

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

MAY 10, 1929.

### I.—GENERAL; PLANT; MACHINERY.

#### Waste-heat dryer calculations and charts.

A. E. R. WESTMAN and R. G. MILLS (J. Amer. Ceram. Soc., 1929, 12, 162—174).—Charts are derived which show the effect of atmospheric humidity conditions and air-discharge temperature on the operation of a typical waste-heat dryer. The output of a dryer could be increased by pre-drying the air with silica gel or increasing the outlet air temperature. This would be economically possible if the latent heat of vaporisation of the water were recovered by condensation.

J. A. SUGDEN.

**Absorption of solvent vapours.** A. ALEŠ (Chem. Listy, 1929, 23, 1—5).—A review of methods used in industry for the recovery of volatile solvents.

R. TRUSZKOWSKI.

**Abolition of the Beaumé hydrometer.** T. WALLIS (Chem. Fabr., 1929, 135—136).—The proposed abolition of the Beaumé hydrometer involves its replacement by a more satisfactory instrument. It is suggested that in future all hydrometers should record the value of  $d$  directly, and should be graduated by immersion in aqueous sulphuric acid from  $d$  1.000 to  $d$  1.841, above this value in zinc chloride solution, and below it in alcohol and petroleum distillate. The standard temperature should be 20°. For other liquids than those used as standards a correction is required on account of the difference in surface tension if an accurate value of  $d$  is required.

C. IRWIN.

#### Use of interferometry in science and technology.

E. BERL and L. RAVIS (Fortschr. Chem. Phys., 1928, 19, No. 7, 1—52; Chem. Zentr., 1928, ii, 1591—1592).

**Crystallisation at rest and in motion.** K. KIEPER (Chem. App., 1928, 15, 185—186; Chem. Zentr., 1928, ii, 1599).

**Determination of drop points.** NAUMANN.—See II.

### PATENTS.

**Concentrating or distilling apparatus.** A. MONTUPET (F.P. 629,395, 27.4.26).—The liquids are allowed to circulate in the tubes of a multiple-effect apparatus in which each heating tube is axially disposed within another tube, so that the circulation of the respective fluid is from below to above in the annular space so formed and from above to below in the inner tubes.

W. J. BOYD.

**Heat treatment apparatus.** J. SCHONBRUNN and G. KELLER, Assrs. to A.-G. BROWN, BOVERI & Co. (U.S.P. 1,697,144, 1.1.29. Appl., 8.9.27. Ger., 10.9.26).—A furnace chamber is provided with interior means of heating, and with a lid or closure which is provided with

an extension or bell of bent conducting material which maintains the chamber closed to atmosphere when the lid is removed.

B. M. VENABLES.

**Treating adsorbent materials.** R. E. MANLEY, Assr. to TEXAS Co. (U.S.P. 1,702,738, 19.2.29. Appl., 26.1.24).—For the removal of adsorbed material from the adsorbent the latter is introduced into a chamber with a vertical helical baffle tapering towards the lower end, and steam is introduced at an intermediate point and air at a lower point tangentially to the baffle, the evolved vapours or liquid being removed from the opposite side at the lower end.

A. R. POWELL.

**Manufacture of disperse systems.** A. G. BLOXAM. From J. R. GEIGY A.-G. (B.P. 307,000, 1.12.27).—A mixture of sulphite-cellulose lye and colloidal silica is a more effective emulsifying agent than either alone, and may be used in neutral or acid solutions. Fish oil emulsified with this mixture is not precipitated by contact with acid leather.

C. HOLINS.

**Filtering apparatus.** A. KÖNIG and G. MÜLLER, Assrs. to STAATLICHE PORZELLAN MANUF. (U.S.P. 1,704,330, 5.3.29. Appl., 11.1.27. Ger., 19.1.26).—See G.P. 433,376; B., 1927, 189.

**Precipitation of solid substances in coarse granular condition from solutions.** F. JEREMIASSEN, Assr. to A./S. KRISTAL (U.S.P. 1,704,611, 5.3.29. Appl., 10.9.25. Norw., 18.9.24).—See B.P. 240,164; B., 1925, 977.

**Catalytic apparatus.** SELDEN Co., Asses. of A. O. JAEGER (B.P. 279,819 and 305,636, 11.10.27. U.S., 27.10.26).—See U.S.P. 1,685,672; B., 1929, 229.

**Gas and liquid contact apparatus. Sprayer for effecting intimate contact between liquids and gases.** W. F. SLATER, Assr. to KIRKHAM, HULETT, & CHANDLER, LTD. (U.S.P. 1,704,518—9, 5.3.29. Appl., [A, B] 30.4.26. U.K., [A] 25.5.25, [B] 2.6.25).—See B.P. 256,351 and 256,358; B., 1926, 857.

**Manufacture of disperse systems.** C. A. AGTHE, Assr. to J. R. GEIGY A.-G. (U.S.P. 1,696,374, 25.12.28. Appl., 22.11.27).—See B.P. 307,000; preceding.

**Centrifugal machines.** T. BROADBENT & SONS, LTD., and B. L. BROADBENT (B.P. 308,033, 6.2.28).

**Furnace wall construction.** F. B. DEHN. From M. H. DETRICK Co. (B.P. 307,567, 28.12.27).

**Regulating combustion in [boiler] furnaces.** SMOOT ENGINEERING CORP., Asses. of C. H. SMOOT (B.P. 282,438 and 300,512, 16.12.27. U.S., 16.12.26).

**Apparatus for heating liquids.** L. F. THOMPSON (B.P. 305,275, 7.7.28).

[Self-cleansing] apparatus for straining liquids. F. BAILEY and F. H. JACKSON (B.P. 307,984, 21.12.27. Addn. to B.P. 246,551).

Apparatus for pneumatic separation or grading of solid materials [by air blast]. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 307,810, 8.12.27).

Absorption refrigerating machines. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of SIEMENS-SCHUCKERTWERKE GES.M.B.H. (B.P. 279,898, 29.10.27. Ger., 30.10.26).

Refrigerator evaporators. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of C. L. HEISLER (B.P. 289,062, 20.4.28. U.S., 22.4.27).

Absorption refrigerating plants. C. PÖHLMANN (B.P. 292,568 and 306,873, 23.8.27).

Refrigerating apparatus. H. R. VAN DEVENTER (B.P. 279,088, 12.10.27. U.S., 12.10.26).

Refrigerating apparatus. GEN. ELECTRIC CO., LTD., and N. E. BARBER (B.P. 307,534, 9.12.27).

Absorption refrigerating systems. ELECTROLUX, LTD., Assees. of [A] C. G. MUNTERS, [B] A. LENNING (B.P. 282,065 and 283,473, [A] 7.12.27, [B] 9.1.28. U.S., [A] 8.12.26, [B] 8.1.27).

Absorption refrigerating machines. B. E. D. KILBURN. From SULZER FRÈRES SOC. ANON. (B.P. 282,056, 6.12.27).

Compressed gas cylinders and the like. C. H. BINGHAM, JUN. (B.P. 307,147, 29.12.27).

Apparatus for continuously producing and drying briquettes. N. FLODIN and G. CORNELIUS (B.P. 306,977, 25.10.27).

## II.—FUEL; GAS; TAR; MINERAL OILS.

Coals from the Jakutski Mountain region. N. M. KARAVAEV and I. B. RAPOPORT (Izvestia Teplotech. Inst. [Moscow], 1928, 10, 32—43).—Results of analyses and various other particulars are given concerning these coals.  
T. H. POPE.

Low-temperature carbonisation of fuels by the Hérens process. C. ROSZAK (Bull. Soc. Ing. Civ. France, 1928, 81, 1064—1104).—The coal, dried and preferably preheated to about 200°, is carried through the distillation chamber by means of a continuous chain grate, and is carbonised principally by the sensible heat of a current of hot gas which is passed down through the coal and the grate, but also by direct radiation from above, the roof of the chamber being externally heated by combustion of part of the gas produced. The process may be used in conjunction with steam-raising plant, the grate then being extended to carry the coke directly into the combustion chamber, wherein also the excess gas may be burnt. To produce a compact coke a heavy endless chain mounted within the chamber rests on the surface of the coal, which is thus compressed as it reaches the softening point.

A. B. MANNING.

Chemical utilisation of coal. C. BERTHELOT (Bull. Soc. Ing. Civ. France, 1928, 81, 1105—1181).—

The methods available for the transformation of coal into motor fuels and fertilisers are outlined and the economic factors involved are discussed in detail.

A. B. MANNING.

Relation between caking and moisture-absorbing power of some Japanese coals. S. IKI (Ind. Eng. Chem., 1929, 21, 239—241).—Dried samples of Japanese bituminous coals of different caking power together with some anthracite and brown coals were kept in an atmosphere of constant humidity and the moisture absorption of each was measured by its increase in weight. The coals were also separated into their  $\alpha$ -,  $\beta$ - and  $\gamma$ -portions, the ulmin of the brown coal being extracted. The chief caking constituent of coal,  $\gamma$ , had the lowest moisture-absorbing power, whilst the constituent of greatest absorbing power was ulmin; this may account for the decrease in moisture absorption with increase in caking properties, and may explain the strong affinity for water shown by weathered coals.

C. B. MARSON.

Oxidation of sub-Moscow coal by atmospheric oxygen at different temperatures. N. M. KARAVAEV and A. K. IVANOV (Izvestia Teplotech. Inst. [Moscow], 1928, 10, 44—50).—Oxidation proceeds differently with the Schtschekinski and Bobrikovski coals of the sub-Moscow basin owing to differences in composition and possibly in the nature of the organic components of the coal. Up to 140° for the former and 80° for the latter the oxidation consists mainly of combination of oxygen; the proportion of carbon monoxide formed increases as the temperature is raised further, the percentage of hydrogen in the coal and, to a considerably less extent, that of hydrogen diminishing and that of oxygen increasing. As a result of the oxidation, the coal loses up to 14% of its heating value.  
T. H. POPE.

Orsat absorption tube. A. H. MOODY and G. E. STEVENS (Chemist-Analyst, 1928, 17, No. 4, 15).—A new type of absorption tube for the determination of carbon dioxide, oxygen, and carbon monoxide in flue gases is described.  
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Composition of tar from low-temperature carbonisation of Utah coal. II. Heavy portions. R. L. BROWN and R. N. POLLOCK (Ind. Eng. Chem., 1929, 21, 234—238; cf. B., 1927, 179; 1928, 393).—The heavy portions of the tar obtained from the carbonisation of Mesa Verde coal at about 700° (by means of superheated steam) which had been collected in air-cooled condensers were analysed. The fractions lighter and heavier than the aqueous distillate consisted of insoluble matter 0.85 and 9.5%, carboxylic acids 2.25 and 2.5%, alkali-soluble (phenolic) 15.0 and 40.0%, tar bases 1.55 and 3.0%, and neutral portions 80 and 45%, respectively. These neutral portions were composed of resin, 18.5 and 20%, wax, 24.1 and 10%, and oil, 57.3 and 69%, respectively. The chief feature observed was the high paraffin wax content of the tar; in the consideration of the economic value of low-temperature carbonisation by-products this is of importance.  
C. B. MARSON.

Detection of benzene with dracorubin. BERGER (Zentr. Gewerbehyg., 1928, 15, 227—228; Chem. Zentr., 1928, ii, 1594).—The dye "dracorubin," which

is soluble in benzene but not in benzenes, is used in the form of test-paper.

A. A. ELDRIDGE.

**Apparatus and methods for separation, identification, and determination of the constituents of petroleum.** E. WASHBURN, J. H. BRUNN, and (Miss) M. M. HICKS (Bur. Stand. J. Res., 1929, 2, 467—488).—A rectifying still with a 20-plate column and with means for independently controlling and measuring the temperatures of the plates is described. Distillation was carried out in a stream of inert gas, with or without boiling. The fractions from the still were further separated in all-glass rectifying stills working *in vacuo*. Finally, molecular stills were used in which distillation can be carried out at any temperature at which the vapour pressure at the distilling surface is not lower than the degree of vacuum attainable. Azeotropic mixtures were broken up by fractional crystallisation or melting. A special apparatus for combustion analysis is described in which the oxygen is purified with unusual care and in which no rubber connexions are employed. It is claimed that the accuracy is such as to permit of determining the formula of any hydrocarbon up to  $C_{100}$  (i.e., carbon and hydrogen each to  $\pm 0.05\%$ ). The change in iodine value of a petroleum oil resulting from heating the oil at different temperatures up to  $370^\circ$  is much less in an inert gas than in air.

W. E. DOWNEY.

**Determination of water in oil fuels.** N. I. TSCHERNOSHUKOV (Izvestia Teplotech. Inst. [Moscow], 1928, 10, 11—12).—The methods examined were: (1) Dean and Stark's, (2) Lysenko's, (3) the centrifugal, and (4) Denemark's. The most accurate method is (1), with a maximum error of 0.2%, and the most inaccurate (2), which gave an error of 98% with a sample of mazut. Method (3) mostly gives excellent results, the greatest inaccuracy being 1% for a mazut. Method (4) is simple, and with a mazut free from paraffin and with naphthas is accurate to 1%; with paraffinous mazuts negative results were obtained in some cases.

T. H. POPE.

**Cracking.** N. A. BUTKOV (Izvestia Teplotech. Inst. [Moscow], 1928, 10, 7—10).—The cracking of various oils has been investigated in a furnace similar to that of Rittman, consisting of a vertical tube-furnace with internal packing heated electrically. The experiments were carried out at constant temperature and pressure, the pressure being developed initially by the introduction of hydrogen, about 1 g. of which was used per 300 c.c. of the prime material. The results obtained from an Embenski solar distillate having  $d_{40}^{15}$  0.8793, flash point  $126^\circ$ , "Akzys" oils 2%, initial b.p.  $225^\circ$ , distillate up to  $270^\circ$  4%, show that, with cracking temperatures and pressures varying between  $478^\circ$  and 0.25 atm. and  $530^\circ$  and 4.7 atm., increase in the pressure effects increase in the yield of benzine (b.p. to  $200^\circ$ ) and diminution of the yield of gaseous products; increase in the temperature at constant pressure results in increased formation of gases. Cracking of decalin, b.p.  $187^\circ$ , at  $478^\circ$  and 5 atm. gives 34% of products b.p. up to  $180^\circ$  and 11 litres of gas per 100 c.c. of the starting material; the total liquid products range in b.p. from  $60^\circ$  to above  $260^\circ$  (74.6% of total). Under 4 atm. pressure, tetralin, b.p.  $205^\circ$ , gives at  $484^\circ$ ,  $533^\circ$ , and  $575^\circ$ , 3.5%, 21.3%,

and 10.7% of liquid products b.p. up to  $200^\circ$ , and scarcely any, 2.5, and 19.0 litres of gaseous products per 100 c.c. of tetralin respectively. At  $533^\circ$  the hydrocarbons formed range in b.p. from  $130^\circ$  to  $200^\circ$  and contain no appreciable amount of naphthalene. At  $575^\circ$  the decomposition assumes a different character, since the b.p. range of the resulting hydrocarbons becomes  $94$ — $200^\circ$  and a large proportion of naphthalene is obtained; thus the decomposition here consists largely of dehydrogenation. At  $467^\circ$  and 5 atm. pinene yields 22.8% of products b.p.  $61$ — $145^\circ$  and 7.0 litres of gas per 100 c.c. of pinene, and limonene at  $450^\circ$  and 5 atm. gives 28.9% of products b.p.  $56$ — $160^\circ$  and 2.5 litres of gas. The thermodynamics of the cracking process is briefly considered.

T. H. POPE.

**Coke formation with fuel oils and lubricating oils.** N. I. TSCHERNOSHUKOV (Izvestia Teplotech. Inst. [Moscow], 1928, 10, 28—31; cf. B., 1929, 85).—Experiments made with Emba naphtha (containing a small proportion of resinous substances), which was heated for 3—6 hrs. at  $150$ — $175^\circ$  in presence of oxygen at 15 atm., show that the substances occurring in naphtha and capable of yielding asphaltenes diminish on prolonged oxidation, no renewal of these substances taking place. The compositions of asphaltenes obtained from Grozni wax-free naphtha, turbine oil, and light spindle oil have the following percentage compositions: C 85.2, 83.05, 81.88; H 7.4, 7.35, 7.12; O 6.7, 8.79, 9.30; S 0.7, 1.21, 1.70%, these corresponding with the formulae  $C_{355}H_{350}O_{22}S$ ,  $C_{172}H_{176}O_{14}S$ , and  $C_{136}H_{142}O_{12}S$ . The acid precipitate of hydroxy-acids, insoluble in light petroleum, formed by oxidising resin-free oil contained C 71.64, H 8.81, O 19.55%, S nil. When colourless Bachinski oil is heated with 3% of sulphur, reaction commences above  $200^\circ$ , and is rapid at  $250^\circ$ , and extremely so at  $270^\circ$ , hydrogen sulphide being liberated and the resulting oil containing 0.35% (at  $220^\circ$ ) or 0.21% (at  $250^\circ$ ) S. The asphaltenes obtained by oxidising this oil (1) before and (2) after purification with floridin contained: C 71.62, 74.82; H 6.82, 7.02; O 13.48, 12.41; S 8.08, 5.75%. In these, as in the asphaltenes referred to above, the atomic proportions of carbon and hydrogen are approximately equal.

T. H. POPE.

**Knock ratings of pure hydrocarbons.** S. F. BIRCH and R. STANSFIELD (Nature, 1929, 123, 490—491).—Diamylene is not such a good anti-knock material as Nash and Howes (B., 1929, 272) suggest, and is certainly not better than trimethylethylene. The observation that polymerisation reduces the anti-knock value was also confirmed for methylcyclohexene and its dimeride methylcyclohexylmethylcyclohexene. The behaviour of cyclohexene depends on the history of the sample, the anti-knock value improving on distillation over sodium and falling on exposure to light and air; the effect is due to the formation of a peroxide. Hydrocarbons containing conjugated double linkings have excellent anti-knock properties, but a diolefine in which the double linkings are not conjugated does not possess marked anti-knock properties. Although an increase in the length of a chain reduces anti-knock properties, the reverse is the case for side chains attached to benzene rings.

A. A. ELDRIDGE.

**Knock ratings of pure hydrocarbons.** A. W. NASH and D. A. HOWES (*Nature*, 1929, 123, 526—527).—Birch and Stansfield's results (preceding abstract) for trimethylethylene and diamylene refer to concentrations by weight; and the authors' results to concentrations by volume; hence strict comparison is impossible. An acid-refined unsaturated spirit has a lower anti-knock value than the original, since quantitative polymerisation of olefines is not attained in practice. Moreover, standardisation of the air : fuel ratio is necessary for comparison. In the olefine series, and possibly also in the aromatic series, ease of oxidation appears to be intimately connected with anti-knock action. A. A. ELDRIDGE.

**Properties and analysis of used crank-case oils.** D. G. PIDGEON and H. E. TESTER (*J. Inst. Petroleum Tech.*, 1929, 15, 91—99).—The extent of dilution of a used crank-case oil is satisfactorily determined by measuring the distillate obtained by passing slightly superheated steam into the sample maintained by external heating at 150° until distillation of oil ceases. The use of wet steam at 100° without external heating gives low results. Dilutions up to 14.0% are found in the used oil from nine different cars operating on the same original lubricating oil. So many factors affect the viscosity that no relationship can be traced between this property and the dilution of the oils. The bulked distillate from a number of dilution determinations was a yellow liquid,  $d^{20}_4$  0.802, which was decidedly unsaturated and consisted largely of heavy fractions of the motor fuel together with cracked products from the decomposition of the oil. Using synthetic mixtures of fresh lubricating oil and up to 15% of such a diluent, it is shown that when a medium-grade oil contains 10% of diluent it has the properties of a spindle oil, and with 15% of diluent those of a light spindle oil. For any particular engine the percentage dilution of the oil reaches an approximately constant and characteristic value, but thereafter the viscosity slowly increases, and adventitious matter and oxidation products accumulate. With certain types of engine the dilution is such that a heavy grade of oil is necessary if the equilibrated diluted oil is to have a safe viscosity. The quantity of adventitious matter is not a function of percentage dilution.

W. S. NORRIS.

**Determination of oxidisability of transformer and turbine oils.** N. I. TSCHERNOSHUKOV (*Izvestia Teplotech. Inst. [Moscow]*, 1928, 10, 13—18).—For practical purposes, transformer and turbo-generator oils may be divided into three classes, according as they give rise on oxidation to a precipitate of (1) asphaltenes or (2) hydroxy-acids, or (3) no precipitate but a greatly increased acidity. Of the many methods devised for examining the oils from this point of view, a number of typical examples, including three rapid methods, are described in detail, and the value of their indications is discussed. The results obtained for a large number of oils by the simple and rapid Butkov test (cf. B., 1929, 119) show that this method is of wide applicability as a means of controlling the quality of the oils in question.

T. H. POPE.

**Determination of drop points by Ubbelohde's method.** E. NAUMANN (*Chem. Fabr.*, 1929, 136—138).

—The determination of the drop point (temperature at which a semi-solid liquefies sufficiently to drop from the constricted end of a tube) by Ubbelohde's method depends for its accuracy on the use of the prescribed dimensions, otherwise capillarity comes into play. The accuracy required is difficult to secure with glass nipples, and these are fragile. Results of comparative tests using brass nipples on vaseline, asphalt, etc. were concordant and agreed with those obtained with accurate glass nipples. The quantity of brass or glass is too small for any error to be caused by variation in heat capacity or conductivity. The end of the tube may be either sharp or rounded without variation in results.

C. IRWIN.

**Electrical conductivity of thin oil films.** WATSON and MENON.—See XI. **Petroleum oils for plants.** DE ONG.—See XVI. "Norit" and "Carboraffin." REICH and VAVRINECK.—See XVII. **Ignition of fire-damp.** SEGAY.—See XXII.

#### PATENTS.

**Hydrogenation of coal.** C. H. LANDER, F. S. SINNATT, and J. G. KING (B.P. 306,564, 17.9.27).—Before subjecting a coal to destructive hydrogenation a material proportion of the fusain and/or durain is removed. The durain may be removed by hand-picking, and the fusain by separation of the finer material from the crushed coal. The coal may also be given a preliminary treatment to modify its caking properties without changing its physical state. It may, for example, be subjected to a preliminary hydrogenation or oxidation, or to the action of gases or vapours at atmospheric or other pressures; or coals which have been subjected to different treatments of the kind described may be blended in suitable proportions. A. B. MANNING.

**Retort setting for heating fuel with gases or superheated steam.** F. B. DEHN. From SOC. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (B.P. 307,250, 16.5.28).—Coal or other fuel is distilled in a retort comprising a number of chambers through which a current of hot gas or steam is passed, and provided with auxiliary gas or steam pipes so arranged that some or all of the chambers may be supplied with gas or steam directly from one or more generators or superheaters, while part of the hot vapours passing out of some of the chambers may be shunted and part only passed into the following chambers. The temperature in each chamber can thereby be adjusted to control the amount and kind of hydrocarbons which are condensed therein and can be collected in oil separators disposed between the chambers. The fuel passes from chamber to chamber counter-current to the steam until distillation is complete, when it is brought into chambers through which cool or low-pressure steam is passed, these chambers serving as heat exchangers.

A. B. MANNING.

**Distillation of carbonaceous materials, tar sands, oil-bearing limestone, etc., and apparatus for subjecting the gases and vapours to the action of liquids.** H. NIELSEN and B. LAING (B.P. 306,429, 19.10.27).—The oil vapours evolved in the distillation of carbonaceous materials are subjected to a process of fractional condensation in a series of scrubbers, in each

of which the scrubbing medium consists of an oil of the same or lower b.p. than the corresponding fraction which it is desired to condense. Each scrubber comprises a horizontal cylindrical casing containing a pair of shafts on which are mounted a large number of discs, which by their rotation throw up the oil in the form of a spray and so bring about an intimate contact of the gas with the scrubbing medium. A number of perforated baffle-plates at the exit end of the casing serve to prevent entrainment of oil globules by the gas. A part of the oil condensed in each scrubber serves as scrubbing medium for the immediately preceding scrubber.

A. B. MANNING.

**Distillation of solid carbonaceous or oil-bearing materials.** H. NIELSEN and B. LAING (B.P. 306,654, 23.12.27).—The material is distilled in a current of heated gas and, before its entry into the distillation chamber, is heated to a temperature, between 100° and 250°, dependent on the volume of gas used, but above that at which the heaviest oil fractions present in the vapour are liable to condense. The walls of the retort, the conduits, and the dust extractor are similarly heated to prevent condensation therein.

A. B. MANNING.

**Low-temperature carbonisation of bituminous and oil-bearing materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,723, 9.3.28).—The material to be carbonised is passed through a sheaf of tubes mounted longitudinally in an inclined cylindrical rotary retort. Hot gases are circulated through the retort, which is lined with heat-insulating material. The heating gases may be introduced and carried away through a centrally disposed guide pipe.

A. B. MANNING.

**Manufacture of coal gas.** SOUTH METROPOLITAN GAS CO., E. V. EVANS, and H. STANIER (B.P. 306,435, 19.11.27).—The gases and vapours produced by the low-temperature carbonisation of coal are mixed with hydrogen or a gas containing hydrogen, *e.g.*, water-gas, and the mixture is subjected to a high temperature in a cracking chamber. The water-gas may be passed through the retort in which the coal is being carbonised, or may be preheated and added to the volatile products of carbonisation as they are leaving the retort. A gas suitable for town supply is produced, and the tar approximates in character to ordinary high-temperature tar.

A. B. MANNING.

**Manufacture of gas from heavy oils.** C. CHILOVSKY (B.P. 279,834, 20.10.27. Fr., 27.10.26).—The apparatus for carrying out the process described in B.P. 255,423, 271,899, and 271,907 (B., 1927, 805, 867) is modified in many details. In particular, the gases after leaving the catalyst are caused to pass back along the outer walls of the vaporisation chamber, the air for partial combustion is preheated by being circulated through a jacket surrounding the apparatus, the catalyst is so constructed that the oil cloud will circulate freely through it in straight channels, and the steam which is added to the cloud of oil is generated in a coil heated by the gases leaving the apparatus. Other modifications relate to the use of the process in conjunction with an internal-combustion engine.

A. B. MANNING.

**Gas producers.** O. MISCH (B.P. 306,614, 28.11.27).

—A vertical shaft is divided into three sections, the uppermost section forming an externally-heated distillation retort, the middle section a water-gas generator, and the lowest section a producer-gas generator. The lower end of the retort is heated by means of a compressed-air burner supplied with hot producer gas, and is maintained at a high temperature, *e.g.*, about 1300°, with a fuel which is converted into coke at 700°, in order that coke should be produced as rapidly as it is used in the gas generators below, so that the process is continuous. The distillation gases and the water-gas are withdrawn by suction at the upper end of the retort. The producer gas is similarly withdrawn by suction at the upper end of the producer section. Tar residues obtained from the distillation gas are returned to the retort to be cracked.

A. B. MANNING.

**Apparatus for fractional distillation of tar, crude oils, petroleum, and other liquids.** O. ELSTERMANN and A. BAUMHÖR (B.P. 307,243, 30.4.28).—The liquid is distilled in a column made up of a number of superimposed funnel-shaped units, which are jacketed and heated to successively higher temperatures by suitable heating medium. The distilled products from each unit pass out through a discharge pipe to a condensing system; the remaining liquid passes into the next lower unit. The liquid entering each unit is ejected in a thin layer over the inner wall of the unit by means of a rotary distributor attached to a common central shaft, and moves down the wall in a helical path. A rotating scraper attached to the central shaft in each unit prevents any sediment sticking to the walls.

A. B. MANNING.

**Separation of the acid constituents from low-temperature tar or its fractions.** C. BUNGE (B.P. 306,738, 27.3.28).—The tar or tar fraction is emulsified with water or distillation liquor and a little soap solution, and the emulsion treated with a solvent for the acid constituents, *e.g.*, an alkali solution, alcohol, etc. The mixture is kept, or the separation of the hydrocarbons and the solution containing the phenols may be effected by centrifuging.

A. B. MANNING.

**Apparatus for preliminary separation of crude benzoles.** SOC. DES ETABL. BARBET (B.P. 295,591, 13.8.28. Fr., 12.8.27).—Crude benzol, free from water, passes through a heater to a rectifying column from which are obtained three distinct fractions, *viz.*, a fraction containing light impurities, a 90–95% benzol, and a residual toluol fraction. Light products are removed from the cooled benzol and the crude benzol fraction is passed to a second rectifying column, where it is exhausted of its xylol. The xylol is withdrawn leaving a residue of still heavier products, which are passed to a discontinuous vacuum rectifier for the production of a solvent naphtha fraction and a tarry residue from which naphthalene can be crystallised.

