discoloured most masonry materials. Insol. soaps were not very durable, but gave no discoloration. Thinned fatty oils gave fairly satisfactory durability, which was improved by addition of a high-m.p. paraffin. Ordinary varnishes, lacquers, and wax emulsions were unsatisfactory. Treatments based on reaction with the masonry or pptn. of an insol. compound at its surface were not very effective. Effective waterproofing

materials retard the deterioration of masonry due to weathering. E. S. H.

**Identification of wood by chemical means. II. Alkalinity of ash and some simple chemical tests for identification of the coloured woods of the genus Eucalyptus.** W. E. COHEN (Counc. Sci. Ind. Res. Australia, Tech. Paper 15, 1935, 23 pp.; cf. B., 1931, 1051).—Data are recorded of tests applied to 540 samples representing 37 species. These species may be divided into 5 groups of different ash alkalinity. The colour obtained by the addition of  $FeCl_3$  to the aq. extracts divides the species into two classes, giving blue (32) and green (5 species) colours, respectively. Addition of 1%  $NH_3$  followed by 1%  $K_3Fe(CN)_6$  to aq. extracts gives a transient purple colour (21 species), a transient pink colour (9), and no colour (10), there being 3 species which give variable results. Dilution, with an equal vol. of  $H_2O$ , of the EtOH extracts of  $H_2O$ -extracted woods produced turbidity with 8 species and clear solutions with 20, the remaining 9 giving mixed results. The application of these four tests divides the woods into a no. of classes containing only a few species in each, and additional confirmatory tests of limited application are available for further identification. A. G.

**Decay of wood and formation of coal.** E. A. RUDGE (Chem. & Ind., 1935, 499—501).—The author's theory of the decay of wood, based on the data that the presence of  $> 20\%$  of  $H_2O$  is necessary and that it is attended by increase of ash content, is that it is ionic in nature and is specifically produced by  $[HCO_3^-]$ . Mixtures of sawdust,  $H_2O$ , and  $CaCO_3$  were subjected to a current of  $CO_2$  at  $60^\circ$  and a dark floating residue and brown solution were produced. The latter on keeping yielded a humus-like ppt. and contained reducing sugars. Similar results were obtained with purified cellulose. It is probable that this is the process of humification in peat, the stages being: uronic acids, sugars, humus. The humus undergoes repeated polymerisation and dehydration with consequent increasing C content. Ash-free vitrain would be formed from the sol. humic fraction and fusain from the leached insol. complexes. (Cf. B., 1934, 675.) C. I.

**Initial inflammability of [fibrous] construction materials.** G. E. LANDT and E. O. HAUSMANN (Ind. Eng. Chem., 1935, 27, 288—291).—Test-prisms are dried at  $105^\circ$  for 24 hr. and heated electrically in a box shielded from air currents. The time from the start of the heating current to the production of a continuous flame is a measure of the flame-carrying tendency. The sp. heat, conductivity, and existence of microscopic channels in the materials influence the speed at which flames develop. T. W. P.

**Veneer glueing with casein and albumin.** P. KRUMIN (Angew. Chem., 1935, 48, 212—215).—Comparative tests show that a 50:50 mixture of casein (*C*) and albumin (*A*) is superior to either *C* or *A* alone as an adhesive for veneer or plywood. Exceptionally good results have been obtained on the large scale by using 4 g. per sq. ft. of the *C-A* adhesive; less should suffice for many commercial purposes. A. B. M.

[Plaster from] polyhalite.—See VII. Painting wood.—See XIII.



## PATENTS.

**Coloured concrete.** G. R. TUCKER (U.S.P. 1,972,207—8, 4.9.34. Appl., [A, B] 12.12.32).—In any kind of concrete the evil effects of finely-divided pigment (even C black) are reduced by the presence of a compound produced by the sulphonation of 100 pts. of  $C_{10}H_8$  with 100 pts. of  $H_2SO_4$  and condensation by means of 12 pts. of formalin. Other org. compounds of a similar nature are claimed. B. M. V.

**Decorative and acoustic composition.** C. J. CERVENY and H. EVANS (U.S.P. 1,971,900, 28.8.34. Appl., 9.5.33).—A wall finish comprises expanded vermiculite and a slow-setting  $CaSO_4$  plaster, the former being exposed on the surface as brilliant particles. B. M. V.

**Finishing of wood.** F. TUCHFARBER, Assr. to R. W. CALLAWAY and A. N. MANN (U.S.P. 1,972,502, 4.9.34. Appl., 13.12.32).—The wood is coated with a colloidal suspension in  $H_2O$  of a resinous material insol. in  $H_2O$  but sol. in org. solvents, one of the latter being applied after the  $H_2O$  has dried out. B. M. V.

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

**Determinations of external heat loss of blast-furnaces.** D. F. MARSHALL (Iron and Steel Inst., May, 1935. Advance copy, 25 pp.; cf. B., 1934, 198).—Heat loss is largely a function of superficial area and is < previous estimates (cf. *loc. cit.*). In all cases studied, > 82% of the heat was lost to the cooling- $H_2O$ , losses by radiation and convection from the shaft and by conduction through the ground being relatively small. E. H. B.

(A) **Theory and practice of cupola fusion.** (B) **Thermochemistry of the cupola furnace and its application in the study of the fusion and over-heating of cast iron.** A. POU MAY, JUN. (Rev. Fond. mod., 1934, 28, 71—75, 85—91; 251—255; Chem. Zentr., 1934, ii, 3033).—A crit. review and discussion. H. J. E.

**Use of electric furnaces in industrial heat-treatment.** III. A. G. LOBLEY (Metallurgia, 1935, 12, 9—13; cf. B., 1935, 360).—Furnaces for bright-annealing and clean-hardening are described. These may be either of the batch type or continuous. The special atm. within the furnace must be suitable to the metal under treatment, e.g., steam for Cu. Cracked  $NH_3$  may usually be substituted for  $H_2$  and burnt  $NH_3$  for  $N_2$ . A partly reducing atm. is obtained by mixing hydrocarbons with air, and a neutral atm. for steel-treating by incomplete combustion of coal gas. E. H. B.

**Attack of metal retorts by combustion gases containing sulphur.** B. WAESER (Chem. App., 1934, 21, 33—34; Chem. Zentr., 1934, ii, 3041).— $SO_2$  is absorbed at 200—300° by  $Fe_2O_3$  on the retort wall and yields  $SO_3$ . This produces a protective coating of  $Fe_2O_3$ ,  $FeSO_4$ , and  $Fe_2(SO_4)_3$ , which persists so long as  $H_2O$  is not allowed to condense. Ni, in general, aids corrosion by gases containing  $H_2S$ , but steels with 12—20% of Cr are resistant up to 500°. H. J. E.

(A) **Resistance measurements of the rate of transition of austenite.** F. WEVER and W. JELLINGHAUS. (B) **Magnetic study of self-hardening steels.** F. WEVER and H. LANGE (Mitt. Kaiser-Wilh.-Inst. Eisenforsch., 1933, 15, 167—177, 179—185; Chem. Zentr., 1934, ii, 3306).—(A) The isothermal transition of austenite (I) in a steel (C 0.89, Mn 0.61%) has been measured between 375° and 140°. Discrepancies in the results of magnetic and resistance measurements are explained by changes in the (I) lattice which precede the  $\gamma$ - $\alpha$  transformation.

(B) Magnetic measurements are recorded of transition phenomena in a Cr-Ni steel (C 0.4, Cr 1—2, Ni 3.2—4.4%) at temp. between the pearlite and martensite points. The cementite formed in this region contains almost the whole Cr content of the steel (cf. B., 1933, 469). H. J. E.

**Heat-resistance of iron castings.** R. ZECH and E. PIVOVARSKY (Giesserei, 1934, 21, 385—388; Chem. Zentr., 1934, ii, 3169).—The optimum heat-resistance (I) in cast furnace bars was obtained with those containing 0.4—0.5% of P. With < 2.6% of C results were unsatisfactory and additions of Ni, Cr, and Mo caused no improvement. These metals increased (I) when 3.2—3.4% of C was present. H. J. E.

**Anomalies in the hardening of castings and their relation to the uniformity of the product.** A. LE THOMAS (Bull. Assoc. techn. Fond., 1934, 8, 225—237; Chem. Zentr., 1934, ii, 3169).—Anomalies are attributed to excess of S and oxidation of liquid Fe. H. J. E.

**Hardening [of steel] without distortion.** A. JUNG (TZ. prakt. Metallbearb., 1934, 44, 191—194; Chem. Zentr., 1934, ii, 3170).—A discussion. H. J. E.

**Diamond in hardness-testing.** P. GRODZINSKI (TZ. prakt. Metallbearb., 1934, 44, 46—49; Chem. Zentr., 1934, ii, 3172—3173).—A crit. discussion of hardness determinations. H. J. E.

**Study of steels at raised temperatures.** H. DUSTIN (Chaleur et Ind., 1934, 15, 201—210; Chem. Zentr., 1934, ii, 3035).—The development of fractures in steel in use at high temp. is discussed. H. J. E.

**Wear of steel rails.** E. COTEL (Rev. Mét., 1935, 32, 137—144).—In plain C steels (C 0.4—0.9%) resistance to wear  $\propto$  C content, and, in the normalised state only, to hardness. Rails containing 0.42% C have worn extremely little in 30 years' severe service in Hungary. E. H. B.

**Tinning of sheet metal for manufacture of gas meters.** J. G. DE VOOGD and A. VAN DER LINDEN (Het Gas, 1934, 54, 323—327; Chem. Zentr., 1934, ii, 3173).—Tinning is not an effective protection against corrosion. Painting with rust-protecting paints is more important. H. J. E.

**Alloy cast irons.** J. E. HURST (Metallurgia, 1935, 12, 15—18).—The hardening and tempering of cast Fe and its alloys which, as cast, have martensitic or austenitic structures are examined. The latter class find extensive application as a result of resistance to corrosion, high electrical resistance, and non-magnetic nature. E. H. B.



**Non-metallic inclusions in ferro-alloys.** B. MATUSCHKA (Iron and Steel Inst., May, 1935, Advance copy, 8 pp.).—In alloy steels the added elements or alloys may be a source of non-metallic inclusions. The inclusions in the usual commercial grades of ferro-W, -Cr, -Mo, -V, -Mn, and -Si, and of Ni and Co are described. E. H. B.

**Alloys of iron and chromium.** V. N. KRIVOBOK (Trans. Amer. Soc. Metals, 1935, 23, 1—60).—The existing literature of Fe-Cr alloys (I) is reviewed. The usual methods for investigating alloy systems indicate that Fe and Cr form a continuous series of solid solutions, but it is shown that age-hardening effects are present in alloys free from C or other impurities. The possible effect of dissolved gases is discussed. Cr has a strong affinity for N<sub>2</sub>, which when present in (I) acts in a similar manner to C in steels, and confers hardenability on the alloys. The compound which N forms with (I) has not yet been identified. W. P. R.

**Spectral analysis, using sensitive lines within the reach of glass instruments. Measurements in the spark spectrum of a cobalt-iron alloy for magnet construction.** W. KRAEMER (Z. anal. Chem., 1935, 99, 410—414; cf. A., 1933, 1110).—Results, and sensitive Co lines, are given for an alloy with C 0.7%, Cr 5%, Co 5%, and Mo 1%. J. S. A.

**Volumetric determination of vanadium in iron and special steels.** A. TRAVERS (Bull. Assoc. techn. Fond., 1934, 8, 383; Chem. Zentr., 1934, ii, 3283).—1 g. of the steel is heated for 1—2 hr. with 50 c.c. of HClO<sub>4</sub>, SiO<sub>2</sub> and WO<sub>3</sub> are filtered off, and the ppt. is washed with 10% HClO<sub>4</sub>. The filtrate is treated with NH<sub>3</sub> and NH<sub>4</sub>Cl, the ppt. being washed, dissolved in dil. H<sub>2</sub>SO<sub>4</sub>, and the solution decomposed with 12 c.c. of conc. H<sub>3</sub>PO<sub>4</sub>. 1 c.c. of H<sub>2</sub>O<sub>2</sub> is added to the hot solution to decompose per-acids of Ti, V, and Mo, and the solution heated until white fumes are evolved, cooled, and titrated at 60° with KMnO<sub>4</sub>. Cr and Ni do not interfere. H. J. E.

**Special steels.** (Sir) R. HADFIELD (Nature, 1935, 135, 741—745).—Progress during the last 25 years is reviewed. L. S. T.

**Carburising of copper steels.** S. EPSTEIN and C. H. LORIG (Met. & Alloys, 1935, 6, 91—92).—A case 0.05 in. deep with a surface hardness of 63 (Rockwell-C) is obtained by quenching after 10 hr. at 940° in a normal carburising pack steels containing 0—3% Cu, the surfaces of which have been machined. Shallow cases are produced on oxidised samples with Cu > 1%. E. H. B.

**Welding of nickel steel.** E. POHL (TZ. prakt. Metallbearb., 1934, 44, 339—343; Chem. Zentr., 1934, ii, 3039).—A review of the advantages of Ni welding-steel. H. J. E.

**Welding of austenitic manganese steels.** A. PORTEVIN and D. SÉFÉRIAN (Rev. Mét., 1935, 32, 108—109).—Austenitic Mn steels may be joined by arc- or C<sub>2</sub>H<sub>4</sub>-welding with austenitic Ni or Ni-Cr steels. Mn steel-mild steel welds were unsuccessful. E. H. B.

**Metallographic and mechanical properties of electric-arc welds.** I. MUSATTI and A. REGGIORI

(Rev. Mét., 1935, 32, 112—113).—Analyses are given of the metal within welds made in a 0.2% C steel with a flux-covered 0.08% C electrode. The greatest hardness is attained by the overheated zone adjacent to the weld. E. H. B.

**Resistance to corrosion by nitric acid of oxy-acetylene welds in stainless steels.** G. FAUSER (Rev. Mét., 1935, 32, 113—114).—The effect of flame conditions, [HNO<sub>3</sub>], and applied stress have been studied from the viewpoint of the application of welded stainless steels to HNO<sub>3</sub> manufacturing plant. E. H. B.

