

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

SEPTEMBER 23, 1927.

### I.—GENERAL; PLANT; MACHINERY.

#### PATENTS.

**Rotary drying apparatus.** J. B. VERNAY (E.P. 262,464, 3.12.26. Conv., 5.12.25).—The apparatus comprises a number of concentric cylinders which are rotated, and through the spaces between which the material passes in zig-zag fashion from the centre outwards. Heated air may be introduced through a hollow shaft, and the material is fed through an opening surrounding the shaft by means of a conveyor device which also divides it into small pieces. If desired, the apparatus may be surrounded by a fixed casing in which a vacuum is maintained.

B. M. VENABLES.

**Drying apparatus.** C. J. HUNT, Assr. to E. G. INFELD (U.S.P. 1,623,540, 5.4.27. Appl., 20.11.26).—A structure contains a tumbling compartment round which air is circulated by being drawn from the sides of the structure and returned to the top. Air is also withdrawn from the lower part of the tumbling compartment.

B. M. VENABLES.

**Method of drying materials.** J. PASQUIERS (E.P. 263,838, 28.12.26. Conv., 28.12.25).—The material is charged into a vessel which has a stirring device near the bottom arranged to give a slight lifting effect. Air for drying is blown through the porous bottom of the vessel, up through the agitated material. The air may be heated by external means or by the "heat of compression" and/or heat may be applied to the vessel.

B. M. VENABLES.

**Drying and storing dried material preparatory to processing.** W. F. HOSFORD, Assr. to WESTERN ELECTRIC Co., Inc. (U.S.P. 1,625,468, 19.4.27. Appl., 26.7.23).—The material is dried in one enclosure and transferred to another enclosure for storage, being maintained all the time independent of atmospheric conditions.

B. M. VENABLES.

**Drying, heating, or the like apparatus.** T. E. WOOD and H. HEYMANN (E.P. 269,064, 4.10.26).—In a dryer of many compartments, suitable for textiles, all the air is delivered by one fan, the delivery of which is divided up into a number of concentric pipes forming annular passages in which steam (for heating) and air alternate. Each chamber is supplied from one air annulus, and provision is made for supplying only new air to each compartment or for partial circulation of old.

B. M. VENABLES.

**Apparatus for recovering solids from press liquids.** H. E. BERGEN (U.S.P. 1,624,385, 12.4.27. Appl., 2.11.25).—The liquid is settled in a vertical cylindrical vessel which has a worm or other conveyor extending right across the bottom and out through a tubular member leading to a gate valve. Beyond

the gate is another independently driven conveyor surrounded by a screen.

B. M. VENABLES.

**Cleaning filtering medium employed in the separation of mixtures of liquids, e.g., oil and water, or mixtures of liquids and solids.** H. M. ALEXANDER (E.P. 268,474, 20.1.26).—Non-inflammable liquids of low b.p. and sp. heat, having sp. gr. greater than that of water, e.g., dichloroethylenes, trichloroethylene, carbon tetrachloride, tetrachloroethane, are forced in liquid or gaseous form through filtering or absorbent media to remove deposited oily matter, after as much as possible of the latter has been previously removed by a reverse flow of steam and/or air. The solvent is subsequently recovered by distillation.

S. S. WOOLF.

**Indicating the presence of submarine objects.** C. W. BONNIKEN and S. BARRATT (E.P. 268,004, 30.11.25, 1.12.25, and 28.9.26).—Flame and smoke are produced at the surface of the water by the ejection from a submerged object of activated phosphine contained in a cylinder. The activating agent is phosphorus dissolved in the phosphine or preferably a nitrogen compound containing oxygen, e.g., nitric oxide, compressed in a similar cylinder and released simultaneously so that the two materials mix as gases. Suitable apparatus and releasing mechanism are described.

W. G. CAREY.

### II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

**Growth of mould fungi in coal.** F. FISCHER and W. FUCHS (Brennstoff-Chem., 1927, 8, 231—233).—Moulds have been observed to grow on raw lignite (not *in situ*), both before and after electrodialysis and with and without the addition of a nutrient medium. Growth occurs also in lignite that has been extracted with boiling water, and is especially vigorous on lignite after extraction with benzene and alcohol.

W. T. K. BRAUNHOLTZ.

**Determination of sulphur in coal.** T. KIDOKORO and Y. KAMIGUCHI (J. Fuel Soc. Japan, 1927, 6, 47—50).—The Probst method (B., 1924, 43) gives higher values than the Eschka method for the sulphur content of Japanese coals, the difference being approximately proportional to the organic sulphur content (difference  $\div$  organic sulphur = 0.242). The error of the Eschka method increases rapidly when the organic sulphur content exceeds 2%. Experiments with semi-cokes indicate that the error is closely associated with the content of non-volatile organic sulphur, and may be greatly diminished if care be taken to oxidise this completely. By the addition of sodium peroxide to the Eschka mixture (up to 2 g. per g. of coal) and subsequently heating for at least 2 hrs. at 800° accurate results are obtained.

A. B. MANNING.



**Improving the quality of Upper Silesian coke.**

G. DÖRFLINGER (Stahl u. Eisen, 1927, 47, 1166—1167).—The coal of the Pochhammer seam in the eastern coal-fields of Upper Silesia is very rich in gas, but has only a poor coking power. A good quality coke can, however, be made from it by the use of narrow retorts and a high temperature if it is previously mixed with about 15% of a bituminous shale or of a fat coal from certain Lower Silesian mines. To test a series of coals for coking power each variety is first separated into the different coal constituents by the float-and-sink method using chloroform-xylene mixtures of different densities; the coking power and percentage of all the fractions are then determined, and from these figures a suitable mixture for feeding to the retorts is readily ascertained.

A. R. POWELL.

**Equilibrium for the reaction  $2\text{CO} = \text{CO}_2 + \text{C}$ .**

A. STANSFIELD (Trans. Amer. Electrochem. Soc., 1927, 51, 423—428).—The results obtained by Rhead and Wheeler (B., 1911, 129) for the equilibrium represented by the above equation are shown to be in error below 850°. The values of Boudouard (A., 1901, ii, 646) are in agreement with the calculated values from 450° to 800°.

L. M. CLARK.

**Constituents of wood spirit and acetone oils.**

IV. H. PRINGSHEIM and A. SCHREIBER [with A. BEISER, W. DOSTER, H. LOOFMANN, G. POJARLIEFF, B. ROSEN, and E. STEINITZ] (Cellulosechem., 1927, 8, 45—66. Cf. Pringsheim and Gorgas, A., 1924, i, 1158; Pringsheim and Bondi, A., 1925, i, 1072).—From the water-soluble portion (15%) of a sample of light oil from wood spirit, b.p. 80—125°, glycol dimethyl ether, allyl alcohol, and crotonyl alcohol were obtained. Glycol dimethyl ether was also present, together with trimethylacetaldehyde, methyl ethyl ketone, methyl propyl ketone, isovaleraldehyde, and cyclopentanone, in the water-soluble fraction (35%) of the heavy oil. The water-insoluble fraction of the heavy oil, b.p. 135—150°, was treated with 50% sodium bisulphite solution, the insoluble portion (53%) was distilled with steam in the presence of dilute sulphuric acid to hydrolyse any esters, and then treated with sodium hydroxide solution to remove acids. On fractional distillation, the product yielded 2-methylcyclopentanone (b.p. 36—38°/11 mm.,  $n_D^{20}$  1.4431) and a fraction, b.p. 49—51°/12 mm., containing an unidentified dimethylcyclopentanone ( $n_D^{20}$  1.4500; semicarbazone, m.p. 185°; oxime, m.p. 58°) and an oil having  $n_D^{20}$  1.4733, which gave a pale red furan reaction, and did not form a semicarbazone. The dimethylcyclopentanone did not condense with cinnamaldehyde or *m*-nitrobenzaldehyde; it therefore contains at least one  $\alpha$ -methyl group. The next fraction contained a ketone  $\text{C}_8\text{H}_{14}\text{O}$ , probably a trimethylcyclopentanone (b.p. 60—63°/12 mm.;  $n_D^{20}$  of freshly distilled sample, 1.4515, increasing after a few days to 1.4588; *p*-nitrophenylhydrazone, m.p. 155—156°; oxime, b.p. 115°/12 mm.,  $n_D^{20}$  1.4940). On oxidation with potassium permanganate and chromic acid the ketone yielded a small amount of *n*-butyric acid. The next fraction of the purified heavy oil consisted of 1:2:4-trimethylbenzene. A portion of the raw heavy oil, b.p. 131—137°, rapidly neutralised sodium hydroxide solution, and, on fractional distillation in a high vacuum