H. S. GARLICK.

**Manufacture of hydrocarbons of low b.p.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,437, 20.10.27).—Components of mineral oils soluble in liquid sulphur dioxide and liquid at ordinary temperatures are converted into hydrocarbons of low b.p. and rich in aromatic compounds by treatment with anhydrous aluminium chloride and/or aluminium metal at above

100° while passing an inert gas (dry hydrogen chloride etc.) through the reaction mixture, with or without application of pressure. H. S. GARLICK.

**Production of light hydrocarbons.** J. M. F. D. FLORENTIN, A. J. KLING, and C. MATIGNON (B.P. 279,488, 20.10.27. Fr., 23.10.26. Cf. B., 1927, 548, 836).—Coal tar is treated with hydrogen under high pressure and at 350–480° in the presence of a dehydrating catalyst, *e.g.*, thoria, and a "substitutive" catalyst, *e.g.*, a metal halide, to hydrogenate the phenols and polynuclear compounds (naphthalene, anthracene).

A. B. MANNING.

**Apparatus for distilling oil.** H. S. PETTINGILL, JUN., Assr. to EMPIRE REFINERIES, INC. (U.S.P. 1,698,507, 8.1.29. Appl., 14.12.22).—Means for scraping the still are claimed.

**Refining petroleum oils.** SHARPLES SPECIALTY Co., Assees. of L. D. JONES (B.P. 280,559, 9.11.27. U.S., 9.11.26).—Petroleum oils containing crystalline wax may be centrifugally dewaxed if, before chilling, sufficient oil-free petrolatum or oil-containing amorphous wax is added to produce a plastic precipitate of sufficient density that the wax is separated from the oil on centrifuging. Substances other than hard asphalt, normally removed by extensive acid or clay treatment, may be retained in or added to the oil; by adding the amorphous wax and chilling they are concentrated in the precipitate.

H. S. GARLICK.

**Treatment of hydrocarbon oil.** C. ARNOLD. From IMPERIAL OIL, LTD. (B.P. 305,108, 30.8.27).—Hydrocarbon vapours are led into the lower part of a rectification tower and passed upwards through a contact zone, to which is fed a descending stream of liquid oil carrying in suspension a finely-divided solid absorbent, *e.g.*, clay, with or without the addition of a reagent for removing sulphur, with which the vapours are brought into intimate contact. The temperature is regulated so that part of the desired product comes into contact with the clay temporarily as liquid, thereafter passing off as vapour to suitable condensing apparatus. The bottom of the tower is connected to a settling tank in which condensate is separated from the clay for return to the distillation zone.

H. S. GARLICK.

**Treatment of mineral oils by heat.** W. J. PERELIS (B.P. 277,983, 22.9.27. U.S., 23.9.26).—In the type of pressure-cracking processes under flow in which, after the heating period, an extended cracking treatment is given in heat-insulated tanks or drums, carbon formation and coke deposition are avoided by agitation either mechanically or by means of a tubular cooling apparatus through which the material is passed under conditions of turbulent flow before entering the tank; in either case the temperature of the material is kept within the cracking range. The agitation may be effected during the heating period by heating the material under low pressure whereby the lighter constituents vaporise, thus increasing the volume to be moved and causing turbulent flow, thereafter increasing the pressure during the cracking reaction.

H. S. GARLICK.

**Cracking of petroleum products.** A. SACHANEN and M. TILITSCHYEYEV (B.P. 307,105, 2.12.27).—Oil, preheated by the heavy residue from the expansion

chamber, is forced under high pressure at sufficient velocity to prevent carbon deposition through two sets of tubes connected in series and disposed in the same chamber maintained at the required high temperature by the admission of furnace gases. The first set of tubes (the heating tubes) are of considerably smaller diameter than the second set (the reaction tubes). The resulting products are led into a coil within and at the bottom of the rectifying column, and thence through a reducing valve into an expansion chamber maintained at a pressure not substantially exceeding atmospheric, in which carbon is deposited. The vapours are led into a rectifying column, where they are cooled by contact with coils carrying untreated oil, the condensate passing through a gas separator into a container. Residues from the bottom of the expansion chamber and condensate from the bottom of the rectifying column are returned to the pipe circuit leading to the heating and reaction tubes.

H. S. GARLICK.

**Distilling or cracking oils and like carbonaceous liquids.** A. L. RISPLER (B.P. 305,263, 3.11.27).—Oil is passed through a heated region of a furnace-fired rotary still in which vaporisation of all but the heavier fractions takes place. The residual carbon and asphaltic matter falls upon crushing members and iron balls which roll loosely within the still, and are sufficiently hot to decompose the asphaltic matter; the resulting carbon is removed by means of a steam blast.

H. S. GARLICK.

**Motor fuel.** E. C. HENNEN, Assr. to U.S. NITO Co., INC. (U.S.P. 1,682,561, 28.8.28. Appl., 7.3.28).—High-heat-test lubricating oil, castor oil, and a benzene solution of *p*-chloronitrobenzene are added to gasoline etc.

R. BRIGHTMAN.

**Preventing knocking of motor fuel.** H. D. ELKINGTON. From GEBR. AVENARIUS (B.P. 277,326, 6.9.27).—Knocking is prevented by the addition of 0.3–0.5% of one or more esters of unsaturated fatty acids of high mol. wt., *e.g.*, an oleic ester, fatty acid glyceryl ester, or vegetable oil, such as olive oil. The anti-detonant may be treated before use with oxygen or ozone.

H. S. GARLICK.

**Emulsions from partially oxidised petroleum wax, and their manufacture.** ALOX CHEM. CORP. (B.P. 287,514, 10.10.27. U.S., 23.3.27).—Normally solid petroleum wax is treated at 160° under pressure with oxygen in the presence of a suitable catalyst, until the acidic contents amount to 30% by wt. The resulting waxes are partially or completely neutralised with alkali either directly or after washing, pressure filtration, or other treatment to remove highly acidic substances, and emulsified with water. The resulting emulsion is suitable for use in textile dressings or for coating paper.

H. S. GARLICK.

**Production of products from brown coal analogous to montan wax.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,111, 3.12.27).—Brown coal is chlorinated and extracted with a solvent containing aromatic hydrocarbons. After evaporation of the solvent the product is melted to expel residual solvent and hydrochloric acid, and may, if desired, be further purified by redissolving.

A. B. MANNING.

**Extraction of montan wax.** A. RIEBECK'SCHE MONTANWERKE A.-G. (B.P. 288,197, 12.3.28. Ger., 4.4.27).—Brown coal is extracted under a small excess pressure, not exceeding 2 atm., produced by correspondingly raising the temperature of the solvent used. The usual apparatus for the extraction at atmospheric pressure is used, modified only by the substitution of a pressure separating vessel for the Florentine flask and the addition of a pressure regulator. A. B. MANNING.

**Plant for washing coal and other minerals.** A. FRANCE (U.S.P. 1,704,185, 5.3.29. Appl., 7.11.27. Belg., 24.2.27).—See B.P. 285,864; B., 1928, 737.

**Apparatus for carbonising coal.** R. L. RODGERS, ASS. to CHARCOLITE CORP. (U.S.P. 1,704,103, 5.3.29. Appl., 28.9.22).—See B.P. 256,038; B., 1926, 812.

**Apparatus for distillation or heat treatment of carbonaceous or other materials. Apparatus for producing coke and gas from solid carbonaceous material. Gas and coke plant apparatus.** F. D. MARSHALL (U.S.P. 1,704,092—4, 5.3.29. Appl., [A—C] 10.4.26. U.K., [A, B] 5.6.25. Renewed [B] 10.12.28).—See B.P. 261,919, 261,927, and 261,975; B., 1927, 244.

**Regenerative heating apparatus [for coke ovens].** J. VAN ACKEREN (U.S.P. 1,704,229 and 1,704,685—7, 5.3.29. Appl., [A] 23.2.23, [B] 6.1.22, [C] 22.4.22, [D] 24.7.22).—See B.P. 211,828, 191,393, 210,085, and 201,130; B., 1925, 90; 1924, 587, 662, 702.

**Rotary [gas] scrubber and washer.** T. J. NODEN (U.S.P. 1,704,663, 5.3.29. Appl., 30.6.24. U.K., 16.7.23).—See B.P. 211,409; B., 1924, 326.

**Petroleum refining process and reagent for removal of sulphur.** AXTELL RES. LABS. (B.P. 282,738, 9.12.27. U.S., 27.12.26).—See U.S.P. 1,645,679; B., 1928, 116.

**Quenching coke by means of water.** A. H. LYMN, N. J. BOWATER, and CHAMBER OVENS, LTD. (B.P. 307,204, 25.2.28).

**Pusher rams for coke ovens.** KOPPERS Co., Assees. of C. B. COLLINS and J. A. B. LOVETT (B.P. 284,311, 16.12.27. U.S., 29.1.27).

**Charging hoppers for gas producers and other furnaces.** J. SAUVAGEOT (B.P. 289,458, 29.3.28. Fr., 29.4.27).

**Cleansing of waste lubricating and other oils [by treatment with water].** C. E. FOX (B.P. 306,771, 1.6.28).

**Lead alkyl compounds** (U.S.P. 1,694,268).—See VII.

### III.—ORGANIC INTERMEDIATES.

**Detection of maltol and salicylic acid.** MERL and BEITTER.—See XIX.

#### PATENTS.

**Manufacture of synthetic organic compounds [from carbon oxides].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,294, 7.7.27).—Oxides of carbon are reduced to ethyl alcohol, higher alcohols, hydrocarbons, and acids at high temperatures and

under normal or high pressures in presence of a catalyst containing cadmium or thallium or their compounds, in addition to considerable amounts of iron, nickel, or cobalt or their compounds. The catalysts are free from metallic halides and the treatment is carried out without an addition of alcohols, esters, or aldehydes. The iron, nickel, or cobalt may be replaced or used in conjunction with one or more elements of the 8th periodic group, and copper, gold, silver, cerium, zirconium, aluminium, vanadium, uranium, chromium, molybdenum, tungsten, manganese, alkali metals, or alkaline-earth metals, or their compounds may be present provided that less than 5% of the catalyst is constituted of difficultly reducible metallic oxides. R. BRIGHTMAN.

**Chlorination of hydrocarbons.** INTERNAT. FIRE-PROOF PRODUCTS CORP., Assees. of F. S. VIVAS (B.P. 286,726, 8.3.28. U.S., 11.3.27).—Heavy oils are removed from commercial petrol by saponification with alkali and the hydrocarbon oil is chlorinated in presence of a catalyst (aluminium chloride or ferric chloride), carbon tetrachloride, and about 1% of aqueous ammonia. The product, principally heptachlorohexane, is a cleaning solvent. C. HOLLINS.

**Manufacture of esters.** IMPERIAL CHEM. INDUSTRIES, LTD., W. GIBSON, and J. B. PAYMAN (B.P. 307,137, 21.12.27).—An aliphatic acid amide is heated with an alcohol, with or without a diluent, in presence of a metallic salt (zinc chloride, mercuric chloride). In the case of formic esters having lower b.p. than the alcohol used the ester is with advantage distilled off as formed. The preparation of *n*-butyl formate, *iso*amyl acetate, glycol monoacetate, and *cyclohexyl* formate (the latter under reduced pressure) is described. C. HOLLINS.

**Continuous manufacture of anhydrous acetic acid from its aqueous solutions.** SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 296,974, 13.1.28. Fr., 10.9.27, and Addn. B.P. 301,415, 3.2.28. Fr., 29.11.27).—Acetic acid is extracted from its solutions in (A) liquid or (B) vapour phase by means of a solvent of b.p. between 120° and 150° (e.g., amyl acetate). The extract is freed from water by means of an entraining liquid (e.g., ethyl acetate), and can then readily be fractionated for the recovery of anhydrous acetic acid. Continuous processes are described. C. HOLLINS.

**Manufacture of *o*-nitro- and *o*-amino-diaryl ethers.** O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 307,130, 15.12.27).—An *o*-dinitro-compound of the benzene series is heated with an alkali phenoxide at 100° for about an hour; one nitro-group is replaced by an aryloxy-group. The nitrodiaryl ether may then be reduced, e.g., with iron and acetic acid. Examples are: *o*-nitro- and *o*-amino-diphenyl ethers; 5-chloro-2-nitrodiphenyl ether, m.p. 85°, and the amine, m.p. 40–41°; 2:5:5'-trichloro-2'-nitrodiphenyl ether, m.p. 97–98°, and the amine, m.p. 74–75°; 5-chloro-2-nitrophenyl  $\beta$ -naphthyl ether, m.p. 109–110°, and the amine, m.p. 108–109°; 5':4'-dichloro-2-nitrodiphenyl ether, m.p. 80–81°, and 5:5'-dichloro-2-aminodiphenyl ether, m.p. 76–77°. C. HOLLINS.

**Production of aromatic carboxylic acids.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,223,

21.3.28).—Aromatic hydrocarbons etc. are carboxylated by the action of carbon dioxide at 80–150°/50–60 atm. pressure in presence of aluminium chloride; considerable amounts of diaryl ketones are obtained as by-products. Examples are: benzoic acid from benzene, *p*-chlorobenzoic acid from chlorobenzene, *p*-toluic acid from toluene, 2:4-dimethylbenzoic acid, m.p. 126°, from *m*-xylene. C. HOLLINS.

**Manufacture of hydrogenated aromatic amino-compounds.** K. and K. S. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,285, 4.5. and 22.11.27).—Secondary and tertiary arylamines of the benzene series and acylated naphthylamines or their nuclear derivatives are hydrogenated in the liquid phase in presence of nickel, cobalt, or copper catalysts. The preparation of the following is described: cyclohexyl-ethylamine and -dimethylamine, and tetrahydro-derivatives of ethyl 2-acetamido-3-naphthoate (hydrolysis gives ar-2-aminotetrahydro-3-naphthoic acid, m.p. 180–182°), of 1-acetamido-2-naphthyl ethyl ether (hydrolysis gives the amine, m.p. 54–55°, b.p. 176–178°/12 mm.), and of 1-acetamido-7-naphthyl methyl ether, m.p. 161° (hydrolysis gives ar-1-aminotetrahydro-7-naphthyl methyl ether, m.p. 72–73°, b.p. 182–183°/12 mm.). Azo dyes from the amines are described. C. HOLLINS.

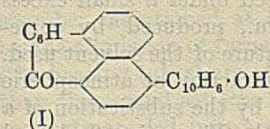
**Manufacture of condensation products of dimethylolurea or dimethylolthiourea.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,875, 27.8.27).—The primary condensation products from carbamide or thiocarbamide and formaldehyde are further condensed in an aqueous medium at 100°, a buffer salt being added to produce a  $pH$  of 4 to 7, preferably 6.3. A continuous process is described. (Cf. B.P. 258,289; B., 1928, 376.) C. HOLLINS.

**Preparation of tetrazoles.** A. BOEHRINGER (B.P. 280,529, 1.11.27. Ger., 10.11.26).—The esters, e.g., the benzenesulphonates, of oximes (especially of cyclic ketoximes) or their Beckmann transformation products are treated with sodium azide or azoimide in absence of concentrated mineral acid. Thus cyclohexanoneoxime *O*-benzenesulphonate with sodium azide yields 2:3-pentamethylenetetrazole,  $\epsilon$ -leucine lactam being formed as by-product. C. HOLLINS.

**Purification of crude anthracene.** A. O. JAEGER, Assr. to SELDEN RES. & ENG. CORP. (U.S.P. 1,693,713, 4.12.28. Appl., 16.1.28).—Furan derivatives, especially methylfurfuraldehyde, readily dissolve carbazole and phenanthrene but very little anthracene in the cold. Two crystallisations from furfuraldehyde or methylfurfuraldehyde at 80° give pure anthracene. Other solvents may be used in combination with these. C. HOLLINS.

**Manufacture of condensation products of the benzanthrone series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,331, 25.8.27).—Benzantrones having free 4- and 3-positions are heated with aromatic hydroxy-compounds having free *o*- or *p*-positions in presence of alkali hydroxide in quantity only slightly in excess of that required for the formation of the phenoxide. Benzantrones and  $\beta$ -naphthol at 200° give 1-(4-benzanthronyl)- $\beta$ -naphthol (I), bright yellow

turning red at 260°, m.p. above 300°. 6-Bromo-2-naphthol gives a similar product, and the product from  $\beta$ -naphthol and 6-chlorobenzanthrone is described. Similar products from  $\alpha$ -naphthol or 2-chloro- $\alpha$ -naphthol and benzanthrone, from  $\alpha$ -naphthol and benzo-



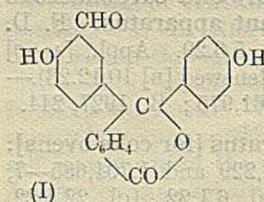
benzanthrone-13-carboxylic acid, and from benzanthrone and *m*-2-xylene (yellow) are described.

R. BRIGHTMAN.

**Sulphonic acids** (G.P. 451,534).—See XV. **Betaine hydrochloride** (B.P. 304,071).—See XVII.

#### IV.—DYESTUFFS.

**Aldehydophenolphthalein and dyes derived from it.** R. N. SEN and K. C. KAR (J. Indian Chem. Soc., 1929, 6, 53–63).—The Reimer-Tiemann reaction with phenolphthalein affords 20%



of aldehydophenolphthalein (I), m.p. 97–99° (phenylhydrazone, m.p. 148°; dioxime, decomp. 235°), which yields a condensation product, phenolphthaleinoin, m.p. 152°, analogous to benzoin. An alkaline solution of I is more

deeply coloured than an alkaline solution of phenolphthalein. Condensation of I with *p*-toluidine (m.p. 140°),  $\beta$ -naphthylamine (m.p. 154°), aminoazobenzene (m.p. 235°), benzidine (m.p. 191°), *o*- and *p*-phenylenediamines (m.p. 218° and 230°), chrysoidine, safranin, and rosaniline gives the corresponding azomethine dyes having the m.p. given in parentheses; each amino-group reacts with 1 mol. of I. A diminution in the intensity of colour of the last three substances is thereby produced. These azomethines dye wool and silk with shades varying from light yellow to bluish-red. Pyronine dyes are obtained by condensing I with resorcinol,  $\beta$ -naphthol, quinol, pyrogallol, and *m*-diethylaminophenol, in presence of warm sulphuric acid. These dyes form red potassium salts, and dye wool and silk with shades varying from light brown to red. Condensation of I with dimethylaniline in presence of hydrochloric acid gives pp'-tetramethyldiaminodiphenylphenolphthaleinylmethane, m.p. 187°, which when oxidised with lead dioxide gives a bluish-green dye (phenolphthalein-green). When I is condensed with *o*-cresotic acid in presence of sulphuric acid and the resulting leuco-compound, m.p. 215°, oxidised with nitrosyl sulphate at 50–60° a red dye is produced. This dye wool with a red shade which deepens to black on after-chroming.

H. BURTON.

**Fluorescence of dyes in Wood's light.** A. SEYEWETZ and J. BLANC (Compt. rend., 1929, 188, 714–715).—A preliminary classification of the fluorescence of dyes in the light of a mercury-vapour lamp, filtered through a screen of Wood's glass (3650 Å. and a slight band at 3500–3700 Å.), is made. Aqueous or aqueous-alcoholic solutions are used, since examination of the powdered dyes does not afford consistent results. Substances such as halogens, alkali halides, silver nitrate,



etc. are found to destroy the fluorescence of fluorescein. Dyed textiles show generally the same fluorescence as the corresponding solution; undyed textiles exhibit but a slight fluorescence. G. A. C. GOUGH.

**Colouring matters from sucrose.** GARINO and TOSONOTTI.—See XVII.

#### PATENTS.

**Manufacture of azo dyes.** I. G. FARBENIND. A.-G. (B.P. [A] 274,128, 11.7.27. Ger., 9.7.26, and [B] Addn. B.P. 275,613, 28.7.27. Ger., 5.8.26).—(A) 6-Halogeno- or 2:6-dihalogeno-*m*-xylydines, obtained by halogenating *as-m*-xylydine in sulphuric acid, are diazotised and coupled in substance or on the fibre with an arylamide of 2:3-hydroxynaphthoic acid, other than the *m*-4-xylylamide or its derivatives. Thus 6-chloro- or 6-bromo-*m*-4-xylydine coupled with the 4-chloro-*o*-anisidide, 5-chloro-*o*-toluidide, or the  $\beta$ -naphthylamide give bright yellowish or neutral red shades very fast to kier-boiling, light, and chlorine. 2:6-Dichloro-*m*-xylydine similarly gives red, scarlet, and claret shades, respectively. (B) 2-Halogeno- or 2-halogeno-6-alkyl-*m*-xylydines are coupled with an arylide of 2:3-hydroxynaphthoic acid. Thus 2-chloro-*m*-xylydine (cf. Nöling, Braun, and Thesmar, B., 1901, 797) and 3-chloro- $\psi$ -cumidine, m.p. 66–67° (obtained by chlorinating  $\psi$ -cumidine sulphate), with the 5-chloro-*o*-toluidide give bluish-red dyeings of good fastness to kier-boiling and chlorine. R. BRIGHTMAN.

**Production of [acid] azo dyes.** K. HEUSNER and M. SIMON, Asses. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,783, 6.11.28. Appl., 7.6.27. Ger., 6.4.25).—Diazo compounds are coupled with  $\beta$ -naphthylsulphamic acids; e.g., sulphanilic acid  $\rightarrow$   $\beta$ -naphthylsulphamic acid (orange), metanilic acid  $\rightarrow$  6-sulpho- $\beta$ -naphthylsulphamic acid (yellowish-orange). R. BRIGHTMAN.

**Manufacture of [acid azo] dyes.** SOC. CHEM. IND. IN BASLE (B.P. 281,713, 3.12.27. Switz., 4.12.26).—Dyes obtained by coupling a pyrazolone with diazotised *m*-aminoacetanilide-4-sulphonic acid, 4-aminoaceto-*o*-toluidide-5-sulphonic acid, or 5-chloro-*m*-aminoacetanilide-4-sulphonic acid, give level yellow shades, fast to light, on wool (cf. B.P. 9968 of 1910). C. HOLLINS.

**Manufacture of azo dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 307,104, 2.12.27).—*p*-Aminoacetophenone, *p*-aminobenzophenone, or a *m*-nitro-derivative of these is diazotised and coupled with acetoacetic arylamides to give green-yellow dyes for oil pigments and size colours. Examples are: *p*-aminoacetophenone  $\rightarrow$  acetoacetanilide; 3-nitro-4-aminoacetophenone  $\rightarrow$  acetoacetic *o*-chloroanilide (product, m.p. 269–270°); *p*-aminobenzophenone  $\rightarrow$  acetoacetic *o*-chloroanilide (product, m.p. 159–160°). C. HOLLINS.

**Manufacture of insoluble [azo] dyes.** O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 307,150, 31.12.27).—Safranisol (3:7-diamino-8-methoxy-5-*o*-anisylphenazonium chloride) is tetrazotised and coupled in substance or on the fibre with a 2:3-hydroxynaphthoic arylamide. The *p*-phenetidine,  $\beta$ -naphthylamide, and *m*-toluidide give deep blacks, the *m*-nitroanilide and *m*-anisidide black-violets, and the anilide a black-brown. Special fastness to chlorine is claimed. C. HOLLINS.

**Manufacture of vat [benzanthrone] dyes.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,874, 22.8.27 and 1.2.28).—Dark blue to violet to black vat dyes result from the alkaline fusion of benzanthronyl-aminoanthraquinones obtained by condensing an amino- or diamino-anthraquinone with mono- or di-chloro-benzanthrones (other than those prepared from 1-chloroanthraquinone) containing no substituents or containing in the 3- or 4-position an etherified hydroxyl group or a 4'- or 3'-benzanthronyl group. Examples are the alkaline fusion of condensation products of 1-aminoanthraquinone with 2-, 9-, 10-, or 11-chlorobenzanthrone, 9-chloro- or 9:9'- or 10:10'-dichloro-4:4'-dibenzanthronyl, dibrominated 4:4'-dibenzanthronyl, 9:9', 10:10', or 11:11'-dichloro-3:3'-dibenzanthronyl, 9-, 10-, or 11-chloro-3-methoxybenzanthrone, brominated 3-methoxybenzanthrone, 9-halogeno-3-phenoxybenzanthrone, 9-chloro-4:3'-dibenzanthronyl, or dibrominated 4:3'-dibenzanthronyl; of 1-amino-4-methoxyanthraquinone with 9-chlorobenzanthrone, 9:9'-dichloro-3:3'- or -4:4'-dibenzanthronyl, 9-chloro-3-methoxybenzanthrone, or brominated 4:3'-dibenzanthronyl; of 2-aminoanthraquinone or 1:6- or 1:5-diaminoanthraquinone with 9-chlorobenzanthrone; of 1:6-diaminoanthraquinone with 9:9'-dichloro-3:3'-dibenzanthronyl or dibrominated 4:3'-dibenzanthronyl; of 1-amino-4-hydroxyanthraquinone with 9:9'-dichloro-3:3'-dibenzanthronyl; of 3-aminoalizarin with dibrominated 4:3'-dibenzanthronyl; and of 1-chloroanthraquinone-2-carboxylic acid with 9-aminobenzanthrone. C. HOLLINS.

**Manufacture of vat dyes.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,432, 1.2.28).—Tetrahalogeno-2:2'-dibenzanthronyls are heated with aminoanthraquinones in presence of copper or other metallic compounds and of an acid-fixing agent. *Tetrabromo-2:2'-dibenzanthronyl*, obtained, e.g., by brominating 2:2'-dibenzanthronyl in chlorosulphonic acid, with 1-aminoanthraquinone in nitrobenzene in presence of sodium carbonate and cupric oxide gives a fast black vat dye; a similar dye is obtained at 320–340° in absence of nitrobenzene; with 1-amino-4-methoxyanthraquinone a blue-violet vat dye is obtained, with 1:6-diaminoanthraquinone, a greyish-black vat dye, with 2-aminoanthraquinone or, e.g., 3-chloro-2-aminoanthraquinone at 300–340°, a greyish-black vat dye, and with 3- or 4-aminoalizarin a blue-black vat dye. 9:9'-Dichlorodibromo-2:2'-dibenzanthronyl, obtained by brominating 9:9'-dichlorodibenzanthronyl with excess of bromine, with 1-aminoanthraquinone gives a blue-black vat dye. R. BRIGHTMAN.

**Manufacture of vat dyes.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,219, 7.7.27).—2-Halogenoanthrones containing a further nucleus attached to the *meso*- and 1-positions by means of a nitrogen atom at least in the 1-position, are heated at 200–250° with a metal, to give bright yellow or orange vat dyes. 3-Halogenopyrazolanthrones, e.g., 3-bromopyrazolanthronone (from 2-bromo-1-aminoanthraquinone), are previously acetylated, the acyl group being removed from the condensation product by hydrolysis if desired. *Acetyl-3-bromopyrazolanthronone*, m.p. 209–211°, affords a yellow acetyl condensation product, hydrolysed by

sulphuric acid to a yellow dye. 4-Bromopyrimidone-anthrone gives a *methyl ether*, m.p. 285—290°, converted when heated with copper and naphthalene into a yellow vat dye. Dyes similarly obtained from 4-iodomethylpyrimidanthrone, m.p. 210—215° (from *C*-methyl-2:1-anthraquinoneoxazole and ammonia through 4-amino-methylpyrimidanthrone), 6-iodocoeramidomine, m.p. 203—205°, 6-bromocoeramidomine, m.p. 210°, and from the *methyl ether*, m.p. 255—260°, of 4-bromopyridonanthrone are described. R. BRIGHTMAN.

**Manufacture of indigoid vat dyes.** I. G. FARBENIND. A.-G. (B.P. 300,114, 8.3.28. Ger., 5.11.27. Addn. to B.P. 282,805, 1928, 849).—In the process of the prior patent isatin  $\alpha$ -chlorides are used in place of  $\alpha$ -arylamides. C. HOLLINS.

**Manufacture of sulphur dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,909, 4.8.27).—Sulphide dyes giving light-fast green shades are obtained by melting with a polysulphide in presence of a copper salt the indophenols obtained by oxidation of *p*-aminophenols with alkyl ethers of *p*-hydroxyaryl-1-naphthylamine-6-, -7-, or -8-sulphonic acids. The dyes from 1-*p*-anisylamino-, 1-*p*-phenetylamino-, and 1-*p*-anisylamino-4-*p*-hydroxyphenylamino-naphthalene-8-sulphonic acid are described. The *p*-anisyl-1-naphthylamine-6-, -7- and -8-sulphonic acids and *p*-phenetyl-1-naphthylamine-8-sulphonic acid are obtained by the process of B.P. 7337/1892 (B., 1893, 350). R. BRIGHTMAN.