**Possibility of altering the composition of the filler metal by means of coatings in oxy-acetylene welding.** E. CREPAZ and S. LOPS (Rev. Mét., 1935, 32, 114).—By suitable coatings on mild steel it is possible to obtain weld metal containing Ni 2.25—2.55%, Cr 0.3—0.4%, or W 3—5%. E. H. B.

**Pick-up of phosphorus from impure acetylene in the welding of steels.** D. SÉFÉRIAN (Rev. Mét., 1935, 32, 114—115).—Steels containing < 0.75% P take up P, during welding, from C<sub>2</sub>H<sub>2</sub> containing > 0.1% PH<sub>3</sub>. E. H. B.

**Building-up of steel rails: the deposited metal.** M. BRUNETEAU (Rev. Mét., 1935, 32, 115).—Building up of worn rails by O<sub>2</sub>-C<sub>2</sub>H<sub>2</sub> welding is favoured, a filler rod of a Cr-Mn steel being most suitable. E. H. B.

**Oxygen-cutting of nickel- and chromium-alloy steels.** L. DE JESSEY (Rev. Mét., 1935, 32, 110).—Ni steels, and Cr and Ni-Cr steels containing < 6% Cr, may be cut with the torch. E. H. B.

**Influence of small additions of cadmium on the filler metal for copper-welding.** L. MATTEOLI (Rev. Mét., 1935, 32, 118).—These have no real advantage. E. H. B.

**Causes of the non-weldability of nickel.** A. BOUTTÉ (Rev. Mét., 1935, 32, 114).—In order to be welded, Ni must contain < 0.02% S and an amount of Mg proportionate to the S content. E. H. B.

**Wrought copper-nickel-aluminium alloys.** D. K. CRAMPTON and H. P. CROFT (Met. & Alloys, 1935, 6, 79—84).—An alloy containing Cu 92, Al 4, Ni 4% has after 80% reduction by cold-work a tensile strength (*T*) > 53 tons per sq. in. and elongation (*E*) > 12%. Alloys containing more Ni and less Al (Cu 91, Ni 7.5, Al 1.5%) are not hardened so much by rolling, but by tempering of the quenched and rolled alloy a *T* > 49 tons per sq. in. and *E* 10% may be obtained. Both types of alloy are readily welded or brazed and are resistant to corrosion. E. H. B.

**Engineering properties of "K-monel" metal.** C. A. CRAWFORD, G. F. GEIGER, and W. A. MUDGE (Met. & Alloys, 1935, 6, 101—105).—K-monel is a temper-hardening alloy consisting of monel metal with 3.5% Al. The alloy is produced in 4 hardness grades: *A*, soft, quenched from 790° after rolling at 1190—930°; *B*, intermediate, *A* tempered at 540°; *C*, intermediate, *A* tempered at 595°; *D*, fully hard, *A* cold-drawn and tempered at 595°. *D* has tensile strength (*T*) < 71 tons per sq. in., elongation (*E*) < 15%, reduction of area (*R*) < 20, Brinell hardness (*H*) < 325, and short-time *T* at 540° (*S*) < 55 tons per sq. in. *A* gives *T* < 54, *E* < 40, *R* < 50, *H* < 225, *S* < 34. The alloy



is resistant to corrosion, non-magnetic, machinable in grades *A—C*, and may be welded or brazed. E. H. B.

**Effect of bismuth as an impurity on the structure and allotropic transformation of tin.** C. W. MASON and W. D. FORGENT (*Met. & Alloys*, 1935, 6, 87—90).—The usual grades of "chemically pure" Sn contain 0.0035% of Bi and exhibit as cast a cored structure resembling a eutectoid when etched in 5% HNO<sub>3</sub> in abs. EtOH. Bi is removed by electrolysis in a bath containing 250 g. of SnCl<sub>2</sub>·2H<sub>2</sub>O, 10 c.c. of conc. HNO<sub>3</sub>, and 990 c.c. of H<sub>2</sub>O. The transformation into grey Sn occurs readily in the purified Sn and in cored metal, but not in Sn containing Bi evenly distributed by annealing. E. H. B.

**Importance to the chemical industry of the prohibited uses of metals.** ANON. (*Chem. Fabr.*, 1935, 8, 179—181).—Cu, Sn, Hg, Pb, Cr, Ni, Co, and their alloys are among the metals the use of which is prohibited in Germany for certain specified purposes. W. P. R.

**Sources of errors in the assaying of gold and platinum.** K. W. FRÖHLICH (*Mitt. Forsch.-Inst. Probieramts Edelmet. Schwäb.-Gmünd*, 1934, 8, 39—46; *Chem. Zentr.*, 1934, ii, 3014).—In dissolving Ag from a Pt—Au—Ag mixture with hot conc. H<sub>2</sub>SO<sub>4</sub>, dissolution of Pt is lessened by addition of As<sub>2</sub>O<sub>3</sub> (1 pt. of As<sub>2</sub>O<sub>3</sub> in 100 pts. of conc. H<sub>2</sub>SO<sub>4</sub>). The Pt : Au ratio should be > 1 : 10. The method gives deviations of ±0.3 and ±3.0% for Au and Pt, respectively. H. J. E.

**Flotation reagents and flotation chemistry.** P. HOLMSEN (*Tids. Kjemi*, 1935, 15, 2—7).—A review of modern theories and practice. R. P. B.

**Electrodeposition of iron for wearing and bearing metal.** D. R. KELLOGG (*Met. & Alloys*, 1935, 6, 97—99).—Steel parts to be plated are cleaned in petrol, then successively made the anode in an alkaline bath and the cathode in H<sub>2</sub>SO<sub>4</sub>. Fe is then deposited either from (i) a cold bath containing 75 g. of Fe<sup>II</sup> NH<sub>4</sub> sulphate (I) per litre, with additions of FeCO<sub>3</sub> and C, using c.d. of 1 amp./sq. dm., or (ii) a hot bath (50—70°) containing 300 g. of (I) per litre. Both baths are stirred. (i) gives a hard coating (200 Brinell), whilst that from (ii) is soft and velvety. Wood should be absent from the baths. The same technique is applicable to cast Fe. E. H. B.

**Prevention of pore formation in galvanic nickel precipitates.** W. ECKARDT (*Oberflächentech.*, 1934, 11, 178; *Chem. Zentr.*, 1934, ii, 3040).—A fine-grained, pore-free Ni deposit is formed at room temp. by adding C<sub>6</sub>H<sub>4</sub>(OH)·SO<sub>3</sub>Na to the plating bath. H. J. E.

**Determination of protective value and strength of galvanic coatings.** J. KORPIUN (*Metallwar.-Ind. Galv.-Techn.*, 1934, 32, 364—368; *Chem. Zentr.*, 1934, ii, 3311).—A review and discussion. H. J. E.

**Lubrication [in wire-drawing].**—See II. **Catalysts for NH<sub>3</sub> oxidation.**—See VII. **Enamelled Fe vessels.**—See VIII. **Metal priming paints.**—See XIII. **Corrosion from zero softened H<sub>2</sub>O.**—See XXIII.

#### PATENTS.

**Apparatus [open-hearth furnace] for production of steel.** H. A. BRASSERT, ASSR. to H. A. BRASSERT &

Co. (U.S.P. 1,954,280, 10.4.34. Appl., 24.7.31).—The furnace (*F*) comprises a wide, shallow hearth fired with gas or oil, a side-wall (*W*) containing a no. of tuyères fed from a common wind-box extending along *W*, and means for tilting *F* until *W* is horizontal and the charge forms a wide, shallow bath thereon. *F* thus acts both as an open-hearth furnace and a converter. A. R. P.

**Manufacture of high-strength cast iron.** G. F. COMSTOCK, ASSR. to TITANIUM ALLOY MANUFACTURING CO. (U.S.P. 1,955,791, 24.4.34. Appl., 22.9.32).—Molten cast Fe is treated with 1—2% of ferrochromium containing 60—75% Cr and 4—8% C and with 1—2% of an alloy of Fe with Ti 15—25, Si 15—25, Al < 1, and C < 1%. The Cr improves the machining properties and the Ti improves the strength by refining the graphite. A. R. P.

**Manufacture of composite metal [steel] articles.** S. L. INGERSOLL, ASSR. to INGERSOLL STEEL & DISC CO. (U.S.P. 1,955,547, 17.4.34. Appl., 8.8.32).—Two thick sheets of non-rusting Cr steel (I) are coated on one side with aq. CrO<sub>3</sub>, placed together with the coated sides in contact, and welded around the edges. The composite ingot is then placed in a mould and an ordinary steel cast around it; the resulting billet is forged and rolled to sheet, the edges are trimmed off so that the sheet separates into two longitudinally, and the resulting rustless coated sheets are pickled and scoured to remove the oxide film on the (I) side. A. R. P.

**Hardening of cast[steel] articles.** F. E. MCCLEARY, K. C. BABO, and H. RAYNER, ASSRS. to CHRYSLER CORP. (U.S.P. 1,954,180, 10.4.34. Appl., 9.1.32).—The hot articles (*A*) are passed through a cooling chamber in which the parts to be hardened are subjected at spaced intervals to the cooling effect of air, oil, or H<sub>2</sub>O sprays the temp. of which is raised as that of *A* decreases. A. R. P.

**High-speed [tungsten] steel.** A. F. BRAID (U.S.P. 1,955,529, 17.4.34. Appl., 1.4.33).—The alloy consists of Fe with W 15—18, Cr 2.5—5, Ti 1—2.5, and C 0.1—0.8%; it has a fine-grained structure, a high elastic limit, and good resistance to frictional wear at high temp. A. R. P.

**Melting of copper to produce dense castings low in oxygen.** R. S. ARCHER and M. A. MAFUSH, ASSRS. to A. O. SMITH CORP. (U.S.P. 1,955,726, 24.4.34. Appl., 21.7.33).—The metal is melted in a graphite pot under a layer of borax flux (*F*), heat being supplied by the passage of a current from the pot to an electrode immersed in *F*. A. R. P.

**[Heat-]treatment of metals [copper].** L. R. CLAPP and R. O. FARMER, ASSRS. to ROLLING PROCESS, INC. (U.S.P. 1,955,576, 17.4.34. Appl., 24.12.29).—Cu sheet is cold-rolled to > 75% reduction and annealed at 250—350° (275°) to obtain a Rockwell hardness of 65—70. A. R. P.

**Concentration of manganese ores.** A. J. WEINIG and R. E. CUTHBERTSON, ASSRS. to CUBAN-AMER. MANGANESE CORP. (U.S.P. 1,955,039, 17.4.34. Appl., 19.11.30).—Mn oxide ores, preferably after roasting at 600° for 15 min., are subjected to froth flotation in presence of a fish oil soap, kerosene, and crude oil. A. R. P.



**Refining of metals [lead].** R. C. RUTHERFORD, Assr. to AMER. SMELTING & REFINING CO. (U.S.P. 1,954,951, 17.4.34. Appl., 11.4.32).—Impure Pb ore containing Bi, Au, Ag, Cu, As, Sb, and Sn is sintered on a travelling grate under conditions in which a small amount of Pb is produced. This is allowed to trickle through the grate into a collecting chamber below the wind-box; it contains the greater part of the impurities in the ore. A. R. P.

**Plating machine.**—See XI.

### XI.—ELECTROTECHNICS.

**Matt glass bulbs.**—See VIII. **Furnaces for heat-treatment.** **Spectral analysis of magnet alloy.** **Welding steels.** **Electrodeposition of Fe. Ni ppts.** **Galvanic coatings.** See X. **Differentiating refined and unrefined oils and fats.**—See XII. **H<sub>2</sub>O sterilisation.**—See XXIII.

#### PATENTS.

**Electrode for electrolytic baths.** P. C. KERK (U.S.P. 1,970,804, 21.8.34. Appl., 24.12.32).—An anode, especially for production of Cl<sub>2</sub>, comprises a Cu core sheathed with Ta or other metal which, if used alone, has a valve action; such action is stated to be prevented by the core even when out of contact with the electrolyte. B. M. V.

**Electrochemical treatment [plating] machine.** A. H. HANNON (U.S.P. 1,970,850, 21.8.34. Appl., 1.10.31).—An automatic plating plant is described in which the conveying means is given an intermittent motion so that the articles are plated both while stationary and moving. B. M. V.

**[Electrical] device for analysing the structure of matter.** E. RUPP, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,971,277, 21.8.34. Appl., 24.6.32. Ger., 26.6.31).—Electrons are reflected from the surface of the sample (*S*) and the interference effects measured. The emissive means and *S* are rotatable by a 2:1 gear in relation to a fixed reception mass, the whole being in a vac. bulb having a ground joint for the rotation. B. M. V.

**Comparison of colours [electrically].** H. H. SHELDON and C. F. LORENZ, Assrs. to SHELDON ELECTRIC CORP. (U.S.P. 1,971,317, 21.8.34. Appl., 6.7.31).—Separate beams of light are emitted from an exact pair of conc.-filament lamps and passed through equal heat-resisting filters and condensing lenses to impinge, respectively, on sample and standard, which are rotated side by side. After reflexion the rays are passed through a common lens and iris diaphragm, diverging thence through a common standard colour-filter, which may be in the form of a tank for solutions, to separate photo-electric cells in the arms of a bridge. B. M. V.

**Electro-optical measurement of concentrations, colours, dispersions, etc. of fluids and similar substances.** W. G. EXTON (U.S.P. 1,971,443, 28.8.34. Appl., 29.4.30).—Light from a single source is divided into 2 beams, one being passed through a calibrated light adjuster (*A*) to a photo-electric cell (*B*); the other beam is passed alternately through a blank (*C*) of clear liquid and the sample (*D*) in a similar container, and falls in each case on a photo-electric cell (*E*). *B* and *E* are

connected in opposition to a millivoltmeter or in a Wheatstone bridge, the difference in readings of *A* in the balance positions giving the result desired. B. M. V.