of the acid oil obtained by acidifying the alkaline extract, *n*-valeric acid, which was present in the raw oil as the methyl ester, and small quantities of *n*-hexoic and *n*-heptoic acids, characterised as the silver salts, were isolated. The higher fractions yielded the lactones of three unknown acids, the silver salts of which were prepared and analysed:  $\text{C}_9\text{H}_{18}\text{O}_4$ , lactone, b.p. 97—100°/3 mm.,  $n_D^{20}$  1.4728;  $\text{C}_{11}\text{H}_{20}\text{O}_4$ , lactone, b.p. 99—100°/0.3 mm.,  $n_D^{20}$  1.4775;  $\text{C}_{13}\text{H}_{22}\text{O}_4$ , lactone, b.p. 113—115°/0.6 mm.,  $n_D^{20}$  1.4884. Another sample of the crude oil yielded an acid fraction, b.p. 143—150°/16 mm., which was neutralised with ammonia and extracted with ether. On addition of silver nitrate to the aqueous layer, silver  $\gamma$ -hydroxyvalerate was precipitated, indicating the presence of valerolactone. The ethereal solution afforded an unsaturated lactone  $\text{C}_{13}\text{H}_{20}\text{O}_2$ , b.p. 95—107°/0.3 mm., which, after prolonged boiling with dilute sodium hydroxide solution, was partly converted into the free acid,  $\text{C}_{12}\text{H}_{22}\text{O}_3$ , isolated as the silver salt. Propionic acid and an acid  $\text{C}_9\text{H}_{16}\text{O}_4$  of unknown constitution were obtained from this lactone by oxidation with potassium permanganate; it may, therefore, be represented by  $\text{CH}_3\text{Me}\cdot\text{CH}:\text{C}_9\text{H}_{14}\text{O}_2$ . The corresponding ketonic acid  $\text{C}_{12}\text{H}_{20}\text{O}_3$ , b.p. 130°/14 mm., was also obtained by oxidation of the lactone. In another experiment 400 c.c. of acid oil obtained from 10 litres of crude heavy oil were carefully fractionated. The fraction of b.p. 202—205° contained  $\Delta^8$ -isohexenoic acid, the silver salt of which was prepared, and which was characterised by oxidation with potassium permanganate, which yielded isobutyric and oxalic acids. The fraction of b.p. 218—223° consisted of  $\Delta^8$ -heptenoic acid, characterised by means of the silver salt and by the formation of glutaric and acetic acids on oxidation. The higher fractions contained the lactone  $\text{C}_{12}\text{H}_{20}\text{O}_2$  already mentioned, together with a lactone  $\text{C}_{14}\text{H}_{24}\text{O}_2$ , b.p. 120—126°/0.3 mm., which was not obtained in a pure state, but was converted into the silver salt of the corresponding acid, and a lactone  $\text{C}_{17}\text{H}_{26}\text{O}_3$ , b.p. 137—147°/0.3 mm., from which the silver salt was prepared. The quantities of these lactones obtained were insufficient for a determination of their constitutions. The light acetone oil used for this investigation had  $d_4^{21}$  0.841,  $n_D^{20}$  1.4029, and contained 13% of water. The dry oil had  $d_4^{21}$  0.848;  $n_D^{20}$  1.4030; 5 c.c. required 2.2 c.c. of 0.1*N*-sodium hydroxide solution to neutralise free acid, and 16.01 c.c. to hydrolyse the esters present. The oil was first freed from acids and esters by means of sodium hydroxide solution. The ketones were separated from traces of aldehydes by converting them into the bisulphite compounds and decomposing the latter in weakly alkaline solution. The product so obtained was then fractionally distilled. The following ketones were identified and characterised by means of the semicarbazone or *p*-nitrophenylhydrazone: acetone, methyl ethyl ketone, methyl *n*-propyl ketone (b.p. 101—103°), ethyl isopropyl ketone (b.p. 113—117°; the *p*-nitrophenylhydrazone has m.p. 84—86°, is unstable in air, and even in a vacuum desiccator rapidly becomes a dark brown viscous mass), and a small amount of ethyl *n*-propyl ketone (semicarbazone, m.p. 113°). A sample of heavy or "yellow" acetone oil was treated with sodium bisulphite solution, and the bisulphite compounds were decomposed with dilute acid.



On fractionation of the resulting oils, the following substances were isolated: 3(?)-methylcyclohexanone, b.p. 64–66°/15 mm.,  $n_D^{20}$  1.4395, 1:3(?)-dimethylcyclohexan-2-one, b.p. 70–75°/15 mm.,  $n_D^{20}$  1.4484, 1:1:3(?)-trimethylcyclohexan-4-one, b.p. 77–80°/15 mm.,  $n_D^{20}$  1.4595, dimethylcyclohexenone (unidentified), b.p. 83–85°/15 mm.,  $n_D^{20}$  1.4671, 1:1:2-trimethyl- $\Delta^2$ -cyclohexen-4-one, b.p. 90–91°/15 mm.,  $n_D^{20}$  1.4827. The sodium bisulphite solution, after removal of the bisulphite compounds and unchanged oil, was made alkaline and steam-distilled under diminished pressure. On extraction of the distillate with ether and removal of the solvent, the extract was fractionally distilled. The following were isolated: heptaldehyde, characterised by preparation of the semicarbazone, m.p. 108–109°; an aldehyde of unknown constitution,  $C_8H_{16}O$ , b.p. 63–64°/18 mm.,  $n_D^{20}$  1.4362 (semicarbazone, m.p. 145–147°); cyclohexanecarbaldehyde, b.p. 76–77°/18 mm.,  $n_D^{20}$  1.4441 (semicarbazone, m.p. 164–166°), and  $\beta$ -cyclohexylpropaldehyde, b.p. 87–89°/18 mm.,  $n_D^{24}$  1.4630 (semicarbazone m.p. 132–134°).

W. J. POWELL.

**Jet and jetonised material.** E. H. C. CRAIG (J. Inst. Petrol. Tech., 1927, 13, 343–362).—Spanish jet showed a much flattened vegetable structure, and appeared to be derived from wood from a conifer. Whitby jet showed far less distinct vegetable structure. When jet was retorted in a Struben still, 3% distilled as water at 100°, 5% as water between 160° and 250°, 19% as light oil ( $d$  0.792) below 300°, waxy oil at 300–320°; the total yield of oil and wax was 57.8 gals. per ton. 7.7% of powdered jet was soluble in kerosene. The examination of several lignites and a torbanite indicated that jetonised material is an intermediate stage between wood (or other vegetable matter) and coal or petroleum. Lignites are more closely related to coal than is jet.

W. N. HOYTE.

**Comparison of qualities of asphalts of German and Mexican origins.** L. ROSNER (Petroleum, 1927, 23, 609–610).—German Elwerath crude oil and Panuco crude oil were distilled to asphalt in a pipe still without vacuum at 400–420°. The respective properties of the oils were: sulphur 0.55% and 5.8%;  $d^{15}$  0.918 and 0.986; wax content, 1.2% and 0.2%, and asphaltum insoluble in benzine 0.1% and 16.4%. The asphalt from the Panuco crude is the more valuable, having higher drop point (Ubbelohde) and lower penetration and breaking points. The respective yields of asphalt were 8.00% and 50.60%.

H. MOORE.

**Action of gases on glasses etc.** FABER.—See VIII.

**Oils in emulsions.** BALDWIN.—See X.

#### PATENTS.

**Coke oven.** T. G. KUS, Assr. to AMERICAN COKE & CHEMICAL CO. (U.S.P. 1,635,679, 12.7.27. Appl., 4.10.19).—A coke oven has regenerators arranged beneath and parallel to the coking chambers, flues connecting alternate regenerators with the bottoms of the combustion chambers, flues connecting intermediate regenerators with the tops of the combustion chambers, and burners in the tops and bottoms of the combustion chambers.