**Finely-divided phenolphthalein and its manufacture.** E. B. PUTT (U.S.P. 1,693,666, 4.12.28. Appl., 11.5.26).—Phenolphthalein is precipitated from an alkaline solution containing gum acacia to retard crystallisation. C. HOLLINS.

**Azo dyes and their preparation.** H. EICHWEDE, ASSR. to GRASELLI DYESTUFF CORP. (U.S.P. 1,694,117, 4.12.28. Appl., 21.12.26. Ger., 23.12.25).—See B.P. 263,816; B., 1928, 183.

**Anthraquinone vat dye.** W. MIEG, ASSR. to GRASELLI DYESTUFF CORP. (U.S.P. 1,690,236, 6.11.28. Appl., 20.1.26. Ger., 21.1.25).—See B.P. 297,133; B., 1928, 849.

**Vat dyes of the 2:2'-indole-thionaphthen-indigo series.** C. KRAUSS, ASSR. to GRASELLI DYESTUFF CORP. (U.S.P. 1,691,660, 13.11.28. Appl., 11.6.27. Ger., 15.6.26).—See B.P. 295,239; B., 1928, 740.

**Derivatives of dibenzpyrenequinone and their preparation.** G. KRANZLEIN, K. ZAHN, P. OCHWAT, and M. CORELL, ASSRS. to GRASELLI DYESTUFF CORP. (U.S.P. 1,693,447, 27.11.28. Appl., 28.12.26. Ger., 30.11.25).—See B.P. 263,845; B., 1927, 870.

**[Manufacture of] vat dye.** H. NERESHEIMER, ASSR. to GRASELLI DYESTUFF CORP. (U.S.P. 1,703,948, 5.3.29. Appl., 22.12.27. Ger., 23.12.26).—See B.P. 282,629; B., 1928, 517.

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

**Colour reactions of lignin.** F. PODBREZNIK (Bull. Inst. Pin, 1928, 233—236, 245—250; Pulp & Paper Mag., 1929, 27, 229—248).—The colour reactions of lignin with various primary and secondary aromatic

amines, phenols, naphthols, and certain oxidising and reducing agents are reviewed and a new characteristic reaction is described depending on the action of hydrochloric acid and potassium chlorate solution, with which pine sawdust and lignin give, on warming, an orange colour turning yellow on addition of ammonia. From the facts that (a) extracted lignins (Willstätter's lignin, phenol-lignin, alkali-lignin), oxidised lignin, and lignin freed from pentosans give the same colour reactions as lignified tissue, and (b) that the alcoholic and ethereal extracts of sawdust do not give some of the most characteristic colour reactions of lignin, it is concluded that these reactions are due in the main to the lignin complex itself, and not, as previously suggested, to methyl-pentosans or aromatic compounds such as vanillin, coniferyl alcohol, etc., though these last-named, which are probably not present in the original wood, but are formed by depolymerisation or hydrolysis of the lignin during the extraction process, may influence the reactions to a certain extent. D. J. NORMAN.

**Alteration of sugar-like substances in sulphite-cellulose cooking.** II. E. HÄGGLUND and T. JOHNSON (Svensk Kem. Tidskr., 1929, 41, 55—59; cf. B., 1929, 202).—The sugars (mainly mannose, xylose, and dextrose, with small amounts of galactose and levulose) formed during the manufacture of cellulose are readily oxidised to carboxylic acids by boiling with the calcium sulphite liquor. In an extract prepared by boiling 332 g. of air-dried wood with 1500 c.c. of a sulphite liquor containing 4.6% of sulphurous acid and 1.32% of lime for 20 hrs. at 135° (maximum temperature), the presence of mannonic and xylonic acids has been definitely proved, the former being identified by its brucine compound and phenylhydrazone, the latter by its double salt with cadmium bromide. Tests for gluconic acid, on the other hand, were negative. H. F. HARWOOD.

**Detergent action of soap.** RHODES and BRAINARD.—See XII. **Treatment of [wool] trade waste.** SNELL. **Treatment of wastes from paper mills.** FALES.—See XXIII.

## PATENTS.

**Manufacture of colloidal solutions of natural silk.** T. MUTO, S. HIDA, and KANEGAFUCHI BOSEKI KABUSHIKI KWAISHA (B.P. 306,699, 18.2.28).—Silk fibre is treated with a solution of a magnesium salt, e.g., nitrate, which is afterwards removed from the colloidal solution by dialysis etc. F. R. ENNOS.

**Production of [non-creasing] cellulosic fabric.** TOOTAL BROADHURST LEE Co., R. P. FOULDS, and J. T. MARSH (B.P. 304,900, 9.1.28).—The fabric is mercerised with caustic soda, washed, and impregnated, while still in the wet swollen condition, with a synthetic resin or the components thereof. After drying at a low temperature it is heated for a short time, and is finally soaped, washed, and dried. F. R. ENNOS.

**Preparation of artificial silk.** S. I. VLES and D. VAN DER WANT (B.P. 306,050, 10. and 12.9.27).—A workable artificial silk yarn is made from untwisted bundles of filaments by treating them with substances which are solvents or swelling agents at the working temperature or a few degrees above it, so that different

parts of each bundle are fixed together by adhesion at regular or irregular intervals throughout its length.

F. R. ENNOS.

#### Production of artificial filaments or threads.

BRIT. CELANESE, LTD., H. DREYFUS, E. KINSELLA, J. BOWER, and W. I. TAYLOR (B.P. 304,674, 24.10.27. Addn. to B.P. 300,998; B., 1929, 203).—Cellulose acetate is spun vertically downwards into heated chambers or cells each of which, containing any desired number of spinning nozzles, is either open at the end remote from the nozzles or is partially closed to ensure even distribution of the air. The chambers are provided with a collector device, so that substantially the whole of the air is drawn through the vicinity of the spinning nozzles.

F. R. ENNOS.

Preparation of artificial textile products [viscose silks] with reduced lustre. G. BONWITT (B.P. 288,222, 7.9.27. Holl., 7.4.27. Addn. to B.P. 285,066; B., 1929, 203).—In applying the process of the prior patent to viscose silks having denier numbers ( $D$ ) of 6 or less, the chlorobenzene particles must be of diameter not greater than  $x$  microns, where  $D^2 = \frac{2}{3}(x^2 + x)$ .

C. HOLLINS.

Treatment of materials consisting of or comprising cellulose derivatives. [Preservation of lustre of acetate silk.] BRIT. CELANESE, LTD. (B.P. 282,722, 23.11.27. U.S., 30.12.26).—Addition of 0.1—30% of a thiocyanate (ammonium, sodium, potassium, calcium) to the bath prevents or diminishes the delustring of acetate silk by boiling liquids.

C. HOLLINS.

Manufacture of cellulose ethers. O. LEUCHS and E. DÖRR, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,694,127, 4.12.28. Appl., 31.10.27. Ger., 8.11.26).—Alkali-cellulose and ethyl chloride are heated in a closed vessel containing a perforated vessel charged with solid sodium hydroxide; the latter absorbs the water of reaction, dissolves gradually, and falls into the reaction mixture.

C. HOLLINS.

Moth-proofing substance. L. E. JACKSON and H. E. WASELL, Assrs. to MUNDATECHNICAL PRODUCTS Co. (U.S.P. 1,694,219, 4.12.28. Appl., 18.11.27. Cf. B.P. 263,092; B., 1928, 227).—Quinoidine, the mixture of amorphous alkaloids remaining in the acid extract of cinchona bark after removal of crystalline alkaloids, is used for moth-proofing.

C. HOLLINS.

Purifying dry-cleaning solvent. A. E. HATFIELD, Assr. to ACHILLE SERRE, LTD. (U.S.P. 1,704,604, 5.3.29. Appl., 30.9.26. U.K., 31.12.25).—See B.P. 266,850; B., 1927, 579.

Manufacture of cellulose ethers. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 306,857, 21.10.27).—See U.S.P. 1,694,127, preceding.

Manufacture of compound yarns or threads. O. RASCH (B.P. 306,007, 10.10.27).

Apparatus for drying webs of woven material. RUDOLPH & KÜHNE GES.M.B.H. (B.P. 288,175, 26.3.28. Ger., 2.4.27).

Pumps for viscose or other liquids. L. FRIEDMANN and N. SCHOPP (A. FRIEDMANN and G. NEMETZ (B.P. 307,225, 22.3.28).

Nitrocellulose solutions etc. (B.P. 307,085 and 307,528). Pigmented products (B.P. 277,949, 277,989, and 307,516).—See XIII. Tanning materials (G.P. 451,913).—See XV.

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

Applications of ozone [for bleaching]. A. VOSMAER (Chem. Weekblad, 1929, 26, 184—186).—An account of the use of ozone for bleaching beeswax, linseed and soya-bean oils, mineral oils, cotton, paper, sugar, and other organic products.

S. I. LEVY.

Bleaching of wood pulp. BAIRD and DOUGHTY.—See V.

### PATENTS.

Increasing wetting and penetrating capacity of liquids for treatment of textiles and leather. H. T. BÖHME A.-G. (B.P. 291,070, 20.9.27. Ger., 27.5.27).—Mixtures (or salts) of pyridine bases with alkylated aromatic sulphonic acids are used as wetting-out agents. Either or both ingredients may be hydrogenated.

C. HOLLINS.

Treatment of textiles. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 306,067, 15.11.27).—Materials consisting either wholly of natural silk or of mixtures of natural silk with cellulose acetate are degummed by heating with an alkaline liquor containing alkali hydroxides, borates, silicates, phosphates, phenolates, cresolates, soap, or a sulphonated soap or oil, the  $p_H$  value of which is maintained at 10—10.5 throughout the treatment; if the temperature is raised above 80° the cellulose acetate may be wholly or partially delustred.

F. R. ENNOS.

Dyeing of animal fibres. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 306,632, 5.12.27).—Deep black shades on wool are obtained by oxidising on the fibre a sulphonated *p*-phenylenediamine or *NN*-di(*p*-aminophenyl)-*p*-phenylenediamine, in which one or both amino-groups carry a naphthyl residue, or a sulphonated naphthylenediamine in which one or both amino-groups carry a *p*-aminophenyl residue. Examples are: 4'-amino-2' (or 3')-sulphophenyl- $\beta$ -naphthylamine; *p*-aminophenyl-Cleve acid; 1:4-di-(6-sulpho- $\beta$ -naphthylamino)benzene; 4-amino-4'- $\alpha$ - and - $\beta$ -naphthylaminodiphenylamine-2-sulphonic acids; 4- $\beta$ -naphthylamino-4'-hydroxydiphenylamine-2-sulphonic acid; 1:4-di-(*p*- $\alpha$ - and - $\beta$ -naphthylamino-*o*-sulphoanilino)benzenes; 1:5-di-(4'-*p*-toluidino-3'-sulphoanilino)naphthalene,  $C_{10}H_6[NH \cdot C_6H_3(SO_3H) \cdot NH \cdot C_6H_4Me]_2$ ; 2:6-di-(4'-amino-2'-sulphoanilino)naphthalene.

C. HOLLINS.

Production of fast dyeings and printings. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 306,637, 6.12.27. Addn. to B.P. 306,632, preceding).—Wool, silk, acetate silk, and mixtures of these are dyed in level shades by oxidising on the fibre a salt of a non-sulphonated *p*-aminoanilino-derivative of naphthalene. Examples are: 2:7-di-(*p*-aminoanilino)naphthalene (bluish-grey to blue-black); 2-*p*-aminoanilino-7-naphthol (blue); *p*-aminophenyl- $\beta$ -naphthylamine (blue-black); *p*-aminophenyl- $\alpha$ -naphthylamine (corinth); 2-amino-7-*p*-aminoanilino-naphthalene (blue to blue-grey).

C. HOLLINS.

**Colouring of cellulose derivatives.** BRIT. CELAN-  
ESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 306,981,  
28.11.27).—Acetate silk etc. is dyed with dehydrothio-  
toluidine either diazotised and coupled in substance or  
on the fibre with suitable non-sulphonated coupling  
components (phenol,  $\beta$ -naphthol, 2:3-hydroxynaphthoic  
acid, dimethylaniline,  $\beta$ -naphthylamines, 2:3-amino-  
naphthoic acid, 1:2-aminonaphthol, ethyl ether, etc.),  
or condensed with a chloronitrobenzene, especially  
1-chloro-2:4-dinitrobenzene (yellow). C. HOLLINS.

**Dyeing of fibres consisting of cellulose mono-  
or di-acetate.** G. B. ELLIS. FROM CHEM. WORKS  
FORMERLY SANDOZ (B.P. 306,877, 27.10.27).—Cellulose  
which has been mono- or di-acetylated but retains the  
original fibrous structure is dyed with the usual acetate  
silk dyes, excluding basic di- and tri-phenylmethane,  
azine, thiazine, and oxazine dyes. C. HOLLINS.

**Dyeing of vat, sulphur, and mineral khaki  
colours on fabrics.** J. WAKEFIELD (B.P. 307,238,  
23.3.28).—The scoured and bleached fabric is batched  
through water containing a little alkali and hyposulphite  
(for vat dyes) or sodium sulphide (for sulphide dyes),  
and dyed in a closed kier, from which air has been  
displaced by steam, by forcing into the kier a fully  
reduced vat solution or a sulphide dye bath or a mineral  
bath. The dye solution is then removed, and the colour  
is developed with an oxidising solution (in the case of  
vat dyes) in the kier. Level shades are produced.  
C. HOLLINS.

**Treatment of materials made of or containing  
cellulose derivatives.** BRIT. CELANESE, LTD., and  
G. H. ELLIS (B.P. 306,611, 28.11.27 and 26.6.28. Addn.  
to B.P. 302,775; B., 1929, 169).—Acetate silk etc. is  
treated with sodium or potassium zincate of such alkali-  
nity as to produce partial hydrolysis; adsorption is  
assisted by addition of swelling agents. The treated  
material has affinity for basic, acid, mordant, direct,  
and vat dyes, and is particularly suited for production of  
discharge effects. The tin-treated material of the prior  
patent shares the last-mentioned property.  
C. HOLLINS.

**Alkaline treatment [mercerisation] of natural  
and artificial cellulosic materials.** IMPERIAL CHEM.  
INDUSTRIES, LTD., and E. CHAPMAN (B.P. 307,239,  
14.4.28).—As a wetting-out agent for use with mer-  
cerising liquors or other strongly alkaline media, a  
mixture of cresol (or other homologous phenol) with  
1—15% of benzyl alcohol, butyl alcohols, amyl alcohols,  
or the synthetic alcohols, b.p. 120—200°, obtained by  
hydrogenation of carbon oxides is found effective.  
C. HOLLINS.

**Production of metallised fabrics.** O. TREBITSCH  
(B.P. 285,921, 24.2.28).—Fibrous materials are treated  
with a metallic salt solution and the metal is precipitated  
thereon by means of a suitable reducing agent. After  
dissolving out any compounds not completely reduced  
by means of a suitable acid solution, the material is  
subjected to mechanical treatment until the precipitated  
metal is consolidated to a tenacious coating.  
F. R. ENNOS.

**Production of fast tints on vegetable fibre.** F.  
STRAUB, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P.

1,704,637, 5.3.29. Appl., 23.5.27. Switz., 27.5.26).—  
See B.P. 271,897; B., 1929, 14.

**Washing, bleaching, and like machines.** J. L.  
RUSHTON, J. LEVER, and H. HILL (B.P. 307,113, 7.12.27).

**Bleaching and dyeing plants.** G. P. GASS, and  
JACKSON & BRO., LTD. (B.P. 307,632, 16.3.28).

**Covers for low-pressure kiers.** W. and J. B.  
SADLER (B.P. 307,621, 2.3.28).

**Printing patterns on plush fabrics etc.** W. C.  
FAIRWEATHER. FROM PLÜSCHWEBEREI GREFRATH A.-G.  
(B.P. 308,123, 14.6.28).

**Decorating the surface of textile materials etc.**  
F. A. H. HEYNERT, and BRIT. BEAD PRINTERS, LTD.  
(B.P. 307,241, 19.4.28).

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

**Kinetics of the contact sulphuric acid process,  
and modern views on adsorption.** M. BODENSTEIN  
(Z. physikal. Chem., 1929, B, 2, 345—348).—Fink and  
Bodenstein's interpretation of their experimental data  
for the rate of combination of sulphur dioxide and oxygen  
on the surface of platinum (B., 1908, 120) requires  
modification to make it accord with modern views on  
adsorption, for it is no longer possible to accept the view  
that a layer of trioxide many molecules thick is adsorbed  
on the surface of the catalyst. If, however, there is a  
unimolecular surface layer of trioxide, and if combination  
occurs only at certain active points or lines, the measured  
reaction velocity will still be the rate of diffusion of the  
reactants to the seat of reaction, but this will be a  
diffusion which is not perpendicular to the surface, but  
along it, and is hindered by the two-dimensional motion  
of the adsorbed trioxide molecules. With this altera-  
tion, the explanation previously advanced still holds  
good.  
R. CUTHILL.

**Action of calcium carbonate and of dolomite on  
zinc sulphate solutions.** L. CAMBI, G. BOZZA, and  
D. MASPERI (Giorn. Chim. Ind. Appl., 1929, 11, 3—8).—  
In the electrolytic production of zinc, calcium carbonate  
is almost universally employed to neutralise the electro-  
lyte, precipitate the iron, and flocculate the silica.  
Results are given of determinations of the velocity of  
reaction of calcium carbonate (mostly marble) and of  
dolomite, ground to various degrees of fineness, on  
2*N*-zinc sulphate solution at 40·2° and 100°. The  
values of the velocity coefficients of the reaction at these  
two temperatures are 0·000282 and 0·00124 for calcite  
and 0·0000884 and 0·000636 for dolomite; for calcite  
the amounts of zinc rendered insoluble at these tempera-  
tures are approximately proportional to these numbers,  
whereas with dolomite the amount is relatively much  
greater at the higher temperature. The results indicate  
that the layer of insoluble matter which forms on the  
calcium or magnesium particles is only slightly permeable  
to the zinc sulphate solution, and that diffusion virtually  
ceases when a very thin layer has been deposited. The  
precipitate formed contains very little calcium or  
magnesium, and consists mostly of zinc hydroxide,  
together with small proportions of zinc sulphate and  
carbonate.  
T. H. POPE.

**Determination of [bismuth in] bismuth hydroxy-iodide.** G. BÜMMING and K. FERREIN (Arch. Pharm., 1929, 267, 142—143).—In the determination of bismuth, the hydroxyiodide, which may be brown in colour, must be boiled with twenty times its weight of nitric acid until no more iodine vapours are evolved; the mixture is then evaporated and ignited and the residue of  $\text{Bi}_2\text{O}_3$  weighed. S. COFFEY.

**Extraction of iodine from Black Sea algæ.** S. KOMARECKYJ (Chem. Listy, 1929, 23, 5—7).—The yield of iodine from algæ is 24—41% less when air is freely admitted during combustion than when the supply is restricted. A mixture of various Black Sea algæ contained 0.0859—0.172% of iodine, or 0.138—0.338% of the ash remaining after combustion. The corresponding figures for *Cystosira* are 0.095 and 0.146% of iodine, and for *Phylophora* 0.645 and 1.01%.

R. TRUSZKOWSKI.

**Absorption of oxygen by dilute alkaline solutions of pyrogallol.** T. J. DRAKELEY and H. NICOL (J.S.C.I., 1929, 48, 62—64; cf. B., 1925, 944).—All alkaline solutions of pyrogallol evolve carbon monoxide when used as an absorbent for oxygen of high purity. For constant concentration of alkali the percentage of carbon monoxide increases as the amount of pyrogallol diminishes, but no stoichiometrical relation was found. Agitation of the solution during absorption of the oxygen is an important factor in minimising the evolution of carbon monoxide.

**Applications of ozone.** VOSMAER.—See VI.

#### PATENTS.

**Preparation of alkyl compounds of lead.** C. A. KRAUS and C. C. CALLIS, ASSTS. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,694,268, 4.12.28, Appl., 13.4.23).—A lead-sodium alloy containing 20% Na is treated with ethyl iodide, and water equivalent to the sodium is added at such a rate as to keep the mixture boiling. Lead tetraethyl is then distilled off in steam.

C. HOLLINS.

**Production of finely-divided sulphur.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,498, 20.2.28. Addn. to B.P. 177,103; B., 1922, 373 A).—A solution of ammonium polysulphide containing a protective colloid, e.g., cellulose pitch, flowing by gravity along the walls of a rotating pipe, is treated with a current of steam flowing in the same direction or in counter-current.

W. G. CAREY.

**Preparation of hydrogen.** H. BOMKE, ASSEE of BERGWERKSVERBAND ZUR VERWERTUNG VON SCHUTZRECHTEN DER KOHLENTÉCHNIK G.M.B.H. (B.P. 279,128, 17.10.27. Ger., 16.10.26).—Magnesium oxide in conjunction with substances absorbing carbon dioxide is used as the catalyst for preparing hydrogen from carbon monoxide, or water-gas, and water vapour. Calcined magnesian lime may be employed, or uncalcined magnesian lime, the carbon dioxide being first set free in the process.

W. G. CAREY.

**Acetic acid** (B.P. 296,974 and 301,415).—See III. **Treatment of pyrites** (B.P. 306,691).—See X. **Electrolytic cell** (G.P. 452,922).—See XI. **Potassium chloride from molasses** (B.P. 304,071).—See XVII.

## VIII.—GLASS; CERAMICS.

**Physico-chemical phenomena exhibited by minor constituents in glass.** W. E. S. TURNER (J.S.C.I., 1929, 48, 65—67 T).—Glass melted at 1400° from mixtures containing moisture, or in tank furnaces the gases of which contain water vapour, retains 0.02—0.08% of water. Arsenious oxide is tenaciously retained in glass melted at 1400°; moreover, except in the presence of large excess of reducing agents, much of it is converted into arsenic oxide, and only at 1500° does this appear slowly to be dissociated into arsenious oxide and oxygen. Ferric oxide is the more stable form in soda-lime-silica glass at 1400—1500°. In solution in glass, melted electrically in a neutral atmosphere, for concentrations between 0.005 and 1.0%  $\text{Fe}_2\text{O}_3$ , the actual ferric oxide in the glasses was found to be 92—94% of the total iron oxide. Under reducing conditions, with sodium tartrate as reducing agent in the mixture, the maximum proportion of ferrous to total iron oxide obtainable was 89%. Glass containing ferrous oxide has a greater transmission in the ultra-violet than when the iron is present as ferric; for a concentration of 0.1% of iron oxide, the difference in the transmission between the ferrous and ferric oxide glasses is about 100 Å. Irradiation by sunlight or electric arc converts the ferrous into ferric oxide. When the glass so irradiated is heated at 600—700° the effect is reversed. (Cf. B., 1929, 245.)

**Determination of the source and means of prevention of stones in glass.** H. INSLEY (J. Amer. Ceram. Soc., 1929, 12, 143—152).—Distinction is made according to their source between devitrification, batch, and refractory "stones." The former may consist of tridymite, wollastonite, diopside, jeffersonite, and  $\text{Na}_2\text{O} \cdot 3\text{CaO} \cdot 6\text{SiO}_2$ . Photomicrographs show the crystalline forms characteristic of each type. "Cordy" devitrification is due to imperfect mixing of the batch or entrainment of the scum. Random distribution of devitrification stones is avoided either by alteration of the cooling schedule or of batch composition. Batch stones are caused by the incomplete dissolution of one of the batch constituents (usually sand or clay) and can be avoided by (1) finer grinding of batch, (2) more intimate mixing of batch, (3) higher melting temperature, and (4) longer melting time. Microscopical examination distinguishes two types of refractory stones: (a) those formed by the action of alkaline vapours on the refractory above the glass level and usually composed of large corundum crystals, and (b) those formed below the level of the glass and composed of smaller corundum crystals associated with nephelite and mullite. Some stones show only partial attack by the glass (quartz grains remaining), and are probably from the cooler parts of the system. Corundum stones dissolve very slowly and are therefore most objectionable. J. A. SUGDEN.

**Physical structure of refractory materials.** T. S. CURTIS (J. Amer. Ceram. Soc., 1928, 11, 904—916).—The relationship of the grog to the matrix of aluminosilicate refractories is investigated, using photomicrographs in natural colour (cf. B., 1928, 693). The products under consideration are divided into three categories, viz., (1) refractory bodies with large developments of mullite, such as firebricks for boiler settings

and glass tank blocks for the slag line; (2) refractory clay wares, in which mullite is relatively unimportant, such as saggars; (3) vitrified products, such as stoneware, sanitary ware, and whiteware, in which mullite is an incidental inclusion in fused felspathic material. In making products of a high refractoriness-under-load value, grog containing 52%  $\text{Al}_2\text{O}_3$  with less than 5% of basic oxides, and completely inverted to mullite and glass, is bonded with clay of a similar composition and fired to cone 17. For the best results the grog should be fired to cone 30—32, the conditions being alternately reducing and strongly oxidising. The addition of 10—20% of an inert mineral fibre improves the rigidity, strength, and toughness of a low-fire refractory and a vitreous body. Careful control of the grain size and the amount of non-plastic additions to vitreous bodies is essential.

A. T. GREEN.

**Effect of heat treatment on diatomaceous earth.**

H. M. KRANER (J. Amer. Ceram. Soc., 1928, 11, 875—883).—Samples of various deposits of diatomaceous earth were fired to temperatures of 1100°, 1260°, and 1300°, and held at these temperatures for 2 hrs. It is shown that the diatom structure may be destroyed without any indication of the deformation of the material. Further, there is a direct relationship between the maintenance of the diatom structure and the purity of a diatomaceous earth. Thus the silica content of such an earth gives an indication of its resistance to the influences of high temperatures. A pure sample shows little deterioration at 1300°.

A. T. GREEN.

**Effect of thermal shock on the transverse strength of fireclay brick.**

C. W. PARMELEE and A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1928, 11, 884—895).—A description of an apparatus designed to give data concerning the effect of repeated thermal shock on the transverse strength of fireclay brick is given. After heating the fireclay brick to a uniform temperature of 1100°, its central portion was cooled by a blast of air at room temperature, the remaining surface being protected by insulating material. The results indicated that stiff-mud bricks were more affected than dry-pressed bricks. Plastic clay products showed deterioration. Brands having a high transverse strength suffered a comparatively large decrease in strength, whilst porous bricks were less affected.

A. T. GREEN.

**Correction of an extreme case of cracking in the drying of brick.**

H. FRÉCHETTE and J. G. PHILLIPS (J. Amer. Ceram. Soc., 1929, 12, 153—161).—Attempts to manufacture stiff-mud brick from a very plastic, low-permeability clay resulted in high drying breakage even when drying was very slow. Laboratory tests showed that this could be completely overcome by preheating the clay to 400—500° (heating above 500° makes the clay too short to work) or by treating the clay with aluminium, ferric, or sodium chloride together with the addition of grog. The temperature range of the preheating treatment is too narrow for rotary kiln operation. Plant-scale tests led to the adoption of chemical treatment of the clay using 1% of ferric chloride, 0.5% of sodium chloride, and 10—15% of grog. The colour of the brick was also improved and a slight tendency to scumming was overcome.

J. A. SUGDEN.

**Physical tests for vitreous enamels.** C. J. KINZIE (J. Amer. Ceram. Soc., 1929, 12, 188—192).—Methods of test are described for resistance to thermal shock, impact, and abrasion. In the first of these water is allowed to drip on to the surface, which is heated to 360°; the resulting cracks are shown up by rubbing with lamp-black. In the impact test a steel ball is allowed to fall repeatedly on the surface from a fixed height; the extent of injury is judged by the loss in weight or by visual examination. It is noted that an enamel with a high resistance to impact usually has a high resistance to cross-bending. The abrasion test consists in coating the inside of a metal cup with the enamel to be tested, partially filling with the abrasive charge, and rotating in a suitable clamp on the end of a horizontal shaft at 63 r.p.m. for 41 hrs. The abrasive charge is made up of water, cleansing and washing powders, and  $\frac{1}{4}$ -in. porcelain balls, and it half fills the cup. The extent of abrasion is measured by the loss in weight sustained by the cup. The results show that there is little or no connexion between resistance to abrasion and other properties of enamels.

J. A. SUGDEN.

**Vitreous enamel and its defects.** A. MALINOVSKY (J. Amer. Ceram. Soc., 1929, 12, 180—187).—Literature dealing with the defects in enamelled cast-iron ware is reviewed. The characteristics of ground coat, enamel, and metal surface are discussed, and the various defects due to the metal surface are illustrated by photographs.

J. A. SUGDEN.

## PATENTS.

**Manufacture of glass.** S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 307,563, 23.12.27).—Batch materials, particularly such as will give a glass of low thermal expansion, preferably water-free, are finely ground and mixed, then pressed into small rods, and sintered together at or above 900°. The rods are melted and worked by holding directly in the flame without the use of a furnace. Alternatively, sintering may be dispensed with by adding a suitable bonding agent before pressing.