**H<sub>2</sub>.**—See VII. **Melting Cu.**—See X.

### XII.—FATS; OILS; WAXES.

**Rancidity and the preservation of fats and oils.** K. STEPHAN (Chem.-Ztg., 1935, 59, 416—417).—The ketonic components of rancid oils may be removed by treatment with semicarbazide hydrochloride and NaOAc or Na stearate, or N<sub>2</sub>H<sub>4</sub> or NH<sub>2</sub>·OH salts and alkali. Oils so treated do not give the Täufel-Thaler reaction for ketones (cf. B., 1932, 515). Camphor (0.3—0.5%) acts as a preservative against rancidity in the case of oils and may be removed by heating or extracting with EtOH. G. H. C.

**Antioxidant properties of vegetable lecithin.** E. I. EVANS (Ind. Eng. Chem., 1935, 27, 329—331).—The effect of vegetable lecithin (*L*) in inhibiting oxidation of fats in presence of a metal catalyst is = that of KCN, and > that of *p*-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub> and diphenylguanidine. Addition of 0.1% of *L* to edible oils affords sufficient protection against autoxidation. E. C. S.

**Chemical structure of fats in relation to their digestibility and palatability.** T. P. HILDITCH (Food, 1935, 4, 350—352).—A discussion.

**Differentiation of refined and unrefined oils and fats by measuring the electrical resistance.** S. H. BERTRAM (Chem. Weekblad, 1935, 32, 296—297).—The sp. resistance is (3—40) × 10<sup>9</sup> for refined and (4—60) × 10<sup>7</sup> ohms for unrefined oils. Measurements are carried out at 20° with oils and at 50° with fats, the temp. coeff. being —0.0336. The difference is due to the presence of mucilage. S. C.

**Micro-phytosteryl acetate test.** L. KOFLER and E. SCHAPER (Fettechem. Umschau, 1935, 42, 21—26).—The test can be used for the detection of vegetable fats in animal fat, and depends on the determination, under the microscope, of the temp. at which the mixture of phytosteryl and cholesteryl acetates (I), obtained from the fat by means of digitonin, become completely molten. (I) and sitosteryl acetate (II) exist in several crystalline forms the optical properties and mode of melting of which are described (cf. A., 1888, 1076). The temp. at which mixtures of (I) and (II) become completely molten, and the corresponding temp. for mixtures of (I) and the acetate of the phytosterol from sunflower-seed oil, are given. The applicability of the test has been examined for a large no. of pure and mixed fats. H. G. M.

**Optimum detergency in blended washing soaps.** R. L. DATTA and S. S. DAS GUPTA (Soap, Perf., and Cosmetics Trade Rev., 1935, 8, No. 4, 34—36).—A soap from a properly selected mixed stock (*S*) (i.e., soap of mixed fatty acids) has a detergent power (*D*) > that of any single fatty acid soap, or > that calc. as the additive effect of the individual components; presumably, several factors, which do not all reach their max. with the same pure fatty acid soap, must be responsible for *D*. The "hardness no." (cf. B., 1933, 1016) is a good guide to the selection of *S*. E. L.



**Practical application of pine oil in soap-making.**

O. DUESBERG (Seifensieder-Ztg., 1934, 61, 632—633, 667—668; Chem. Zentr., 1934, ii, 3331).—Purified pine oil (I) may be added to soaps of all consistencies. Owing to its alcoholic and dehydrating character various precautions must be taken; these are discussed and a no. of recipes for soaps containing (I) are given.

H. N. R.

**"Trolhetta" oil in the liquefaction and superfatting of liquid soaps etc.** C. STIEPEL (Seifensieder-Ztg., 1934, 61, 631—632; Chem. Zentr., 1934, ii, 3330—3331).—The use of trolhetta oil (I) (an artificial fatty "acid ester") for superfatting liquid soaps is discussed. (I) is stable to cold and keeps well. Addition of 8% of (I) to a linseed oil soft soap causes salting out of a soap paste.

H. N. R.

**Autoclave process of oil splitting. IV. Successive hydrolysis.** D. NAKAE, H. NORBORI, and S. SASADA (J. Soc. Chem. Ind., Japan, 1935, 38, 7—8 B; cf. B., 1935, 317).—From determinations of the acid and sap. vals. of the products of saponification of soya-bean oil (at 8 atm.), using CaO etc. as catalysts, it is concluded that the hydrolysis of glycerides may proceed in stages, but is certainly not a quadrimol. reaction. Intermediate products (mono- and di-glycerides) are most prominent when MgO is used as catalyst, and more in evidence when aged ZnO is employed in place of fresh ZnO.

E. L.

**Hydrolysis of linseed oil by fat-splitting ferments.** R. F. BOAN (J. Proc. Sydney Tech. Coll. Chem. Soc., 1933—4, 6, 16—17).—The free acids in a very acid linseed oil were due to ferments produced by development of oöspora following storage in cans containing honey and H<sub>2</sub>O.

G. H. C.

**Ultramicroscopical study of irradiated drying oils.** K. E. McCLOSKEY and W. G. FRANCE (Ind. Eng. Chem., 1935, 27, 160—162).—Photomicrographs are given to show the formation of colloidal particles in commercial and optically empty linseed and tung oil by exposure for a few hr. to irradiation by a Hg-vapour lamp; addition of small proportions of AcOH retards their development. Air-blowing the oils for several days produces similar sub-microns.

S. M.

**Test for sesamé oil.** L. PAVOLINI (Olii min., Ol. Gras., Col. Vern., 1934, 14, 41—43; Chem. Zentr., 1934, ii, 3331).—The oil is treated successively with Ac<sub>2</sub>O, an EtOH solution of furfuraldehyde, and H<sub>2</sub>SO<sub>4</sub>. In absence of sesamé oil (I), olive oil etc. gives a red-brown colour; when (I) is present a deep brick-red colour, passing through green to greenish-blue, is produced.

H. N. R.

**Rendering German rapeseed oil edible.** R. DIETERLE (Seifensieder-Ztg., 1934, 61, 637—638; Chem. Zentr., 1934, ii, 3330).—The oil is edible if great care is taken in its extraction; details of the necessary purification are given.

H. N. R.

**Critical solution temperature in alcohol of castor oil.** D. ANDERSON (J. Soc. Chem. Ind. Victoria, 1934, 34, 914—916).—The crit. solution temp. (I) of pure castor oil in EtOH (*d*<sup>15</sup> 0.8303) determined by the B.E.S. method is 2.8°. The (I) varied with *d* of EtOH as

follows: 0.8303, 0.6°; 0.8306, 0.7°; 0.8307, 0.8°; 0.8343, 12.7°, and 0.8351, 14.7°.

E. H. S.

**Technical value of sulphur oils.** J. N. ZAGANARIAS (Praktika, 1933, 8, 389—394; Chem. Zentr., 1934, ii, 3198).—The neutralisation and Ac vals. of fresh and old S oils from olive kernels are compared. In soap manufacture old oils must be blended with fresh.

H. N. R.

**Crystallising point of wax solutions.** G. BAER-LOCHER (Seifensieder-Ztg., 1934, 61, 655—656; Chem. Zentr., 1934, ii, 3330).—The crystallising point (I) is recommended for the characterisation of wax solutions (II). An apparatus, similar to that of Beckmann for mol. wt. determination, is described. (II) are the more homogeneous the nearer the f.p. approaches (I).

H. N. R.

**Lubricants.**—See II. **Sizes for paper.**—See V. **Waterproofing masonry.**—See IX. **Sterilisation of oils.**—See XX.

## PATENTS.

**Edible fats and fatty oils.** PROCTER & GAMBLE Co. (B.P. 428,048, 30.9.33. U.S., 3.10.32).—Superior cooking fats (particularly for bakery purposes) are characterised by the presence of 0.25—10% of free fatty acids, formed *in situ* by partly hydrolysing (or saponifying and acidifying) an edible fat.

E. L.

**Manufacture of soaps, shaving creams, and similar masses.** HENKEL & Co., G.M.B.H. (B.P. 427,147, 17.5.34. Ger., 20.5.33).—The addition to soaps, shaving creams, cosmetics, and (wax) polishes of hydroxyaryl compounds having an alkyl or cycloalkyl group of < C<sub>6</sub> attached to the nucleus by CO or CH<sub>2</sub>, e.g., hydroxy-laurophenone or -dodecylbenzene or their homologues, is claimed.

H. A. P.

**Oleo-emulsion of graphite.**—See II. **Wetting etc. agents.**—See III.

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

**Formation and deterioration of linseed oil films on normal exposure.** ANON. (Paint Var. Prod. Man., 1935, 12, No. 5, 22—26).—The various theories concerning the mechanism of the drying and subsequent breakdown of linseed oil films are discussed.

D. R. D.

**Relationship between oil content, particle size, and particle shape.** H. WOLFF and G. ZEIDLER (Paint Var. Prod. Man., 1935, 12, No. 5, 14—20).—The particle size is  $\mu = 4^{1-\log k-n}$ , where *k* and *n* are the consts. in Wolf's turboviscosity equation ( $TV - TV_0 = kp^n$ ).

D. R. D.

**House paints. Effect of climate, wood types, and priming practices.** J. MARSHALL, J. W. LIFF, and H. R. YOUNG (Ind. Eng. Chem., 1935, 27, 147—152; cf. Browne, B., 1933, 799).—Panels of white, western yellow, Arkansas yellow, and long-leaf southern pines, red cedar, and Douglas fir were coated with 6 paint systems (white-Pb, Pb-ZnO, lithopone, and lithopone-titanox) and exposed at 3 widely separated U.S. stations; the degree of flaking was observed at intervals (graphs given). The effect of climate is pronounced; for dry atm. and where daily temp. variations are small those



paints which yield the less flexible, harder films are desirable. Failure was generally accelerated (a) when the edges and back of the panel were not also painted, and (b) with southerly exposures at 45° (I) instead of vertically (II). Al priming paint appeared to be advantageous with (I), but not with (II). White pine and red cedar wood showed min. film disintegration; pre-weathering of the wood had little effect. S. M.

**Painting characteristics of hardwoods.** F. L. BROWNE (Ind. Eng. Chem., 1935, 27, 42—47).—A series of parallel exposure tests in 5 localities of white-Pb paint, Pb-Zn paint, and a Ti-Pb-Zn-stand oil enamel on a comprehensive range of hardwoods (I) (*i.e.*, from broad-leaved trees) is fully described, and the results are summarised and illustrated. It is shown that the  $d$  of the wood and the type and size of the pores are the most significant properties of (I) affecting their painting. S. S. W.

**Luminous highway paint.** W. G. SCHNELL (Amer. Paint J., 1935, 19, No. 23, 52—53).—The use of luminous paints containing radioactive ingredients for traffic signs on roads is suggested. A new process for obtaining conc. Pa preps. from American radioactive minerals (B., 1935, 590) will probably render this economically possible. D. R. D.

**Metal priming paints. Inhibiting qualities and influence of reactions within the paint film.** H. A. NELSON (Ind. Eng. Chem., 1935, 27, 35—41).—The different theories of metal corrosion are reviewed. It is shown that the changes occurring in oils and resins within paint films supply such essential external agents for corrosion as H<sub>2</sub>O, CO<sub>2</sub>, and acids, together with a cathodic depolarising agent (O) in the form of H<sub>2</sub>O<sub>2</sub>. Priming paints should contain basic pigments of good neutralising properties, that form reaction products adding to the physical properties, *e.g.*, impermeability, of the film, and tend to counteract the effects of H<sub>2</sub>O<sub>2</sub> etc. The behaviour in this connexion of metallic Zn, Pb<sub>3</sub>O<sub>4</sub>, Spanish Fe<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub>, basic Pb chromate, blue Pb, graphite, and Al powder in various vehicles is summarised. S. S. W.

**Leafing of aluminium paint.** P. H. FAUCETT (Paint Var. Prod. Man., 1935, 12, No. 5, 12).—Orientation of the leaf-like Al particles parallel to the painted surface is facilitated by ample thinning. D. R. D.

**Colour-permanency of synthetic and natural iron oxides.** H. R. HARLAN (Amer. Paint J., 1935, 19, No. 31, 18).—The light-fastness of paints containing various Fe<sub>2</sub>O<sub>3</sub> pigments has been compared. Artificial raw sienna yielded the most permanent paints. Indian-red and French ochre being also satisfactory. Natural raw sienna gave the least permanent paints. D. R. D.

**Zinc sulphide pigments for interior paints.** H. A. NELSON (Off. Digest, 1935, No. 145, 177—182).—The pigment properties of various commercial lithopones have been compared and explanations are offered of the observed differences. D. R. D.

**Testing dry colours [pigments].** W. E. POMEROY (Off. Digest, 1935, No. 145, 162—164).—Standard methods are briefly reviewed. D. R. D.

**Cellulose acetate lacquers.** C. R. FORDYCE (Paint, Oil, and Chem. Rev., 1935, 97, No. 9, 36—38).—A review. D. R. D.

**Plasticisers [for cellulose nitrate].** R. L. SHUMAN (Paint, Oil, and Chem. Rev., 1935, 97, No. 9, 34—35).—Historical. D. R. D.

**Effect of dehydration of nitrocellulose on "orange peel" of sprayed lacquer films.** C. A. HOCHWALT and P. E. MARLING (Ind. Eng. Chem., 1935, 27, 190—192).—The pitting of nitrocellulose (I) films ("orange-peel" effect) is partly due to the H<sub>2</sub>O content of the alcohol with which technical (I) is usually wetted. The defect can be reduced considerably by washing the wet (I) with an anhyd. alcohol, *e.g.*, C<sub>5</sub>H<sub>11</sub>-OH (PhMe may be used for a second washing), and avoiding the use of very volatile solvents. (Cf. U.S.P. 1,961,120; B., 1935, 320.) S. M.