A. B. MANNING.

**Coke oven.** E. COPPÉE & C<sup>ie</sup>. (E.P. 253,887, 4.6.26 and 273,630, 2.5.27. Conv., 22.6.25 and 4.9.26).—The wall of the oven comprises an even number of groups of vertical flues, each of which is connected to its neighbour by a communication chamber. These chambers are arranged alternately at the top and bottom of the wall. The whole of the air for combustion is admitted in the outer group of flues, but the gas stream is divided, part entering each flue in the first group and part into the path of the burning gases through the ducts serving a group of flues. The means for admitting the gases to the upper communication chambers of two adjacent groups of flues is situated near to the dividing wall between the groups. At the base of the outer groups only, primary gas may be admitted through a duct short enough to preclude the formation of graphite.

R. A. A. TAYLOR.

**Apparatus for coking coal.** URBANA COKE CORP., Assees. of S. W. PARR and T. E. LAYNG (E.P. 256,192, 19.6.26. Conv., 30.7.25).—Coal is fed by screw conveyor through an inclined cylinder heated by waste gas or by gas or oil burners at a temperature below the plastic stage of the coal (cf. E.P. 249,886; B., 1927, 35), and thence into a heat-insulated hopper or direct into retorts of calorised iron or iron-chromium-nickel alloy. These are heated by gas or oil burners and the gases after combustion in outer vertical flues pass into direct contact with the retorts and thence to the coal preheater. The process is particularly applicable for the production of coke from non-coking coals.

A. C. MONKHOUSE.

**Fuels for use in internal-combustion engines and for other purposes.** E. G. E. MEYER (E.P. 269,222, 3.12.25. Cf. E.P. 262,363; B., 1927, 134).—Liquid hydrocarbons of relatively high b.p. and substantially devoid of constituents boiling below 100°, and of which 80–100% distils over at temperatures up to 300°, are used mixed with a small quantity of volatile basic material (e.g., ammonia) and ethyl ether.

R. A. A. TAYLOR.

**Production of gas.** O. U. BEAN (E.P. 269,004, 14.6.26).—Steam is admitted to a chamber, and so heated that it is dissociated into its elements. A catalyst may be used to promote this reaction. A hydrocarbon vapour is then admixed with the hydrogen and oxygen, and the mixture is subjected to the influence of the elevated temperature, fluctuations in pressure, and, if necessary, of a catalyst. A gaseous fuel resembling natural gas results, together with the possible formation of light hydrocarbons and carbon black. Free oxygen appears in the gaseous product, and may be separated.

R. A. A. TAYLOR.

**Low-temperature distillation of bituminous substances.** KOHLENVEREDLUNG GES.M.B.H. (E.P. 244,456, 5.12.25. Conv., 11.12.24).—A protective coating of aluminium or of an aluminium alloy lines the iron retort and prevents corrosion of it by the products of distillation.

R. A. A. TAYLOR.

**Distillation of coal at low temperatures.** COMP. DES MINES DE VICOIGNE, NŒUX, ET DROCOURT (E.P. 255,411, 20.1.26. Conv., 17.7.25).—Fine coal and a tar or oil are mixed and moulded. They are then distilled in chambers by the passage over them of a hot



atmosphere of steam or other gas poor in oxygen, the temperature rising progressively to 500–600° in 3–12 hrs., when a good smokeless fuel results.

R. A. A. TAYLOR.

**Manufacture of amorphous carbon etc.** J. W. MACDONALD, Assr. to W. L. RUCKER (U.S.P. 1,626,418, 26.4.27. Appl., 14.12.25).—Ozokerite, paraffin, distillates, etc. are burnt by melting in a vessel which communicates by means of a valved pipe with a burner contained in a closed housing. C. O. HARVEY.

**Polymerisation of oils.** BARRETT Co., Assees. of S. P. MILLER (E.P. 246,491, 22.1.26. Conv., 23.1.25).—The cooled oil is introduced into a vessel in which it is agitated with sulphuric acid, aluminium chloride, or other polymerising agent, the requisite amount of which is added by means of an automatic device. After treatment the mixture is withdrawn, and the spent polymerising agent, together with any precipitated tar, is separated from the oil, which may then be washed and neutralised with alkali. The oil is treated in successive batches, all the operations, however, being automatically controlled so that in effect the process is continuous.

A. B. MANNING.

**Expelling sulphur dioxide gases from mixtures of sulphur dioxide gases and oil.** ALLGEM. GES. FÜR CHEM. IND. M.B.H. (E.P. 269,118, 14.5.26. Conv., 12.4.26).—The mixture flows through a vertical pipe, externally heated by steam or hot air, and then passes into a superposed horizontal drum, so that the sulphur dioxide is driven off by the heated oil in the vertical pipe and is entirely separated from the oil in the drum.

W. G. CAREY.

**Apparatus for cooling coke.** HUMPHREYS & GLASGOW, LTD., Assees. of W. T. BOSLER (E.P. 264,796, 2.11.26. Conv., 20.1.26).

**Heating walls for coke ovens.** KOKSOFFENBAU U. GASVERWERTUNG A.-G. (E.P. 270,232, 25.8.26. Conv., 28.4.26).

**Generating the supplementary steam for gas producers.** RASSELSTEINER EISENWERKS-GES., A.-G., and A. BIEN (E.P. 272,725, 9.9.26).

**Gasification of solid fuels.** L. CHAVANNE (E.P. 266,656, 10.2.26. Addn. to E.P. 247,571).

**Compositions for roadways** (E.P. 268,950).—See IX.

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

**Calcium sulphate. III. New methods of analysis and use of plaster.** L. CHASSEVENT (Ann. Chim., 1927, [x], 7, 43–68).—A calorimetric method (cf. B., 1927, 676) for the analysis of plasters is developed, by which the course of the heating of the plaster may be followed, and a knowledge of its ultimate properties rapidly obtained. Anhydrous calcium sulphate, dehydrated below 300°, is rehydrated practically instantaneously, and a measurement of the heat evolved in g.-cal. during the first 2–3 min. (X) is due to this cause, so that the weight in grams (*p*) of anhydrous calcium sulphate in the sample is given by the expression  $p = (X - 20)/20$ , the heat of dissolution of the hemi-

hydrate being 20 g.-cal. Calcium sulphate dehydrated between 300° and 600° requires 7 days for complete rehydration, and the period necessary for any particular sample may be determined either (1) by measuring the electrical resistance which, when the plaster contains no calcium sulphate dehydrated above 300°, does not increase after several hours; or (2) by a determination of the increase in weight of a known weight of the powder kept under water and dried at 50–60° after periods of 2, 7, and 15 days; all increase in weight between 2–7 days corresponds with the hydration of calcium sulphate which has been heated between 300° and 600°, and any increase after 7 days with that heated above 600°, 1 g. of anhydrous calcium sulphate requiring 0.26 g. of water for hydration. By this method the influence of the method of heating on various samples has been investigated. With samples prepared from precipitated calcium sulphate with insufficient heating a smooth temperature-time curve is obtained showing that hydration is continuous, but as the period of heating is increased a step curve results, the initial evolution of heat corresponding with the hydration of the anhydrous calcium sulphate to the hemihydrate, no further evolution of heat then occurring until crystallisation of the dihydrate sets in. With a plaster prepared from gypsum at 145° crystallisation sets in rapidly owing to the presence of seeds of the dihydrate being present, but removal of these by heating at 200° lengthens the period before crystallisation sets in. A good commercial plaster shows a steep step curve. A sample of dead plaster (which had been kept for 2 years) does not set owing to the formation of a skin of the dihydrate over each grain, which prevents diffusion of the supersaturated solution of the hemihydrate throughout the mass, each grain setting separately. The plaster may be restored by complete dehydration at 220°. The lack of hardness in set plaster has been investigated, and may be due either to the presence of overburnt particles which rehydrate only slowly, or to the addition of too much water, the best proportion being 45–60 g. per 100 g. of plaster. By mixing plaster with only slightly more than the theoretical quantity of water required for hydration at 80–90° and subjecting the mass to pressure (varying with the conditions and the size of the particles employed), samples having an apparent density greater than 2, a high resistance to compression, and an appearance like marble have been obtained. An attempt thus to prepare artificial alabaster was unsuccessful, the samples always being opaque and having a density less than 2.3. If plaster is hydrated with boiling water, care being taken that the temperature of the mass never falls below 63°, and the liquid mass is stored in steam-jacketed vessels, it remains plastic for many hours, but on allowing the temperature to fall to 40° it sets in the usual manner. By this means a smaller quantity of water is required for hydration, the plaster may be stored during use, and machinery employed to mix and work it.