A. COUSEN.

**Metal-glass union.** MANHATTAN ELECTRICAL SUPPLY Co., Assees. of W. F. HENDRY (B.P. 293,292, 9.12.27. U.S., 2.7.27).—To obtain a seal between glass and metal, the glass contains as an ingredient either the same metal or one of its salts, such glass being used alone or joined to a body of ordinary glass.

A. COUSEN.

**Production of enamel.** F. C. R. MARKS. From LEICHTMETALL-VERWERTUNGS-GES.M.B.H. (B.P. 307,259, 15.6.28).—The major portion of an enamel for iron articles consists of lepidolite, or chemically similar material, natural or synthetic, which is ground to extreme fineness and floated simply with water upon the objects. A ground-coat enamel is dispensed with. Substances such as silica, aluminite, lime, or magnesia may be added to vary the coefficient of expansion, the m.p., or the viscosity. The enamel is fired on at 1000°.

F. SALT.

**Production of a coloured coating similar to ceramic enamels.** USINES DE LA BASSE-MEUSE, SOC. ANON., and O. RUPPEL (B.P. 306,615, 26.11.27).—A cement enamel suitable for application in the cold

state to cement etc. consists of 1—3 pts. of Portland cement mixed with 1 pt. of slag cement, together with 10—15% of colouring matter and 0.1—0.5% of an organic acid such as valeric acid. Articles coated with the enamel are treated in drying rooms or stoves with moist air containing up to 5% CO<sub>2</sub>. The setting period can be shortened by treating the articles, after they have been in the stoves for 2 days, in baths containing 100 pts. of water, 8—10 pts. of a carbonate soluble in water, and 1—2 pts. of a soluble silicate. F. SALT.

**Opacifying substance for adding to enamelling compositions.** I. KREIDL (B.P. 283,130, 17.11.27. Austr., 4.1.27).—A powerful opacifying agent for enamels consists of a synthetically prepared alkali or alkaline-earth silico-aluminate, in which the alkali or alkaline-earth oxide is present in the proportion of 1 mol. to 2 mols. of silica. In an example, kaolin is introduced into a boiling solution of caustic soda and the product is dried. F. SALT.

**Production of non-splinterable or safety glass.** J. E. THORNTON (B.P. 308,117 and 308,119, 25.5.28 and 29.5.28).

**Detergents etc.** (B.P. 307,141).—See XII.

## IX.—BUILDING MATERIALS.

**Fire-resistance of hollow load-bearing wall tile.** S. H. INGBERG and H. D. FOSTER (Bur. Stand. J. Res., 1929, 2, 1—334).

**Fusibility of mixtures of ferric oxide and lime.** JACQUÉ.—See X.

### PATENTS.

**Method and apparatus for burning lime and natural cement.** E. C. R. MARKS. FROM LOUISVILLE CEMENT CO. (B.P. 306,856, 21.10.27).—Raw material for calcination is moved by suitable means (gravity etc.) through a preheating zone, in which it is exposed to hot gases the temperature of which is increasing instead of decreasing, thence through a combustion zone, in which the dried and preheated material is rapidly raised to calcination temperature, and finally through a roasting zone. In the combustion and roasting zones the products of combustion and the material move in the same direction; in the preheating zone the reverse is the case. The hot gases from the exit end of the roasting zone are passed to the exit end of the preheater. Apparatus is described, consisting of a rotary kiln and a preheating chamber. F. SALT.

**Preventing evaporation from concrete during curing.** H. P. HAYDEN, Assr. to BARBER ASPHALT Co. (U.S.P. 1,684,671, 18.9.28. Appl., 12.10.25).—An adherent film, impervious to water, is applied to the concrete before curing. F. G. CLARKE.

**Treatment of artificial stone, cement, etc.** W. MAGUIRE (B.P. 307,132, 16.12.27).—A weathered or aged appearance is imparted to artificial stone, cement, etc. by applying a layer of soluble material to the surface during the moulding process. The soluble substance, e.g., magnesium sulphate, is subsequently dissolved out, leaving a pitted surface on the stone. The pitted surface may be coloured and then scraped or brushed to produce special effects. F. SALT.

**Impregnation of wood with substances toxic to animal and bacterial life and fungus growths.** C. B. LIPMAN and A. GORDON, Assrs. to UNIV. OF CALIFORNIA (U.S.P. 1,693,930, 4.12.28. Appl., 9.9.26).—Copper arsenate or other metal salt solution is injected (from a reservoir with a head of about 20 ft.) into a hole bored transversely three quarters through the trunk of the living tree. This is followed, preferably, by an injection of glucose solution to ensure reduction. Two to three days after the injection the tree is felled, and the wood is found to be uniformly impregnated. C. HOLLINS.

**Handling, mixing, and pouring concrete.** H. P. PARIS (B.P. 308,006, 9.1.28).

**Concrete and like mixers.** J. F. ROBB (B.P. 308,007, 9.1.28).

**[Making pressure-tight joints for] drying, impregnating, and similar treatment of timber.** C. GOODALL (B.P. 308,102, 24.4.28).

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

**Eutectic cast iron.** A. MITINSKY (Rev. Mét., 1929, 26, 84—87).—The properties of cast iron depend on the relative proportions and distribution of metal and graphite. The mechanical resistance to external stresses is a maximum when the graphite is distributed regularly in a non-laminated form. Cast iron with excellent mechanical properties can be obtained (1) by diminishing the total carbon content to less than 3%, the desired amount of graphitisation being produced by hammering or by raising the silicon content; (2) by decreasing the silicon content, graphitisation being obtained by pouring into a pre-heated mould. When the graphite is uniformly distributed and is non-laminated, the presence of ferrite increases the ductility, shock-resistance, and machinability. Solidification of the eutectic cast iron gives the most uniform distribution of all the elements and also leads to the formation of the best graphite, and, as its m.p. is a minimum, it is more easily heat-treated than are other cast irons. The pure eutectic iron-carbon contains 4.3% C, but this is decreased by the presence of silicon and phosphorus. It is possible to calculate approximately the carbon content of the eutectic of a commercial cast iron of known composition. M. E. NOTTAGE.

**Heat-treatment and volume changes of grey cast iron between 15° and 600°.** J. W. DONALDSON (Rev. Mét., 1929, 26, 78—84).—Four series of cast irons with varying content of one of the elements, carbon, manganese, phosphorus, or silicon, were used. The heat treatments were carried out at 450° and at 550° for periods of 5 days of 8 hrs. each, the specimens being allowed to cool overnight. As a consequence of the decomposition of the combined carbon, the tensile strength and the hardness were affected to an extent depending on the temperature, duration of heating, and composition of the cast iron, equilibrium being reached after 200 hrs. The stability of the combined carbon, and consequently the resistance to heating, diminished with increase in carbon content from 2.71 to 3.51%; increase of manganese content from 0.52 to 2.43%

and of phosphorus from 0.20 to 1.06% increased the resistance. The cast iron was stable when less than 1.10% Si was present, the stability increasing as the silicon content decreased to 0.65%; the critical content of silicon for the decomposition of combined carbon lies between 1.10 and 1.17%. In the heat treatments at 550° expansion during the first heating is due to the decomposition of the combined carbon; the penetration of air along the laminae and consequent oxidation in their neighbourhood produces a secondary expansion. When the total carbon content is low the expansion is small; increase in the manganese content is accompanied by an increase in the expansion, whilst increase in phosphorus decreases the expansion; 1.1% of silicon and less produces small expansion, which becomes more marked and is accompanied by oxidation of the iron when it increases from 1.2 to 1.5% and becomes very pronounced in cast irons containing 1.5–2.2%.

M. E. NOTTAGE.

**Influence of heat treatment below the A1 point on the properties of technical iron.** W. KÖSTER (Arch. Eisenhüttenw., 1928–9, 2, 503–522; Stahl u. Eisen, 1929, 49, 357–358).—The solid solubility of cementite in  $\alpha$ -iron increases with rising temperature between 600° and the A1 point, so that steels with less than 0.9% C age-harden after quenching from above 600°, the maximum effect being obtained with 0.09–0.1% C aged at room temperature. In this case the yield point increases by 60%, the tensile strength by 55%, and the hardness by 65%, whereas the elongation decreases by 50% and the reduction in area by 10%. The decomposition of the supersaturated solid solution begins to be appreciable in 1 hr. at 50°, and is completed at 200–250°; the precipitated carbide particles first separate as black points throughout the grains, but with rising temperature they migrate to the grain boundaries. The coercivity increases slightly with increase of quenching temperature below A1, but the remanence undergoes a marked decrease; on ageing or annealing the quenched steel the values for these properties return more or less rapidly to the normal. A. R. POWELL.

**Useful and deleterious action of gases in steel.** F. RAPATZ (Z. Metallk., 1929, 21, 89–92).—The brittleness produced in a steel by hardening at too high a temperature is due to the presence of oxygen; a thoroughly deoxidised steel shows no signs of brittleness even after quenching from a temperature well above the normal. Oxygen causes also the formation of white spots in the fracture of hardened steel. Nitrogen in small quantities has little effect on the properties of steel, but in conjunction with sufficient carbon it tends to increase the hardness of surfaces. The effect of gas inclusions on the crystal structure is briefly discussed and previous work on the subject reviewed.

A. R. POWELL.

**Determination of gases in metals, particularly of oxygen in steel.** W. HESSENBRUCH (Rev. Mét., 1929, 26, 93–114).—The influence of elements other than iron on the results obtained is reviewed. Metallic elements such as manganese and aluminium, the oxides of which have less tendency to dissociate than ferrous oxide, will reduce it and so retard the reduction by

carbon. At 1200° the formation of water vapour has not been proved; sulphides of carbon and hydrogen are formed in small quantities, but influence the result only when the sulphur content is greater than 0.05%. The formation of sulphur dioxide has not been detected, whilst ammonia, methane, and phosphine cannot exist at high temperatures and low pressures. By adding fluxes which lower the m.p. of iron, the extraction of the dissolved gases is rendered complete, but not the reduction of oxides at 1200°; hence an apparatus which can be used at a higher temperature is necessary. A high-frequency furnace suitable for the purpose is described. Owing to surface oxidation, the sample should be in the form of a single lump. The analysis can be made in 20–40 min., the total content of oxygen, hydrogen, and nitrogen being determined in one operation.

M. E. NOTTAGE.

**Recovery of austenitic steels.** A. MICHEL and P. BÉNAZET (Compt. rend., 1929, 188, 912–915).—Special steels which may be rendered austenitic by tempering at high temperatures fall into two classes, according to their behaviour on recovery:—(1) There is a sudden dilatation accompanied by the liberation of heat at a temperature which depends on the composition of the steel and the rate of heating and corresponds with the  $\gamma \rightarrow \alpha$  transformation. (2) Above a certain temperature there is a gradual negative anomaly in the dilatation, which is more accentuated when the rate of heating is rapid. It is shown that the two types of recovery are analogous, and that the difference is due only to the different rates of reaction, the passive resistance being too feeble in the first type to inhibit the  $\gamma \rightarrow \alpha$  transformation during the heating process.

J. GRANT.

**Microscopic composition and structure of basic slag after different heat treatments and their relation to the citric acid solubility.** H. SCHNEIDER-HÖHN (Stahl u. Eisen, 1929, 49, 345–352).—Microscopical examination of the structure of basic slag with and without addition of silica after various annealing and quenching treatments has shown that these do not affect the nature of the different constituents or their relative proportions; hence it appears that the constituents of basic slag are in equilibrium with one another at all temperatures. The following constituents have been identified: silicocarnotite ( $5\text{CaO} \cdot \text{P}_2\text{O}_5 \cdot \text{SiO}_2$ ), crystallised lime, magnesia, ferrous oxide, ferric oxide, and alumina, and compounds and solid solutions of these oxides. In slag to which no silica has been added hilgenstockite ( $4\text{CaO} \cdot \text{P}_2\text{O}_5$ ) is also found. The lower citric acid solubility of the phosphoric acid in the last-named type of slag is attributed to the depressing effect of soluble lime salts on the solubility of silicocarnotite. The rapid disintegration of quenched basic slags without added silica is attributed to hydration and carbonation of the lime crystals. The usual methods of determining free lime in basic slags are shown to be untrustworthy.

A. R. POWELL.

**Fusibility of mixtures of ferric oxide and lime.** L. JACQUÉ (Compt. rend., 1929, 188, 917–919).—Mixtures of lime (5–60%) and ferric oxide are readily fused by rapid heating below 1500°, the greatest fusi-



bility being at 1220° for 20–30% of lime. The ferric oxide is partly decomposed, particularly in a non-oxidising atmosphere, with the formation of magnetite.

J. GRANT.

[Determination of copper.] H. F. BRADLEY (Chemist-Analyst, 1928, 17, No. 4, 14).—Low's short iodide method is modified. The ore (0.5 g.) is moistened with water, a little potassium chlorate being added if sulphides are present, and decomposed with 5 c.c. of concentrated nitric acid and a few drops of hydrochloric acid. Digestion is continued for 6 min.; if brown fumes appear, a few drops of a mixture of hydrochloric acid and hydrogen peroxide are added. Then concentrated sulphuric acid (7 c.c.) is added, and the mixture boiled until it fumes freely. After cooling, 3 c.c. of water followed (cautiously) by 133 c.c. of concentrated ammonia solution are added. To this slightly acid solution a concentrated solution of ammonium acetate is added in excess (5 c.c.) of the amount necessary to produce a red colour. After cooling, 0.5 g. of sodium fluoride is added, and the liquid stirred until the red colour disappears; 3 g. of potassium iodide are added, and the titration is performed in the usual way.

#### CHEMICAL ABSTRACTS.

**Brass for screw-cutting.** P. DEJEAN (Rev. Mét., 1929, 26, 45–67).—This alloy is hot-rolled, annealed, and cold-drawn, with or without intermediate annealings. The method of manufacture fixes its composition within narrow limits. Hot-rolling of a brass containing more than 61.5–62% Cu is difficult, whilst cold-drawing of one containing less than 57.5–58% Cu may produce cracks either during the process or long afterwards; hence the copper content should be between 58.5 and 61%. The narrowness of the permissible zone of composition is due to the fact that the  $\alpha$ -constituent is malleable when cold but cracks when hot, whilst the  $\beta$ -constituent is plastic when hot but only slightly malleable when cold. The plasticity decreases with increase in copper content for temperatures above 500°, and this effect becomes more marked the higher is the temperature. All brasses undergo a transformation at about 475°; this change starts at a little above 300° and is probably connected with the  $\beta$ -constituent, which increases the plasticity above 480° and decreases it below this temperature. The increase in strength and elastic limit and the decrease in elongation with cold-hardening are the more rapid the greater is the copper content. The strength and the elastic limit decrease, whilst the elongation increases, with higher temperatures, the rate of decrease reaching a maximum between 300° and 400° and becoming very slight between 550° and 800°. The orientation due to rolling and drawing has only commenced to disappear at 600° for brass containing 61.4% Cu, whilst it has completely disappeared with 56.8% Cu; in the former case grain growth has not commenced at 700°, whilst at 850° it is very noticeable; in the latter case extensive grain growth takes place at 800°, and this produces a sudden decrease in the elongation when the annealing temperature is above 700°. The best annealing temperature is 500°, and it should not be raised above 700°. The addition of 1–2% Pb is necessary to prevent the alloy from machining badly.

M. E. NOTTAGE.

**Nickel-chromium [plating] process.** W. PFANHAUSER (Chem.-Ztg., 1929, 53, 207–208).—A chromium plating which will entirely protect iron, steel, or brass from corrosion can be obtained only by 4–6 hrs.' treatment in the bath. As this is quite uneconomical for technical work, the article is usually plated with nickel first, but, to obtain adherent coatings with no tendency to peeling, it is essential that the nickel layer be thick enough and relatively free from hydrogen, and that the surface to be plated be completely free from grease and oxide. With a nickel coating 0.02–0.025 mm. in thickness a sufficiently protective chromium coating is obtained by electrolysis for 10–15 min. in the usual plating solution.

A. R. POWELL.

**Age-hardening of standard silver.** M. HAAS and D. UNO (Z. Metallk., 1929, 21, 94–96).—Dilatometric and micrographic examination of standard silver after quenching from 720–800° and ageing above 200° has confirmed Norbury's work (B., 1928, 267). Decomposition of the supersaturated solid solution becomes visible in the microstructure at 180°, and at 360° agglomeration of the precipitated particles is rapid. The maximum hardness value is obtained when the grain boundaries are surrounded by numerous precipitated particles of copper; this first occurs at about 290°.

A. R. POWELL.

**Recovery of platinum from dunite rock at Onverwacht, Transvaal.** T. K. PRENTICE and R. MURDOCH (J. Chem., Met., Min. Soc. S. Afr., 1929, 29, 157–166).—The dunite ore of the Onverwacht mine contains platinum in association with chromite, hornblende, diopside, and hortonolite. By stage-crushing and concentration on Wilfley, curvilinear, and corduroy tables an extraction of 83% of the platinum metals in a concentrate weighing 1–2% of the ore and assaying about 30 oz./ton is readily obtained. Of this recovery about 65% is in the form of a product which may be dressed by hand-panning and acid treatment to obtain a concentrate assaying about 82% of platinum metals. The remainder is in a concentrate which is rich in chromite and metallic iron from the machinery; this product is agitated in barrels with zinc amalgam and an acid solution of copper sulphate whereby the platinum mineral is taken up by the amalgam leaving a residue of chromite etc. The discharge from the barrels passes over bateas, amalgamated plates, and curvilinear tables and the recovered amalgam is cleaned, pressed, and retorted. The residue in the retorts is crushed, cleaned with acid, panned to remove slimes, and cleaned from iron by magnetic separation. All the by-products from the cleaning and the concentrates from the curvilinear tables are returned to the barrel to be re-treated, the amalgam being worked up as before. More than 97% of the platinum in the original concentrate is recovered by this amalgamation process.

A. R. POWELL.

**Corrosion. XIV. Protection against corrosion of iron in steam boilers.** A. THIEL and H. LUCKMANN (Korrosion u. Metallschutz, 1928, 4, 169–177; Chem. Zentr., 1928, ii, 1610).—Experiments on the production of protective coatings of ferrous oxide with (a) water and dilute sodium hydroxide solution at 100° in nitrogen or

air, (b) water at 100° and air, (c) sodium chloride or sulphate solutions at 100° in absence of air, (d) sodium hydroxide and various salts at 200° are recorded.

A. A. ELDRIDGE.

**Method of testing opacity of protective [metal] coatings against corrosion.** J. COURNOT (Rev. Mét., 1929, 26, 76—77).—Filter paper impregnated with ferricyanide and an alkali chloride is soaked in water and placed on the cleaned surface. The pores are indicated by transparent spots which are blue in colour where the base metal is a ferrous alloy and brown where it consists of copper or its alloys. The paper is then washed with water to remove excess of ferricyanide. In the case of ferrous alloys which have been plated with chromium or nickel over copper, if the holes pass through both layers blue spots are formed, the alkali ferrocyanide masking the coloration due to the copper, whilst if they pass only through the copper, brown spots are formed. The method is not so good for deposits of zinc and cadmium, as the ferrocyanides of these metals mask to some extent the blue or brown colorations. The method can also be used for testing the opacity of thin sheets of tin or aluminium used for wrapping food products, the sheet being placed on a polished steel plate and the ferricyanide paper on top; the formation of blue spots indicates the presence of holes.

M. E. NOTTAGE.

**Electric annealing furnaces for metals.** M. TAMA (Z. Metallk., 1929, 21, 77—86).—An illustrated description of various types of electric muffles for annealing metal rods, tubes, and bands.

A. R. POWELL.

**Penetration of hydrogen into metal cathodes and its effect on the tensile properties of metals and their resistance to repeated stresses. Effect of non-electrolytic baths and nickel plating on these properties.** F. C. LEA (Proc. Roy. Soc., 1929, A 123, 171—185).—Static tensile tests, single-blow impact tests, and repeated-stress experiments in torsion and under direct stress were carried out on various metal cathodes in acid and alkaline baths, both when current was and was not flowing. The results indicate that nascent hydrogen liberated at a cathode penetrates the crystal boundaries of mild steel (0.14% C), nickel, and a rustless steel containing about 15% Cr and 10% Ni, but the penetration does not influence the tensile strength or the resistance to repeated stress. It does, however, diminish the elongation of specimens of mild steel very considerably, although it does not lower the resistance of mild steel specimens to the Izod impact test. The effect of polishing mild steel is to prevent, or modify the action of, hydrogen penetration between the crystals. Acid corrosion reduces the resistance to torsional stress, but a caustic soda solution, with or without the presence of hydrogen, protects the specimen and the fatigue range is raised. It is considered that the presence of hydrogen at the crystal boundaries affects the continuity of slip and causes a crack to occur as in quenched and tempered materials, and that in all such materials the failure is at the boundaries, whilst the failure of plastic materials occurs by cracking within the crystal. Some tests were made on nickel-plated specimens, and the results are compared with those for mild steel and drawn nickel bar. Under direct repeated

stress the effect of the deposited surface is to lower the fatigue range very considerably and to make the results erratic. A tool mark, having a depth equal to the thickness of the nickel deposit and sharp at the bottom, lowers the fatigue range in bending by about the same amount as the nickel plating, and it is concluded that the nickel is deposited under stress or acts as a discontinuity like the tool mark, or else that a crack is first produced in the deposited nickel and causes the lowering of the repeated-stress range.

L. L. BIRCHUMSHAW.

**Action of calcium carbonate and dolomite on zinc sulphate solutions.** CAMBI and others.—See VII.

#### PATENTS.

**Induction [melting] furnace.** E. F. RUSS (B.P. 305,388, 2.3.28).—In an induction furnace for melting copper, bronze, etc., the melting channel, opening at different heights into the melting chamber, ascends throughout its course and surrounds the upper half of the primary coil.

J. S. G. THOMAS.

**Reduction of iron ores or oxides.** J. W. HORNSEY (B.P. 306,561, 18.8.27).—The ore is fed into a rotary preheating cylindrical furnace in which it is heated by combustion of the gases from the reducing cylinder, then into the reducing cylinder where it is mixed with a solid fuel and reduced to iron sponge. Fuel and air are introduced into the discharge end of the reducing cylinder by means of a water-jacketed conveyor in such a way that the air does not come into contact with the material being reduced.

A. R. POWELL.

**Roasting and reduction of metallic [iron] ores.** F. L. DUFFIELD (B.P. 281,338, 28.11.27. U.S., 29.11.26).—The roasting and reduction of iron ores is carried out simultaneously in two rotating furnaces each of which is alternately used as the roasting and as the reducing furnace. Reduction is effected in one furnace by means of a current of reducing gas, and the waste gases from this furnace are burnt with air in the roasting furnace to supply the necessary heat.

A. R. POWELL.

**Hardening iron and steel articles.** DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 291,423, 18.1.27. Ger., 4.6.27).—Iron or steel articles are carburised in molten cyanide mixtures without addition of finely-divided carbonaceous material. The cyanide mixture is heated to 920° or higher, avoiding frothing, and the surface of the melt may be protected by a layer of graphite.

C. A. KING.

**Production of castings of cast iron mixed with steel.** J. ROBINSON (B.P. 296,080, 16.7.28. India, 25.8.27).—A molten mixture of cast iron and steel is mixed without loss of heat by immersing an aluminothermic mixture in the metal contained in a ladle or other container.

C. A. KING.

**Manufacture of steel.** H. J. VAN ROYEN (B.P. 285,814, 16.2.28. Ger., 21.2.27).—Steel is rendered non-susceptible to cold deformation and ageing by previously deforming it as extensively as possible as soon as the metal has cooled below the Ar1 point, after which it is normalised.

C. A. KING.

**Continuous [steel] annealing and cleaning process.** H. M. NAUGLE and A. J. TOWNSEND, Assrs. to

**COLUMBIA STEEL CO.** (U.S.P. 1,704,015, 5.3.29. Appl., 1.7.25).—Sheet steel is heated quickly and uniformly to annealing temperature, which is maintained only to obtain correct microstructure, at which point the steel is cooled rapidly. C. A. KING.

**Reduction process and apparatus.** T. F. BAILY (U.S.P. 1,704,029, 5.3.29. Appl., 6.3.26).—The charge is fed to a shaft furnace by means of an aerating pump, and gases from the reaction chamber are conveyed to a combustion chamber surrounding the shaft. C. A. KING.

**Treatment of pyrites.** S. I. LEVY and G. W. GRAY (B.P. 306,691, 10.2.28).—Pyrites or pyrrhotite is heated in chlorine so as to volatilise sulphur and form ferrous chloride and chlorides of any non-ferrous metals present. The ferrous chloride formed is then heated in pure dry air whereby it is decomposed into ferric oxide and ferric chloride; the latter volatilises and is passed over further quantities of pyrites to effect chlorination as before. Both processes may be carried out in the same furnace, partial chlorination being effected by means of chlorine at 400–600° followed by oxidation with dry air at 600–1100°. The low-temperature process results in the retention of zinc, copper, and other chlorides by the ferric oxide residue, whereas in the high-temperature operation these chlorides are volatilised and may be separated from the sulphur by fractional condensation. A. R. POWELL.

**Roasting of zinc sulphide ores.** S. ROBSON (B.P. 306,569, 24.10.27).—Zinc sulphide ore or concentrate is mixed with 20–25% of its weight of crushed sinter from a previous process and the mixture is roasted in an apparatus so designed that a current of air is drawn through the heated mass in such a way that only sufficient air is supplied to effect the roasting and a porous sintered mass of oxide is produced together with a gas having a high content of sulphur dioxide. A. R. POWELL.

**Reduction of ores, oxides, etc. [of volatile metals, e.g., zinc].** H. E. COLEY (B.P. 306,425, 14.9.27).—In the process in which volatile metals are reduced from their ores by spraying liquid hydrocarbons on to the heated mass, the vapours issuing from the furnace are passed upwards through a water-jacketed tower down which a spray of water from a series of fine jets is allowed to fall. The sludge from the tower is filter-pressed and re-distilled with carbonaceous material to recover the metal. A. R. POWELL.

**Treatment of ores etc. containing platinum.** S. C. SMITH (B.P. 306,566, 21.10.27).—Ores containing platinum metals in association with copper, nickel, and iron sulphides are smelted to obtain a matte which is ground finely and roasted, with or without previous bessemerisation. The roasted product is leached with sulphuric acid to remove the greater part of the copper and the residue is washed, dried, and reduced to metal by heating it with carbonaceous material or in a stream of reducing gas. The metal sponge so obtained is digested with sulphuric acid, which dissolves nickel and iron. The residue from this treatment may be roasted again and leached to remove the remaining copper and the reduction process repeated. The final product con-

tains all the platinum metals in a form ready for refining by the ordinary methods. A. R. POWELL.

**Metallic [nickel-tungsten] alloy.** H. L. COLES and J. G. DONALDSON, Assrs. to GUARDIAN METALS CO. (U.S.P. 1,702,765, 19.2.29. Appl., 13.6.22).—The alloy contains 14% Ni, 2–5% C, and not more than 86% W. A. R. POWELL.

**Composite metal plate.** H. L. COLES and J. G. DONALDSON, Assrs. to GUARDIAN METALS CO. (U.S.P. 1,702,766, 19.2.29. Appl., 4.2.26).—The plate comprises a core of an alloy containing 10% Ni, 14% Cu, and more than 70% W inside a casing of a metal having a high heat conductivity, the whole being heat-treated to effect superficial alloying between the outside of the core and the inside of the casing. A. R. POWELL.

**Manufacture of steel products.** R. A. HADFIELD (U.S.P. 1,704,385, 5.3.29. Appl., 7.7.26. U.K., 29.4.26).—See B.P. 273,855; B., 1927, 658.

**Sintering zinc ores.** H. J. STEHLI (B.P. 307,595, 7.2.28).—See U.S.P. 1,661,813; B., 1928, 337.

**Conveyer furnaces [for heat-treating metals etc.].** BRAYSHAW FURNACES & TOOLS, LTD., and S. N. BRAYSHAW (B.P. 307,963, 15.12.27).