**Manufacture of lithographic varnish in an atmosphere of carbon dioxide.** M. OGURA (J. Soc. Chem. Ind., Japan, 1935, 38, 129—132 B).—The construction of a pot for heating linseed oil (I) in an atm. of CO<sub>2</sub> is described. The  $d$ ,  $n$ ,  $\eta$ , and sap., acid, and I vals. of the product obtained by heating (I) at about 290°, 300°, and 320°  $\pm$  3° during various times are tabulated and compared with the corresponding properties of the products obtained by means of an open pot. The former products are lighter in colour than the latter. The product obtained by heating (I) at 300° for 8 hr. is a varnish with  $\eta$  70—80 poises and low acid val., and is light in colour. It is suitable for printing and lithographic purposes. The product obtained at 320° may be used for plate printing, but is a little too "long." H. G. M.

**[Lacquer] thinner production.** P. H. FAUCETT (Paint Var. Prod. Man., 1935, 12, No. 5, 7—12, 36).—Practical advice on the formulation and testing of lacquer thinners is given. D. R. D.

**Effect of butyl alcohol on viscosity of [cellulose nitrate] lacquer and alkyd resin finishes.** C. BOGIN (Paint, Oil, and Chem. Rev., 1935, 97, No. 9, 45—48).—The  $\eta$  of a large no. of lacquers of stated composition has been determined. The reduction in  $\eta$  produced by the addition of a const. proportion of different solvents falls in the order: BuOH > BuOAc > xylene > turpentine > naphtha. There is a slight variation in the order in certain cases, but BuOH is always the best. D. R. D.

**Coal-tar solvents.** H. H. SCHNOOR (Paint, Oil, and Chem. Rev., 1935, 97, No. 9, 49—50).—The manufacture of solvents from coal tar is described. D. R. D.

**Lacquer solvents.** E. F. METZINGER (Paint, Oil, and Chem. Rev., 1935, 97, No. 9, 42—44).—The advantages and disadvantages attending the use of *sec.*-alcohols, their esters, and the ketones derived by their oxidation are considered. D. R. D.

**Lacquer testing.** H. E. EASTLACK (Paint, Oil, Chem. Rev., 1935, 97, No. 9, 22—27).—Standard tests are reviewed. D. R. D.

**Coating composition films. Physical properties and durability.** J. K. HUNT and W. D. LANSING (Ind. Eng. Chem., 1935, 27, 26—29).—Intrinsic durability (D)



—one factor determining the useful life of a coating film—was correlated with various film properties. *e.g.*, distensibility at different R.H., thermo-plasticity,  $H_2O$ -vapour permeability,  $\eta$  (for films of cellulose derivatives), etc., and particularly with the change in these properties with age. Details of the tests and results on the various clear films examined are described, it being stressed that pigmented films will require further investigation. The factors affecting *D* are outlined and the possibility of predicting *D* from the physical properties above-mentioned is discussed. S. S. W.

**Nature and constitution of shellac. X. Compatibility of French varnish with nitrocellulose solutions.** W. H. GARDNER and B. GROSS (Ind. Eng. Chem., 1935, 27, 168—170).—Gradual increments of  $C_6H_6$  and PhMe were added to solutions containing both shellac and nitrocellulose in mixtures of EtOAc with either MeOH or EtOH until separation did not take place during either the mixing or subsequent setting of the films. The tolerances for  $C_6H_6$  and PhMe (triangular diagrams given) increase with the age of the shellac varnish; the age can, in fact, be determined in this way. With MeOH the effect of ageing developed twice as rapidly as with EtOH; in  $OH\cdot[CH_2]_2\cdot OEt$  the effect of storage was nil. The change is influenced by the mol. wt. of the alcohol and, as the chemical composition of the varnishes remains unaltered, it is probably colloidal. S. M.

**Detection of Congo copals.** W. NAGEL and M. KÖRNCHEN (Fettechem. Umschau, 1935, 42, 34—36; cf. B., 1934, 1069).—The resin acids in Congo copals (I) are esterified to a considerable extent by  $MeOH-HCl$ , and by  $EtOH-H_2SO_4$ , and cannot be separated from fatty acids by this means. There is no essential difference between the action of these two reagents. The colophony (II) obtained from (I) also contains substances which can be esterified. The proportion of these decreases with rise in the temp. at which (II) was produced. The acid obtained by the saponification of these esters is not agathidicarboxylic acid (cf. A., 1931, 231). The corresponding ester from Manila copals probably contains some agathic diester. The proportion of ester formed from the EtOH-sol. portion of various copals is given. H. G. M.

**Acid value of resin esters.** W. H. HEART (Metallbörse, 1934, 24, 1162—1163; Chem. Zentr., 1934, ii, 3187).—Glycerol (I)-colophony esters for weather-resisting varnishes must have an acid val.  $< 4-6$  and be free from glyceryl mono- and di-resinates. The use of a 1—2% excess of (I) yields neutral esters. H. N. R.

**Oil-sol. metallic salts.**—See III. **Resin acids of spruce. Sizes for paper.**—See V. **Waterproofing masonry.**—See IX. **Plasticity and elasticity. Rubbone.**—See XIV.

#### PATENTS.

**[Cellulose acetate] composition.** E. W. REID, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,969,183, 7.8.34. Appl., 23.9.31).—Compositions comprising cellulose acetate dissolved in a mixture of  $CH_2Cl-CH_2Cl$  and MeOH and/or EtOH are claimed. S. S. W.

**Cellulose derivative compositions.** BRIT. CELLULOSE, LTD., and W. H. MOSS (B.P. 427,621, 24.8.33).—The use of triphenylacetin as a plasticiser is claimed. S. M.

**Light-filtering overcoating.** T. F. MURRAY, JUN., Assr. to EASTMAN KODAK CO. (U.S.P. 1,969,473, 7.8.34. Appl., 16.1.32).—Nitrocellulose lacquers are protected from decomp. by ultra-violet light by a superimposed coating of cellulose acetate (or other vehicle) containing about 2—5% of the  $Ph_3$  compound of Bi, Sb, N, As, or P. J. L.

**Plastic and coating compositions etc.** J. W. C. CRAWFORD, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 427,727, 27.9.33).—Polymerides of lower alkyl esters of methacrylic acid are plasticised by ethers of mono- and poly-hydric alcohols ( $\geq C_{22}$ ), m.p.  $\geq 80^\circ$ , b.p.  $\leq 200^\circ$ , *e.g.*, anethole, cetyl Et ether. S. M.

**Production of [vinyl] plastic materials.** J. W. C. CRAWFORD, J. MCGRATH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 427,494, 25.10.33).—A vinyl ester or lower alkyl acrylate is heated with continuous agitation in an aq. suspension of a colloid, *e.g.*, sol. starch, gums, glycol-cellulose, which prevents coalescence. The product is granular or globular. S. M.

**Manufacture of articles from hardenable artificial resins.** H. D. ELKINGTON. From A. NOWACK A.-G., and R. HESSEN (B.P. 427,885, 31.7.33).—A mechanical mixture of hardenable resin and fillers, together with pigments etc. if desired, is pressed through heated mixing nozzles and directly introduced into a mould, or first passed through a filling space and/or heating space of an injection apparatus, the prep. of a moulding mixture as a separate stage being eliminated. S. S. W.

**Manufacture of linoleum.** A. B. MILLER, Assr. to ARMSTRONG CORK CO. (U.S.P. 1,969,272, 7.8.34. Appl., 5.4.32).—The gelled drying oil binder in scrap linoleum etc. is recovered by heating the scrap with a suitable resinous flux, *e.g.*, rosin, introducing a further amount of compatible drying oil, *e.g.*, linseed oil, removing the solid constituents, *e.g.*, fillers, from the mixture by centrifuging or filtration while hot, and finally oxidising the binder constituents to give linoleum cement. The rosin may be retained in the final cement, or reclaimed by an intermediate extraction with EtOH. S. S. W.

**Finishing wood.**—See IX.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Plasticity and elasticity, especially of unvulcanised rubber.** J. HOEKSTRA (Chem. Weekblad, 1934, 31, 754—758).—The mechanical properties of plastic materials are discussed, with particular reference to the use of the balance plastometer and the significance of the recovery curves of unvulcanised rubber after compression to a given thickness. Methods of studying the behaviour of such materials when subjected to shear forces are outlined. H. F. G.

**Thermal properties of rubber compounds. II. Heat generation of pigmented rubber compounds.** C. E. BARNETT and W. C. MATHEWS (Ind. Eng. Chem.,



1934, 26, 1292—1296; cf. B., 1934, 465).—A testing machine is described for measurement of the resilience ( $R$ ) (% of energy restored after impact) and depth of indentation when a block of vulcanised compounded rubber is struck by a "hammer" carried on a pendulum arm. The quotient % energy lost/indentation varies inversely as the flexing life determined by a machine comprising two parallel plates, one of which moves in a circle parallel to the other, between which a specimen block of the rubber is compressed; with this machine, time failure is directly  $\propto$  the temp. developed. With ZnO as compounding ingredient the optimum particle size varies with the proportion incorporated. With C (channel) black increase in the % causes rapid decrease in  $R$  and increase in hardness. Tests were also made with various grades of Thermatomic black, lithopone, whiting, barytes, and Dixie clay. With another type of testing machine in which a rubber block is pounded repeatedly with a definite load, the thermal conductivity of the compounding ingredient may interfere appreciably with the simple hysteresis effect. D. F. T.

**Vulcanisation of rubber. VI. Thermochemistry.** J. T. BLAKE (Ind. Eng. Chem., 1934, 26, 1283—1286; cf. B., 1930, 829).—The heat evolution during vulcanisation of mixtures of rubber, *e.g.*, with 1—8% of S, was investigated by means of a thermocouple embedded in a cylinder of the various compositions. Heat evolution occurs at all % of S, but beyond 3% of S the temp. rise becomes progressively greater and is an approx. linear function of the S content. The results agree with the view that two reactions, *viz.*, (I) the formation of soft vulcanised rubber and (II) further conversion of this into ebonite, occur concurrently. The presence of an accelerator such as  $\text{NH}_2\text{C}(\text{NHPH})_2$  favours the (isothermic) reaction (I) and leads to a reduction in the extent of the (exothermic) change (II). Vulcanisation with Se and/or tetramethylthiuram disulphide is isothermic and presumably leads only to stage (I) of the vulcanisation process;  $m\text{-C}_6\text{H}_4(\text{NO}_2)_2$  behaves similarly. If, however, Se is used in conjunction with S, the Se still enters into reaction (I), leaving more S available for stage (II) so that the heat liberated is  $>$  with S alone. D. F. T.

**Rubber vulcanisation. Formation of dimethyl-dithiocarbamic acid derivatives in vulcanisation with tetramethylthiuram di- and mono-sulphides.** A. F. SHEPARD (Ind. Eng. Chem., 1934, 26, 1200—1201).—When rubber is vulcanised with tetramethylthiuram di- or mono-sulphide and S,  $\text{NMe}_2\text{CS-SH}$ ,  $\text{NHMe}_2$  is formed in considerable proportion. If ZnO is also present the Zn salt is obtained instead of the  $\text{NHMe}_2$  salt. The last-named is also formed when a mixture of rubber and the thiuram disulphide is heated for 38 min. at 138°. (Cf. B., 1932, 316.) D. F. T.

**Graphic comparison of the effectiveness of hot vulcanisation at variable temperatures.** (Miss) S. KRONSTEIN (Caoutchouc et Gutta-Percha, 1935, 32, 17,145—17,147).—A table of figures is calc. giving the relative velocity of vulcanisation at 110—160° in 5° intervals, for a range of temp. coeffs. between 1.8 and 2.8 (with intervals of 0.1). The method of application of such a set of factors to the computation of the effective-

ness of vulcanisation with variable temp. by measurement of the area subtended by the velocity-time curve against the time axis is demonstrated. D. F. T.

**Rubber regeneration and regenerates.** F. KIRCHHOF (Chem.-Ztg., 1935, 59, 433—437).—A review.

**Rubbone.** H. P. and W. H. STEVENS (Bull. Rubber Growers' Assoc., Mar., 1935, Reprint, 4 pp.).—Rubbone is a viscous resinous product, approx.  $\text{C}_{10}\text{H}_{16}\text{O}$ , obtained by aerating a solution of rubber (20 pts.) in white spirit (80 pts.) containing Co linoleate ( $\frac{1}{3}$  pt.) at 80° until a sample shows rapid sedimentation. After clarifying by settling or centrifuging, the resin is isolated by distillation in steam or vac. At  $>100^\circ$  it undergoes thermo-setting; the hardening can be accelerated by the addition of certain substances. It also dries on exposure to air. Various possible commercial applications are indicated. D. F. T.

#### PATENTS.

**Manufacture of rubber compositions from latex and like aqueous rubber dispersions.** U. PESTALOTTA, and SOC. ITAL. PIRELLI (B.P. 427,562, 30.10.33).—Increased hardness in the products is obtained by adding to the aq. rubber dispersion an insol. hydratable oxide such as MgO, *e.g.*, in aq. suspension and, if desired, together with a suitable dispersing agent. Hydration of the oxide to hydroxide at  $\leq$  room temp. occurs at least in part after coagulation. D. F. T.

**Preservation of rubber.** E. I. DU PONT DE NEMOURS & Co. (B.P. 427,495, 25.10.33. U.S., 25.10.32).—Age-resisting properties are imparted by incorporating  $\text{NN}'$ -diphenyl-2 : 6- or -2 : 7-naphthylenediamine. D. F. T.

#### XV.—LEATHER; GLUE.

**[Hide-]bating preparations. I. Ageing.** A. ULČEK. II. **Combination of pancreatin with hide substance.** A. ULČEK and J. POSPÍŠIL (Gerber, 1933, 59, 47—50; 99—100, 111—114; Chem. Zentr., 1934, i, 3297; ii, 2344).—I. The apparent enzymic activity of proteolytic bating materials containing  $(\text{NH}_4)_2\text{SO}_4$ , as determined by their action on elastin, diminished during storage, but no diminution was observed in their action on casein.

II. The hydrolytic effect of the enzyme towards casein was progressively lowered by previous contact with hide powder ( $H$ ), due to combination of the pancreatin with  $H$ . D. W.