J. W. BAKER.

### PATENTS.

**Removal of cyanides from masses containing them.** C. B. JACOBS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,623,599, 5.4.27. Appl., 10.3.21).—The residue from the treatment of a mixture of alkaline



carbonate, carbon, and ferric oxide with nitrogen at high temperatures is treated at 40° with sufficient mother-liquor from a previous extraction to dissolve the alkali cyanide present. The extracting liquid is removed from the residue by centrifuging, followed by pressing at about 270 atm. T. S. WHEELER.

**Removing deterrent salts from pickling liquors.** G. S. MORGAN (U.S.P. 1,626,623, 3.5.27. Appl., 30.6.26).—Sulphuric acid is added to the solution, to precipitate iron sulphate, until the acid content reaches 35–50% of the solution; the liquor is then drawn off and the precipitate dissolved in hot exhausted liquor and cooled to form copperas. H. ROYAL-DAWSON.

**Production of articles containing silicon carbide.** GEER. SIEMENS & Co. (G.P. 438,065, 13.5.24).—A plastic mass containing tragacanth, alone or mixed with water-glass, and powdered silicon carbide, with or without the addition of other material, such as mixtures of carbon with silica, silicon oxychloride, or silica, is moulded into the desired shape. L. A. COLES.

**Preventing explosions in chambers used for grinding calcium carbide.** GRÄFLICH SCHAFF-GOTSCH'SCHE WERKE G.M.B.H. (G.P. 437,750, 18.9.25).—Combustion gases obtained by passing dry air through glowing coke are passed through the chambers at a temperature above 150°. L. A. COLES.

### VIII.—GLASS; CERAMICS.

**Testing glass vessels for holding drugs according to the specifications of the German Pharmacopœia, 6th ed.** L. KROEBER (Apoth.-Ztg., 1926, 41, 1406–1407; Chem. Zentr., 1927, 1, 785–786).—Glass vessels in which no formation of fine crystals takes place for at least  $\frac{1}{2}$  hr. on filling with freshly prepared and filtered 0.1% narcotine hydrochloride solution, are suitable for most purposes, whilst vessels in which no crystals appear after 3 days are suitable for the most stringent use. Medicine bottle glass should be suitable for general use if it does not discolour a test solution containing 3 c.c. of 0.1N-hydrochloric acid and 5 drops of methyl red per litre, on heating for  $\frac{1}{2}$  hr. on the water-bath. L. A. COLES.

**Abstraction of sulphur dioxide from flame gases by glasses and glazes.** A. FABER (Gas- u. Wasser-fach, 1927, 70, 419–422).—Summarising available evidence, it could be said that the surface action on glasses and glazes which occurred with gases containing sulphur and under fixed firing conditions, depended upon the concentration and the state of oxidation of the sulphur compounds. The composition of the glass was of importance, whilst the velocity of the gas and the temperature had been found to affect the results obtained. The conclusions of Travers on the action of sulphur trioxide on glass (B., 1921, 510A) had been confirmed by Berdel, Ortmann, Zschimmer, and others. In technical furnaces the action of sulphur could be avoided by adjusting the firing and working conditions, i.e., by a reducing atmosphere at first, followed by oxidising conditions. A. COUSEN.

**Influence of wet grog on the properties of saggars and other refractory goods.** J. KONARZEWSKI (Trans.

Ceram. Soc., 1926, 25, 386–393).—Two sets of grog fireclay bricks, one with dry grog and the other with wet, were prepared and fired in a commercial kiln to cone 10. Mechanical analysis of the grog showed it to contain 70% on the 8-in. sieve and 26% on the 20-in. sieve. Data concerning the contraction (wet to dry, dry to fired, wet to fired), sp. gr., porosity, tensile strength, cold crushing strength, and resistance to deformation at high temperatures, were obtained. The results show that the use of wet grog improves the properties of firebricks. The explanation appears to lie in the fact that clay sticks to wet grog much better than dry grog, and, in consequence, the particles of the brick are cemented more closely together during firing. A. T. GREEN.

**Refractory linings for cement kilns.** G. MARTIN (Trans. Ceram. Soc., 1926, 25, 394–399).—A brief description of the process of burning cement in relation to the refractory linings of the rotary kiln is given. A cheap firebrick of fairly dense texture is satisfactory for the lining of the drying zone. The abrasive action experienced in the preheating zone makes a dense, non-porous firebrick essential for this portion. In the calcining zone the lining must be capable of withstanding the corroding action of lime at high temperatures. A satisfactory firebrick for this zone contains 62% SiO<sub>2</sub> and 32% Al<sub>2</sub>O<sub>3</sub>, a more highly siliceous product being undesirable. The brickwork in the clinkering zone is subjected to intense corrosion and abrasion by alkaline materials in continual movement, and to severe strains set up by the rotation of the kiln. There is diversity of opinion concerning the class of product for this zone. Alternate layers of high and low alumina brick have been used; the more siliceous bricks, being more deeply corroded, serve as holders for the attached cement clinker. Bauxite bricks, in a large-scale experiment, proved very satisfactory. In another instance, however, this material gave trouble through shrinkage. A kiln lined with firebricks, on which was placed a layer of material consisting of 2 pts. of small cement clinker and 1 pt. of cement, has given good service. A. T. GREEN.

### IX.—BUILDING MATERIALS.

**Calcium sulphate.** CHASSEVENT.—See VII.

**Linings for cement kilns.** MARTIN.—See VIII.

#### PATENTS.

**Magnesite cement.** J. AGERUP (E.P. 268,989, 17.5.26).—Ferrous chloride is rendered anhydrous by drying the crystallised chloride or by rapid evaporation of a solution of the salt, and the ground product intimately mixed with magnesium oxide and suitable fillers, forming a magnesite cement which can be stored or transported without deterioration and used without skilled labour. B. W. CLARKE.

**Manufacture of slag cement.** J. S. HARDING, Assr. to W. R. CASPARIS and S. E. STEPHENSON (U.S.P. 1,627,237, 3.5.27. Appl., 13.1.23).—Lime is mixed with 70% or more of undried granulated slag and 3% or less of gypsum; after thoroughly drying the mixture, about 5% of cement clinker is added, and the whole is pulverised. H. ROYAL-DAWSON.



**Tar or tarry compositions applicable for roadways and the like.** R. O. CHILD (E.P. 268,950, 23.3.26).—Coal tar or coal-tar pitch, dissolved in a suitable solvent, is agitated at 82° or less with partially coagulated casein, prepared according to E.P. 238,586 (B., 1925, 851) by treatment with sulphated oil with or without the addition of formaldehyde, the casein being rendered homogenous by a preliminary treatment with a 5% Glauber's salt solution if necessary. B. W. CLARKE.

**Production of a plastic composition and articles therefrom.** A. J. SLOSSER, Assr. to POMPEIAN FLOORING Co. (U.S.P. 1,627,296, 3.5.27. Appl., 20.4.26).—A liquid binder solution of magnesium chloride is added slowly to a mixture containing approximately 45% by weight of vegetable fibrous material, 30% of asbestos fibre, and 25% of caustic calcined magnesia, producing a plastic mass which is spread or moulded into the desired form and allowed to set and harden.

H. ROYAL-DAWSON.

**Manufacture of plastic composition.** J. F. HAGGERTY (E.P. 260,597, 18.9.26. Conv., 27.10.25).—Finely-divided fibrous material, *e.g.*, newspaper pulp, saturated with water, in the form of a flocculent mass in which the dry weight of the fibres is not more than 10% of the weight of water, is mixed with plaster of Paris, so that the quantity of dry fibrous material is approximately 5% of that of the plaster. The product may be moulded, and is lighter and tougher than ordinary gypsum products. B. W. CLARKE.

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

**Influence of boundary films on corrosive action.** L. H. CALLENDAR (Proc. Roy. Soc., 1927, A 115, 349—372; cf. B., 1926, 328).—The local corrosion of metals in normal waters is governed by boundary films of a solid, liquid, or gaseous nature present on the metal surface, and is liable to occur wherever a metal tends to form oxide films or scales of a higher potential than the metal itself. The location of the primary cathode and anode areas on the metal depends upon the distribution of these films, which are determined by the presence of foreign substances on the metal surface (dust, grease, moisture, etc.) and by irregularities in the surface itself. When the metal is immersed in an electrolyte, the oxide film is the primary cathode and metal passes into solution at unoxidised parts of the surface, but subsequently the original location of the cathode and anode areas may be altered by variations in the oxygen concentration, by the spreading of the products of corrosion over the metal surface, and by other factors. The rate of corrosion is indicated by the "boundary resistance," *i.e.*, the resistance in ohms of the boundary films between the electrodes and the electrolyte, obtained by subtracting the resistance of the electrolyte from the total internal resistance of the experimental corrosion cell. The electrical method used for measuring the changes in resistance and *E.M.F.* was similar to that previously described (*loc. cit.*), the corrosion cell consisting of an aluminium anode and a platinum or oxidised aluminium cathode with various dilute salt solutions as electrolytes. The normal cathodic oxide film formed

in air is so thin that it has little effect on the rate of corrosion, but the thicker films formed by heating give a high boundary resistance and must tend to reduce the rate of corrosion. Oxidising electrolytes also retard corrosion by increasing boundary resistance. With aluminium, the boundary resistance increases both with the thickness of the film and with the increasing dilution of the electrolyte. L. L. BIRCUMSHAW.