**Furnaces [for heating tubes etc.].** "ILVA" ALTI FORNI E ACCIAIERIE, and A. AURELI (B.P. 307,853, 12.12.27).

**Electrolytically depositing metal on tubes.** J. STONE & Co., C. J. LYTH, and F. J. PIKE (B.P. 307,726, 12.12.27).

**Metal-glass union** (B.P. 293,292).—See VIII. **Attaching rubber etc. to metal** (B.P. 307,180 and 307,628).—See XIV.

## XI.—ELECTROTECHNICS.

**Electrical conductivity of thin oil films. I. General nature of the phenomenon.** H. E. WATSON and A. S. MENON (Proc. Roy. Soc., 1929, A, 123, 185–202).—Static friction measurements have been made by means of an apparatus somewhat similar to that used by Hardy (cf. *ibid.*, 1925, A, 108, 1). With "wiped films" the result obtained by Hardy, that the coefficient of friction  $\mu$  decreases considerably with increase of pressure, was confirmed. Microscopical examination and measurements of the thickness of these non-conducting films suggested that they consist of a number of small oil globules between two adsorbed films of air or water vapour on the metal surfaces. There is no relation between the value of  $\mu$  and the thickness of the film. Air films have a high insulation resistance, and when they break down the resistance usually becomes very low. "Flooded films" of paraffin oil will withstand a high potential gradient (over 100,000 volts/cm.) as long as their thickness is greater than about 15  $\mu$ . Thinner films (below 11  $\mu$ ) break down frequently on application of 2–40 volts and become partially conducting. The breakdown thickness does not vary much with the voltage. The film becomes conducting in two stages: in the first the resistance is of the order of 1 megohm, in the second it is very low. With both films the *E.M.F.* increases with the current for small values of the latter, but Ohm's law is not obeyed; with

larger currents the *E.M.F.* becomes nearly constant, and on still further increase it decreases.

L. L. BIRCUMSHAW.

**Electric annealing furnaces.** TAMA. **Nickel-chromium plating.** PFANHAUSER.—See X.

PATENTS.

[Terminals for] **electric heating devices.** BRIT. THOMSON-HOUSTON CO., LTD., H. W. H. WARREN, and R. G. GREEN (B.P. 305,160, 11.9.28).—A paste of finely powdered graphite and, if desired, carborundum is pressed round the ends of the heating unit and then treated so as to harden it; *e.g.*, the paste made up with a bonding agent such as acacia gum is pressed round the unit and heated at 2000° while embedded in coke and sand.

J. S. G. THOMAS.

**Electrolysis in pressure-resisting vessels.** W. VOGT (G.P. 453,275, 13.3.25).—Water or other electrolyte is electrolysed under such a pressure that one of the gases produced is denser than the surrounding liquid; *e.g.*, oxygen at 100° and 1100 atm. is denser than the electrolyte. The electrodes employed consist of conical shells having points on the upper surfaces and joined together with vertical sheet-metal strips.

J. S. G. THOMAS.

[Bi-polar] **electrolytic cell.** R. EDGEWORTH-JOHNSTONE (B.P. 307,093, 4.11.27).—Closely-positioned electrodes in the form of sheets or plates, normally insoluble and upon which metals are electrolytically deposited, are held together in pack formation and provided with perforations for passage of electrolyte and escape of gases.

J. S. G. THOMAS.

**Electrolytic cell, especially for electrolysis of alkali chlorides.** SIEMENS & HALSKE A.-G. (G.P. 452,922, 22.10.25).—The electrolyte vessel is closed by a glass cover which absorbs chemically active radiation, and current is supplied by conductors passing through the cover and reinforced immediately below the cover.

J. S. G. THOMAS.

**Device for controlling electrolytic operations.** J. PORZEL (U.S.P. 1,700,178, 29.1.29. Appl., 1.9.23).—The sides and ends of a frame of insulating material extend beyond the faces of the anode or cathode, which is enclosed and supported within the frame.

J. S. G. THOMAS.

**Colloidal diaphragms for electrolytic cells.** G. F. JAUBERT (B.P. 281,674, 30.11.27. Fr., 1.12.26).—A diaphragm, permeable to electrolyte but impermeable to gaseous products of electrolysis, is prepared by depositing a hydrogel of a colloidal organic or inorganic substance upon a porous diaphragm, *e.g.*, wire gauze. Thus the gauze may be immersed successively in aqueous solutions of magnesium sulphate and sodium silicate. Suitable hydrogels comprise magnesium or iron silicate, kaolins, clays, magnesium or aluminium phosphate, graphite, india-rubber, etc.

J. S. G. THOMAS.

[Electrolyte for Leclanché type] **galvanic cell.** MANNESMANN LICHT A.-G. (F.P. 626,678, 23.12.26. Ger., 13.4.26).—Potash end-liquor either alone or mixed with other salt solutions is used, and the efficiency of the electrolyte is increased, if desired, by addition of mercury salts.

J. S. G. THOMAS.

**Electrolyte for accumulators.** W. M. JOWETT (Austral. P. 1755, 8.5.26).—Magnesium, aluminium, potassium, and ammonium sulphates and glycerin are added to sulphuric acid of *d* 1.27. J. S. G. THOMAS.

**Negative electrode for zinc electric accumulators.** A. POUCHAIN (B.P. 303,823, 10.5.28. Ger., 10.1.28).—The surface of a support of a metal which is a better conductor than zinc is covered with zinc only where zinc would be deposited during charging of the accumulator, the rest of the surface being covered with insulating material, *e.g.*, india-rubber or cellulose. The electrode is then coated with a solution of gum and sodium silicate.

J. S. G. THOMAS.

**Alkaline [electric] accumulator.** F. JIRSA and B. KRAUPNER (G.P. 453,129, 18.6.25).—The accumulator comprises an iron electrode and an anode composed of the higher oxides of silver, produced by immersing a silver electrode in an alkaline electrolyte containing telluric hydroxide or its derivatives.

J. S. G. THOMAS.

**Manufacture of photo-electric cells.** GEN. ELECTRIC CO., LTD., and N. R. CAMPBELL (B.P. 306,996, 1.12.27).—A photo-electric cell having a greatly increased sensitivity to red light comprises a cathode composed of a surface, *e.g.*, a silvered portion of the wall of the bulb, covered with a very thin invisible layer of photo-sensitive material, *e.g.*, potassium, which has been sensitised after its formation, by an electric discharge in hydrogen. Preferably the thin layer is formed by heating a thick visible layer.

J. S. G. THOMAS.

**Electron emitter.** E. E. SCHUMACHER, Assr. to WESTERN ELECTRIC CO. (U.S.P. 1,700,454, 29.1.29. Appl., 8.7.24).—A metallic sleeve fitted to a core of refractory metal is coated with an alkaline-earth oxide, the metal of the sleeve being such that its oxide, when heated in air, reacts with the alkaline-earth oxide to form compounds which, when heated *in vacuo*, decompose to form alkaline-earth oxide.

J. S. G. THOMAS.

**Production of moulded articles suitable as electric insulating material.** SCHLEFFERWERKE AUSDauer A.-G. (B.P. 286,732, 9.3.28. Ger., 10.3.27).—Filling materials are cold-pressed at very high pressures with about one sixth of their weight of a viscous artificial resin obtained as described in B.P. 286,731 (B., 1929, 294). Subsequent hardening is effected by heating at about 80° between porous stone slabs.

S. S. WOOLF.

[Impregnating paper] **insulating material for electrical condensers.** J. E. G. LAHOUSSE (F.P. 629,408, 29.4.26).—Paper is impregnated with the isomerisation products of frankincense or colophony, *e.g.*, abietic acid or pimaric acid. The m.p. is reduced by using a mixture of abietic acid with its sodium salt, wax, or paraffin.

J. S. G. THOMAS.

**Galvanic cell.** A. SCHMID (B.P. 281,698, 1.12.27. Ger., 2.12.26).

**Lead electrode for electric accumulators.** A. POUCHAIN (B.P. 303,824, 10.5.28. Ger., 10.1.28).

**Pasting battery plates.** F. B. DEHN. From VESTA BATTERY CORP. (B.P. 307,618, 27.2.28).

**Filaments for electric incandescence lamps.**

FALK STADELMANN & Co., Assees, of NEUE GLÜHLAMPEN GES.M.B.H. (B.P. 301,014, 24.7.28. Ger., 23.11.27. Addn. to B.P. 297,052).

**X-Ray tubes [with metallic envelope].** S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 307,377, 5.12.27).

**Induction furnace (B.P. 305,388).**—See X. **Deposits of organic substances for dispersion (B.P. 307,585).**—See XIV.

## XII.—FATS; OILS; WAXES.

**Extraction of orujo [olive press-residue] with carbon disulphide.** J. BOHLE (Chem.-Ztg., 1929, 53, 165—167, 187—189).—The preparation of orujo before extraction is described. After grinding and sifting (10 mm. mesh) the orujo, which contains on the average 10—12% of oil and 25% of moisture, should be dried to a water content not exceeding 16%; careful operation with rotating drums heated by direct fire is satisfactory, but investigation is suggested of methods of drying by exhaust steam under vacuum; drying by hot air involves serious mechanical difficulties, and steam-drying (without vacuum) is too expensive. The employment of trichloroethylene as solvent is discussed; the oil obtained is not of such good quality as sulphur olive oil, being poor in colour and not amenable to bleaching. Such oil cannot be used for the production of white soap, contains a greater proportion of non-oleaginous material (colouring matter, waxes, etc.), and solidifies in conditions under which sulphur oil remains liquid. Attention is drawn to the considerable fire risks attending the use of carbon disulphide in the large extractors (capacity 17,000—18,000 litres) in current use, especially when discharging spent pulp; the risk of explosion is increased when very wet orujo is used, as balling-up may occur, causing the solvent to be retained in pockets after the steaming-off. The usual type of plant consists of extractors coupled in pairs; a description is given of an improved lay-out of a battery of extractors designed to give a maximum throughput with minimum risk of explosion. E. LEWKOWITSCH.

**Detection of coconut and palm-kernel oils by testing for lauric acid.** J. GROSSFELD and A. MIERMEISTER (Z. Unters. Lebensm., 1928, 56, 423—437).—The method of identifying lauric acid in alcoholic liquors (B., 1929, 145) has been applied to the examination of fats for coconut and palm-kernel oils as follows:—(A) 1—100 mg. of the fat are saponified with 2.5 c.c. of 0.5*N*-alcoholic potash solution and the alcohol is removed by evaporation. The residue is dissolved in 2 c.c. of water mixed with 2 c.c. of glycerin solution (300 g. per litre), heated for 5 min. on the boiling water-bath, 2.0 c.c. of magnesium sulphate solution (150 g. of the crystallised salt per litre) are added, and the hot solution is filtered clear. A cloudiness immediately on cooling or a white flocculent precipitate after some time indicates the presence of 10% or more of lauric acid (20% of coconut oil) in the fat. (B) If test A is negative, 1.0 g. of fat is saponified by heating with 0.4 c.c. of 50% aqueous potash solution and 2 c.c. of glycerin; 200 c.c. of water and 50 c.c. of glycerin solution (300 g. per litre) are added to the soap solution, which is then

heated to boiling and 10 c.c. of magnesium sulphate solution as above are added. It is heated for 10 sec. more, filtered clear, and cooled. A flocculent precipitate indicates 2.5% or more of lauric acid. (c) If test B is negative the filtrate is acidified with 5 c.c. of dilute hydrochloric acid and shaken with 60 c.c. of ether. The ether extract is evaporated to dryness, the residue dissolved in 2.5 c.c. of alcoholic 0.5*N*-potash solution and re-tested as in test A. A flocculent precipitate indicates 0.5% or more of lauric acid (1% or more of coconut oil). Myristic acid and higher fatty acids give negative results. Decoic acid gives a negative result when less than 4 mg. are present, and nonoic acid when less than 30 mg. are present. Lauric acid was found in small quantities in butter fat, hardened arachis oil, hardened and unhardened cotton-seed oil. It was not found in cacao butter, soya-bean oil, or hardened whale oil. Of rancid samples two cotton-seed oils and an oleostearin contained no lauric acid, but one of hardened arachis oil and another of hardened cotton-seed oil gave positive tests. A self-extracted arachis oil gave a negative result, but after oxidation of the fatty acids with potassium permanganate a faintly positive test was obtained. A quantitative separation of lauric acid from the higher fatty acids by distillation and precipitation of the latter by magnesium sulphate was not found possible. W. J. BOYD.

**Oil of [seeds of] *Pistacia lentiscus*.** F. L. VODRET (Annali Chim. Appl., 1929, 19, 76—84).—This optically inactive oil, obtained in 12.4% (in some years 14%) yield from seeds of plants grown in Sardinia, has:  $d^{20}_D$  0.9182, sp. viscosity at 22° 10.65,  $n^{22}_D$  1.4671, butyrefractometer index 61.8, solidification point —6°, Tortelli thermo-value 42.0, solubility in alcohol at 95° 5.5:100, deep yellow fluorescence in Wood's light, acid value 28.05, saponif. value 209, acetyl acid value 21.09, acetyl saponif. value 228.6, Reichert-Meissl value 0.96, Hehner value 91.9, iodine value 81.57, absolute iodine value 100.56. The percentage composition is approx.: palmitic acid 24.82, stearic acid 11.63, oleic acid 47.62, linoleic acid 6.25, glycerol 9.53, unsaponifiable substances (phytosterol, resins, etc.) 0.96, linolic and isolinolic acids traces. From the phytosterol an acetate, m.p. 119—121°, was prepared. Tannin is detectable and, under Livache's conditions, 100 pts. of the oil absorb 0.15 pt. of oxygen in 48 hrs. A number of colour reactions of the oil are given, and the changes in characters produced by boiling are described (cf. Sernagiotto and Vita, B., 1915, 1061). T. H. POPE.

**Sulphonated oils. I. Preparation of pure sodium ricinoleosulphate and its isolation from commercial sulphonated oils.** K. NISHIZAWA and K. WINOKUTI (Chem. Umschau, 1929, 36, 79—81).—Ricinoleosulphuric acid was prepared by treating pure ricinoleic acid dissolved in anhydrous ether with chlorosulphonic acid (cf. Grün and Woldenberg, A., 1909, i, 284); the hydrochloric acid and ether may be removed in the vacuum desiccator, but better yields of a purer product were obtained by salting out the aqueous solution of the ester with hydrochloric acid and extracting impurities with ether. The ester was then neutralised with alcoholic caustic soda and the sodium salt obtained

in a state of purity after repeated crystallisation from alcohol. The composition agrees with the formula  $\text{SO}_4\text{Na}\cdot\text{C}_{17}\text{H}_{32}\cdot\text{CO}_2\text{Na}\cdot\text{H}_2\text{O}$ . It is readily soluble in water, and almost insoluble in organic solvents; it is sparingly soluble in cold 95% alcohol, but the solubility increases rapidly with decreasing concentration or rising temperature. By similar methods ricinoleo-sulphuric acid and its sodium salt were isolated from three commercial sulphonated oils.

E. LEWKOWITSCH.

**Determination of organically combined sulphuric acid in sulphonated oils.** C. RIESS (Chem. Umschau, 1929, 36, 77—79, and Collegium, 1928, 657—661).—The organic sulphate in a sulphonated oleic acid and in a commercial Turkey-red oil was determined by Herbig's gravimetric method and by the more convenient volumetric method (American official method, except that 5—6 g. of oil were used and refluxed for  $\frac{1}{2}$  hr.); the results from both methods agreed within practical limits, whether hydrochloric or sulphuric acid was used for the hydrolysis (volumetric). Traces of sulphate could be found in the oil separated from the Turkey-red oil, after boiling with acid, by extraction with ether and washing with dilute brine; the amount retained was slightly greater when sulphuric acid was employed for the hydrolysis (cf. B., 1928, 646).

E. LEWKOWITSCH.

**Determination of iodine values. II. Action of iodine chloride on fatty acids with conjugated double linkings.** E. T. GELBER and J. BÖESEKEN (Rec. trav. chim., 1929, 48, 377—385).—The addition of iodine chloride (Wijs' solution) to linoleic acid proceeds in two stages. The first addition is rapid, whereas the second is slow and only tends to go to completion after a long time and with a large excess of iodine chloride (cf. B., 1927, 427). Free iodine is produced at the beginning of the reaction. Ingle's theory that the iodine results from the hydrolysis of the iodochloride is probably incorrect, since free iodine is obtained even when the determination is carried out in water-free media. Under these conditions saturation of both double linkings occurs. Determination of the iodine value of linoleic acid by Marshall's solution (J.S.C.I., 1900, 19, 231) gives correct values. No free mineral acid is produced, and any possible substitution is thus avoided. When elæostearic acid is acted on by Wijs' solution two of the three double linkings are saturated rapidly, the third only after several days, a large excess of iodine chloride being essential. The production of free iodine is explained by assuming that the first stage of the addition of iodine chloride to a conjugated doubly unsaturated higher fatty acid is a 1:4-addition of chlorine. This is a rapid reaction, and it is followed by the saturation of the remaining double linking with iodine chloride. When a carboxyl group is adjacent to a double linking as in sorbic acid, no free iodine is produced. Elæostearic acid combines with four atoms of chlorine to give a tetrachloro-oleic acid (first stage), which is then saturated with iodine chloride.

H. BURTON.

**Detergent action of soap.** F. H. RHODES and S. W. BRAINARD (Ind. Eng. Chem., 1929, 21, 60—68).—For the determination of the detergent power, a piece of

cotton cloth is soiled by immersion in a mixture of lamp-black, lubricating oil, and tallow in carbon tetrachloride, freed from the excess of liquid, dried at 80° for 1 hr., and allowed to age for 11 hrs. The cloth is then sewn up in the form of a bag which is filled with glass beads, and is placed in a rubber-covered cage inside a glass cylinder which holds the soap solution; after heating the latter to the desired temperature, washing is carried out by rotating the cylinder. The brightness of the soiled cloth is measured before and after washing by means of an integrating photometer whereby indices of detergent power for the various soap solutions are calculated. For short periods of washing (30 min.) the detergent effect of distilled water is about one half that of a 0.25% solution of soap, but on increasing the period to 5 hrs. the cleansing action of the former increases whilst that of the latter diminishes, probably owing to the increased degree of dispersion of the dirt. The detergent effect for each washing reaches a maximum after a certain time, viz., 7½ min. with the conditions obtaining in these tests. Increasing the concentration of soap above 0.25% produces little change in the detergent effect. Although solutions containing 0.05 and 0.1% of soap remove about the same amount of dirt in 5 washings as the more concentrated solutions, the rate of removal in the first few washings is slightly lower; 0.01% soap solutions are distinctly less efficient. Soap solutions 24 hrs. old show about the same ultimate efficiency as those freshly prepared, although less dirt is removed in the early stages. Variations in temperature between 20° and 60° have little effect on the detergent power.

F. R. ENNOS.

**Viscosity of soap solutions.** B. WALKER (Allgem. Oel- Fett- Ztg., 1928, 25, 448—450; Chem. Zentr., 1928, ii, 1633).—The viscosity of 0.3% (of fatty acid) soap solutions at 20° was 52—56.2 sec. (water at 20°, 50.8 sec.). Generally, potassium soaps gave higher values than sodium soaps, tallow and lard excepted. Values for 30°, 40°, and 50° are recorded.

A. A. ELDRIDGE.

**Determination of iodine value [of fats].** J. J. A. WIJS (Z. Unters. Lebensm., 1928, 56, 488—490).—See B., 1929, 179.

**Applications of ozone.** VOSMAER.—See VI. **Determination of fat in malted milk.** BALLARD.—**Glycerides in butter.** DE'CONNO and SCOPINARO.—See XIX.

#### PATENTS.

**Detergent, cleansing, and polishing compositions.** BRITISH DYESTUFFS CORP., LTD., J. BADDILEY, and E. CHAPMAN (B.P. 307,141, 23.12.27).—Pastes or aqueous solutions of complex sulphonic acids or their salts ("wetting-out" agents, e.g., sulphonated mineral oils) with the addition of inorganic acid detergents (e.g., nitre-cake) are claimed. E. LEWKOWITSCH.

**Oil presses.** J. JAKOBSEN (B.P. 307,770, 12.12.27).

**Manufacture of dry, non-caking, readily-soluble soap in the form of threads.** A. WELTER (B.P. 307,549, 14.11.27).

**Butter etc.** (B.P. 307,167).—See XIX.

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

**Effect of light on paints containing lead.** G. ZEIDLER and W. TOELDT (Farben-Ztg., 1929, 34, 1547—1549).—The nature and extent of colour changes occurring when iron plates coated with a range of paints containing lead pigments were suspended in an atmosphere of hydrogen sulphide for 15 min. are detailed. The panels were next exposed to each of three sources of artificial light and the relative bleaching action was observed. The results indicate the unreliability of fading and weathering tests carried out with the mercury-vapour lamp as source of artificial light. Although rapid in action, the latter does not reproduce the effect of the sun's rays, and more significant results are to be obtained with the slower-working "Nitalamp."

S. S. WOOLF.

**New raw materials for the [cellulose] lacquer industry.** A. NOLL (Farben-Ztg., 1929, 34, 1486—1490, 1549—1552).—The properties and uses of a number of modern solvents, diluents, plasticisers, and synthetic resins are detailed.

S. S. WOOLF.

**"Gum-running."** E. PYHÄLÄ (Farben-Ztg., 1929, 34, 1552—1553).—A new process for "gum-running" is described wherein hard copal resins are melted in the presence of white oil (liquid paraffin) which is subsequently distilled off and recovered. The gum kettle, which is heated by means of a bath of molten zinc, is closed by a cover fitted with suitable outlet and condensing apparatus etc. The gum swells in the white oil, which functions as a protective agent, taking up and removing from the system oily ingredients liberated from the melting gum. The losses in gum running are reduced and a much lighter product is obtained. It is considered that uniform varnishes can be prepared by mixing, in the cold, solutions at varnish consistency of "tempered" copal, ester gum, and prepared tung and linseed oils, in suitable varnish solvents.

S. S. WOOLF.

**Finnish turpentine. VII. isodiprene, a new terpene of the sylvestrene group.** O. ASCHAN (Bidr. K. Finlands Natur. Folk, 1926, H. 80, No. 6, 18 pp.).—Of Finnish turpentine, about 60% has b.p. 160—170°. The fraction of b.p. 163—167° contains an unidentified terpene, pinonene. The fraction of b.p. 167—170° contains isodiprene,  $d_4^{20}$  0.8561,  $n_D^{20}$  1.47536,  $R_{44-84}$  (monohydrochloride,  $d_4^{20}$  6.9775,  $n_D^{20}$  1.48175,  $R_{50-57}$ ). Elimination of hydrogen chloride affords sylvestrene. When distilled with acetic anhydride and concentrated sulphuric acid the terpene gives a characteristic reddish-violet colour. Terpenes of the sylvestrene group are classified as follows:—(1) those which give an intense blue colour with acetic anhydride and sulphuric acid, (2) *m*-menthadienes which give a violet colour, and take up only 1 mol. of hydrochloric acid without difficulty, (3) dicyclic terpenes containing the same double ring as carone. CHEMICAL ABSTRACTS.

**Ash content of podophyllum resin.** L. D. HAVENHILL (J. Amer. Pharm. Assoc., 1929, 18, 129—130).—Eight commercial samples of the resin gave 0.18—0.89% of ash and 5 others gave 0.65—0.89%; the comparative densities of the samples ranged from

0.6 to 1.8. Experiments with different precipitants indicated that alum water offers no advantage over dilute hydrochloric acid, and that yellow resins are produced by alum water. The author is of the opinion that the maximum ash content should be fixed at 0.5%, and the raising of the ash limit to 1.5% in the U.S.P. VIII is not justifiable.

E. H. SHARPLES.

**Disposal of wastes.** MOHLMAN and BECK.—See XXIII.

## PATENTS.

**Printing ink.** A. R. TRIST (B.P. 307,535, 9.12.27 and 9.10.28).—A printing ink for use with printing plates having mercurised non-printing areas consists of an intimate mixture of partly polymerised linseed oil containing about 7½% of fatty acids, pigments, and mercury, with or without the addition of an aqueous dispersing agent for the oil, e.g., an aqueous solution containing 2½—5% of borax and 5—10% of shellac.

S. S. WOOLF.

**Manufacture of pigments and pigmented products.** E. C. DE STUBNER (B.P. 277,949, 30.8.27, U.S., 23.9.26).—A pigment is precipitated in an aqueous medium and the precipitate, after preliminary washing, is dehydrated by washing with a non-aqueous water-miscible liquid compatible with the product to be pigmented, until the water has become replaced by this liquid. For application to lacquers the pigment can be precipitated on a soluble cellulose derivative of fibrous cellular structure, e.g., cellulose nitrate, and 86—95% ethyl alcohol used as dehydrating agent. For application to paints, printers' inks, etc. the precipitated pigment is dehydrated with acetone, pyridine, etc.

S. S. WOOLF.

**Manufacture of coloured or pigmented products having a cellulosic base.** E. C. DE STUBNER (B.P. 277,989, 22.9.27, U.S., 7.7.27, and 307,516, 30.8.27. Cf. preceding abstract).—Pigmented soluble cellulose derivatives are prepared by precipitating a preformed pigment, e.g., ultramarine blue, zinc sulphide, etc., from a colloidal dispersion on to a soluble cellulose derivative. In a preferred process the pigment is electrically of the same sign as the cellulose derivative to be pigmented, and a substance of opposite sign is first deposited in the latter, thus promoting deposition of the pigment. A pigmented soluble cellulose derivative suitable for use in lacquers, printing inks, etc. is obtained by precipitating a pigment, e.g., lead chromate, or depositing a metal, e.g., copper, silver, antimony, gold, on a cellulose derivative of cellular structure.

S. S. WOOLF.

**Lacquers or varnishes.** BRIT. THOMSON-HOUSTON Co., LTD., H. W. H. WARREN, and R. NEWBOUND (B.P. 306,914, 31.8.27).—A "glyptal" resin is plasticised by the addition of oleic acid, butyl phthalate, tolyl phosphate, etc. and dissolved in a mixture of solvents with graded b.p., with or without the addition of colouring matter or pigment. After application, the lacquer so obtained is baked for ½ hr. at 100° and subsequently for ¾ hr. at 200°.

S. S. WOOLF.

**Nitrocellulose products [lacquers etc.].** W. J. JENKINS, and IMPERIAL CHEMICAL INDUSTRIES, LTD. (B.P.

307,528, 10.11.27).—The use of derivatives of  $\beta$ -hydroxypropionic acid in nitrocellulose lacquers is claimed.

S. S. WOOLF.

**Nitrocellulose solutions and coating compositions.** W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 307,085, 26.8.27 and 26.6.28).—Monoalkyl ( $C_1$ — $C_5$ ) ethers of butylene glycols, especially of isobutylene glycol, are used as solvents for nitrocellulose, particularly for coating lacquers and enamels.

C. HOLLINS.

**Manufacture of synthetic resins and of varnishes therefrom.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 306,501, 22.11.27).—Crude solvent naphtha free from bases is treated with ferric chloride in the presence of about 10% of phenolic substances. Iron is removed by dilution with benzene, xylene, etc., and after removal of the precipitate the solution is treated with quicklime and fuller's earth and filtered. On evaporation of the solvent, preferably *in vacuo*, pale resins are obtained which are soluble in drying and stand oils, and hence suitable for use in varnishes.

S. S. WOOLF.

**Manufacture of coloured varnishes.** SOC. CHEM. IND. IN BASLE (B.P. 285,058, 7.2.28. Switz., 10.2.27).—Nitrocellulose or phenol-aldehyde varnishes are coloured with non-sulphonated monoazo dyes of the type: nitrated or halogenated arylamine  $\rightarrow$  arylamine. Examples are: *p*-nitroaniline  $\rightarrow$  cresidine (scarlet), aniline (orange), *m*-aminoacetanilide (yellow), diphenylamine; 2:5-dichloroaniline  $\rightarrow$  aniline (yellow), *m*-toluidine (yellow); *m*-nitroaniline  $\rightarrow$  aniline (yellow); 4-nitro-*o*-anisidine  $\rightarrow$  dimethylaniline (yellow). The shades are fast to light.

C. HOLLINS.

**Cellulose nitrate solution.** M. B. HOPKINS and H. E. BUC, Assis. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,694,097, 4.12.28. Appl., 3.4.22).—Nitrated non-benzenoid hydrocarbons (petrol,  $C_6$ — $C_{12}$ ) are used with alcohols and benzene to dissolve cellulose nitrate.

C. HOLLINS.

**Impregnating solution.** L. V. REDMAN and H. C. CHEETHAM, Assis. to BAKELITE CORP. (U.S.P. 1,693,939, 4.12.28. Appl., 24.4.23. Renewed 23.2.28).—Phenol-aldehyde resins for impregnating purposes are dissolved in furfuraldehyde together with hexamethylenetetramine as hardening agent; the ammonia evolved reacts with the furfuraldehyde.