**Damage to sheep pelts by crude naphthalene.** P. HUC (Halle aux Cuirs [techn.], 1934, 150—151; Chem. Zentr., 1934, ii, 2344).—Red or brown patches due to oxidation of phenolic impurities may develop on the skin as well as on the wool. Treatment with  $\text{NaHSO}_3$  may effect bleaching. J. S. A.

**The "first bath" of the two-bath chrome-tanning process.** L. MEUNIER and P. CHAMBARD (J. Soc. Leather Trades Chem., 1935, 19, 116—118).—The author's results (B., 1924, 684) have been confirmed by Innes (B., 1935, 419). D. W.

**Measurement of the "acidity" of vegetable-tanned leather by the acetone method.** W. R. ATKIN (J. Soc. Leather Trades Chem., 1935, 19, 186—



190).—Sound and rotted leathers (*L*) of known history were tested for free mineral acid by the Procter-Searle (*A*), Innes (*B*), Atkin-Thompson (*C*), and  $\text{COMe}_2$  (*D*) methods. Free acid was detected in the rotted *L* by methods *A*—*C*, whereas *D* showed them to be free from acid and to contain protective salts. Method *D* yields erroneous results also in presence of  $\text{MgSO}_4$  or  $\text{NaHC}_2\text{O}_4$ . Protective buffer salts must be determined by titrating the *L* extract to  $p_{\text{H}}$  3.0, using the glass electrode.

D. W.

**Acetone method for determination of sulphuric acid and buffer salts in vegetable-tanned leather.** R. F. INNES (J. Soc. Leather Trades Chem., 1935, 19, 183—185; B., 1934, 975).—Free  $\text{H}_2\text{SO}_4$  can be detected in new leathers (*L*) by the  $\text{COMe}_2$  method, but not in old and much rotted *L* owing to the formation, by the action of  $\text{H}_2\text{SO}_4$  on the *L* constituents, of a compound which is non-ionisable in 90%  $\text{COMe}_2$ . Free  $\text{H}_2\text{SO}_4$  could not be detected by this method in new *L* to which  $\text{H}_2\text{SO}_4$  and glycine or  $(\text{NH}_4)_2\text{SO}_4$ , respectively, had been added. Buffer salts, if present, can be detected by this method.

D. W.

**Gravimetric determination of the acid [radicals] in dyed chrome[tanned] leather.** M. SCHUSTER (Collegium, 1935, 181—183).—The leather is moistened with  $\text{COMe}_2$ , boiled with 25 c.c. of 0.1*N*- $\text{NaHCO}_3$ , left overnight, again boiled, the liquid poured off, the residue boiled with 6 lots of  $\text{H}_2\text{O}$ , and the combined extracts mixed with filter-paper pulp. 30 c.c. of 0.1*N*- $\text{H}_2\text{SO}_4$  and 20 g. of  $\text{NaCl}$  are added, the mixture is boiled and filtered, the filter washed with aq.  $\text{NaCl}$ , and the filtrate decolorised by boiling with  $\text{H}_2\text{O}_2$  and then titrated with 0.1*N*- $\text{NaOH}$ . The acid is calc. as %  $\text{H}_2\text{SO}_4$ . Concordant results are obtained.

D. W.

**Changes occurring during the neutralisation of chrome[tanned] leathers.** R. F. INNES (J. Soc. Leather Trades Chem., 1935, 19, 190—197).—The apparent difference in the ratio  $\text{SO}_4:\text{Cr}$  in one- ( $L_1$ ) and two-bath chrome-tanned leathers ( $L_2$ ) is brought about by the partial replacement, in  $L_2$ , of the  $\text{SO}_4$  by other acid radicals, e.g.,  $\text{S}_2\text{O}_3$ ,  $\text{S}_4\text{O}_6$ , etc. It is due neither to "free" S insol. in the degreasing solvent, nor to the presence in  $L_2$  of a compound containing nearly double the amount of  $\text{SO}_4$  present in  $L_1$ . The  $\text{HNO}_3$  method of determining  $\text{SO}_4$  in  $L_2$  yields erroneous figures. The  $\text{SO}_4$  in chrome-tanned leathers can be replaced by other acid radicals, e.g.,  $\text{PO}_4$ ,  $\text{BO}_3$ ,  $\text{SiO}_3$ ,  $\text{S}_2\text{O}_3$ , etc., without affecting the pliability of the leather.

D. W.

**Deterioration of vegetable-tanned leather on storage. VII. Protective action of salts (contd.).** R. F. INNES (J. Soc. Leather Trades Chem., 1935, 19, 109—117; cf. B., 1934, 420).—Even in the absence of  $\text{H}_2\text{SO}_4$ , the deterioration (*D*) of pyrocatechol-tanned leathers was accelerated by the presence of Fe and Cu compounds, respectively, but not by Mn salts. *D* of pyrogallol-tanned leathers was also accelerated by the presence of Fe, but not to the same extent. *D* was prevented by the presence of 5% of tartrates if the Fe content was  $\geq 0.1\%$ . The accelerating effect of Fe on *D* in presence of  $\text{H}_2\text{SO}_4$  was  $>$  that of Cu.

D. W.

**Refractive index of vegetable-tanned leathers.** R. H. MARRIOTT (J. Soc. Leather Trades Chem., 1935, 19, 133—140).—The *n* of good leathers (*L*) is  $<$  *n* for poor *L*, increases as the degree of tannage is increased, is independent of the  $\text{H}_2\text{O}$ -sol. matter but is reduced by washing the *L*, and is about 1.5797—1.5838. *n* varies from layer to layer, the grain and flesh layers showing a max. The outline of the fibres of a section of *L* mounted in a medium of low *n*, e.g., Canada balsam, is thin for good- and thick for poor-quality *L*.

D. W.

**Sampling of leather for analysis.** M. C. LAMB (J. Soc. Leather Trades Chem., 1935, 19, 129—133).—A new method of sampling is proposed in which a strip ( $\frac{1}{2}$  in. wide) is cut  $\frac{1}{4}$  in. from the outside edge of the centre line of the hide or bend, the bull side of a belly, and edge of a shoulder. Comparative analytical results are quoted for samples provided by the new and old methods of sampling the same leathers.

D. W.

**Practice and theory of protective materials in the leather industry.** K. BITTNER (Collegium, 1935, 183—186).—Known methods and materials for the protection of the wood, cement, brickwork, etc. are discussed.

D. W.

**Chemical-technical problems in the tannery.** G. GRASSER (Chem.-Ztg., 1935, 59, 413—416).—A discussion.

**Erratum.**—On p. 514, col. 1, line 15 from bottom, of "B" abstracts, 1934, for 958 read 598.

**Veneer glueing.**—See IX.

## XVI.—AGRICULTURE.

**Phosphate fertilisers by the calcination process. Factors affecting the reaction between water vapour and Florida hand-pebble phosphate.** D. S. REYNOLDS, K. D. JACOB, H. L. MARSHALL, and L. F. RADER, JUN. (Ind. Eng. Chem., 1935, 27, 87—91).—The removal of F and conversion of P into the citrate-sol. state by heating rock phosphate containing 5—10% of  $\text{SiO}_2$  in presence of  $\text{H}_2\text{O}$  requires  $<$  0.7 mg. of  $\text{H}_2\text{O}$  per c.c. of furnace gas per g. of rock per min. A moderately oxidising atm. is sufficient, but effective contact between vapour and solid must be maintained. A temp. of 1400° is necessary and this should not be much exceeded or the charge will begin to fuse. Rapid cooling from  $>$  1200° is necessary for a high yield of citrate-sol. P, probably because  $\text{Ca}_3(\text{PO}_4)_2$  undergoes an enantiotropic change at this temp. No increase in citrate-sol. P occurs until F in excess of the fluorapatite equiv. is removed. Afterwards the increase is  $\propto$  F removal.

C. I.

**Phosphate fertilisers by the calcination process. Experiments with different phosphates.** H. L. MARSHALL, D. S. REYNOLDS, K. D. JACOB, and L. F. RADER, JUN. (Ind. Eng. Chem., 1935, 27, 205—209).—In the laboratory process for removing F from phosphate rock and increasing the citrate-sol. P to 85% by heating with  $\text{H}_2\text{O}$  vapour at 1400°, rocks containing  $<$  4% of  $\text{SiO}_2$  give poor results. Some rocks containing much  $\text{SiO}_2$  fuse at 1400° and therefore also give poor results. This may be due to high Fe and Al contents. In the



former case addition of 10% of  $\text{SiO}_2$  gives improved results. Phosphate rocks from various Pacific Islands contains much less F than that indicated by the formula  $[\text{Ca}_{10}\text{F}(\text{OH})(\text{PO}_4)_6]$ , and a portion of the P is present as hydroxyapatite  $[\text{Ca}_{10}(\text{OH})_2(\text{PO}_4)_6]$  or  $\text{Ca}_3(\text{PO}_4)_2$ . In these cases an increase in citrate-sol. P results from heating at  $1400^\circ$  in a dry atm. With  $\text{H}_2\text{O}$  vapour  $\text{SiO}_2$  is also needed. As shown by pot tests, the plant-food val. of the P in these calcined phosphates is as high as that of the P in superphosphate. They are obtained as sintered clinkers and require only grinding. C. I.

**Manurial trials with slag phosphate, "triple superphosphate," and dicalcium phosphate.** E. TRUNINGER (Landw. Jahrb. Schweiz, 1934, 48, 495—520; Chem. Zentr., 1934, ii, 3164—3165).—Comparative trials on meadowland are recorded. The reaction of the soils had no great influence on the action of the P fertilisers.

A. G. P.

**Behaviour of small amounts of phosphate fertiliser in soils of different composition.** L. MEYER and U. VON RENNENKAMPFF (Phosphorsäure, 1934, 4, 24—42; Chem. Zentr., 1934, ii, 3165).—Wrangell's method indicates the fate of additions of P fertilisers and yields reliable information as to the fixative properties of the soil, and its ability to maintain supplies of this nutrient for the plant.

A. G. P.

**Reversion of superphosphate in soil.** W. LUKASZEWICZ (Rocz. Nauk. roln. leśn., 1934, 32, 79—86; Chem. Zentr., 1934, ii, 3165).—Reversion of superphosphate in soils occurs principally during the first day of application and results in the formation of  $\text{CaHPO}_4$  (I) and  $\text{Ca}_3(\text{PO}_4)_2$  together with insol. phosphates of Al, Fe, Mg, and Mn. In soils poor in salts (I) is the principal product.

A. G. P.

**Distribution and action of phosphatic fertilisers.** S. GERICKE (Phosphorsäure, 1934, 4, 79—97; Chem. Zentr., 1934, ii, 3165).—The efficiency of P fertilisers is lowered by distribution through a large amount of soil and reaches a max. when conc. in a layer at a depth appropriate to the individual crop.

A. G. P.

**Nitrogenous composition of ammoniated peat and related compounds.** L. A. PINCK, L. B. HOWARD, and G. E. HILBERT (Ind. Eng. Chem., 1935, 27, 440—445).—Ammoniation of peat (cf. B., 1933, 1026), lignin, and starch gives amorphous products containing 12.2, 9.1, and 5.4% N, respectively, most of which, classed as "amide"-N (arbitrary classification), is extracted with  $\text{H}_2\text{O}$ . Similarly treated, glucose gives a liquid which when heated affords  $\text{NH}_3$ ,  $\text{NH}_2\cdot\text{CO}_2\text{NH}_4$ , and 2:6-dimethylpyrazine, and gives an amine on reduction, as well as a granular substance which contains a mixture of pyrazines (also isolated from ammoniated starch) and urea. Urea is formed in every case. Further ammoniation of the N fractions gives polymerides which probably comprise the "insol. N."

J. L. D.

**High-nitrogen [fertiliser] material from urea-ammoniated peat.** R. O. E. DAVIS, W. SCHOLL, and R. R. MILLER (Ind. Eng. Chem., 1935, 27, 69—71).— $\text{NH}_2\cdot\text{CO}_2\text{NH}_4$  and peat, with or without added liquid  $\text{NH}_3$ , were heated in a bomb at  $180^\circ$  for 4 hr. Max. conversion into urea occurred with 275% excess  $\text{NH}_3$ .

Presence of  $\text{H}_2\text{O}$  in peat increases the amount of  $\text{NH}_3$  required for max. conversion. With a urea content of < 60% the finished product is non-hygroscopic. The greater part of the N is sol. and the remainder active under proper conditions.

C. I.

**Soils containing urea.** A. J. J. VANDE VELDE (Natuurwetensch. Tijds., 1935, 17, 57—63).— $\text{AcOH}$  and  $\text{CHCl}_3\cdot\text{CO}_2\text{H}$  are more active than  $\text{CH}_2\text{Cl}\cdot\text{CO}_2\text{H}$  and  $\text{CCl}_3\cdot\text{CO}_2\text{H}$  in accelerating the hydrolysis of urea in 0.1N-acid at  $100^\circ$ .  $\text{Na}^+$  and  $\text{SO}_4^{2-}$  increase the rate of hydrolysis,  $\text{K}^+$  has a slight positive effect, and  $\text{NH}_4^+$  retards hydrolysis.

S. C.

**Denitrification [of soil] in sunlight and its retardation.** N. R. DHAR, S. P. TANDON, and S. K. MUKHERJI (J. Indian Chem. Soc., 1935, 12, 67—81).—Apart from the well-known anaërobic denitrification of soils, a loss of N in presence of  $\text{O}_2$  has frequently been observed, but is not yet satisfactorily explained. Further, the loss of N observed when a manure is composted under aerobic conditions is > that under anaërobic conditions. The rate of loss is decreased by the presence of large amounts of C compounds. The amount of N lost by exposure of solutions of  $\text{NH}_4$  salts to air in the light is > that in the dark, but both light and dark reactions may be catalysed by such substances as  $\text{ZnO}$  and  $\text{TiO}_2$ . Solutions of  $\text{NH}_4\text{Cl}$  and  $\text{NaNO}_2$ , mixed with sterilised and unsterilised soils, also decompose readily with evolution of  $\text{N}_2$  in sunlight in open beakers. The action is retarded by cane sugar. It is concluded that in the processes of ammonification and nitrification in the soil or in solution,  $\text{NH}_4\text{NO}_2$  (I) is produced if  $\text{O}_2$  is available, and is decomposed into  $\text{N}_2$  and  $\text{H}_2\text{O}$  by heat or light. It is suggested that such substances as molasses might be added to the soil to retard the formation and decomp. of (I) and also to increase N fixation as shown by the increase of  $\text{NH}_3$ -N in the soil.