**Comparison of the alloying elements chromium, nickel, molybdenum, and vanadium in structural steels.** H. J. FRENCH (Trans. Amer. Soc. Steel Treat., 1927, 11, 845—884).—The low-alloy structural steels are shown to be interchangeable to a large degree, and the relation fatigue limit—tensile strength for the various steels may be made by suitable treatment to fall on the same curve. Molybdenum produces uniform hardness over large sections. The load-carrying capacity at high temperatures and resistance to chemical reagents of various high-chromium-nickel alloys are given. A chart shows the effect of chromium in decreasing the attack of hydrogen sulphide on iron; with increasing nickel the loss of weight rises. Under certain conditions nickel and chromium increase the resistance of steels to wear, and cast railway crossings are now being made of steel containing 0.50% C, 0.75% Cr, and 2.75% Ni. T. H. BURNHAM.

**Tensile properties of boiler plates at temperatures between 20° and 600°.** G. URBANCZYK (Stahl u. Eisen, 1927, 47, 1128—1135).—The mechanical properties between 20° and 600° of four typical boiler plate steels have been determined after annealing for 1 hr. at 920° and cooling slowly. The elastic limit remains constant up to 220° and then falls slowly with rise of temperature, the tensile strength increases slowly to a maximum at about 275° and then falls rapidly, and the elongation decreases slowly to a minimum at 250—275°, then increases slowly. The impact strength reaches its maximum at 120° and a minimum at 500—600°. Tables showing the composition and variation of the mechanical properties with the temperature are given together with graphs for all the steels tested. A. R. POWELL.

**X-Ray studies on the decomposition of austenite.** O. E. HARDER and R. L. DODWELL (Trans. Amer. Soc. Steel Treat., 1927, 11, 975—985).—X-Ray analysis of powdered austenitic steels showed that the austenitic structure was partially destroyed by filing. The presence of undissolved carbides was objectionable as they gave a large number of lines interfering with the  $\alpha$ - and  $\gamma$ -iron lines. Although micro-examination indicated a definite recrystallisation from  $\gamma$ - to  $\alpha$ -iron after 70 hrs.' immersion in liquid air, no increase in intensity of the  $\alpha$ -pattern was found. It is suggested that the  $\alpha$ -crystal nuclei do not grow to sufficient size to give an X-ray diffraction pattern. X-Ray and micro-examinations also gave discordant results on the effect of stress on austenitic structure. When the  $\alpha$ -pattern was present in quenched steels its intensity increased on tempering at 100°. The  $\gamma$ -pattern completely disappeared on tempering at 190° except in manganese and high-nickel steels. The  $\gamma$ -lattice contracted on immersion in liquid oxygen and the  $\alpha$ -lattice increased after tempering at 190°, so that there was a hysteresis effect in change of para-



meter with change of temperature. It is unlikely that cementite is dissolved as a unit in austenite or martensite. Density determinations from X-ray data indicate that carbon atoms cannot replace iron atoms in the  $\gamma$ -lattice but must occupy positions inside it. T. H. BURNHAM.

**Growth of cast iron.** W. SCHWINNING and H. FLÖSSNER (*Stahl u. Eisen*, 1927, **47**, 1075—1079).—Grey cast iron begins to grow after heating at 450°, and the growth is much more marked at 550°. Repeated heating at 550° for periods of 3 hrs. followed by cooling results in a slow permanent growth at first, accompanied by a gradual change in the microstructure characterised by an intergrowth of the pearlite and cementite regions and a slow decomposition of the cementite lamellæ along their edges. After a certain number of heatings (10—20), depending on the composition of the iron, rapid decomposition of the cementite occurs accompanied by a marked increase in the volume of the metal and in the number and size of the graphite inclusions. At 650° this decomposition is extremely rapid, and takes place during the first 2—3 heatings. A. R. POWELL.

**Light aluminium alloys.** L. LOSANA and E. FROVA (*Giorn. Chim. Ind. Appl.*, 1927, **9**, 111—115).—Various ternary and quaternary systems containing aluminium as principal component have been studied. Aluminium alloys rich in silicon and containing also copper give fused parts of good mechanical properties. That with 3% Cu and 2% Si exhibits a tensile strength of about 17 kg./mm.<sup>2</sup> and an elongation of 6%; these results are superior to those given by the aluminium-copper alloy with 6% Cu, the latter being also the heavier. Aluminium alloys rich in silicon and containing also zinc or tin have low breaking strengths and are, in many cases, brittle. Silicon-rich aluminium alloys containing magnesium have very low densities and excellent tensile strengths, but their elongations are poor. With aluminium-copper-magnesium-silicon alloys a tensile strength of 20 (17—18) kg./mm.<sup>2</sup> and an elongation of 3—3.5 (about 8)% are obtainable. To binary aluminium-copper alloys not more than 2% of zinc can be added without reducing the elongation excessively, whereas addition of tin lowers the breaking stress without raising the elongation markedly, and introduction of magnesium to the extent of 2% increases the tensile strength, but lessens the elongation somewhat. Hence, addition of zinc or tin to the ordinary aluminium-copper alloys containing 6% Cu is of no great practical advantage. T. H. POPE.

**Analysis of antimony alloys.** H. VIGNAL (*Ann. Chim. anal.*, 1927, [ii], **9**, 193—196).—For the determination of metals other than tin 1 g. of alloy is digested with gentle heat with 40 c.c. of 50% tartaric acid and 15 c.c. of nitric acid, and 10 c.c. of 50% sulphuric acid and about 15 c.c. of water are added to the clear mixture. After 12 hrs. the liquid is filtered and the lead salt weighed. Copper is then determined by adding caustic soda in excess, boiling, adding 0.5 g. of lactose in small portions, and boiling till the precipitate of cuprous oxide is well formed. To determine iron and zinc the filtrate is treated with hydrogen sulphide. The precipitate, now free from alkali salts, is added to the copper precipitate, calcined, dissolved in nitric acid, and the solution diluted

and electrolysed, when any remaining lead is practically entirely deposited. 10 c.c. of 50% sulphuric acid are then added and electrolysis is continued until the copper is completely deposited. Iron and zinc left in solution are determined by the usual methods. The alkaline filtrate containing the tin and antimony is neutralised till the precipitated sulphates are only redissolved with difficulty, 30 g. of oxalic acid are added, and the mixture is boiled for 1—2 hrs. with hydrogen sulphide passing through. When precipitation appears complete a few drops of ammonium sulphide are added, the mixture is filtered hot, washed, the precipitate redissolved in hydrochloric acid, potassium chlorate added, the whole boiled, filtered, a few more crystals of chlorate are added, and boiling is continued till no odour of chlorine remains. After cooling to 25—30° and addition of 2—3 g. of potassium iodide, the solution is titrated with sodium thiosulphate (49.5 g. per litre); antimony sulphide is then precipitated. Antimony may be determined directly if lead and tin are present, or only lead, but not in the presence of copper. In the first case the alloy is dissolved in nitric acid, and after calcination the oxides are treated with melted potassium hydroxide in a nickel crucible, redissolved in hydrochloric acid, and the solution is boiled and treated as above. In the absence of tin, antimony oxide is not completely insoluble in nitric acid, so that 0.1—0.2 g. of pure tin should be added. For the determination of tin when over 50% is present, dissolution is effected as for an alloy of lead, tin, and antimony, and the hydrochloric acid solution of oxides, with 1 g. of iron wire, boiled under a reflux condenser until the iron is completely dissolved, antimony precipitated, and tin in the stannous state. 5—6 c.c. of antimony chloride are added (about 0.2 g. Sb) and more iron wire, and boiling is continued. After adding boiling water and filtering, the precipitate of antimony is rapidly washed, reheated, and the stannous chloride titrated in the boiling filtrate with 0.5N-potassium permanganate solution.