C. HOLLINS.

**[Spray]-lacquering process [to form drops].** ELEKTRIZITÄTSWERK LONZA (B.P. 297,711, 12.1.28. Switz., 25.9.27).

**Condensation products of dimethylolurea etc.** (B.P. 306,875).—See III. **Moulded articles** (B.P. 286,732).—See XI.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Coagulation phenomena in *Hevea* latex.** VII. **Phenomena in alkaline latex.** O. DE VRIES and N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, No. 34; Arch. Rubbercultuur, 1928, 12, 454—471).—The power of coagulation to coalesce the floccules in *B*-mixture (B., 1925, 17) is destroyed not only by heat, quinosol, or acidifying to the second stable zone, but also by the

addition of alkali such as ammonia or sodium hydroxide. Acidification shortly after the addition of the alkali restores the coalescing activity only in part. The lower limit of concentration for killing coagulation is 0.15*N*-ammonia or 0.015*N*-sodium hydroxide, the  $p_H$  approximating to 8 with both. Sodium acetate does not destroy coalescing power as it is unable to attain this degree of alkalinity. On account of this effect of alkali, ammonia-preserved latex has no coalescing power.

D. F. TWISS.

**Coagulation phenomena in *Hevea* latex.** VIII. **Rubber obtained by freezing latex.** O. DE VRIES and N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, No. 37; Arch. Rubbercultuur, 1928, 12, 675—685).—Coagulation by freezing occurred only at temperatures considerably below 0°, several days being required for complete effect even at  $-15^\circ$ . The coagulum was yellower than the normal; the viscosity, hardness, and rate of vulcanisation of the rubber were somewhat greater than the standard, whilst the nitrogen content, 0.31%, although a little below the normal, was considerably higher than for rubber from washed cream obtained by centrifuging. The serum had  $p_H$  6.2 and possessed coalescing activity.

D. F. TWISS.

**Preserving [rubber] latex with borax.** N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, No. 33; Arch. Rubbercultuur, 1928, 12, 441—453).—For use as an anti-coagulant, 1.2—1.5 g. of borax is needed per litre of latex, whilst as a preservative the minimum proportion is 30 g. per litre. Latex treated with borax has a lower viscosity than ordinary latex. The rubber from borax-treated latex dries more slowly, and is more plastic than rubber from untreated latex; the viscosity of such rubber and the time of vulcanisation are initially lower than for ordinary rubber, but gradually change in the direction of the normal values.

D. F. TWISS.

**Determination of the rubber content in latex preserved with sodium phosphate and formalin.** W. SPOON and N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, No. 36; Arch. Rubbercultuur, 1928, 12, 659—674).—Investigation of the influence of various factors in the coagulation of latex preserved with 0.2% of trisodium phosphate and 0.2% of formalin leads to recommendation of the following method for determination of the rubber content. After thorough shaking of the latex, 200 g. in an aluminium bowl are coagulated by the gradual addition of 30 c.c. of 10% acetic acid or of 5% formic acid with constant stirring. The bowl is then covered and left undisturbed for 2—3 hrs. The coagulum is kneaded and then milled in a standardised manner, the wet crêpe being dried overnight in the air and then for 2 hrs., with occasional exposure of a fresh surface, in a vacuum dryer; it is finally kept in a desiccator over quicklime, and next day weighed to the nearest centigram. Each determination is made in duplicate.

D. F. TWISS.

**Cream from [rubber] latex.** O. DE VRIES, R. RIEBL, and N. BEUMÉE-NIEUWLAND (Comm. Proefstat. Rubber, No. 35; Arch. Rubbercultuur, 1928, 12, 559—575).—Concentrated latex cream ("primary cream") obtained by centrifuging latex containing



ammonia was diluted with water, the mixture being again centrifuged to give a "secondary cream," which by the addition of ammoniated water and further centrifuging was made to yield a "tertiary cream." Rubber from the secondary and tertiary creams was remarkably little different in composition (ash, aqueous and acetone extracts, and nitrogen) from that from the primary cream. The tensile strength of the rubber from the creams was low; the rate of vulcanisation was lower than the standard, especially for the rubber from the "washed" creams, which also showed a greater plasticity and a lower viscosity, this feature being particularly marked in the "tertiary" rubber. Rubber from the "primary skim" had high viscosity, normal plasticity, rather rapid vulcanisation, and normal tensile strength but high slope; the rubber from the "secondary skim" was variable in character. Examination of a number of samples of rubber from different "primary" creams revealed rather wide variations in composition and in vulcanisation behaviour, and did not support the view that centrifuging would yield an especially uniform rubber. D. F. TWISS.

**Plasticity determinations in crude rubber. VI. Changes in plasticity on keeping.** O. DE VRIES (Comm. Proefstat. Rubber, No. 32; Arch. Rubber-cultuur, 1928, 12, 411—422).—Samples of smoked sheet showed a considerable decrease in plasticity when kept for from one to four years; a sample of pale crêpe showed no such decrease. Samples of crêpe and smoked sheet after twelve years' storage had become somewhat hard and brittle, but tensile strength and slope after vulcanisation indicated no great deterioration, the only marked alteration being the decrease in viscosity. Shipment from Java to America and back had no definite influence on the rubber. The changes in plasticity on storage cause a much greater degree of variability than the combined factors in the preparation of the rubber, and it is possible that modern standardised methods of preparation are tending to a product of more constant characteristics. D. F. TWISS.

**Physical influence of selenium-red on accelerated rubber mixtures.** R. DITMAR (Chem.-Ztg., 1929, 53, 239).—Selenium-red (12 pts.) in various mixtures containing also rubber (100 pts.), sulphur (3 pts.), zinc oxide (10 pts.), whiting (40 pts.), and organic accelerator (2 pts.) had no physical influence on some, but on others it had a hardening effect dependent on the accelerator present. The vulcanised products showed satisfactory resistance to deterioration by ageing. D. F. TWISS.

#### PATENTS.

**Production of creams and pastes from aqueous dispersions of rubber or rubber-like materials.** DUNLOP RUBBER Co., LTD., and R. C. DAVIES (B.P. 306,994, 1.12.27).—Creams or paste-like mixtures are obtained from aqueous dispersions such as of rubber by adding, under agitation, a concentrated sodium cellulose xanthate gel containing, e.g., 25—30% of cellulose. The dispersions may have been compounded previously; compounding ingredients also may be incorporated in, or during the addition of, the cellulose xanthate or in the resulting creams or pastes. The final mixtures

may be used for such purposes as moulding, spreading, etc., or may be coagulated. D. F. TWISS.

**Production of coherent deposits of organic substances from aqueous dispersions thereof by electrodeposition.** ANODE RUBBER Co., LTD. From S. E. SHEPPARD and C. L. BEAL (B.P. 307,585, 21.1.28).—In the electrophoretic deposition of organic substances such as rubber from aqueous dispersions, the temperature of the bath and particularly of the deposition surface is kept low by cooling. D. F. TWISS.

**Manufacture of ingredients for the compounding of rubber.** IMPERIAL CHEM. INDUSTRIES, LTD., A. J. HAILWOOD, W. J. S. NAUNTON, and A. SHEPHERDSON (B.P. 307,155, 11.1.28).—Rubber-compounding ingredients such as fillers, reinforcing agents, colours, and accelerators are coated with a thin film of suitable material, e.g., oils, higher fatty acids, or paraffin wax. These may be applied to the compounding ingredients in the form of solutions or dispersions in media in which the latter are substantially insoluble. Compounding ingredients so treated can be mixed into rubber with exceptional ease. D. F. TWISS.

**Production of rubber.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,375, 5.12.27 and 19.6.28. Addn. to B.P. 300,719; B., 1929, 66).—Emulsions of synthetic rubber, with or without the presence of natural latex, and after removal of part of the stabilising agent, are coagulated by proteolytic enzymes such as papain, pancreas extract, or banana juice. The results may be modified by the addition of substances, e.g., hydrocyanic acid or hydrogen sulphide, which assist the enzymes; the properties of the product can also be improved by imparting such a  $p_H$  that coagulation may be retarded and by introducing saline buffering substances. D. F. TWISS.

**Manufacture of artificial rubber.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,808, 12.9.27).—In the production of rubber-like polymerides of butadiene, this hydrocarbon is produced by way of  $\alpha$ -butylene glycol, aldol, acetaldehyde, and acetylene, the last-named being formed from methane, e.g., by passage through an electric arc, so as to ensure the absence of substances harmful to the subsequent processes. D. F. TWISS.

**Making rubber conversion products.** A. E. WHITE. From B. F. GOODRICH Co. (B.P. 307,134, 20.12.27. Cf. B.P. 249,172; B., 1926, 453).—Rubber is converted into isomeric products by heating a solution containing also phenol with a strong non-oxidising mineral acid or a sulphonic acid or sulphonyl chloride. The resulting solutions may be applied as adhesive paints or the acid-freed products may be employed for the production of articles by moulding. D. F. TWISS.

**Preparation of halide additive products, polymerides, and oxides of rubber.** GOODYEAR TIRE & RUBBER Co., Assees. of H. A. BRUSON (B.P. 285,071, 16.12.27. U.S. 11.2.27).—If rubber, in solution or in the solid state, is treated with metallic salts, especially halides, e.g., stannic chloride, antimony pentachloride, titanium tetrachloride, or ferric chloride, additive compounds are obtained; the metallic chloride can then be

removed, *e.g.*, by treatment with alcohol, acetone, or water. The remaining polymeric hydrocarbon is obtained in the form of white powder or flakes. If air or oxygen is admitted during the reaction, powdery insoluble oxides or polymerides of rubber are produced.

D. F. TWISS.

**Rubber-like substances.** E. W. HULTMAN, Assr. to F. P. DUNCKLEE, J. MONTELEONE, and W. R. SIMONS (U.S.P. 1,704,194, 5.3.29. Appl., 29.10.27).—Mineral oil substantially free from constituents boiling below 205° is treated in the liquid state below 205° with a polymerising agent, *e.g.*, cerium oxide or tin oxide; it is then cooled, *e.g.*, to 17°, and treated with an activated reducing gas (produced for example by subjecting carbon monoxide and/or methane to contact with platinum or nickel at about 205°). It is then subjected to a halogenating reagent, such as chlorine, bromine, or boron trifluoride, when a soft rubbery and vulcanisable product is obtained and can be separated from the bulk of the oily material.

D. F. TWISS.

**Attachment of india-rubber or the like to metal or other surfaces.** DUNLOP RUBBER CO., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 307,180, 8.2.28).—Coverings of gauze or mesh work are fixed to the surfaces; the facing material is then applied in the form of solution or aqueous dispersion, *e.g.*, by spreading, spraying, or electrodeposition. The dispersion of rubber or similar materials may be in a concentrated and/or compounded condition and vulcanisation may be effected concurrently with drying.

D. F. TWISS.

**Vulcanisation of rubber or the like coverings of metal rolls.** C. MACINTOSH & Co., LTD., and H. W. WOLTON (B.P. 307,628, 10.3.28).—In the vulcanisation of rubber coverings on metal rolls an electric current at low potential but high amperage is passed through the central spindle or an alternating current is passed through a wire coil introduced into the interior of the hollow metal roll. Evenness of heating and of vulcanisation are thus obtained.

D. F. TWISS.

## XV.—LEATHER; GLUE.

**Chemistry of the liming process [for hides].** A. STEIGMANN (Collegium, 1928, 653—657).—Cystine was treated with milk of lime, alone and with 8% solution of sodium hydroxide, ammonia, ammonium chloride, and calcium chloride, respectively. After 8 days sodium hydroxide had liberated sulphur from the cystine, whereas no free sulphide was present in the cystine treated with calcium hydroxide alone. This is attributed to the formation of a calcium hydroxide-cystine complex. The hydrolysis of cystine was accelerated by sodium hydroxide and ammonia, and hindered by the addition of calcium chloride to the milk of lime. Solutions of cystine, tyrosine, and albumin when treated with sodium sulphide solution yielded additive products.

D. WOODROFFE.

**Periodic and continuous tanning processes.** B. SCHWARZBERG (Collegium, 1928, 661—667).—In most tanning processes the liquors are strengthened periodically, and the rate of tannage is greatest immediately after each strengthening, and then gradually

diminishes. A continuous process of strengthening to maintain a constant rate of tannage could be arranged by providing for a slow continuous flow of tan liquor through each pit, paddle, or drum. The liquor in the last-named is allowed to run off at the same rate as the strengthening liquor is run into it.

D. WOODROFFE.

**Determination of the volatile acids in tannin baths.** A. PONTE (Boll. Uff. Staz. Sperim. Ind. Pelli, 1929, 7, 57—63).—This method, based on that applied by Rota to wines, consists in diluting a certain volume of the bath with 10 c.c. of 96% alcohol and water to a total volume of 200 c.c., and distilling 100 c.c. of the liquid until exactly 72 c.c. of distillate are collected. This quantity of distillate, which contains one half of the total volatile acid, is titrated and the acid found multiplied by two. If a sulphited bath is tested in this way the distillate will contain sulphur dioxide. In this case the titrated solution is acidified with sulphuric acid and titrated with 0.02*N*-iodine solution, and a correction applied to the result of the acidimetric titration.

T. H. POPE.

**State of combination of acid sulphate in chrome [tanned] leather.** H. B. MERRILL and J. G. NIEDERCORN (Ind. Eng. Chem., 1929, 21, 252—253).—Portions of well-washed chromed hide powder were each shaken for 2 hrs. with different amounts of *N*-sodium hydroxide or *N*-sulphuric acid, then washed for 1 hr., and dried. The leather as tanned contained a chromium complex of 32.3% acidity. Treating the leather with acid did not much increase the acidity of this complex. Partial neutralisation of the leather with alkali, however, decreased the total acid sulphate in direct proportion to the amount of alkali added until the acidity of the chromium complex was one half of its original value. The chromium-bound and protein-bound acid contents both decreased with increasing neutralisation of the leather. When the chromium-bound acid had been reduced to 17.4% acidity, the leather no longer contained protein-bound acid. Further neutralisation reduced the acidity of the chromium complex.

D. WOODROFFE.

**Consistency of animal glue.** D. BROUSE (Ind. Eng. Chem., 1929, 21, 242—247).—The viscosity of solutions of a standard, a higher-grade, and a lower-grade animal glue, respectively, was determined at different temperatures and shown to be a hyperbolic function of the temperature. The better the grade of glue or the greater the concentration the greater is the viscosity. The viscosity-temperature curve was altered with respect to the axes by changes in concentration or by using a different grade of glue, but its shape remained the same. The temperature of gelation increased as the grade of glue improved, but the different grades showed the same sharp change from viscosity to plasticity, only at different temperatures. The usual methods for expressing consistency or viscosity in the form of an equation do not apply, and the Herschel expression is favoured. There is no difference in the fundamental properties underlying glueing technique. If the gelation temperature must be reached quickly, a low concentration of a high-grade glue or a more concentrated solution of a lower-grade glue can be used. For long

assembly periods, a low-grade glue or a dilute solution of a high-grade glue may be used. D. WOODROFFE.

**Preparation of standard gelatin.** J. M. HUDSON and S. E. SHEPPARD (Ind. Eng. Chem., 1929, 21, 263—264).—The following specifications are suggested. The gelatin should be prepared from well-limed calfskin and only the first extract taken at about 54°; this should be dried at a concentration > 5%, de-ashed to an ash content < 0.05%, and should have a constant isoelectric point  $p_H$  4.7—4.9 and a viscosity of about 4 centipoises at 5% concentration, or 6 centipoises at 7% concentration and 40°. The jelly strength should be about 300 g. (Bloom) for a 7% solution chilled by melting ice for 16—24 hrs. The gelatin should be nearly colourless, free from grease, fats, or heat-coagulable protein, and have a minimum absorption of blue light at a definite thickness of a 5% solution. D. WOODROFFE.

**Food gelatin values relative to concentrations.** M. BRIEFER and J. H. COHEN (Ind. Eng. Chem., 1929, 21, 264—265).—The jelly strengths of different solutions (1—10% concentration) of various grades of gelatin were found to vary directly as the concentration. These determinations are only of value commercially at about 3% concentration, at which the jelly strength-concentration curve is not linear. Gelatin should be graded on a 3% concentration basis instead of on a 6.67% value as at present. D. WOODROFFE.

**Treatment of tanning wastes.** FALES.—See XXIII.

#### PATENTS.

**Method of depilating hides.** J. PEINY (F.P. 621,577, 16.9.26).—The flesh side of the hides is painted with a solution, at 35°, of sodium chloride, sulphur compounds, *e.g.*, alkali or arsenic sulphides, and sodium hydroxide or carbonate, to which may be added calcium carbonate. The hair is loose in 0.5—3 hrs.

D. WOODROFFE.

**Process for tanning hides and skins.** J. HELL (G.P. 451,988, 13.4.24).—Hides or skins are treated for about 20 hrs. with pastes of suitable alkalinity, obtained by mixing insoluble prepared or natural carbonates, *e.g.*, chalk, limestone, dolomite, magnesite, precipitated chalk, with solutions of suitable salts or mixtures of salts, *e.g.*, calcium or magnesium chloride or magnesium sulphate, to which may be added formaldehyde, quinone, or other known tannins. The product is dark coloured, but very soft and tough. D. WOODROFFE.

**Manufacture of sulphonic acids with tanning properties.** M. MELAMID (G.P. 451,534, 26.3.22).—Anthracene oil or soft pitch is strongly oxidised, the product extracted with mineral oil, the solvent distilled off, and the residue sulphonated and condensed or esterified with either formaldehyde or acetylene respectively and an aromatic sulphochloride.

D. WOODROFFE.

**Manufacture of tanning materials from sulphite-cellulose liquors.** C. and C. P. HÜTTENES (G.P. 451,913, 12.4.25).—Sulphite-cellulose liquors, from which the sugars may have been removed by fermentation, are treated with an alkali or alkaline-earth hydroxide and/or

sulphide to remove heavy metals, heated, concentrated to  $d$  1.26, treated with alum and sulphuric acid, or with sodium, ammonium, or chromium sulphate to precipitate the lime, and finally concentrated to form a pulverulent mass when cold. D. WOODROFFE.

**Dressing of leather, skins, etc.** HANSEATISCHE MÜHLENWERKE A.-G., and B. REWALD (B.P. 306,672, 23.1.28).—A lecithin or phosphatide other than egg yolk is mixed with an oil, which may be sulphonated, emulsified with water, which may contain a small quantity of alkali, ammonia, or soap, and the emulsion employed in fat-liquoring of leather. The oil may be replaced by an aqueous swelling or a solution of lecithin, to which has been added an aliphatic or aromatic sulphonic acid and/or albumin etc. D. WOODROFFE.

**Impregnated material [leather].** L. F. WHITNEY and W. E. WHITNEY, ASSRS. to RAJET CO. (U.S.P. 1,682,652, 28.8.28. Appl., 15.1.24).—Chrome-tanned leather is impregnated at 55—60° with a solution of cellulose nitrate or acetate in a low-boiling solvent, such as tetrachloroethane, acetone, or methyl alcohol, containing a filler, *e.g.*, paraffin wax, carnauba wax, an anti-shrinking agent, such as glycerin or ethylcarbamide, and a high-boiling solvent or gelatinising agent, *e.g.*, ethyl phthalate, phenyl phosphate. R. BRIGHTMAN.

**Manufacture of glue, gelatin, etc. in the form of globules or pellets.** BRIT. GLUES & CHEMICALS, LTD., and R. B. DREW (B.P. 306,622, 1.12.27).—A concentrated solution of the material is delivered in the form of drops on a moving surface upon which a film of ice has been frozen or ice shavings or snow has been delivered. D. WOODROFFE.

**Treatment of gelatin.** J. T. DIXON (B.P. 306,439, 18.11.27).—An alkaline solution of gelatin or glue is mixed with 30—50% of a solution of a saponified fatty compound, *e.g.*, castor or linseed oil, previously sulphonated if desired, with or without the addition of a tanning or hardening agent (a chromate, formaldehyde, or hexamethylenetetramine). The product may be subsequently treated in a hardening bath. Finally it is mixed with 1—2% of phenol or sodium or potassium iodide, spread, rolled or moulded, and dried.

D. WOODROFFE.

**Increasing wetting capacity of liquids** (B.P. 291,070).—See VI.

#### XVI.—AGRICULTURE.

**Routine mechanical analysis of soils.** P. KÖTTGEN and H. HEUSER (Z. Pflanz. Düng., 1929, 13A, 137—159).—The relative merits of sedimentation and pipette methods and the use of sieves for large particles are discussed. In pipette methods the streaming motion of the suspension caused by the withdrawal of the sample affects the downward fall of the particles. This effect is slight with large particles, but marked with the finer ones. The size and shape of the jet of the pipette and the period occupied in sampling therefore influence the composition of the sample. A wide horizontal jet minimises the disturbance of the motion of the smaller particles. The use of glycerin and glycerin-water suspensions, by decreasing the rate of sedimentation,

allows accurate values to be obtained for larger particles by the pipette method. Details of apparatus and laboratory technique for pipette sampling, filtration of suspension, and sieve-washing are described.

A. G. POLLARD.

**Method of water control for sand cultures.** O. V. S. HEATH (Ann. Bot., 1929, 43, 71—79).—An apparatus for measuring the moisture contents of sand cultures is described. It consists of a porous porcelain candle full of water, buried in the sand, and with a mercury manometer attached. By calibrating the manometer in terms of sand moisture content, measurements of the latter may be made to 1.2% of the weight of sand.

E. A. LUNT.

**Phosphate and lime contents of Oldenburg soils.** S. GERICKE (Z. Pflanz. Düng., 1929, 8B, 1—15).—The soils examined are largely acidic and respond to liming in pot experiments. The marsh and sandy soils are characterised by an apparent sufficiency of total phosphate, of which, however, only a relatively small proportion is root-soluble.

A. G. POLLARD.

**[Soil] reaction experiments with mustard and oats in connexion with nutrient-requirement trials.** L. HELLER (Z. Pflanz. Düng., 1929, 8B, 37—40).—The importance of determinations of the effect of soil reaction, lime content, and lime requirement on the growth of individual crops as a necessary complement to fertiliser-requirement experiments is emphasised.

A. G. POLLARD.

**Determination of small quantities of nitrates in soils and plants.** J. BLOM and C. TRESCHOW (Z. Pflanz. Düng., 1929, 13A, 159—190).—The method is based on the formation of 5-nitro-*m*-4-xyleneol, its subsequent distillation in steam, and colorimetric determination after rendering alkaline with sodium hydroxide. Organic matter in the sample is removed by heating with sulphuric acid and permanganate. No loss of nitrate occurs during this process, and there is no appreciable oxidation of ammonia or of amino-acid to nitrate. Excess of permanganate may be removed by means of oxalic acid before addition of the nitroxyleneol. With 0.05 mg. of nitrate ion an accuracy of  $\pm 2\%$  is attainable.

A. G. POLLARD.

**Cold- and hot-fermented manure.** GERLACH and SEIDELL (Z. Pflanz. Düng., 1929, 8B, 15—37).—Published literature is critically reviewed. Hot-fermented manure has rather less dry weight and total nitrogen, but more water-soluble nitrogen than cold-fermented manure. The work of Krantz and of Löhnis and Ruschmann on the fermentation process was not confirmed. The decomposition in soil of hot-fermented manure was slightly slower than with the cold-fermented product, and crop yields were rather lower.

A. G. POLLARD.

**Determination of the nitrogen fixation by legumes by means of the nitrogen-base ratio.** A. STORCK and A. RIPPEL (Z. Pflanz. Düng., 1929, 13A, 158—159).—Comparison is made of the nitrogen-base ratio (see Rippel and Ludwig, A., 1926, 439) of legumes at two successive stages of growth, e.g., before and after flowering. Relative differences in the nitrogen equi-

valent in the two cases are indicative of the extent of nitrogen fixation.

A. G. POLLARD.

**Chemical effect of gypsum, sulphur, iron sulphate, and alum on alkali soil.** W. P. KELLEY and A. ARANY (Hilgardia, 1928, 3, 393—420).—Gypsum precipitated the soluble carbonate as calcium carbonate, whilst the other materials either decomposed the carbonate or converted it into hydrogen carbonate. The exchange complex was also affected. Acid formed by the oxidation of sulphur or hydrolysis of ferrous sulphate or alum causes calcium to pass into solution, whence the sodium content of the exchange complex is decreased.

CHEMICAL ABSTRACTS.

**Fertiliser experiments in the Vladimir district.** L. L. BALASHEV (Trans. Sci. Inst. Fert., Moscow, 1926, No. 37, 5—60).—Loam soils respond best to phosphorus, and light soils to nitrogen and potassium; manganese oxides were not advantageous. Potassium fertilisers lower the starch content of potatoes.

CHEMICAL ABSTRACTS.

**Fertiliser experiments in the Tver district.** A. V. KAZAKOV and S. L. SHAPIRO (Trans. Sci. Inst. Fert., Moscow, 1926, No. 37, 61—91).—The sandy and loam soils respond primarily to phosphorus.

CHEMICAL ABSTRACTS.

**Field experiments with fertilisers in the Tula district.** A. V. KAZAKOV (Trans. Sci. Inst. Fert., Moscow, 1926, No. 35, 9—42, 43—83).—Experiments on podzolised loams, sandy loam, grey forest soils, dark forest soils, degraded and leached chernozem during the period 1903 to 1925 are summarised. Fertilisation experiments with phosphate are recorded.

CHEMICAL ABSTRACTS.

**Fertiliser experiments in the Moscow district.** L. L. BALASHEV (Trans. Sci. Inst. Fert., 1926, No. 35, 83—153).—Grain crops respond to fertilisers in the order: nitrogen > phosphorus > potassium; potatoes, flax, and clover respond in the order: potassium > phosphorus > nitrogen. Meadow hay responds primarily to potassium.

CHEMICAL ABSTRACTS.

**Phosphate requirement of barley at different periods of growth.** W. E. BRENCHLEY (Ann. Bot., 1929, 43, 89—110).—Experiments have been carried out in water cultures to investigate the effect of the withdrawal of the phosphorus supply on the barley plant at varying stages during its growth. The minimum period from germination during which phosphates must be supplied in order to obtain normal growth is six weeks; during this period phosphate absorption is sufficient to enable the plant to make its maximum dry weight.

E. A. LUNT.

**Plant nutrition. I. Effect of manurial deficiency on the respiration and assimilation rate in barley.** F. G. GREGORY and F. J. RICHARDS (Ann. Bot., 1929, 43, 119—161).—The effect of nitrogen, phosphorus, and potassium deficiency on the respiration and assimilation rates of the barley plant has been investigated. The following results have been obtained: nitrogen-deficient plants exhibit a subnormal respiration and a slightly subnormal assimilation rate; phosphate-deficient plants a normal respiration and a

slightly supernormal assimilation rate; potassium-deficient plants a supernormal respiration and a subnormal assimilation rate. E. A. LUNT.

**Influence of nutrients on the value of bast-fibre plants (flax and nettle).** I. H. FABIAN (*Faserforsch.*, 1928, 7, 1—56; *Chem. Zentr.*, 1928, ii, 1605).—Lack or excess of potassium, nitrogen, or phosphorus is unfavourable for the production of fibre. Diminution of fibre yield occasioned by nitrogen can be to a certain extent compensated by means of potassium. The most favourable fertilisation for the production of fibre is that which best stimulates growth, but does not afford the maximum production of stem. A. A. ELDRIDGE.

**Specifications for petroleum oils to be used on plants.** E. R. DE ONG (*J. Econ. Entomol.*, 1928, 21, 697—702).—The presence of 0.05% of sulphur may be injurious; oil emulsion containing free sulphur is not recommended. Specifications are given of oils for various purposes. CHEMICAL ABSTRACTS.

#### PATENTS.

**Fertilisers.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,230, 30.3. and 31.8.28).—Siliceous material, e.g., river sand, is heated above 250° with a quantity of phosphoric acid corresponding to 1.5—8 times the weight of silica present. The product may be treated with ammonia or mixed with other fertilisers. L. A. COLES.

**Preparation of fertilisers.** F. ROTHE and H. BRENEK, Assrs. to RHENANIA VER. CHEM. FABR. A.-G. (U.S.P. 1,704,218, 5.3.29. Appl., 11.6.25. Ger., 23.6.24).—See B.P., 235,860; B., 1925, 731.

### XVII.—SUGARS; STARCHES; GUMS.

**Drying of sugar beet and extracting the sugar from it in a diffusion battery.** N. L. KARAVAEV and A. P. PALKIN (*Bull. Sakhatrest*, 1928, No. 5, 40—44, and *Acta Univ. Asia Med.*, 1928, [vi], 3—12).—Sliced sugar beet sun-dried to 8—10% H<sub>2</sub>O can be stored indefinitely without increase of invert sugar. Extraction of the dried beet slices requires 50% longer time than with raw beets, but the diffusion juice is twice as concentrated. CHEMICAL ABSTRACTS.