M. S. B.

**Effect of lime and neutral calcium salts on solubility of soil potassium.** M. PEECH and R. BRADFIELD (Amer. Soil Survey Assoc., Rept. 14th Ann. Meet. Bull., 1934, 15, 101—106).—Electrodialysed clay partly saturated with K and Ca showed no conversion of exchangeable K (I) into non-exchangeable forms after 2 years. In clay-biotite mixtures saturated to varying extents with Ca the decline in (I) with ageing decreased with rising  $p_{\text{H}}$ . Adsorbed K in colloidal clay lowered the (I) of biotite in the mixtures. Similar but smaller effects were observed in clay-orthoclase mixtures. Addition of  $\text{CaO}$  to acid soils decreased the  $[\text{K}^+]$  of the soil solution. On Ca-saturated soils liming liberated adsorbed K (II).  $\text{CaSO}_4$  liberated (II) regardless of the Ca-saturation of the soil. Adsorption of K by clay from  $\text{KOAc}$  was > that from  $\text{KCl}$ . The effects of Mg resembled those of Ca except that excess of  $\text{MgCO}_3$  gave lower vals. for (II) than did  $\text{CaCO}_3$ .

CH. ABS. (p)

**Compost manure.** A. W. R. JOACHIM and S. KANDIAH (Trop. Agric., 1934, 83, 277—293).—Analytical data show the course of decomp. and nature of composts from street refuse, night soil, etc. Addition of  $\text{CaO}$  offers no advantage in composting.

A. G. P.

**Meadow-manuring trials in Quartino (Tessin).** F. WERNER (Landw. Jahrb. Schweiz, 1934, 48, 572—



587; Chem. Zentr., 1934, ii, 3164).—Neubauer's method indicated P and K requirements more accurately than did hay analyses. The P of fertilisers was utilised more satisfactorily when K was used, and still more so when K and N were supplied simultaneously. Similarly the K of fertilisers was assimilated more effectively in presence of P and N. A. G. P.

**Has manuring an influence on scab formation in potatoes?** E. HÖHNE and G. CHÉLARD (Phosphorsäure, 1934, 4, 161—167; Chem. Zentr., 1934, ii, 3031).—The influence of soil reaction on the incidence of scab is less important than is usually supposed. The generous use of green or cattle manures and the sowing of resistant varieties provide the best protective measures. A. G. P.

**Plum-fruit sawfly and its control.** H. W. MILES (J. Min. Agric., 1935, 42, 129—133).—Spraying with nicotine (8 oz. per 100 gals. with a spreader) 10 days after petal fall gave satisfactory control. Two applications within a week are recommended. A. G. P.

**Zinc sulphate as corrective for bronzing of tung trees.** H. MOWRY and A. F. CAMP (Florida Agric. Exp. Sta. Bull., 1934, No. 273, 34).—Bronzing is due to Zn deficiency and is remedied by application of  $ZnSO_4$  to soil or in spray form to the plant. A. G. P.

**Action of urea on  $CaH_2PO_4$ .**—See VII. **Utilising agricultural waste.**—See XVIII. **Derris root etc.**—See XX. **Pest-destroying agents.**—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

**Impurities in white sugars. VII. Distribution of impurities in the sugar crystal.** J. C. KEANE, J. A. AMBLER, and S. BYALL (Ind. Eng. Chem., 1935, 27, 30—33; cf. B., 1932, 1099).—Sugar (*S*) crystals of uniform size were suspended in *S* solution of varying concn., so that amounts varying from 4.3 to 30.0% of the crystals were dissolved. > 50% of the ash,  $SO_4^{2-}$ , Cl<sup>-</sup>, Na, K, and total N was found in the outer 5% of the crystal, whilst colour, Ca, and  $SO_3^{2-}$  are more uniformly distributed. The colour of *S* candy made from this *S* was markedly improved by removal of the outer layer, suggesting that this is a possible means of improving the colour of white *S*. E. C. S.

**Colouring substances of cane sugar. I.** I. SAKUMA and I. MOMOSE (J. Soc. Chem. Ind., Japan, 1935, 38, 161—163 B).—The absorption curves of saccharetin (I) and of the colouring substance (II) isolated from sugar cane attacked by a fungus (*Colletotrichum falcatum*, Went) have been measured at various  $pH$ . At  $pH$  7.0 the ratio of the absorption at 4720 Å. to that at 5900 Å. is 1.886 for (I) and 2.64 for (II). H. G. M.

**Hygrosopicity of sugars and sugar mixtures.** J. H. DITTMAR (Ind. Eng. Chem., 1935, 27, 333—335).—Graphs are given by means of which the %  $H_2O$  in equilibrium with glucose, fructose, sucrose (I), invert sugar (II), and mixtures of (I) and (II) at different R.H. can be determined. E. C. S.

***Aërobacter aërogenes* as cause of ropiness in maple syrup.** F. W. FABIAN and H. H. BUSKIRK

(Ind. Eng. Chem., 1935, 27, 349—350).—Bacteria agreeing in all essential details with *A. aërogenes*, isolated from maple sap, produced a ropiness (*R*) in the syrup (*S*). None of the known cultures of *Escherichia* or *Aërobacter* produces *R*. AcOH added to *S* caused no *R*, but lactic acid caused slight *R*. E. C. S.

**Evaluation of textile starch.** W. SECK (Melliands Textilber., 1934, 15, 368—371; Chem. Zentr., 1934, ii, 3335).—A crit. discussion of  $H_2O$ , ash, and  $\eta$  determinations. H. J. E.

**Cellulose from bagasse.**—See V. **Liquid sugar and spirits. Alcohols by fermentation.**—See XVIII. **Heather honey.**—See XIX. **Sterilisation [of starch].**—See XX. **Starch nitrates.**—See XXII.

## PATENT.

**Centrifugal separation [of sugar liquors].**—See I.

## XVIII.—FERMENTATION INDUSTRIES.

**Modern chemistry applied to the fermentation industry.** H. FINK (Z. Spiritusind., 1935, 58, 155—156, 158—159).—A lecture.

**Determination of barley extract.** H. LÜERS and P. VON MILLER (Woch. Brau., 1935, 52, 153—154).—Finely-ground barley, mixed with  $H_2O$ , is treated with a solution of superclastase to liquefy the starch and the temp. is raised gradually to 85°. After subsequent boiling and cooling to 45°, diastase "Merck" is added and the temp. held at 45—50° for 10 min. and then raised gradually to 75°. The solution is cooled, its vol. adjusted, and, after filtration, the *d* is determined and corr. for increments due to the enzyme prep. The operations throughout are carried out under specified controlled conditions. The results are in good agreement with those obtained by Graf's method. I. A. P.

**Source of danger for beer stability.** I. JANENSCH (Woch. Brau., 1935, 52, 154—156).—Beer which froths over from transport casks is contaminated with organisms of beer disease as well as with harmless air-borne organisms. It should be collected separately and filtered or pasteurised before further use. I. A. P.

**Effect of cold and freezing storage on wine composition.** M. A. JOSLYN and G. L. MARSH (Ind. Eng. Chem., 1935, 27, 33—35).—The subject of the clarification of wine (*W*) by cooling is reviewed. The effect of storage of red and white *W* at room temp. for 34 days, at 0° for 41 and 12 days (with and without decantation or filtering), and at -17° for 12 days on *d*, and on the % of EtOH, total and volatile acid, tartaric acid, cream of tartar, ash, extract, N, tannin and colouring matter, and reducing sugar is investigated. E. C. S.

**Liquid sugar as source of spirits.** W. L. OWEN and R. L. MOBLEY (Amer. Wine J., 1935, reprint, 4 pp.).—Fermentation (*F*) of solutions of maize syrup (I) with yeast in presence of inorg. nutrients [( $NH_4$ )<sub>2</sub>SO<sub>4</sub>, CaHPO<sub>4</sub> (?)] gave low yields of EtOH, but *F* in presence of "corn solubles" (II) (containing protein, mineral matter, and carbohydrates) gave much increased yields, which with (II), activated C, and added molasses (10—11.33%) were very satisfactory. With the high efficiency of *F* obtained,



it is possible that (I) may prove a cheaper source of EtOH than grain. I. A. P.

**Utilisation of agricultural wastes. II. Influence of nitrogenous substrate on production of butyl and isopropyl alcohols by *Clostridium butylicum*.**

O. L. OSBURN and C. H. WERKMAN (Ind. Eng. Chem., 1935, 27, 416—419; cf. B., 1935, 432).—The effect of addition of peptone, malt sprouts (I), "corn steep" (II), and various hydrolysed proteins on the production of Pr<sup>o</sup>OH and BuOH (III) from by-product glucose (IV) syrup by fermentation with *C. butylicum* is investigated. Addition of the growth-stimulating factor (cf. A., 1934, 563) present in yeast extract or (II) increases the yield of (III) from maize mash from 3 to 14%. With mixtures of (II) and (I) or maize-gluten as sources of N, 4% aq (IV) yields 20—25% of (III) and 4—9% of Pr<sup>o</sup>OH.

E. C. S.

**Purification of fermentation gases by carbonic acid-washing and by active charcoal filters.**

F. REINDEL, A. FREY, and E. MALENKE (Z. Spiritusind., 1935, 58, 147—148).—In agricultural distilleries, the use of active C filters for purifying fermentation gases from potato mashes offers at present no notable advantages over the usual washing process. I. A. P.

**Fermentation of hemicellulose.**—See V. **Apple pectin.**—See XIX.

**XIX.—FOODS.**

**Temperature inside loaves [rolls, and cakes].**

A. VAN RAALTE and R. M. N. A. MALOTAUX (Chem. Weekblad, 1935, 32, 256—257).—The method of distinguishing rolls etc. fresh from the oven from those taken from a refrigerator, by measuring their temp., is not reliable, since the temp., particularly at the surface, of rolls from the oven may fall > 2° below atm. owing to rapid evaporation of H<sub>2</sub>O. D. R. D.

**Brown milk.** R. STROHECKER (Deuts. Nahrungsm.-Runds., 1934, 130; Chem. Zentr., 1934, ii, 3195).—The colour is due to enzymic oxidation of tyrosine by *B. fluorescens*. Methods of prevention are discussed. H. J. E.

**Simplified molecular constants of Belgian milk.**

VIVARIO and STAINIER (J. Pharm. Belg., 1934, 16, 631—633, 647—649; Chem. Zentr., 1934, ii, 3064; cf. B., 1932, 366).—Due allowance must be made for the lactose content (I) of the milk. In the neighbourhood of Brussels (I) is 4.9—5.0%.

H. N. R.

**Milk of abnormal f.p.** H. OESTERMANN (Pharm. Zentr., 1935, 76, 300—301; cf. B., 1935, 171).—Analyses of milks with f.p. depressions of 0.499—0.505° and 0.493—0.498°, obtained from two healthy cows (one being the calf of the other), are given. E. H. S.

**Microbiology of butter. I. Yeast and mould count of butter as a measure of creamery sanitation.**

F. W. WOOD and H. R. THORNTON. **II. Growth of moulds in and on butter.** H. R. THORNTON and F. W. WOOD (Canad. J. Res., 1935, 12, 286—294, 295—305).—I. Data showing yeast (I), mould (II), and bacterial (III) counts of butter and cream before and after pasteurisation indicate that (III) nos. should supplement the

customary (I) and (II) counts in characterising creamery sanitation. After melting for 4 hr. butter should show little change in microbiological population.

**II. Mould infection of salted butter from contained H<sub>2</sub>O is unlikely.** It arises probably from moisture condensed on the surface. Use of parchment soaked in brine gave best protection against moulding. Mould counts and the development of mouldiness were not related. A. G. P.

**Effect of malvaceous seeds on stored-egg quality.**

F. W. LORENZ and H. J. ALMQUIST (Ind. Eng. Chem., 1934, 26, 1311—1313).—The eggs of hens fed on seeds of malvaceous plants (e.g., cotton, hollyhock), crude kapok oil, or crude or partly refined cottonseed oil develop a pink coloration in the white during storage. The oil from these seeds gives the Halphen test (H), as does also the fat from the yolk of pink-white eggs. The inclusion in the feed of materials likely to give rise to pink-white can be detected by applying H to the feed. E. C. S.

**Growth of moulds on chilled meat under various air conditions.**

G. KAESS (Z. ges. Kälte-Ind., 1934, 41, 96—102; Chem. Zentr., 1934, ii, 3327).—The effect of variations of temp. and humidity on the mould growth is recorded. H. J. E.

**Determination of nitrates in meat. II.**

F. T. VAN VOORST (Chem. Weekblad, 1935, 32, 295—296).—The method described previously (B., 1933, 282) is modified. 10 g. of minced sample are extracted with 100 c.c. and then with 4 × 50 c.c. of boiling H<sub>2</sub>O, the filtered extract is made up to 500 c.c., and 25 c.c. are treated with 10 c.c. of 0.5% AgNO<sub>3</sub> solution and 2 c.c. of 0.01N-K<sub>4</sub>Fe(CN)<sub>6</sub> and filtered. 10 c.c. of the filtrate (made up to 50 c.c.) are evaporated to dryness, treated with phenolsulphonic acid, and the nitrophenol is determined colorimetrically after making alkaline with NH<sub>3</sub>. S. C.

**Determination of pentosans in foodstuffs.**

O. FERNÁNDEZ and M. DE MINGO (Anal. Fis. Quím., 1935, 32, 382—390).—The pentosans are converted by dil. HCl into furfuraldehyde, which is determined as 2:4-dinitrophenylhydrazone. The results are compared with those given by the method of Krüger and Tollens (A., 1896, ii, 393). F. R. G.