D. G. HEWER.

**X-Ray investigation of the internal stress in metals [copper].** S. SEKITO (*Sci. Rep. Tôhoku Imp. Univ.*, 1927, **16**, 343—355).—Copper wire was cold-drawn, and the internal stress present then, and after annealing at different temperatures, was determined from the breadth of the lines in its X-ray spectrum. When reduced to 2% of its initial cross-section by drawing, the internal stress corresponds to a widening of 0.3% of the original lattice-parameter, the directions of greatest stress being [111] and [100]. The breadth of the spectral lines decreases as the annealing temperature is raised.

W. HUME-ROTHERY.

**Determination of very low concentrations of volatile oils in emulsions. [Separation of minerals].** E. J. BALDWIN (*Ind. Eng. Chem.*, 1927, **19**, 78—81).—Distilled water is introduced into a 500 c.c. separating funnel, and 10 g. of galena, sifted to 35—48-mesh, are introduced for each 100 c.c. of water taken. The desired amount of oil is added, and the whole shaken for 5 min. Sufficient of the emulsion is blown off into a flask to leave 5—10 c.c. of emulsion with the ore in the funnel. 15 c.c. of purified ether are then introduced into the funnel, which is stoppered and shaken. The separated ether layer is blown into a distilling tube, another



10 c.c. of ether are added to the funnel, and the operation is repeated. Four more portions of 10 c.c. of ether are subsequently added, in each case the ether layer being added to the distilling tube as the ether is distilled off. Distillation is effected by immersing the tube in water at 40°, and is continued until the residue just fills a 2 c.c. bulb fused on to the bottom of the distilling tube. It is next transferred to a 10 c.c. container, which is then filled with alcohol, mixed, and placed in a stoppered Erlenmeyer flask. The oil in the original emulsion is determined by comparing the turbidity produced by 5 c.c. of the alcohol-ether solution in 100 c.c. of gelatin solution with that produced by a standard alcohol-ether solution. The gelatin solution contains 1 g. of gelatin and 5 c.c. of glacial acetic acid in 2 litres of water. The readings are made in the dark with a Kober nephelometer-colorimeter. The oil remaining in the emulsion after ore treatment is determined in a similar manner, and the oil adsorbed by the galena calculated by difference. The determinations were concordant within 1%. The method might be used for determination of fats in food products, with the substitution of acetone for alcohol in some cases.

H. MOORE.

**Relation between temperature and Hall effect in some alloys.** J. OBATA (Japan. J. Phys., 1927, 4, 43—47).—The Hall effect in fields from 0—20 kilogauss has been determined at room temperature and at the temperature of liquid air for two samples of manganin (Cu 85.40, Mn 10.03, Ni 4.11, Fe 0.42; and Cu 80.88, Mn 12.72, Ni 4.88, Fe 1.27); an alloy Cu 87.35, Ni 9.21, Al 2.80, Fe 0.50; constantan (Cu 59.62, Ni 37.77, Mn 1.39, Fe 0.65); copper (99.52%); phosphor bronze; gold (99.12%); and nickel. The corresponding values of the Hall constant excluding gold and nickel are, respectively, —0.00055, —0.00052, —0.00032, —0.00116, —0.00046, —0.00051. Nickel and the above alloys (except phosphor bronze, in which the constant remains unchanged) are characterised by a positive temperature coefficient of the Hall effect, whilst gold and copper have a small negative coefficient.

R. W. LUNT.

## PATENTS.

**Metallurgical process. [Recovery of copper and the precious metals from concentrates.]** W. E. GREENAWALT (U.S.P. 1,634,497, 5.7.27. Appl., 6.12.26).—Concentrates containing copper are roasted and leached to remove the greater part of the copper and the residue is smelted to obtain a copper matte containing the precious metals. This matte is roasted and leached to extract the remaining copper, and the residues are treated for the recovery of the precious metals.

A. R. POWELL.

**Treatment of [precious metal] ores for the removal of sulphur, tellurium, arsenic, and the like.** A. J. SUMMERTON (Austral. P. 22,041, 2.3.25. Conv., 15.3.24).—The finely-ground ore is mixed with a dilute acid and the pulp is passed over an amalgamated copper plate above which is arranged a parallel sieve-like anode. By passing a current between the plate, as cathode, and the anode the nascent hydrogen liberated

at the plate causes the arsenic, tellurium, and sulphur to be removed from the ore as hydrides which are oxidised by the nascent oxygen evolved at the anode while the precious metals adhere to the amalgamated plate.

A. R. POWELL.

**Alloy.** G. O. SMITH, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,634,343, 5.7.27. Appl., 18.9.24).—An alloy which retains considerable resiliency at red heat and is suitable for high-temperature springs contains not more than 30% Cu, 60—95% of metals of the iron group, and 5—40% of metals of the tungsten group.

C. A. KING.

**Treatment of ores with chloride solutions.** N. C. CHRISTENSEN (U.S.P. 1,621,969, 22.3.27. Appl., 18.5.26).—Ground lead ore is treated with hot sodium chloride solution containing ferric chloride, and, when chloridisation is complete, the pulp is cooled, neutralised with calcium carbonate, and filtered, the lead chloride formed being extracted from the residue with clean, hot sodium chloride solution, from which it is recovered by cooling.

T. S. WHEELER.

**Gas for cutting and welding purposes.** J. R. ROSE, Assr. to CARBO-HYDROGEN Co. OF AMERICA (U.S.P. 1,628,066, 10.5.27. Appl., 3.8.18).—The gas to be used in conjunction with oxygen consists of a mixture of hydrogen with 20% by vol. of methane.

F. G. CROSSE.

**Soldering articles.** E. THOMA (U.S.P. 1,630,771, 31.5.27. Appl., 17.7.24. Conv., 7.9.22).—A process for soldering articles formed of linked metal rings comprises cleaning the material, electrolytically depositing a suitable solder of low m.p. on it, coating with a flux and with graphite to prevent flowing of the molten solder, and heating to cause the solder to unite each link. For silver mesh a solder containing silver and cadmium is employed.

T. S. WHEELER.

**Centrifugal separator and amalgamator.** W. C. COLEMAN (U.S.P. 1,632,950, 21.6.27. Appl., 6.9.24).—An outer rotor provided with a mercury chamber is divided by an inner rotor into two compartments communicating through a passage at the lower part of the outer rotor, the passage being constricted vertically to retard the flow of material through the mercury chamber.

H. HOLMES.

**Chromium-plated carbon article [mould].** F. M. BECKET, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,630,688, 31.5.27. Appl., 1.12.24).—Moulds or chills for use in casting metals consist of graphite electrolytically coated with chromium.

T. S. WHEELER.

**Manufacture of malleable cast iron.** M. KUBO (U.S.P. 1,634,586, 5.7.27. Appl., 12.6.22).—See E.P. 202,464; B., 1923, 1027 A.

**Beneficiation of [iron] ores.** CROMWELL & MURRAY Co., Assees. of C. P. MCCORMACK (E.P. 251,959, 19.4.26. Conv., 5.5.25).—See U.S.P. 1,575,852; B., 1926, 412.

**Case-hardening of steel articles.** F. PACHER (U.S.P. 1,635,384, 12.7.27. Appl., 21.11.25. Conv., 17.11.24).—See E.P. 242,978; B., 1927, 369.



## XI.—ELECTROTECHNICS.

## PATENTS.

**Insulating liquid.** C. J. RODMAN, ASSR. to WESTINGHOUSE ELECTRIC AND MANUF. CO. (U.S.P. 1,622,809, 29.3.27. Appl., 25.10.21).—A mixture of a hydrocarbon insulating oil with a chlorinated methane derivative, *e.g.*, carbon tetrachloride, and a chlorinated benzene derivative, *e.g.*, hexachlorobenzene, is of value as an insulating oil for switches, and the like. It is practically non-inflammable, and gives little gas when arcing occurs.

T. S. WHEELER.

**Manufacture of high electrical resistances.** J. BRADLEY, and METROPOLITAN-VICKERS ELECTRICAL CO., LTD. (E.P. 273,025, 30.3.26).—Synthetic or other resin, celluloid, shellac, pitch, etc. is dissolved in a volatile solvent, and the solution incorporated with a filler, *e.g.*, wood flour, chalk, or asbestos, and with finely-divided, electrically conducting material, *e.g.*, carbon, silicon. The solvent is evaporated and the mixture moulded to shape.