**Treatment of beet juices with sodium carbonate, and the juice alkalinity.** V. STANEK (*Z. Zuckerind. Czecho-Slov.*, 1929, 53, 173—177).—Sodium carbonate added for the reduction of the calcium content of juices during their clarification should be introduced before the first carbonatation, e.g., to the juice in the measuring tanks. If triple carbonatation is applied, the alkali should be mixed with the second carbonatation juice before turning on the gas, the colloidal calcium carbonate formed being thus entrained by the carbonatation scums. In practice, it is found that the quantity of sodium carbonate added should be somewhat more than the equivalent of the calcium present, otherwise the desired final alkalinity will not be realised. J. P. OGILVIE.

**Solubility of carbon dioxide in sugar factory juices, and its formation through some amino-acids.** V. MAJER (*Z. Zuckerind. Czecho-Slov.*, 1929, 53,

213—229).—Diffusion, first carbonatation, second carbonatation, and boiled clarified juices were compared in respect of carbon dioxide absorption. It was found that carbon dioxide is present in such juices in three forms: (a) in solution, (b) combined with alkalis, and (c) combined with aspartic and glutamic acids as carbamates. When boiled in aqueous solution, these last-named compounds slowly decompose, which fact probably explains the gradual separation of calcium carbonate observed during the boiling-up of second carbonatation juices in the factory. J. P. OGILVIE.

**Pressure evaporation [in the beet factory].** K. VESELY (*Z. Zuckerind. Czecho-Slov.*, 1919, 53, 204—206).—In pressure evaporation in white sugar manufacture, colour formation is due to defects in the design of the apparatus, whilst other factors are incorrect clarification and filtration, and the absence of sulphites. There is no question as to the suitability of such apparatus under proper operating conditions. J. P. OGILVIE.

**Rapid filtration of syrup through sand.** G. J. T. HEYNING (*Archief Suikerind. Nederl.-Indië*, 1928, 36, 1179—1182).—The filter consisted of a cylindrical tank filled with sand (having grains about 2 mm. diam.) to a depth of about 1 metre, the syrup being allowed to percolate downwards. Before commencing the filtration, air was expelled from the medium by passing juice upwards through it, in which manner also later cleaning was accomplished. The insoluble matter in the syrup before and after this sand filtration was found to be 0.028 and 0.005%, using a laboratory centrifugal for the determination. J. P. OGILVIE.

**Alkaline defecation-carbonatation process for clarifying cane juices.** K. DOUWESDEKKER and P. C. NIKOLA (*Archief Suikerind. Nederl.-Indië*, 1928, Med. 13, 721—749).—Sufficient lime is added to the cold juice to impart a slightly alkaline reaction, after which carbon dioxide is pumped in until an acidity corresponding to about 1000 mg. CaO per litre is reached, or about  $p_H$  6.7. The main quantity of lime is then added until the alkalinity is between 700 and 800 mg. After being allowed to settle, the clear juice is treated with carbon dioxide, filtered, and sent to the evaporators, whilst the mud is heated, settled, and filtered, the clear liquids being added to the acid juice before the addition of the main quantity of lime. J. P. OGILVIE.

**Modification of the sulphitation process of clarification of cane juices.** H. H. E. SUSSBACH (*Archief Suikerind. Nederl.-Indië*, 1928, 36, 1187—1189).—The raw juice is neutralised by the addition of a suitable quantity of milk of lime ( $d$  1.1), and allowed to remain at 55° for about 5 min., which conditions are stated to be suitable for colloid separation. A further addition of milk of lime is then made, so as to complete the usual amount, following which the juice is sulphited in the usual manner. A better clarification and less glucose decomposition than ordinarily are claimed. J. P. OGILVIE.

**Acidity determination in sulphitation, using the  $p_H$  value.** M. DEN HARTOGH (*Archief Suikerind. Nederl.-Indië*, 1928, 36, 1175—1179).—In the juice sulphiting operation, treatment with the gas is carried

on till the is  $p_H$  7.1—7.2, using the colorimetric method of determining hydrogen-ion concentration, and bromothymol-blue as the indicator. For syrup sulphitation control is effected by means of chlorophenol-red, so as to obtain an indication of  $p_H$  5.3—5.5. A comparator in connexion with a rotating disc containing the standard buffer solutions recommended for such determinations is described. J. P. OGILVIE.

**Refining qualities of Philippine raw sugars.** P. SENGSON (Sugar News, 1928, 9, 807—811).—It is advised to maintain the polarisation between 96.5 and 97.0 in the production of raws for the American refiners. The water content of such sugars has now been reduced to an average of 0.785%, whilst the ash (direct incineration) is 0.246%. Improvements have been effected in the clarity of the re-dissolved sugars, and also in the filtration rate. It has further been possible to reduce the percentage of "total smalls" (grains having diameter from 0.15 to 0.60 mm.), so that this figure, which in 1924—5 was 54%, is now about 25%, the limit acceptable to the refineries. J. P. OGILVIE.

**Comparative examination of "Norit" and "Carboraffin" [in sugar refining].** K. REICH and G. VAURINECK (Z. Zuckerind. Czecho-Slov., 1929, 53, 285—289).—Analytical figures observed for "Norit Supra 3X" and "Carboraffin" were, respectively, loss of weight at 150°, 12.73, 11.10; ash, 6.50, 2.98; water-soluble ash, 2.27, 1.57%; reaction, alkaline, neutral;  $Fe_2O_3$ , 0.68, 0.34%. Inverting effect was observed by heating 2 g. of each carbon with 100 c.c. of a 50% solution of sugar for  $\frac{1}{2}$  hr. at 90°, and determining the copper reduced from 50 c.c. of Fehling's solution, the figures found being 30.9 and 58.6 mg. (blank 26.0 mg.). To charge a Danek filter of 13 sq.m. surface, the weights of the two carbons to give the same thickness of layer were 30 kg. and 20 kg. After a factory run lasting 49 hrs. using such filters, the colour removed by the two media was, respectively, 29.9 and 39.4%. Boiling the used carbon for 1 hr. with water showed only a slight revivification with the "Norit," but restored the "Carboraffin" to 50% of its original decolorising power. J. P. OGILVIE.

**Instrument for determining the specific gravity of refined sugar.** V. YANOVSKI (Bull. Sakharotrest, 1928, No. 1, 53—60). CHEMICAL ABSTRACTS.

**Colouring matters formed by the caramelisation of sucrose and by the action of lime on invert sugar.** M. GARINO and A. TOSONOTTI (Giorn. Chim. Ind. Appl., 1929, 11, 8—13).—The constituents of caramel prepared by heating sucrose and termed caramelan, caramelen, and caramelin by Gélis (Ann. Chim. Phys., 1858, [iii], 52, 352) are not individual compounds but mixtures. Caramelan may be freed from caramelen by repeated treatment with 84% alcohol, followed by evaporation of the solution obtained to dryness. This procedure gives a caramelan completely soluble in 84% alcohol and of constant colouring power if entirely dissolved, but when only a small quantity of the alcohol is used the substance dissolved has far less colouring power (about 260 Stammer units instead of 800). Caramelan tenaciously retains alcohol, which is

removed only by repeated boiling with water or by vacuum evaporation almost to dryness. Caramelen does not dissolve any large amount of caramelin, and may be freed from this by means of cold water, in which caramelin is insoluble; the colouring power of caramelen is about 18,000 Stammer units. Caramelin is probably not produced during the manufacture of sugar. The products obtained by caramelisation of dextrose or invert sugar resemble those formed from sucrose chemically, but differ in other ways, the colouring power, for instance, being enormously lower. Glucic and apoglucic acids, formed when invert sugar is heated in presence of an alkali, are described. Certain physical and chemical properties of all the above products have been investigated, the most noteworthy results being that solutions of both caramelan and the calcium salt of glucic acid exhibit marked surface activity, and that the intensity of colour of a caramelan solution is greatly increased on addition of alkali. T. H. POPE.

**Alteration of sugar-like substances in sulphite-cellulose cooking.** HÄGGLUND and JOHNSON.—See V. Furfuraldehyde derivatives. YAMADA.—See XVIII. Fluorescence of honey. ORBÁN and STITZ. Hydroxymethylfurfuraldehyde in honey. FIEHE and KORDATZKI.—See XIX.

#### PATENTS.

**Saturation of sugar juice.** A. G. VERHEUL (Dutch P. 16,981, 12.12.23).—The production of carbon dioxide by fermentation of molasses and the saturation of the raw juice are so adjusted as to balance one another.

W. J. BOYD.

**Decolorisation of sugar juice by means of hyposulphites.** SOC. IND. DES DÉRIVÉS DU SOUFRE (F.P. 628,128, 26.1.27).—A solid hyposulphite is added at intervals during concentration, a total of 15—20 g.  $Na_2S_2O_4$  per ton of sliced beets being used. The evaporated liquor retains its pale colour and can be very highly concentrated owing to the reduction in viscosity due to the salts.

W. J. BOYD.

**Purification of molasses [for manufacture of spirit and yeast].** A./S. DANSK GAERINGS IND. (B.P. 283,970, 20.1.28. Austr., 21.1.27).—The diluted molasses is partly or entirely freed from betaine and related products by the action of betaine-decomposing bacteria. This process may be combined with, or may follow, the usual production of lactic acid by bacteria. After decantation or filtration a further purification may be effected by precipitating the alkaloids with tannin, removing the excess of the latter by means of lime, precipitating the colouring matters by producing a precipitate of aluminium hydroxide, and removing the sulphuric and carbonic acids by adding a barium compound, e.g., barium aluminate. Excess of barium and aluminium ions may then be removed by addition of water-glass or phosphoric acid. The liquid is finally pressed through a filter.

W. J. BOYD.

**Separation of betaine hydrochloride and potassium chloride from waste molasses.** Y. TAKAYAMA (B.P. 304,071, 13.3.28).—Crude potassium chloride is crystallised from concentrated waste liquor of beet molasses by addition of hydrochloric acid (as gas or

solution) and removed by filtration, and betaine hydrochloride containing potassium chloride is crystallised by concentration of the filtrate. This is dissolved in water, and the solution neutralised with lime, filtered, and concentrated to crystallise the potassium chloride, the free betaine remaining in solution. The potassium chloride crystals are removed, hydrochloric acid is added, and after further concentration crystals of betaine hydrochloride are obtained containing little ash. Instead of adding hydrochloric acid after removal of the potassium chloride, dilute sulphuric acid equivalent to the calcium chloride present may be added, the calcium sulphate filtered off, and the betaine hydrochloride obtained on concentration of the filtrate. W. J. BOYD.

### XVIII.—FERMENTATION INDUSTRIES.

**Strengthening of yeast by fermentation under pressure.** SCHUSTER (Woch. Brau., 1929, 46, 99).—Small- and large-scale fermentations confirmed the observation of Windisch (B., 1928, 685; 1929, 69) that fermentation under pressure leads to increase of size and improvement in appearance of the yeast. The yeast, however, did not settle so quickly or firmly as in the control fermentations. When subsequently used under ordinary conditions it gave good vigorous fermentation and returned to its original appearance. The strengthening effect is only of secondary importance to the possibilities of the recovery and utilisation of carbon dioxide. F. E. DAY.

**Furfuraldehyde derivatives in fermentation products.** M. YAMADA (Bull. Chem. Soc. Japan, 1929, 4, 31—35).—Hydroxymethylfurfuraldehyde-*p*-nitrophenylhydrazone was prepared from the distillate (160—180°) from 50% aqueous dextrose solution. The colour reactions with aniline + acetic and barbituric acids and the phloroglucinol test showed that the distillation of lævulose solutions also yields small quantities of hydroxymethylfurfuraldehyde. Arabinose solutions yield furfuraldehyde, whilst saké, shōyu (soya sauce), and shōchu (Japanese whiskey) yield both in varying proportions, and fusel oil mainly furfuraldehyde. The proportion of furfuraldehyde is increased by repeated distillation, owing to decomposition of the hydroxymethyl derivative. The substances in the distillates from fermented liquids which give a red coloration with the aniline-acetic acid reagent are mixtures of furfuraldehyde with its hydroxymethyl and methyl derivatives, derived from the partial decomposition of pentoses, hexoses, and methylpentoses respectively (cf. Hastie and Dick, B., 1928, 797). F. E. DAY.

**Apparatus for determining water content of barley and malt.** F. WINDISCH (Woch. Brau., 1929, 46, 96—97).—The apparatus depends on the much greater dielectric capacity of water as compared with other grain constituents, and consists of a condenser which can be filled with a definite quantity of material, together with suitable means for determining the change in the capacity of the condenser. Figures are quoted comparing the results of seven methods of determining the water content of two varieties of wheat. Though all methods are only of relative value, and so far the proposed method yields consistent results, besides

having the advantage that it may be carried out in one minute, it cannot be accepted for barley and malt analysis without further investigation. F. E. DAY.

**Titration [of worts and beers] by stages.** P. KOLBACH (Woch. Brau., 1929, 46, 91—96).—A critical consideration of the principles involved in titration by stages. The results of the method may be of value simply as a measure of the buffering over a given range of  $p_H$  or of the amount of a buffering substance or substances if these are almost exclusively responsible for the buffering over a range of  $p_H$ . By the titration of worts by stages, before and after treatment with baryta, it is shown that the titration value between  $p_H$  5.67 and 7.07 is a measure of the amount of phosphates, whilst titration above  $p_H$  7.07 gives a measure of the protein degradation products, though this is more accurately determined by a formol titration. On the other hand, below  $p_H$  5.67 the cause of buffering is not important, but the degree of buffering is so, since it affects the fall of  $p_H$  during fermentation. It is suggested that determinations of  $p_H$ , titration values with alkali to  $p_H$  7.07 and with acid to  $p_H$  4.27, and a formol titration will give more information regarding a wort than the usual titration by stages, with little more trouble. The initial values can be corrected for the small variation of the observed  $p_H$  from 5.67. Since beers of low  $p_H$  and less strongly buffered are more stable than those having the converse properties, determinations of  $p_H$ , titrations to  $p_H$  5.67 and 7.07, and formol titration should give as much information as can at present be obtained by such methods. F. E. DAY.

**Ropiness in wine.** G. MEZZADROLI and E. VARETON (Giorn. Chim. Ind. Appl., 1929, 11, 14—17).—When treated with norit, ropy wine loses its ropiness, but this returns after a few days. Treatment of the wine with sulphur dioxide prevents the appearance of further turbidity, but does not diminish the ropiness. When these two reagents are used together the ropiness disappears completely and permanently; vigorous agitation of the wine, a low temperature, and protracted action of the reagents assist the action. T. H. POPE.

**Evaluation of wine distillates and wine brandies.** G. BÜTTNER and A. MIERMEISTER (Z. Unters. Lebensm., 1928, 56, 492—498).—Results of the examination of a number of still wines, wine distillates, and brandies by the method of Graff (B., 1927, 953) are given. It is concluded that the method is useful, especially for the detection of grosser adulterations. W. J. BOYD.

**Manufacture, composition, and evaluation of Samos wine.** A. KICKTON and P. BERG (Z. Unters. Lebensm., 1928, 56, 397—422).—Samos wine is usually made by addition of spirit to the must at an early stage in the fermentation, but sometimes concentrated must or must from dried grapes is used. Its characteristics are low total acidity (0.28—0.42 g. per 100 c.c.), glycerin, and sugar-free extract (1.7—2.5 g. per 100 c.c.), and approximately equal contents of lævulose and dextrose. Total extract is usually 21—24 g. per 100 c.c. Of late years the alcohol content has risen from 11—12.5 g. to 14—15 g. per 100 c.c. in consequence of new regulations regarding the composition of wine imported into Germany. W. J. BOYD.

**Butyric acid fermentation.** A. HEIDUSCHKA and C. REYMANN (Pharm. Zentr., 1929, 70, 87—92).—The production of butyric acid, under various conditions of temperature and sugar concentration, by a typical butyric acid organism belonging to the *Amylobacter* group has been studied. The medium employed was ordinary nutrient broth to which was added 2—10% of dextrose, and chalk sufficient to neutralise 20% above the theoretical maximum yield of acid. Portions each of 2 litres were employed, and fermentation was allowed to proceed till evolution of carbon dioxide ceased. The following figures show the maximum and minimum production of each acid, calculated as percentage of the theoretical yield of butyric acid, together with the corresponding conditions of temperature and dextrose concentration. Total acid, 121.4% at 37°, 2% ; 22.4% at 47°, 5%. Lactic acid, 73.5% at 27°, 2% ; 2.0% at 47°, 5%. Acetic acid, 59.4% at 37°, 2% ; 11.0% at 42°, 10%. Butyric acid, 56.3% at 37°, 5% ; 8.2% at 47°, 5%. Under the conditions for maximum production of butyric acid, 37° and 5% of dextrose, the butyric acid constitutes 60.5% of the acid produced, with 11.4% of lactic and 28.1% of acetic acids. The trace of alcohol produced has no practical significance.

F. E. DAY.

## PATENTS.

**Apparatus for continuous manufacture of absolute alcohol.** E. RICARD, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,704,213, 5.3.29. Appl., 23.1.24. Belg., 16.4.23).—See B.P. 214,581 ; B., 1925, 185.

**Molasses** (B.P. 283,970).—See XVII.

## XIX.—FOODS.

**Determination of the degree of heating of milk.** P. WEINSTEIN (Z. Unters. Lebensm., 1928, 56, 457—467).—The effect of time and temperature of heating of milk on the oxidase (Rothenfusser), aldehyde-reductase (Scharlinger), catalase, and amylase reactions and on the creaming and albumin-sediment tests has been studied in order to utilise these as indicators of the previous heat treatment. Milk pasteurised at 85° for 1 min. gives a negative, milk heated at 70° for 30 min. a positive Rothenfusser test. The latter gives a negative Scharlinger reaction. Regulation-sterilised milk gives a positive Rothenfusser reaction, decolorises Scharlinger's reagent within 10—11 min., shows a low catalase value not exceeding 8—10 c.c. of oxygen for 100 c.c. of milk, and separates cream on keeping. Insufficiently sterilised milk shows a higher catalase value, and may show a positive amylase reaction. Milk heated at not above 55° has a strong amylase reaction and a catalase value equal to that of normal milk. These tests are also applicable as indicators of the efficiency of sterilisation by the Tödt method of flash-heating. W. J. BOYD.

**Chlorine index of goat's milk. Systems of rapid analysis.** J. M. CLAVERA and D. GUEVARA POZO (Anal. Fis. Quim. [Tec.], 1929, 2, 14—22).—The determination of the chlorine index (g. Cl/litre) of goat's milk by a rapid method (Weiss, Lebensm. u. Hyg., 1922, 12, 133 ; Sirot and Joret, B., 1928, 391) is recommended for control of its purity. Four quickly determined values,

which, in the case of goat's milk in Granada and Andalusia, are normally as follows:  $d^{15}$  1.029—1.032, fat 32—55 g./litre, ash 6.5—8.5 g./litre, chlorine 0.9—1.25 g./litre, then suffice for the estimation of its quality, and the type of adulteration practised, e.g., simple addition of water, or addition of water, sugar, and salt, may be readily diagnosed. R. K. CALLOW.

**Chemistry of sour milk.** L. L. VAN SLYKE (N.Y. Agric. Exp. Sta. Tech. Bull., 1928, No. 140, 1—14).—A review. CHEMICAL ABSTRACTS.

**Determination of fat in malted milk powders.** C. W. BALLARD (J. Amer. Pharm. Assoc., 1929, 18, 122—123).—A modification of the Röse-Gottlieb method recommended in "Methods of analysis," Association of Official Agricultural Chemists, pp. 262 and 275, is described in which the Röhrig tube is made with a detachable portion which facilitates the manipulation of the sample. E. H. SHARPLES.

**Presence of mixed glycerides in butter from cows' milk.** E. DE'CONNO and E. SCOPINARO (Annali Chim. Appl., 1929, 19, 55—65).—When butter fat, purified by fusion and filtration, is dissolved in hot acetone containing one third of its volume of chloroform, the solution deposits when cooled to the ordinary temperature (about 13°) about 10% (on the weight of dissolved fat) of a white crystalline solid, which after six crystallisations has a constant m.p. (51°) and the composition of a myristodipalmitin. When cooled to —15° the filtrate yields a myristodistearin, m.p. 57.5°, to the extent of about 4% of the weight of the original fat. The residual stable, oily liquid yielded no further fractions, but showed saponification, iodine, and volatile acid values higher than those of the original fat, and thus contained all the glycerides of saturated acids of low mol. wt. (volatile) and of unsaturated liquid acids, mainly oleic. Hence the existence of the liquid glyceride, butyropalmito-olein, recorded by Bell ("The Chemistry of Foods," II, 44) and by Blyth and Robertson (Proc. Chem. Soc., 1889, 5), is not confirmed. T. H. POPE.

**Cotrone cheese.** E. DE'CONNO and M. FRATTURA (Annali Chim. Appl., 1929, 19, 65—75).—Analysis of this Calabrian cheese gave the following mean percentage composition: water 27.816, acidity as lactic acid 1.308, fat 22.886, fatty acids 4.185, nitrogenous substances ( $N \times 6.25$ ) 32.142, ash 8.193, sodium chloride 5.341. The fat had acid value 25.747 and saponif. value 237.55. The nitrogen exists in the following forms: soluble 2.361, insoluble 2.783, protein 1.382, ammoniacal 0.128, non-protein 0.989, amino- 0.850. The coefficients of maturation, solubilisation, and decomposition are, respectively, 0.458, 0.268, and 0.190. These results differ from the data given by König for this product. T. H. POPE.

**Calcium of cheese.** K. BLUNT and E. SUMNER (J. Home Econ., 1928, 20, 587—590).—Average values are: Swiss cheese 1.05, Cheddar cheese 0.71, cottage cheese 0.077% Ca. CHEMICAL ABSTRACTS.

**Commercial lecithins and lecithin preparations.** J. SONOL (Rev. fac. quim. farm. Univ. La Plata, 1928, 4, No. 2, 3—40).—For the preparation of lecithin, brain or fresh egg-yolk is dried with acetone and extracted



with absolute alcohol at 50°, the alcohol being renewed three times every 12–24 hrs. After 12 hrs. at 0° the alcoholic filtrates are filtered and distilled in a vacuum. Before removal of the alcohol is complete the upper (yellowish) layer is separated from the lower (reddish) layer, freed from alcohol, dissolved in a small amount of ether, and precipitated with acetone; the procedure is repeated three times, and the lecithin freed from acetone in a vacuum. Analytical data for commercial lecithins and lecithin preparations are recorded.

CHEMICAL ABSTRACTS.

**Rôle of carbohydrates and proteins in staling of bread.** L. KARÁCSONYI (Z. Unters. Lebensm., 1928, 56, 479–484).—In staling of bread the carbohydrate material soluble in water but insoluble in alcohol decreases, probably owing to alteration of the starch gel. The amount of protein water-soluble material remains unchanged.

W. J. BOYD.

**Determination of hydroxymethylfurfuraldehyde in honey and artificial honey.** J. FIEHE and W. KORDATZKI (Z. Unters. Lebensm., 1928, 56, 490–492; cf. Troje, B., 1925, 1004; Fiehe, B., 1929, 146).—A solution of 100 g. of the honey in water is treated with zinc acetate and potassium ferrocyanide and filtered. The filtrate is continuously extracted with ether thrice renewed for 12 hrs. and the ether extract is then mixed with an equal volume of light petroleum and some anhydrous sodium sulphate, left for 24 hrs. with occasional shaking, and then filtered. The filtrate is evaporated at a moderate temperature, the residue extracted in 20 c.c. of water, and the solution filtered. 5 c.c. of the solution were titrated with alkaline iodine solution by Troje's method (*loc. cit.*) and 5 c.c. were precipitated with phloroglucinol. The rest of the solution was titrated with alkaline copper solution by Lenk's method (Z. angew. Chem., 1917, 30, 49). It was found that the iodometric method cannot be used for the determination of hydroxymethylfurfuraldehyde in honey, but that the other methods are suitable for this purpose.

W. J. BOYD.

**Fluorescence of honey in ultra-violet light.** G. ORBÁN and J. STITZ (Z. Unters. Lebensm., 1928, 56, 467–471).—Numerous samples of honey examined all showed luminescence in ultra-violet light depending on the absorption in the ultra-violet region, on the colour of the honey, and on the thickness of the layer of honey. Honey heated to 100° has weaker luminescence while hot, but recovers its original intensity on cooling to 30°. On evaporating some of the water from the honey the increase in luminescence is greater than can be explained by increase in concentration, and is a function of the viscosity. Slightly caramelised honey luminesces more strongly in thin layers, but more faintly in a test-tube than normal honey. Strongly caramelised honey luminesces very faintly in thin layers and becomes brown in colour. It is difficult to distinguish natural from artificial honey by means of its luminescence, but it is at once evident whether a particular kind of honey has been adulterated.

W. J. BOYD.

**Detection of maltol and of salicylic acid in presence of maltol.** T. MERL and H. BEITTER

(Z. Unters. Lebensm., 1928, 56, 472–474).—Powdered malt coffee (20 g.) is boiled for a short time with 50 c.c. of chloroform and 3 g. of blood charcoal and filtered after cooling. 10 c.c. of the filtrate are shaken with 1–2 c.c. of water and, after separation of the water, filtered into a centrifuge glass. 1 c.c. of a freshly diluted ferric chloride solution (2 drops of a 10% solution in 20 c.c. of water) is added, the mixture emulsified by shaking, and the liquids are separated by centrifuging. The presence of maltol is shown by the characteristic violet coloration of the aqueous layer. Another 10 c.c. portion of the filtrate is tested for salicylic acid if necessary, as follows: After evaporation of the chloroform 5 c.c. of phosphoric acid (*d* 1.7) and 15 c.c. of water are added to the residue and 15 c.c. of the liquid distilled off. The distillate is evaporated to dryness on the water-bath with a few drops of 5% barium hydroxide solution, cooled, and treated with 10 drops of glacial acetic acid and 3 drops of Mandelin reagent (a 5% solution of ammonium vanadate in 95% sulphuric acid). 0.025 mg. of salicylic acid can be detected by the indigo-blue coloration formed.

W. J. BOYD.

**Flesh proteins of various animals.** K. BECK and E. CASPER (Z. Unters. Lebensm., 1928, 56, 437–457).—Determinations of the nitrogen distribution in various proteins have been carried out by the Van Slyke method. These include edible gelatin (gold leaf), "glucose" obtained from edible gelatin and from Liebig's meat extract by Striegel's method (Chem.-Ztg., 1917, 41, 313), and the coagulated muscle-protein of the ox, calf, pig, sheep, horse, goose, and cod. The extracts of these muscle tissues were examined for content of albumose, glucose, total nitrogen, amino-acid nitrogen, creatinine, ash, and phosphoric acid. The glucose from Liebig's meat extract was found to be similar to that of gelatin. The extract from cod flesh was shown to differ from those from the other varieties of flesh in that higher proportions of albumose and glucose were present.

W. J. BOYD.

**Changes in composition during ripening and storage of melons.** J. T. ROSA (Hilgardia, 1928, 3, 421–443).—In the late stages of development and the ripening process the total and soluble solids and sugar, and the sp. gr. of the juice increase. Reducing sugars (lævulose and dextrose in approximately equal proportions) decrease; sucrose increases proportionately more rapidly. The total amount of pectic substances is unchanged, but the protopectin decreases rapidly. Fruits picked when immature show on storage an eventual small decrease in sugars. The total amount of pectic substances decreases slightly, and protopectin is changed to pectin. Unripe fruits, when exposed to the action of ethylene, show no increase in sugar content, but an acceleration in the conversion of reducing sugars into sucrose. The action of the ethylene is attributed to an activation of enzymic reactions.

CHEMICAL ABSTRACTS.

**Available carbohydrate content of some fruits and vegetables.** M. BELL, M. L. LONG, and E. HILL (J. Metabol. Res., 1925–1926, 7–8, 195–197).—Values for fresh and canned products are recorded.

CHEMICAL ABSTRACTS.

**Content of alcohol-soluble material in cinnamon.**

J. PRESCHER (Z. Unters. Lebensm., 1928, 56, 474—478).—Data are given for the alcohol extract, ash and sand contents of various samples of cinnamon. The alcohol extract of Ceylon cinnamon varied from 13.40 to 9.60%, that of cassia cinnamon from 12.63 to 6.55%.

W. J. BOYD.

**Food gelatin.** BRIEFER and COHEN.—See XV. **Determination of water content of barley.** WINDISCH.—See XVIII. **Disposal of wastes.** MOHLMAN and BECK.—See XXIII.

## PATENTS.

**Food product and method of making same.** S. GELFAND (U.S.P. 1,697,312, 1.1.29. Appl., 19.12.27).—A cultured milk product is emulsified into an emulsion containing oil and yolk, and sufficient of a solution of an edible organic acid is added to stabilise the product and prevent further activity of micro-organisms.