**Ratio between the calcium and phosphorus contents in *Merlangus vulgaris* and *Scyllium canicula*.**

A. DE CLERCQ (Natuurwetensch. Tijds., 1935, 17, 49—56).—A study of the P and Ca contents of *M. vulgaris* (I) and *S. canicula* shows that they are suitable for human consumption. In (I) P and Ca decrease with increase in wt.; male are richer in both constituents than female fish. No seasonal variation was noted. S. C.

**Peroxidase in darkening of apples.**

A. K. BALLS and W. S. HALE (Ind. Eng. Chem., 1935, 27, 335—337).—The browning of apple slices is prevented by spraying with substances containing SH groups, e.g., glutathione, cysteine hydrochloride, or strongly proteolytic pineapple juice (I). The action of (I) is due to the activator of bromelin. E. C. S.



**Viscosity of apple pectin. Effect of commercial diastatic enzymes at 40°.** G. L. BAKER and R. KNEELAND (Ind. Eng. Chem., 1935, 27, 92—94).—Four preps. of diastase (*D*) lowered the  $\eta$  of a pectin (*P*) solution between  $p_H$  3.3 and 5.0 at 40°. Hydrolysis was at a min. at  $p_H$  3.3. In using *D* to remove starch from preps. of *P*, conditions should be chosen which are least detrimental to *P*.  
E. C. S.

**Determination of water content of fruit products.** A. BEYTHIEN (Z. anal. Chem., 1935, 99, 391—397).—H<sub>2</sub>O is most accurately determined from the *d* of a 10% aq. extract by the Beythien-Simmich method (B., 1910, 1223), correction being made for EtOH and insol. matter. For process control, bulk *d* or measurement of *n* is advocated.  
J. S. A.

**Thixotropy of heather honey.** G. W. S. BLAIR (J. Physical Chem., 1935, 39, 213—219).—Heather-honey (*H*) "gel" shows rigid properties since, at low shearing stresses rapidly applied, the  $\eta$  of the system  $\equiv$  that of an elastic solid, but the elastic limit is not sharp. The change to sol form on stirring is continuous, and although there is still an increase in  $\eta$  at low stresses the final deviation from true fluidity is not great. A semi-quant. test for thixotropy is described which distinguishes the behaviour of *H* from that of clover honey or golden syrup. It may be useful in deciding whether excess H<sub>2</sub>O in honey is due to unripeness and may lead to fermentation, or whether it is natural owing to a heather origin. It is possible that honey of unmixed origin, derived from *Erica* alone, is not thixotropic.  
M. S. B.

**Food storage and transport.** F. KIDD (Nature, 1935, 135, 739—741).—Improvements in the technique of storage by the use of low temp. and of different atm. are described.  
L. S. T.

**Lucerne hay for milk production.** I. R. JONES, P. M. BRANDT, and J. R. HAAG (Oregon Agric. Exp. Sta. Bull., 1934, No. 328, 30 pp.).—Milk yields from cows receiving long lucerne hay (I) were < those from a chopped hay diet and, in turn, much < those from a chopped hay-grain ration. The inferiority of (I) was due to insufficient consumption of total nutrients, and probably also to lack of P.  
A. G. P.

**Chemical analyses of New South Wales pasture plants.** C. E. AHRENS (Agric. Gaz. N.S. Wales, 1934, 45, 639—640).—The nutritive val. and protein ratio (I) of 18 species are given. (I) varies from 1:1.97 in burr trefoil to 1:5.90 in prairie grass.

NUTR. ABS. (*m*)

**Feeding value and nutritive properties of citrus by-products. I. Digestible nutrients of dried grape-fruit and orange cannery refuses and the feeding value of grape-fruit refuse for growing heifers.** W. M. NEAL, R. B. BECKER, and P. T. D. ARNOLD (Florida Agric. Exp. Sta., 1935, No. 275, 26 pp.).—The products are characterised by low crude fibre, protein, and fat contents, high N-free extract, high digestibility coeffs., and good palatability. Data of feeding trials are recorded.  
A. G. P.

**Influence of loft- and frame-drying on the mineral content of dried fodder.** E. CRASEMANN

(Landw. Jahrb. Schweiz, 1934, 48, 588—597; Chem. Zentr., 1934, ii, 3195).—The loss of mineral constituents is greater in frame- than in loft-drying under favourable conditions.  
H. N. R.

**Haddock meal. Effect of manufacturing process on nutritive values.** H. S. WILGUS, JUN., L. C. NORRIS, and G. F. HEUSER (Ind. Eng. Chem., 1935, 27, 419—422).—Dry-rendered meals had a protein val. (I) for chicks > that of wet-rendered, and 50% more vitamin-G (II). Flame-dried meal was of inferior val. (II) was increased by steam-drying in vac. as compared with that at atm. pressure, but (I) was not affected. The (II) content was best preserved when the raw scrap was not preground.  
E. C. S.

**Vegetable lecithin. Digestibility etc. of fats. Edible rapeseed oil.**—See XII.

#### PATENTS.

**Production of meat extract from whales, seals, and other mammiferous sea-animals.** J. O. NYGAARD (B.P. 427,455, 24.10.33. Addn. to B.P. 426,357; B., 1935, 523).—The final extraction may be made with cold and boiling H<sub>2</sub>O, and with H<sub>2</sub>O at intermediate temp.  
E. B. H.

**Production of tea entirely or almost entirely free from theine.** T. C. W. GRETHE (B.P. 427,191, 31.10.33. Ger., 17.11.32. Addn. to B.P. 384,197; B., 1933, 171).—Tea, after moistening with H<sub>2</sub>O and extraction by a theine solvent, is treated with H<sub>2</sub>O vapour at > 65°.  
E. B. H.

**Edible fats and oils.**—See XII. **Pest-destroying agents.**—See XXIII.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Effect of sterilisation on solutions of [B.P.] calcium chloride.** C. E. COULTHARD and G. F. HALL (Quart. J. Pharm., 1935, 8, 96—97).—Sterilisation by autoclaving or tyndallisation of aq. CaCl<sub>2</sub> (up to 10%) produces marked deposition (mainly CaCO<sub>3</sub>) when the CaCl<sub>2</sub> is not of sufficiently high purity (although within B.P. specification).  
F. O. H.

**Sterilisation of injection of bismuth, B.P.** C. E. COULTHARD (Quart. J. Pharm., 1935, 8, 98—99).—Preps. packed under non-sterile conditions are sterilised by autoclaving and, if not heavily infected, by tyndallisation.  
F. O. H.

**Sterilisation by dry heat at 150°, with special reference to oils.** C. E. COULTHARD (Quart. J. Pharm., 1935, 8, 90—93).—Dry heat at 150° for 1 hr. sterilises starch, straw, paraffin, and olive oil etc. infected with highly resistant spores. The oils are not significantly decomposed by the treatment.  
F. O. H.

**Sterilisation of oils.** R. A. O'BRIEN and H. J. PARISH (Quart. J. Pharm., 1935, 8, 94—95).—Heating at 150° for 1 hr. sterilises heavily infected oils (medicinal paraffin, almond, olive); tyndallisation (1 hr. at 80° on 3 successive days) is not reliable.  
F. O. H.

**Assay of strong ointment of mercuric nitrate [B.P.].** C. H. HAMPSHIRE and G. R. PAGE (Quart. J. Pharm., 1935, 8, 75—80).—Proposed methods of assay were found not to be superior to that of the B.P.



method; the latter, however, is advantageously modified by substituting titration with 0.1N-NH<sub>4</sub>SCN for gravimetric determination of Hg as HgS. F. O. H.

**Pharmacopœial tests. II. Chiniofon, codeine, simple solution of iodine, sodium phosphate.** G. R. PAGE (Quart. J. Pharm., 1935, 8, 81—89).—Methods are given for the determination of I and NaHCO<sub>3</sub> in chiniofon and for the detection of < 0.2% of NaF in Na phosphate. Codeine monohydrate is sol. in approx. 75 pts. of Et<sub>2</sub>O at 15.5°. The I content of EtOH-I becomes const. after keeping for 8 months, but the acidity continues to increase slightly. The rate of reaction and final composition are not appreciably affected by light.

F. O. H.

**Assay and stability of commercial pepsin.** K. BULLOCK (Quart. J. Pharm., 1935, 8, 13—30).—The B.P., U.S.P., D.A.B., and edestin methods of pepsin (I) assay are compared and modifications suggested. Errors due to variations in the substrate (egg-white) are avoided by using standard (I) preps. or averaging the results of many tests. Commercial (I) is generally of the activity claimed.

F. O. H.

**Coloration of pyrimidone by some oxidising agents.** M. WAGENAAR (Pharm. Weekblad, 1935, 72, 564—566).—Pyrimidone (I) gives a semi-permanent violet colour with K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> solution (II). The test is very sensitive and 0.5% of (I) in antipyrine can be detected. (II) has the advantage as an oxidising agent that further oxidation and bleaching of the violet compound takes place very slowly.

S. C.

**Manufacture of ichthyol preparations from [Polish] native raw materials.** S. GEBSKI (Rocz. Farm., 1933, 11, 5—77; Chem. Zentr., 1934, ii, 3006—3007).—The possibilities of obtaining ichthyol preps. from such materials as linseed oil (I), poppyseed oil (II), a wood-oil distillate, gas oil, and anthracene oil have been investigated. (I) and (II) were unsuitable, but, by methods of sulphuring and sulphonation (described) successful preps. were obtained from the others. The efficiency of the preps. ∝ the proportion of sulphide-S in the dried substance.

H. N. R.

**Seeds of *Strophanthus emini*.** ANON. (Quart. J. Pharm., 1935, 8, 61—70).—The seeds of *S. emini* are distinguished from those of other species by colour tests with H<sub>2</sub>SO<sub>4</sub>, PhOH-HCl, furfuraldehyde, and resorcinol-HCl, but the derived glucosides, tinctures, etc. are chemically and pharmacologically similar to those from seeds of *S. kombé*.

F. O. H.

**Strophanthin of *Strophanthus emini*.** I. D. LAMB and S. SMITH (Quart. J. Pharm., 1935, 8, 71—74).—Strophanthin (I) from the seeds of *S. emini* resembles *k*-(I) in its general characteristics, responses to B.P. tests, and cardiotoxic activity; differentiation is achieved by the composition and colour reactions (cf. preceding abstract).

F. O. H.

**Chemical determination of the value of plant material (derris root, *Lonchocarpus* root, etc.) containing rotenone.** P. A. ROWAAN (Chem. Weekblad, 1935, 32, 291—295).—The polarisation method for the determination of rotenone (I) is not accurate. In the preferred method, 50 g. of material (passing a 0.5-mm.

sieve) are extracted at room temp. with 500 c.c. of CHCl<sub>3</sub> and the extract is evaporated. The residue is dissolved in 20 c.c. of boiling CCl<sub>4</sub> and the solution cooled in an ice-chest, when (I) crystallises completely as the CCl<sub>4</sub> solvate containing 72% of (I), which is filtered off and dried at room temp. S. C.

**[Composition of] tobacco smoke.** A. WENUSCH (Pharm. Zentr., 1935, 76, 297—300).—The smoke (S) of cigarettes (Oriental) and cigars consists of discreet particles of nicotine (I) salts, resins, resin acids, and higher hydrocarbons together with air, CO, CO<sub>2</sub>, H<sub>2</sub>O vapour, etc. S is not an aq. cloud and (I) is present in only small amounts in cigarette S, which has an acid reaction, but in considerable quantities, varying with the origin of the tobacco and the length of the cigar, in cigar S (cf. B., 1935, 381; 1934, 780).

E. H. S.

**French pine oil.** L. DESALBRES (Bull. Inst. Pin, 1935, 48—49).—The surface tensions at 18° of aq. solutions (1:1000) of "double-distilled oil" (solubility < 1:1000), "Dertol no. 90" (I), Yarmor pine oil, "Rayon oil," and pure terpineol are, respectively, 48.4, 47.6, 47.6, 47.3, and 48.0 dynes/sq. cm. (I) is claimed as an efficient substitute for the American oils.

E. H. S.

## PATENTS.

**Manufacture of *l*-2-ketogulonic acid [and its esters].** T. REICHSTEIN (B.P. 427,286, 15.10.34. Switz., 25.10.33).—Derivatives of *l*-sorbose (I) with CO-compounds are subjected to a mild oxidation [alkaline KMnO<sub>4</sub> or K<sub>3</sub>Fe(CN)<sub>6</sub>] and the resulting acids hydrolysed or esterified, using an acid catalyst. *E.g.*, *l*-diisopropylidene-sorbose, m.p. 77—78°, [α]<sub>D</sub><sup>25</sup>—18.1° in COMe<sub>2</sub>, is oxidised (aq. KOH-KMnO<sub>4</sub>), and the resulting acid, m.p. 75—77° (clear at 95°) [K salt, m.p. > 300° (decomp.)], is hydrolysed by heating with H<sub>2</sub>O at the b.p. Alternatively, the (CHPh)<sub>2</sub> derivative of (I) is oxidised (KOH-KMnO<sub>4</sub> in aq. C<sub>5</sub>H<sub>5</sub>N) and the resulting *l*-2-ketodibenzylidene-gulonic acid, m.p. 202—204°, boiled with dil. aq.-alcoholic HCl. *d*-Gulonic acid has m.p. 170° (corr., decomp.), [α]<sub>D</sub><sup>25</sup>—48° in H<sub>2</sub>O (*Me*, m.p. 155—157°, [α]<sub>D</sub><sup>25</sup>—25° in MeOH, and *Et* ester, [α]<sub>D</sub><sup>25</sup>—14.5° in EtOH).