J. S. G. THOMAS.

**Protective coating for electrical conductors.** H. T. TILLQUIST (G.P. 438,472, 30.6.25).—Iron tubes etc. are protected by a coating of electrolytically-precipitated chromium, which is very resistant to the action of moisture and vapours.

J. S. G. THOMAS.

**Incandescence electric lamp.** J. A. HEANY (U.S.P. 1,635,320, 12.7.27. Appl., 28.6.18).—Leading-in wires made of a copper-lead alloy containing 5–10% Pb are connected to the lamp filament positioned in a lead-glass stem in the lamp.

J. S. G. THOMAS.

**Application of coatings to wires, cables, and the like, and composition therefor.** A. E. P. GIRARD and M. J. P. ROUMAZEILLES (E.P. 253,906, 14.6.26. Conv., 16.6.25).—See F.P. 611,899; B., 1927, 579.

**Method of electrically testing [the dielectric strength of] insulating layers [on wires].** INTERNAT. GENERAL ELECTRIC CO., INC., ASSEES. of ALLGEM. ELEKTRICITÄTS-GES. (E.P. 260,997, 3.11.26. Conv., 3.11.25).

**Electric cells.** J. W. CARPENTER, and EDISON SWAN ELECTRIC CO., LTD. (E.P. 273,837, 14.4.26).

**[Regulating position of electrodes in] electric furnaces.** BRITISH THOMSON-HOUSTON CO., LTD., ASSEES. of J. A. SEEDE (E.P. 263,792, 20.12.26. Conv., 24.12.25).

**[Device for] electrode operation in electric furnaces.** INTERNAT. GENERAL ELECTRIC CO., INC., ASSEES. of ALLGEM. ELEKTRICITÄTS GES. (E.P. 264,851, 20.1.27. Conv., 20.1.26. Addn. to E.P. 259,615).

## XII.—FATS; OILS; WAXES.

**Reactivity of chloramine (sodium salt of *p*-toluenesulphochloroamide) with fats.** B. M. MARGOSCHES and M. FRISCHER (Chem.-Ztg., 1927, 51, 519).—The behaviour of fats with chloramine under varying conditions was studied, the chloramine being determined in acid solution by adding potassium iodide and titrating the liberated iodine, and in alkaline bicarbonate solution with arsenious acid. The iodine value of a fat, calculated from the chloramine which reacted with it, varied with the nature and proportion of the solvents used for the

chloramine and for the fat, with the acidity or alkalinity of the liquid, and with the time and temperature of mixing, but under certain conditions results were obtained in substantial agreement with those given by the Hübler method.

F. R. ENNOS.

**Detergent action of soaps.** P. H. FALL (J. Physical Chem., 1927, 31, 801–849).—The relative detergent action of various concentrations of olive oil, palm oil, tallow, "Green Arrow," and silicated "Green Arrow" soaps has been determined at 40° and 70° by a method which measures the amount of finely-divided manganese dioxide suspended by a detergent solution in a given time. The optimum concentration for all five soaps, at both temperatures, lies in the range 0.2–0.4%, and the detergent values decrease with an increase in temperature. Olive oil soap solutions are less effective at the optimum than the remaining soaps, which resemble each other closely under the same conditions. The method, which is fully described and discussed, has also been applied to sodium oleate, sodium silicate solutions, and to sodium hydroxide and alkaline salts. The relative detergent values of soaps differ with different dirt, owing to specific adsorption. If the ratio of soap to dirt is less than 1:25 or 1:30, the dirt is not stabilised, and flakiness and floating occur. Solutions of sodium silicates are similar to soap in peptising solid dirt, the silicates with a high silica content being the most effective at concentrations greater than 0.15%. The optimum concentrations for all the silicates lie in the range 0.0125–0.05%, and the detergent powers, which decrease with a rise in temperature, are less than those of the soaps. Addition of sodium silicate to soap solutions the concentrations of which are below the optimum enhances the detergent power of the soap more than does the addition of sodium hydroxide, carbonate, or phosphate. These latter are also able to peptise certain samples of manganese dioxide, the optimum concentration lying in the range 0.012–0.025%. Change of temperature has an effect similar to that mentioned above. The lowering of surface tension is not a safe criterion for determining maximum detergent action. The methods previously used for measuring this property are summarised and that of McBain (A., 1924, ii, 155) and co-workers is adversely criticised. A new method utilising the action of salts on suspensions of clay in soap solutions was also unsatisfactory. A *résumé* of previous theories of detergent action is given.

L. S. THEOBALD.

**Utility of the Bertram method [for determination of the higher saturated fatty acids].** J. VAN LOON, also S. H. BERTRAM (Chem. Weekblad, 1927, 24, 319–320, 320; cf. B., 1927, 450).—(A) The procedure laid down by Bertram cannot lead to accurate results if fatty acids are present having more than 9 and less than 16 carbon atoms in the saturated chain, and unless the oxidation of the unsaturated acids present gives rise to no monocarboxylic acids between the acids  $C_9H_{18}O_2$  and  $C_{16}H_{32}O_2$ . The oxidation of the unsaturated acids present in parsley oil, for example, gives rise to lauric acid, which is not separated by the method laid down by Bertram. (B) A reply to the above. Saturated acids containing between 9 and 16 carbon atoms in the chain are, of course, not separated. Unsaturated acids having



the double linking removed from the carboxyl group by 8 or more saturated carbon atoms do not occur in edible oils. S. I. LEVY.

**Oils in emulsions.** BALDWIN.—See X.

**Rancidity in lard.** HUSA and HUSA. **Whale oil in lard.** GRONOVER and BLECHSCHMIDT.—See XIX.

#### PATENTS.

**Manufacture of soft soaps.** L. G. LEFFER and N.V. INTERNAT. QEEP Co. (E.P. 268,980, 3.5.26).—Oils, fats, fatty acids, or resins are saponified under pressure with the optimum quantity of potassium hydroxide solution to produce soap without further treatment. During the saponification,  $\frac{1}{2}$ –2% of aliphatic or aromatic hydrocarbons, and/or 6–15% of cruciferous plant oil, *e.g.*, rape seed oil, and/or 5–20% of oils or fats containing unsaponifiable matter, *e.g.*, sheanut butter, are added, in order to influence the setting power of the soap. Substantially neutral soaps stable to heat and cold within wide limits are obtained. S. S. WOOLF.

**Production of a detergent paste.** M. BRANDEGGER (G.P. 437,245, 12.2.24).—A paste suitable for removing tar, resin, and oil stains, etc. is prepared by mixing vegetable starch with an emulsion of an organic solvent with water, with or without the addition of a dissolving agent, and subsequently hydrolysing part of the starch.

L. A. COLES.

**Cod-liver oil** (U.S.P. 1,629,074).—See XIX.

### XVI.—AGRICULTURE.

**Determination of the lime requirement of soils by means of the state of saturation with calcium.**

A. GEHRING, A. PEGGAU, and O. WEHRMANN (Z. Pflanz. Düng., 1927, A8, 321–331).—A number of details have been further studied in regard to the technique of the authors' method for ascertaining the lime requirement of soils by determination of the exchangeable calcium after treatment with excess of calcium hydroxide and conversion of the excess into carbonate by passing in carbon dioxide and boiling. The conclusion is reached that in general the method gives satisfactory results, but that some points in connexion with it are not yet clear, and that, with certain soils, errors are not excluded. The method, in its present form, does not seem to be applicable to humic soils. C. T. GIMINGHAM.

**Determination of the reaction of soil.** R. GANSEN, H. PFEIFFER, A. LAAGE, H. HALLER, K. UTESCHER, and M. TRÉNEL (Z. Pflanz. Düng., 1927, A8, 332–344).—Investigation of a large number of soils has shown satisfactory correlation between the exchange acidity, the  $p_H$  value, and the molecular ratio of the total bases and of calcium oxide to 1 mol.  $Al_2O_3$  combined with 3 mols.  $SiO_2$ . The molecular ratio of  $CaO$  to 1 mol.  $Al_2O_3 + 3$  mols.  $SiO_2$  in that part of the soil which is decomposed by boiling concentrated hydrochloric acid appears to be suitable for detecting a shortage of lime and for determining the reaction of a soil. The determination of silica is liable to introduce errors; the most suitable method is by extraction with 2% sodium hydroxide solution. C. T. GIMINGHAM.