W. J. BOYD.

**Treatment of butter, margarine, and similar oil-in-water or water-in-oil emulsions.** J. E. NYROP (B.P. 307,167, 26.1.28. Addn. to B.P. 297,256; B., 1928, 825).—Homogenised emulsions of fatty matter containing a relatively small amount of protective substances (e.g., casein) when spray-dried at low temperatures in the absence of oxygen yield a pasty product, which when kneaded with water will absorb it and reproduce a material of butter-like structure and consistency.

E. LEWKOWITSCH.

**Treatment of the yolk of eggs.** H. BELHOMMET (B.P. 304,902, 10.1.28).—About 4% of sugar or other suitable substance soluble in water is added to the fresh liquid yolk, from which 50% of its weight of water is then evaporated. The dried yolk can be preserved almost indefinitely and reconstituted in the liquid condition by replacing the water. [Stat. ref.] W. J. BOYD.

**Preservation of flour, grain, grain products, etc.** R. A. LEGENDRE (U.S.P. 1,702,735, 19.2.29. Appl., 22.3.28. Fr., 2.4.27).—The moisture in the product is maintained at about double the normal and its alkalinity is kept within the range  $p_{\text{H}}$  7—10.

A. R. POWELL.

**Treatment of fruit to prevent decay.** W. R. BARGER, Assr. to U.S.A. (U.S.P. 1,704,072—3 and 1,704,456, 5.3.29. Appl., 14.11.27).—Citrus or other fruit is treated with a solution of cadmium sulphate and/or chloride.

**Preservation of edible products.** A. G. M. STABACK, and STABAVITE SYND., LTD. (B.P. 304,253, 18.7.27 and 13.12.27. Cf. B.P. 212,979; B., 1924, 489).—The products are covered with a thin layer of gum such as gum tragacanth to which glycerin has been added with or without a preservative, e.g., oil of cloves, an alkali nitrate, or a bisulphite. Sulphuric acid or sodium bicarbonate may be added to the gum solution.

W. J. BOYD.

[Apparatus for] **pasteurising or sterilising milk or other liquids.** N. J. NIELSEN (B.P. 307,854, 12.12.27).

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Volumetric determination of santonin in pharmaceutical preparations.** A. LANGER (Apoth.-Ztg., 1928, 43, 815; Chem. Zentr., 1928, ii, 1597).—Benzene is preferred to chloroform for extraction after preliminary treatment with light petroleum saturated with santonin. A weighed aliquot portion of the filtrate is evaporated; the residue is dried at 80°, weighed, treated with a solution of santonin in light petroleum, dissolved in warm benzene and 93% alcohol, heated under reflux with 0.05*N*-potassium hydroxide, and titrated back with hydrochloric acid.

A. A. ELDRIDGE.

**Colloidal nature of iron scale salts.** D. LOESER (J. Amer. Pharm. Assoc., 1929, 18, 124—128).—Experiments on the injection of non-colloidal, partly colloidal, and completely colloidal ferric salts into white rats show that colloidal iron is much less toxic than non-colloidal iron, the toxicity decreasing as the proportion of the salt in the colloidal state increases. Dialysis experiments with a series of iron scale salts indicated that these salts are partly or wholly colloidal in nature, the amount present in the colloidal state depending on the nature and mode of preparation of the salt.

E. H. SHARPLES.

**Determination of ascaridole in chenopodium oils.** E. KNÄFFL-LENZ and A. HOFMANN (Arch. Pharm., 1929, 267, 117—128).—Biological tests on worms, fish, or white mice are unreliable on account of the antagonistic action of other constituents of the oil. The sp. gr. and refractive index may serve as an index of the ascaridole content of oils of similar origin, but this fails when applied to oils of different origins. The titanous reduction method described by Paget (B., 1926, 462) gives reliable results when an arbitrary factor is introduced, the latter depending on the conditions of the experiment and particularly on the amount of hydrochloric acid used. Ascaridole cannot be determined iodometrically. The intensity of the brown coloration produced by the action of hydrochloric acid on a 1% alcoholic solution of the oil is proportional to the ascaridole content, and this forms the best method of procedure, the oil under examination being compared with a standard ascaridole solution. The colour reaction obtained by heating with phenolphthalein (Langer, A., 1921, i, 259) gives satisfactory results with oils containing above 60% of ascaridole, but fails with oils of poor quality; the same remarks apply also to similar reactions with dimethylaminobenzaldehyde or diphenylamine.

S. COFFEY.

**Bamba oil and the terpenes and higher-boiling fractions of cajuput oil.** D. B. SPOELSTRA (Rec. trav. Chim., 1929, 48, 372—376).—A specimen of bamba oil,  $d_{4}^{20}$  0.9326,  $n_{\text{D}}^{20}$  1.4816,  $\alpha_{\text{D}}$  +33.6°, saponification value 1.8, ester value 3.0, was found to contain *d*- $\alpha$ -pinene (47%), *d*-limonene and dipentene (5%), *d*- $\alpha$ -terpineol (15%), dillapiol (24%), and a small amount of a solid, m.p. 113—118°. *l*- $\alpha$ -Pinene, *l*-limonene, and dipentene were isolated from the lower-boiling fraction of cajuput oil. The higher-boiling fractions of the original oil gave, after hydrolysis with 10% alcoholic potassium hydroxide solution, *i*- $\alpha$ -terpineol, a sesqui-

terpene fraction, b.p. 131—138°/22 mm.,  $d_4^{17.5}$  0.9054,  $n_D^{20.7}$  1.5023,  $\alpha_D +4.3^\circ$ , consisting of a mixture of mono- and di-cyclic hydrocarbons which when dehydrogenated with sulphur affords azulene and cadalene. A sesquiterpene alcohol fraction, b.p. 160—165°/20 mm.,  $d_4^{15.6}$  0.9758,  $n_D^{17.5}$  1.5086, was also obtained. This has the composition  $C_{15}H_{26}O$ , but when it is dehydrated by heating with formic acid a mixture of mono- and di-cyclic hydrocarbons,  $C_{15}H_{24}$ , b.p. 134—139°/16 mm.,  $d_4^{19}$  0.9186,  $n_D^{20}$  1.5126, results. Dehydrogenation of this mixture with sulphur yields cadalene. H. BURTON.

**Finnish turpentine.** ASCHAN.—See XIII.

#### PATENTS.

**Manufacture of derivatives of hydroxy-compounds containing mercury in the nucleus.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 307,532, 8.12.27).—Phenols are mercurated in presence of thiocyanates, ferrocyanides, or ferricyanides, or mercurated phenols are treated with these salts. The products,  $OH \cdot Ar \cdot Hg \cdot X$ , in which  $X = SCN$  or  $\frac{1}{4}$  or  $\frac{1}{8}Fe(CN)_6$ , are bactericides and fungicides.

C. HOLLINS.

**Manufacture of pharmaceutical products [salts of heterocyclic bases].** I. G. FARBENIND. A.-G. (B.P. 298,240, 5.10.28. Ger., 6.10.27).—The salts of heterocyclic bases with carboxylic acids derived from hydroxylated diaryl ketones are almost tasteless, non-irritant, and sufficiently soluble in 30% alcohol. Examples are 3-phenyldihydroquinazoline 2-p-hydroxybenzoylbenzoate, m.p. 187—190° (+EtOH), o-2':4'-dihydroxybenzoylbenzoate, m.p. 119°, and o-4'-hydroxy-naphthoylbenzoate, m.p. 120°.

C. HOLLINS.

**Manufacture of benziminazolonearsinic acids.** I. G. FARBENIND. A.-G. (B.P. 281,703, 2.12.27. Addn. to B.P. 256,243; B., 1927, 670).—The process of the prior patent is extended to the preparation of arsenic acids by the diazo reaction from 5(6)-aminobenziminazolones carrying an alkyl, alkylenyl, or aralkyl group in position 1. 1-Methylbenziminazolone-5-arsinic acid, the 1-ethyl compound, and the intermediate 2:4-dinitroethylaniline, m.p. 119°, 4-nitro-2-aminoethylaniline, m.p. 138—140°, and 5-nitro-1-ethylbenziminazolone, are described.

C. HOLLINS.

**Preparation of a therapeutic product.** C. JAEGER (B.P. 290,195, 18.4.28. Switz., 9.5.27).—A therapeutic product having properties similar to those of cod-liver oil and exceptionally rich in vitamins is prepared by exposing dried banana meal in thin layers to ultraviolet light.

E. LEWKOWITZCH.

**Manufacture of *Raphanus* preparations for medical use.** CHEM. FABR. VORM. SANDOZ (B.P. 292,984, 27.6.28. Ger., 28.6.27. Addn. to B.P. 235,883; B., 1927, 268).—The comminuted roots of *R. sativus* (radish) are treated with adsorptive charcoal and dried as described in the prior patent.

L. A. COLES.

**Manufacture of the hormone of the sexual organs.** SCHERING-KAHLBAUM A.-G. (B.P. 285,402, 6.1.28. Ger., 15.2.27. Addn. to B.P. 265,166; B., 1928, 348).—Material extracted from fresh sexual organs

as described in the prior patent is suspended in aqueous alkaline solution and extracted with ether; the ether residue is then extracted with cold acetone or with methyl alcohol, and the residual lipoids are hydrolysed after removal of the solvent.

L. A. COLES.

**Optic nerve and retina extract.** E. H. STUART, Assr. to E. LILLY & Co. (U.S.P. 1,700,691, 29.1.29. Appl., 8.3.27).—The optic nerves and/or retinas of animals are extracted with cold and then repeatedly with hot alcohol, the extracts mixed, and the alcohol is evaporated. The residue may be dissolved in water.

B. FULLMAN.

**Manufacture of alkamine esters of [N-substituted] o-aminobenzoic acids.** O. EISLEB, Assr. to WINTHROP CHEM. Co. (U.S.P. 1,704,660, 5.3.29. Appl., 11.10.26. Ger., 28.10.25).—See B.P. 260,605; B., 1928, 107.

**Manufacture of *i*-menthol.** K. SCHÖLLKOPF, Assr. to RHEINISCHE KAMPFER-FABR. G.M.B.H. (U.S.P. 1,704,630, 5.3.29. Appl., 26.6.26. Ger., 23.11.21).—See B.P. 189,450; B., 1923, 743 A.

**Production of 1-propenyl-3-ethoxy-4-hydroxybenzene [alkyl ethers of protocatechuic aldehyde].** F. BOEDECKER (U.S.P. 1,704,494, 5.3.29. Appl., 19.11.27. Ger., 14.2.27).—See B.P. 284,199; B., 1928, 547.

**Process for arsenating organic compounds.** E. SCHELLER, Assr. to DEUTS. GOLD- & SILBER-SCHNEIDANSTALT VORM. ROESSLER (U.S.P. 1,704,106, 5.3.29. Appl., 8.11.26. Ger., 15.11.24).—See B.P. 261,026; B., 1928, 140.

**Moth-proofing substance** (U.S.P. 1,694,219).—See V.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Photographic sensitivity.** I. Effect of heat on sensitivity curve of photographic plates. II. Sensitivity of photographic plates at various temperatures. O. MASAKI (Mem. Coll. Sci. Kyōtō, 1929, A, 12, 1—12, 13—22. Cf. A., 1924, ii, 807; Kellner, B., 1926, 387).—I. The effect of a preliminary heating for 1 hr. at 80° on the sensitivity of photographic plates to various wave-lengths has been investigated. With panchromatic and other slow plates, the sensitivity, as measured by the inertia, is increased for all wave-lengths, the effect near a maximum point on the sensitivity-wave-length curve being greater on the side corresponding with longer wave-lengths, *i.e.*, the maximum point is shifted towards the red. The sensitivity of fast plates shows the same shift, but is usually diminished by heating. With all plates and for all wave-lengths the contrast is increased by heating, and for panchromatic and orthochromatic plates the increase is greatest for long wave-lengths. Plates which have been heated gradually regain their normal sensitivity on keeping at the ordinary temperature. Drying the plate has very little effect on its sensitivity.

II. The relation between the temperature of preliminary heating, *t*, and the inertia and contrast,  $\gamma$ , for a given plate and a given wave-length has also been

investigated. The inertia varies most between about  $30^\circ$  and  $70^\circ$ , whilst  $\gamma$  and  $t$  are connected by the empirical expression  $\gamma = e^{at^2 + k}$ , where  $a$  and  $k$  are constants depending on the wave-length of the light to which the plate is exposed, on the time of development, and on the properties of the emulsion. If  $D_m$  is the maximum density at  $0^\circ$ ,  $E$  the length of exposure, and  $D$  the density of the developed plate, it is further found empirically that  $D = D_m e^{at^2} \{1 - e^{-(bt^2 + c)E}\}$ , where  $b$  and  $c$  are constants of the same nature as  $a$ ,  $b$  being negative for some rapid plates and positive for other plates. This formula is valid from  $10^\circ$  to  $80^\circ$ . R. CUTHILL.

**Sensitisation of photographic emulsions by colloidal materials.** B. H. CARROLL and D. HUBBARD (Bur. Stand. J. Res., 1928, 1, 565—588).—Photographic sensitivity is greatly increased by the presence of ultra-microscopic particles on the silver halide grains of an emulsion, and various theoretical explanations are discussed. Emulsions, after ripening and washing, were treated with gelatin-protected sols of silver iodide, silver sulphide, metallic silver, and metallic gold with the object of introducing sensitivity nuclei. Gelatin emulsions with excess of silver salts may be developed normally with certain precautions. The grains carry a positive charge, and are particularly adapted to sensitisation by the negatively charged metallic colloids. All the colloids retarded after-ripening to such an extent that the practical effect was desensitisation. Colloidal silver iodide produced a marked increase in contrast with chemical development, and an increase in speed with physical development. These effects are attributed to increased absorption of the developer. Colloidal gold and silver increased the speed of emulsions with excess of silver. The results are explained in terms of orientation of the photolysis at the sensitivity nuclei formed by the colloidal particle.

C. J. SMITHELLS.

#### PATENTS.

**Photographic film.** H. BRADSHAW, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,703,470, 26.2.29. Appl., 4.3.27).—The rear side of the cellulose film base is coated with a layer of a cellulose carbamate.

J. W. GLASSETT.

**Photographic etching and light-sensitive resist therefor.** A. B. DAVIS, Assr. to KEYSTONE WATCH CASE Co. (U.S.P. 1,703,512—3, 26.2.29. Appl., [A] 30.10.26, [B] 15.11.28).—The resist contains (A) a protein (*e.g.*, glue) and (B) an amino-compound, together with an aliphatic monohydroxy-aldehyde, *e.g.*, aldol, and preferably dichromate.

F. G. CLARKE.

**Colour photography and cinematography.** L. DUFAY (B.P. 307,437, 7.9.27).—A negative taken through a green, violet, and orange network screen is printed on to a sensitised support associated with a screen coloured yellow, blue, and red, the printing taking place through both screens without necessity for the registration of the patterns. The transmission properties of the coloured elements of the positive are so arranged that each element will transmit the light from two of the elements of the green, violet, and orange screen, but will stop the light from the third, namely, that approximating to its complementary colour.

J. W. GLASSETT.

**Reproduction of original cinematograph colour record positive films having a support goffered in lenticular elements.** SOC. FRANÇ. DE CINÉMATOGRAPHIE ET DE PHOTOGRAPHIE FILMS EN COULEURS KELLER-DORIAN (B.P. 285,035, 8.2.26. Fr., 9.2.27).—The contrast of the original positive film is lowered by converting the developed silver either into silver chloride, bromide, or iodide by means of a suitable halogenising bath, or into a blue-toned image by means of a mordant dyeing process. In this manner it is possible to counter-balance the increase in contrast which normally takes place during the reproduction of such films when using undiffused light.

J. W. GLASSETT.

**Reversal process for developing photographic silver sensitised films.** I. G. FARBENIND. A.-G. (B.P. 287,542, 16.1.28. Ger., 25.3.27).—The exposed film is given a short development to ensure that ample silver halide remains for the formation of a strong positive, is then reversed in the usual manner by bleaching and re-development, and finally the image is brought to normal density by treatment with a uniformly acting reducing solution. The following solutions, which are recommended for the reducing process, may also be used for bleaching the negative silver. (A) Iodine 1 g., potassium iodide 2 g., water 200 c.c.; (B) thiocarbamide 4 g., water 100 c.c.; 8 pts. of A are used to 100 of B.

J. W. GLASSETT.

## XXII.—EXPLOSIVES; MATCHES.

**Chemical and ballistic stabilities of BAm and BD powders.** I. ANON. II—IV. DESMAROUX. V. MARQUEYROL (Mém. Poudres, 1928, 23, 79—112, 113—115, 116—123, 124—127, 128—146).—I. The relative advantages of amyl alcohol and diphenylamine as stabilisers for B powder were investigated on three lots of BM powder (French naval nitrocellulose powder) that had been made from the same nitrocotton. One lot (A) was stabilised with 8% of amyl alcohol and two lots (B and C) with 2% of diphenylamine. All these were steeped in cold water for 24 hrs., whilst B had also four steepings of 8 hrs. each at  $80^\circ$ , and C four of 24 hrs. each at  $50^\circ$ . The powders were then placed in cartridge cylinders and subjected to hot dry storage at  $110^\circ$  and hot moist storage at  $60^\circ$  and  $75^\circ$ . Samples were withdrawn at intervals and tested for colour, flexibility, transparency, loss of weight, gas formation, residual velocity, and vivacity. After 175 and 185 days' treatment ballistic tests were carried out on these powders in the 16 cm. gun M.93/96, and the results compared with those obtained from the unheated powder. Samples B and C showed a slight increase in velocity, pressure, and regularity, which was more marked in C than in B, while A had lost about 10% of its velocity and 20% of its pressure. In these tests the weights taken of the treated and untreated powders were equal, but if the results from the treated powders are calculated to an equal weight of the powders before treatment, A, B, and C would all show a slight decrease in ballistic properties.

II—IV. Samples of gas withdrawn from the cylinders after 85 days' heating at  $75^\circ$  were analysed without giving any positive evidence that nitric oxide had

been formed. When samples of A and B were heated in flasks, 0.7% NO was found in the gas from A and 3% from B.

V. Samples of A, B, and C were heated in wide-mouthed bottles, both open and corked, at 75°, both in dry and moist atmospheres. The evolved gases showed that diphenylamine and nitrosodiphenylamine are the best stabilisers at present available for powders with low content of solvent. In thick powders the superiority of diphenylamine over amyl alcohol is not so marked. This fact may be attributed to the action of the solvent.  
S. BINNING.

**Transformation products formed from centralite during storage of SD powder.** H. LÉCORCHÉ and P. L. JOVINET (Mém. Poudres, 1928, 23, 147—154).—The products either not volatile in steam or rendered non-volatile during dissolution of the powder were examined. The only example of the former class present was a mononitro-derivative of centralite (diphenyldiethyl-carbamide), whilst of the second class *p*-nitrophenylethyl-nitrosoamine was detected. The acids formed during the decomposition of the powders are neutralised by bases resulting from the hydrolysis of centralite, which might be effective in nitroglycerin powders, where even feebly basic stabilisers are excluded, if it were not for the fact that apparently it volatilises along with the nitroglycerin.  
S. BINNING.

**Thermodynamic treatment of explosive reactions. II.** A. SCHMIDT (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 90—93).—Considerations previously advanced (B., 1929, 303) are now extended to explosives the oxygen content of which is sufficient for complete gasification, but not for complete combustion to carbon dioxide and water. Nitrocellulose is taken as an example of this class, and it is shown that the composition of its explosion gases is determined, for all practical purposes, by the water-gas reaction, and is independent of the charge-density. The same holds true for the heat of explosion and the explosion temperature. The heat of explosion as calculated by the thermodynamic method is slightly lower than that obtained by calorimetry in an explosion bomb.  
S. BINNING.

**Vapour pressure of nitroglycerin and nitroglycol.** P. NAOUM and K. F. MEYER (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 88—90).—Determinations by a static method gave at 20° vapour pressures of 0.009 and 0.30 mm. Hg for nitroglycerin and nitroglycol respectively. By an aspiration method similar to that used by Marshall and Peace (cf. J.S.C.I., 1916, 35, 298) the vapour pressures at 20° and 35° were 0.011 and 0.036 for nitroglycerin and 0.035 and 0.115 for nitroglycol. The lower values for nitroglycerin obtained by Marshall and Peace are attributed to excessively rapid aspiration. The difference in volatility between nitroglycol and nitroglycerin is greater than would be expected from their relative vapour pressures.  
S. BINNING.

**Ignition of fire damp by explosives.** A. SEGAY (Compt. rend., 1929, 188, 867—868).—The addition of about 25% of salt to amatol lowers the temperature and luminosity of the flame produced on explosion, but since the amount of heat liberated is unaffected it is

assumed that the duration of the flame is increased. The presence in the flame of gases produced as a result of complete detonation (carbon dioxide, nitrogen, water vapour) also inhibits inflammation of fire damp.

J. GRANT.

#### PATENT.

**Manufacture of [book] matches.** S. and H. RAKOWITZKY (GEBR. S. & H. RAKOWITZKY) (B.P. 288,620, 8.12.27. Austr., 15.4.27).

### XXIII.—SANITATION; WATER PURIFICATION.

**Sludge digestion and  $p_H$  control.** H. W. CLARK and G. O. ADAMS (Ind. Eng. Chem., 1929, 21, 258—260).—Laboratory-scale experiments on the digestion of Lawrence sludge showed that the addition of sodium nitrate in amounts equivalent to 2.5 pts. of nitrogen per 100,000 reduced the time required to obtain complete digestion. The volume of gas obtained per gram was also reduced while the proportion of nitrogen therein was above normal owing to reduction of the nitrate. An increase in the hardness of sewage and sludge was also found to reduce the digestion period. Gas production reached a maximum when small amounts of acetic acid and 1% of a strong sulphite-paper waste liquor were added, these being the only experiments in which more gas was obtained than from the control. Addition of calcium carbonate had very little effect on gas production, whilst the addition of 1% of strong wool-scouring liquor definitely retarded it. Other sludges were found to differ very considerably with regard to the digestion process, chemical composition being an important factor. In some cases addition of calcium carbonate was necessary before fermentation would proceed at all rapidly, whilst in others it was unnecessary. Apparently some sludges have more available alkalinity than others, though there is always a tendency to accumulate calcium carbonate as digestion proceeds owing to the gasification of the organic matter. Generally speaking, calcium carbonate is unsuitable as an agent for  $p_H$  control owing to its slight solubility being insufficient to offset the acids produced by the fermentation process. The  $p_H$  value of digesting sludge may be rapidly raised by removing carbon dioxide by aeration.  
C. JEPSON.

**Effect of pressure on sludge digestion.** M. C. WHIPPLE, G. M. FAIR, and L. KLEIN (Ind. Eng. Chem., 1929, 21, 254—256).—A 2:1 mixture of fresh sewage solids and Imhoff tank sludge incubated at 20° under pressures corresponding to 0.6, 1.0, and 1.8 atm. showed very little difference in the time required to obtain complete digestion, though the rate of fermentation in the early stages showed a slight advantage for diminished pressure. The rate of methane production at increased pressure was reduced at first, possibly due to the presence of entrained gases, but there would not appear to be any evidence that deep tanks and the consequent increased pressures would have disadvantages outweighing their advantages with regard to economy of space etc.  
C. JEPSON.

**Complete sludge-washing plant in a single unit.** A. ANABLE (Ind. Eng. Chem., 1929, 21, 223—226).—When economy of floor space is the governing factor, the

standard five-thickener counter-current decantation plant with a washing efficiency of 99% may be replaced by a single multiple-storey tank with a washing efficiency of 97.7%. This unit consists of four superimposed trays, each section being fitted with a Dorr thickener and connected to the one immediately below by a central, sealed opening which permits downward passage of sludge, but prevents the mixing of the wash-waters in the various compartments. The clean wash-water is introduced into the bottom chamber and the sludge to be washed into the top one; the washed sludge is discharged at the bottom and the strong liquor flows over a peripheral sill at the top. C. JEPSON.

**Disposal of industrial wastes.** F. W. MOHLMAN and A. J. BECK (Ind. Eng. Chem., 1929, 21, 205—210).—The wastes from a Chicago maize products factory had a biochemical oxygen demand equivalent to the normal requirements of a population of 350,000. By removal of suspended solids the demand was reduced to a population equivalent of 250,000. The chief waste responsible for this demand was water from the steeping of grain prior to grinding, and as a loss equivalent to 2% of the ground maize was incurred it was considered economical to recover. The steep water was re-circulated until it contained 7% of dissolved solids and was then further concentrated in vacuum pans to a syrupy liquid containing 45%, in which form it could be worked up with other products as a stock food. The condensate from the vacuum pans had a population equivalent of 70,000, but as 80% of the oxygen-consuming substances are contained in the first 15% of distillate, it is hoped shortly to reduce the demand by 50% or more. In another instance the wastes from an oil and paint works seriously interfered with the operation of the Calumet sewage disposal plant, the average daily flow of 1.25 million gallons being approximately 3% of the normal sewage flow. The chief source of trouble was an intermittent discharge of sulphuric acid (5%), but others included copper and arsenic from the manufacture of Paris-green and sodium sulphite from the manufacture of  $\beta$ -naphthol. It has been decided to reduce the effect of the acid by installing a balancing tank and to spread the discharge evenly over the 24 hours, to remove the copper, which has a deleterious effect on nitrification, by means of iron filings with recovery of copper, and to keep out the sulphite liquor and use it as a neutralising agent, discharging the sulphur dioxide through a high stack. It is hoped to reduce the volume to be dealt with to 60,000 gals. per day. C. JEPSON.

**Treatment of industrial wastes from paper mills, [wool scouring,] and tannery on Neponset River.** A. L. FALES (Ind. Eng. Chem., 1929, 21, 216—221).—Two paper-making plants and a wool-scouring and tannery plant discharge their waste waters into the river Neponset. In winter and spring the effluents are discharged after partial purification, but in summer and autumn full treatment is insufficient to keep the river free from putrefaction, and sodium nitrate solution is added when required to prevent this condition. The full treatment at the paper-making plants consists of chemical precipitation followed by filtration, whilst at

the other plant the wool-scouring liquor is cracked with acid to separate the grease, and the aqueous layer resulting therefrom after mixing with the tannery waste is coagulated with aluminium sulphate and dealt with on sand filters. C. JEPSON.

**Chemical treatment of [wool] trade waste.** F. D. SNELL (Ind. Eng. Chem., 1929, 21, 210—213).—Experimental work indicates that, after removal of grit by settlement for an hour, coagulation by means of aluminium sulphate is a more effective method of dealing with wool-scouring waste than the more common one of cracking with sulphuric acid. The effluents thus obtained may be discharged direct to the stream without serious nuisance, and the fat recovered from the sludge by acidification of the cake obtained from either a plate-and-frame press or a vacuum filter. The aluminium thus redissolved may be re-used as a coagulant after neutralisation. The minimum amount of coagulant required to give a satisfactory effluent is 80 lb. per 1000 gals., but anything up to twice this quantity may be required to get a good press-cake for the recovery of fat which may be expected to have a value equivalent to the cost of removal. C. JEPSON.

**Chemical and biological correlations in a polluted stream.** W. RUDOLFS (Ind. Eng. Chem., 1929, 21, 256—258).—A direct relation was found in the Raritan river between the biochemical oxygen demand, the numbers of bacteria and plankton, and the ammoniacal nitrogen content, each increasing in rough proportion until tidal water was reached, when all decreased. The rate of oxygen depletion was greater in summer than in winter, though during the year under review no absolute depletion was recorded. The rate of flow during the year was 40% in excess of the average for the preceding five years, and oxygen saturation depends very largely on the incidence of rainfall. C. JEPSON.

**Sterilisation of water by chlorine.** F. DIÉNERT and P. ETRILLARD (Compt. rend., 1929, 188, 826—829).—In a review of recent experiments no evidence is found for the theory that the bactericidal action of chlorine is due to the emission of "abiotic" rays when that element combines with organic matter present in the water. In one case chlorine was added to river water sterilised by means of a Chamberland filter; when sufficient hyposulphite was added to destroy free chlorine the water had no harmful effect on *B. coli*. G. A. C. GOUGH.

**Bacteriological investigation into the state of pollution of the Clyde at Port Glasgow, Greenock, and Gourock.** D. ELLIS (J. Roy. Tech. Coll., Glasgow, 1929, 2, 129—142).

## PATENT.

**Removing oil from sewage or sludge.** K. IMHOFF (U.S.P. 1,703,041, 19.2.29. Appl., 18.6.27. Ger., 2.12.26).—The sewage or sludge is passed through a tank fitted at the bottom with means for supplying compressed air. The floating oil is collected in lateral channels separated from the main tank by floating beams which have overflow apertures communicating with the sewage level. C. JEPSON.