H. A. P.

**Manufacture of 4-alkyl-, 4-cycloalkyl-, and 4-aralkyl-dihydroresorcinols.** F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 427,297, 17.12.34. Ger., 20.1.34. Addn. to B.P. 416,892; B., 1934, 1050).—The process of the prior patent is applied to the corresponding 4-substituted resorcinols. *E.g.*, the Na salt of 4-β-phenylethylresorcinol is reduced in H<sub>2</sub>O (H<sub>2</sub>-Niekieselguhr at 80°/15 atm.) to the H<sub>2</sub>-compound, m.p. 126°. Other examples describe the prep. of 4-*hexyl*-, m.p. 64°, 4-*propyl*-, m.p. 70°, and 4-cyclohexyl-dihydroresorcinol, m.p. 149°.

H. A. P.

**Manufacture of compounds of 1-phenyl-2:3-dimethyl-4-isopropyl-5-pyrazolone [with barbituric acids. Pharmaceuticals].** F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 427,296, 27.11.34. Ger., 8.1.34).—The components are caused to interact in an inert diluent; the reaction is applicable only to *CC*-disubstituted barbituric acids having > 1 unsaturated substituent.



Examples are the *compounds*: 1-phenyl-2:3-dimethyl-4-isopropylpyrazolone + allylisopropyl-, m.p. 128°, *C*-phenyl-*C*-ethyl-, m.p. 161°,  $\beta$ -bromoallylisopropyl-, m.p. 134—135°, and diethyl-barbituric acid (equimols.), m.p. 138—139°.

H. A. P.

**Conditioning of tobacco and similar materials.**

G. D. HARRIS, ASSR. TO INDUSTRIAL DRYER CORP. (U.S.P. 1,971,405, 28.8.34. Appl., 2.11.29. Renewed 29.1.34).—Tobacco which is so dry as to be brittle is made flexible by contact with circulating air which has been moistened by passage over heated H<sub>2</sub>O, cooled, and again moistened.

B. M. V.

**Desiccating liquids and semi-solids.**—See I. C<sub>5</sub>H<sub>5</sub>N compounds.—See III. [Surgical] threads etc.—See V. Shaving creams etc.—See XII. Theine from tea.—See XIX.

**XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.**

**Grain isolation by dyes.** LÜPPO-CRAMER (Phot. Korr., 1934, 70, 113—114).—Dyes (I) which fog AgBr emulsions (II) are without action on AgI emulsions, due to greater stability of the colloid-AgI complex. The extent to which the fogging of (II) by (I) is removable by bathing in solutions of, *e.g.*, acridine-yellow or quinoline-red has been determined when using various kinds of (I).

J. L.

**Production of fogging by dyestuffs.** LÜPPO-CRAMER (Phot. Ind., 1934, 32, 974; Chem. Zentr., 1934, ii, 3212).—Photographic layers which are coloured by fog-producing dyes (I) still give a faint fog on development, even after pretreatment with CrO<sub>3</sub> mixture to remove Ag nuclei. The (I) interfere with the action of gelatin as a protective colloid.

H. J. E.

**[Photographic] over-sensitisation.** LÜPPO-CRAMER (Phot. Korr., 1934, 70, 129—131; Chem. Zentr., 1934, ii, 3212).—Over-sensitisation (I) is attributed to removal of excess Br ions, which are always present in commercial emulsions. (I) is best carried out by washing in H<sub>2</sub>O.

H. J. E.

**PATENTS.**

**Manufacture of photographic silver halide emulsion.** SOC. CHEM. IND. IN BASLE (B.P. 428,305, 9.12.33. Switz., 10.12.32).—Emulsions with better gradation and range of sensitivity are produced by withdrawing [and cooling to stop ripening (*R*)] < 2 portions of the emulsion from a batch of emulsion during the *R* process, the portions being reunited afterwards by mixing in a common vessel. Alternatively, the emulsion may be run off slowly and continuously from the *R* tank. The process may be repeated for a second *R* of the solidified, comminuted, and washed emulsion.

J. L.

**Light-filtering overcoating.**—See XIII.

**XXII.—EXPLOSIVES; MATCHES.**

**Preparation and properties of starch nitrates.** III. Explosive properties. J. HACKEE and T. URBAŃSKI (Z. ges. Schiess- u. Sprengstoffw., 1935, 30, 98—101; cf. A., 1934, 282).—The N contents of 12 samples varied from 13.43 to 6.49%. In 10 samples

detonation velocities ranged from 6190 to 1000 m./sec., Pb expansions from 430 to 125 c.c., and fall-hammer results from 0.825 to 8.5 kg./m. Compressions of 7 × 10.5-mm. crushers of 9 samples ranged from 2.05 to 0.24 mm. Nitrates of < 9% N are practically non-explosive.

W. J. W.

**Chemical stability of explosives.** A. HAID, F. BECKER, and P. DITTMAR (Z. ges. Schiess- u. Sprengstoffw., 1935, 30, 66—68, 105—108).—After being dried in vac. over P<sub>2</sub>O<sub>5</sub> at 50°, the explosive is introduced into a flask provided with a manometer, and heated at 75°. Daily readings of the pressures developed give an indication of the progress of decomp. Curves are shown for a no. of explosives. Some comparative results obtained in the 100° stability test and by Hansen's method are shown.

W. J. W.

**Modification of the Bergmann-Junk-Mayrhofer stability test for smokeless powders.** J. VON MEERSCHIEDT-HÜLLESSEM (Z. ges. Schiess- u. Sprengstoffw., 1935, 30, 73—75).—With the apparatus described previously (B., 1934, 798) there is risk of explosion when the test-tube is withdrawn. In a modified type the tube is not removed after the 2 hr. heating, but is cooled and rinsed out while in the thermostat.

W. J. W.

**Energy and capacity for work of explosives on detonation.** A. SCHMIDT (Z. ges. Schiess- u. Sprengstoffw., 1935, 30, 75—79, 101—104, 145—151).—Tables show how, for const. explosion temp. and const. gas vol. (*i.e.*, sp. pressure), the capacity for work  $\propto d$ . The capacity for work of the explosion gases depends on the vol. and heat capacity of the explosion gases, when the heat of explosion is const. In the Pb block test, the influence of the tamping, the amount and *d* of the explosive, and the velocity of detonation are discussed.

W. J. W.

**Rapid deflagration and double detonation of explosives.** E. AUDIBERT and L. DELMAS (Ann. Mines, 1934, 5, [13], 280—306; Chem. Zentr., 1934, ii, 2341).—Rapid deflagration may occur under some conditions with any explosive, and is attributed to faulty ignition or obstructed propagation of the explosive reaction. Premature release of pressure, before attainment of that min. val. leading to detonation, gives rise to an apparent double detonation.

J. S. A.

**Determination of the brisance of explosives.** T. URBAŃSKI (Z. ges. Schiess- u. Sprengstoffw., 1935, 30, 68—71).—Using a modified Kast apparatus, tests were made on a no. of NH<sub>4</sub>NO<sub>3</sub> explosives and on C<sub>6</sub>H<sub>2</sub>Me(NO<sub>2</sub>)<sub>3</sub> to compare the results with other properties of the explosive, *e.g.*, velocity of detonation (I). For explosives of *d* 1.10—1.15 and with (I) between 2500 and 500 m./sec., (I) may be calc. from the equation  $V^n = KS_p$ ,  $V^n$  being (I),  $S_p$  the compression of the Cu cylinder (7 × 10.5 mm.), and *K* and *n* consts.,  $\approx 24$  and 0.5, respectively.

W. J. W.

**Analysis of process solvents.**—See V.

**XXIII.—SANITATION; WATER PURIFICATION.**

**Practical aspects of coagulation with ferric chloride.** C. G. HYDE (J. Amer. Water Works Assoc., 1935, 27, 631—659).—The use of FeCl<sub>3</sub> as a coagulant



for  $H_2O$  and sewage and as a conditioning agent for sludge is comparatively recent and its corrosive nature renders its application difficult. Its nature, use, and efficiency are discussed and a method of analysis is recommended. C. J.

**Significance of ammonia in the chlorine-consuming capacity of water. IV. Influence of physical factors (light and temperature) on chlorination of pre-ammonised water.** M. L. KOSCHKIN and E. M. SPECTOR (*Z. Hyg.*, 1935, **116**, 688—696; cf. B., 1934, 653).—The increment in  $Cl_2$  consumption of  $H_2O$  due to rising temp. or increased exposure to light is smaller in pre-ammonised than in untreated samples. Under both conditions the bactericidal action of  $Cl_2$  increases with temp. A. G. P.

**Behaviour of oxidising agents with activated carbon [in water purification].** A. S. BEHRMAN and H. GUSTAFSON (*Ind. Eng. Chem.*, 1935, **27**, 426—429).—The action of activated C in removing free  $Cl_2$  from  $H_2O$  by reduction to  $Cl^-$  is only an example of its general tendency to adsorb oxidising agents, with or without decomp. or interaction. Examples are: (1) adsorption of  $H_2O_2$  and  $O_3$  with catalytic decomp.; (2) adsorption of  $KMnO_4$  with reduction of the latter and retention of  $MnO_2$ ; (3) adsorption of I in which no reaction occurs. An activated C may be prepared containing 25—30% I. C. I.

**Water sterilisation with ultra-violet light.** T. BAUMGÄRTEL (*Chem.-Ztg.*, 1935, **59**, 417).—An apparatus capable of handling 2000 litres of  $H_2O$  per hr. is described. G. H. C.

**Potability of water from the viewpoint of fluorine content.** H. V. SMITH (*Amer. J. Publ. Health*, 1935, **25**, 434—441).— $H_2O$  containing  $> 0.8$  p.p.m. of F causes mottled dental enamel in children of a susceptible age. No satisfactory method of removing excessive amounts is available at moderate cost, but, in the discussion, J. M. SANCHIS suggests that certain ions, e.g.,  $Ca^{++}$ , may have an inhibitory effect on the toxicity of F. Methods of determination depending on the effect of F' on the colour produced when alizarin reacts with Zr in HCl solution are recommended. C. J.

**Spectrographic determination of minor chemical constituents in various water supplies in the United States.** M. M. BRADECH and F. H. EMERY (*J. Amer. Water Works Assoc.*, 1935, **27**, 557—580).—All  $H_2O$  examined contained Ca, Na, Mg, Si, Al, Fe, Cu, and B in chemically determinable amounts, Sr up to 10 p.p.m., and traces of Ba, K, Ag, and Zn. Ti, Sn, Pb, Cr, Mn, F, and Ni were present in most samples. The supply at Denver may contain 0.2—0.3 p.p.m. of sol. Ag and 0.5 p.p.m. of Mn, whilst Lake Michigan  $H_2O$  contains 0.2 p.p.m. of Zn. The highest amount of B (1.0 p.p.m.) was found in the supply of Bismarck, and only two supplies contained an appreciable amount of Pb, those of Baltimore and Cincinnati having 0.18 and 0.15 p.p.m., respectively. C. J.

**Oilfield water analysis. V. Determination of potassium.** W. R. WIGGINS and C. E. WOOD (*J. Inst. Petroleum Tech.*, 1935, **21**, 200—203; cf. B., 1935, 432).

—The K is pptd. by means of Na 6-chloro-5-nitro-toluene-3-sulphonate. The K salt is reduced, diazotised, and coupled with R-salt to produce a sol. red dye, and K determined by measuring the colour intensity in the Lovibond colorimeter or by direct comparison against standard amounts of K. This method is not accurate for solutions containing  $< 1\%$  of KCl. Alternatively, the pptd. K salt is reduced with excess of  $SnCl_2$  or  $TiCl_2$  and the excess of reagent determined volumetrically. The latter reagent gives the more accurate results.  $NH_3$  should be absent, but Ca and Mg give only slight positive errors. Other metals do not interfere. C. C.

**Corrosion from zero softened waters.** R. L. DERBY (*J. Amer. Water Works Assoc.*, 1935, **27**, 627—630).—The advantages of zero zeolite-softened  $H_2O$  are largely counterbalanced by its corrosive properties, especially when heated. The corrosion is probably due to the contact of a rather highly mineralised alkaline solution with an exposed metal or to local electrolysis at the junction of dissimilar metals. It is recommended that the piping etc. should be of the same metal throughout and that a lining of  $CaCO_3$  should be formed by raising the hardness by 2.0—3.0 grains by by-passing a little of the untreated  $H_2O$  or by slightly supersaturating the  $H_2O$  with  $CaCO_3$ . C. J.

**Creamery sanitation.**—See XIX.

#### PATENTS.

**Manufacture of disinfectants.** H. BÄHNKE and E. MELHOSE (B.P. 426,450, 23.1.34. Ger., 16.5. and 10.7.33).—The use of saponin (I) as an emulsifying agent for xylenols or halogenated xylenols is claimed. E.g., a disinfectant solution is obtained by addition of *m*-5-xyleneol (75 pts.) and 1:3:2:5- $C_6H_2Me_2Cl-OH$  (25 pts.) in MeOH (200 pts.) to (I) (50 pts.) in  $H_2O$  (800 pts.) with good stirring. H. A. P.

**[Agents for] destruction of pests.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 426,398, 29.9.33).—The use of additive compounds of inorg. salts with esters, ethers (including alkylene oxides), CO-compounds, amides, amines, sulphides, thiols, and non-acidic nitriles is claimed. E.g., weevil-infected corn is mixed with a  $CaCl_2-HCO_2Me$  compound and kept in a closed space, or the vapours evolved on adding  $MgCl_2-HCO_2Me$  to  $H_2O$  are used as fumigant. H. A. P.

**Sewage-treatment apparatus.** D. C. SCOTT (U.S.P. 1,971,796, 28.8.34. Appl., 28.10.31).—The bottom of the activating tank is preferably corrugated in the form of parallel shallow troughs. In each of these is a longitudinal rotor carrying dipping buckets which are inverted on the down journey and at the lowest point are tripped upright so that air is spilled out of them under the sewage. B. M. V.

**Production of distilled water.** W. L. and H. L. McLAUGHLIN, and V. BALLOU, Assrs. to NAT. PURE WATER CORP. (U.S.P. 1,972,350, 4.9.34. Appl., 16.6.32).—An elaborate system relying on the cold of the  $H_2O$  to condense the steam, but embodying otherwise correct heat exchanges and purification of the steam, is described. B. M. V.