**Colloidal fraction of certain soils having restricted drainage.** W. L. POWERS (Soil Sci., 1927, 23, 487–

491).—In experiments with the separated clay fraction of soils, the rapidity of flocculation and the volume of coagulum increased with the valency of the flocculating ion. Flocculation by acids and by calcium salts was more rapid when the suspension was previously made alkaline with ammonia. Improvement in the physical condition of certain soils following treatment with sulphur is attributed, not to the sulphuric acid produced, but to increased calcium brought into solution by the acid. Titration curves of each of the soils examined had similar characteristics. Cataphoresis trials showed that between  $p_H$  10 and  $p_H$  2.5 the colloid migrates as with a negative charge, and without any indication of an isoelectric point. A. G. POLLARD.

**Measurement of "suction forces" in colloidal soils.** F. HARDY (Soil Sci., 1927, 24, 71–75).—The method of Joffre and McLean (B., 1925, 858) is adversely criticised in that it is affected by, but takes no account of, the rate of permeation of water through soil in the immediate neighbourhood of the instrument.

A. G. POLLARD.

**Replaceable bases in some Oregon soils.** R. E. STEPHENSON (Soil Sci., 1927, 24, 57–64).—Acid soils lose replaceable calcium by leaching following the exchange of hydrogen ions for calcium in the colloid complex. Similarly, alkali soils result from the exchange of sodium for calcium ions. Heavy soils have a greater base exchange capacity than light soils, but not necessarily a larger amount of replaceable bases. It is considered that the development of both acidity and alkalinity in soils may be considerably retarded and in some cases obviated by suitable soil management.

A. G. POLLARD.

**Possible rôle of iron-depositing bacteria in the formation of hard-pan.** C. S. MUDGE (Soil Sci., 1927, 23, 467–470).—From samples of hard-pan, bacteria were isolated which precipitated iron in pure culture solutions. The probability that such organisms are directly concerned with the formation of hard-pan is discussed.

A. G. POLLARD.

**Reaction and degree of saturation of soils.** J. VON CSIKY (Z. Pflanz. Düng., 1927, 9 A, 1–19).—Data are presented for the  $p_H$  in aqueous and potassium chloride solution suspensions, and for the exchangeable and hydrolytic acidity of certain Hungarian soils. Although, in some cases, a considerable degree of base unsaturation is shown, the  $p_H$  figures are considerably higher than those obtained under similar circumstances with soils of more humid countries such as Germany and Scandinavia. This is explained by the poverty of Hungarian soils in humus, and by the fact that the soils of the humid regions receive greater quantities of saline fertilisers. Whilst individual data do not give trustworthy information as to the lime status of soils, yet with closely comparable soils fertile soils show a higher  $p_H$  and less exchangeable and hydrolytic acidity than infertile soils.

G. W. ROBINSON.

**Comparison of different methods for the determination of the degree of saturation of soils.** S. GERIQUE (Z. Pflanz. Düng., 1927, 9 A, 20–34).—The author has compared the baryta method of Hissink, the barium chloride method of Bobko and



Askinasi (B., 1926, 1025), and the lime-water method of Gehring (B., 1926, 207) for determining the base saturation capacity of soils. The baryta method gave the highest figures, followed by the lime-water method, whilst the barium chloride method gave the lowest figures. There is no evidence as to which method is the most trustworthy, but it is pointed out that Gehring's method is most comparable with practical procedure in the liming of soils. G. W. ROBINSON.

**Pipette method for mechanical analysis of soils, its theoretical basis, and suggestions for a simplified apparatus.** P. KÖTTGEN (Z. Pflanz. Düng., 1927, 9 A, 35—46).—A discussion of the pipette method, and of the methods to be used for the preliminary dispersion. A simple apparatus is described, consisting of a cylinder and pipette with bent orifice. The suspension is forced into the pipette by blowing air into the cylinder by means of a rubber bulb. G. W. ROBINSON.

**Uniformity and utility of data in soil solution analyses.** W. B. BOLLEN and R. E. NEIDIG (Soil Sci., 1927, 24, 69—70).—A plea for the adoption of a standard expression of soil solution analysis on the basis of equivalent parts per million. A. G. POLLARD.

**Lysimeter studies.** W. M. HIGBY (Soil Sci., 1927, 24, 51—56).—Drainage waters from concrete lysimeter tanks were examined. Early portions of the percolate correspond approximately to soil solutions. Applications of lime and manure, alone and in combination, increased the leachable calcium, magnesium, nitrate, sulphate, and potassium in relative quantities of the above general order. The loss of nitrate was approximately doubled by this treatment. The amount of phosphate in the drainage water was small in all cases, but decreased with the lime and manure treatments. A. G. POLLARD.

**Effect of manuring with Chili saltpetre on the sugar beet.** J. SOUČEK and F. KRAUS (Z. Zuckerind. Czechoslov., 1927, 51, 417—421).—Sodium nitrate to the amount of 300 kg./hectare (2.4 cwt./acre) was applied in three different ways: (1) total amount at sowing; (2) half at sowing and half at singling as a top-dressing; and (3) all at singling, the crops being lifted at the end of September and later at the beginning of November. It was found that with early harvesting the roots were not ripe, and their sugar content was lower than that of the unmanured crop. Both for farmer and factory the most advantageous procedure is late harvesting with the divided addition. Later manuring (e.g., all at singling) was regarded as unremunerative. J. P. OGILVIE.

**Assimilation of phosphates measured by the bacterial fixation of nitrogen.** G. TRUFFAUT and N. BEZSSONOFF (Compt. rend., 1927, 185, 85—86).—Cultures of nitrogen-fixing bacteria growing in a medium free from phosphates were supplied with various natural phosphates as their sole source of phosphorus, the amount of fixed nitrogen being determined after 26 days. It is thus shown that the biological inferiority of fluophosphates is due to their insolubility and not to any toxic action of the fluorine. After dissolution in sulphuric acid their metabolic value increases tenfold. E. A. LUNT.

**Chemistry of basic slags.** E. VANSTONE (J. Agric. Sci., 1927, 17, 143—152).—Two basic slags, one a low-soluble slag containing 18% of calcium phosphate, and the other a high-soluble slag containing 30% of calcium phosphate, were extracted for long periods with boiling 10% ammonium chloride solution. The low-grade slag lost 70% of its lime and very little phosphate, the residue containing about 40% of calcium phosphate. With the high-grade slag there was also a considerable loss of lime and, in addition, of phosphate. The residue after 96 hrs.' extraction of low-grade slag corresponded with the formula  $3.3 \text{ CaO}$ ,  $1.0 \text{ P}_2\text{O}_5$ ,  $4.5 \text{ SiO}_2$ ; that from the high-grade slag corresponded with the formula  $4.09 \text{ CaO}$ ,  $1.0 \text{ O}_2\text{P}_5$ ,  $1.3 \text{ SiO}_2$ . It is concluded that a silicophosphate is present in the high-grade slag, but not in the low-grade slag. Attention is directed to the effect of dressings of slag on the exchangeable calcium of soils. The phosphate in fluor slags is probably present as fluorapatite. G. W. ROBINSON.

**Determination of nitrates in plant materials.** G. T. PYNE (J. Agric. Sci., 1927, 17, 153—161).—Determinations of nitrate by distillation with magnesium oxide and Devarda alloy at  $100^\circ$  are untrustworthy in the presence of organic matter. Two methods are proposed for use with plant juices. In the first, the juice, cleared by addition of an equal volume of alcohol and subsequent filtration, is diluted and reduced with finely divided Devarda alloy in the presence of sufficient sodium hydroxide to give a  $0.1N$ -solution. After reduction, excess of hydrochloric acid is added, and the ammonia is distilled off under reduced pressure with calcium oxide. In the second method, the cleared plant juice is treated with titanous hydroxide formed by adding titanous chloride to the juice to which sufficient alkali has previously been added. After reduction, ammonia is distilled off as above under reduced pressure at  $40$ — $50^\circ$ . G. W. ROBINSON.

## XVII.—SUGARS; STARCHES; GUMS.

**Desiccation of sugar samples in tins.** K. ŠANDERA (Z. Zuckerind. Czechoslov., 1927, 51, 309—313).—Tins with lever-top lids are prescribed for use by the Prague sugar sample distributing office, and have been found to render good service, meeting all requirements for storage and transport. Under ordinary conditions, with atmospheric moisture at 65% and temperature at  $15$ — $25^\circ$ , the loss of weight is negligible, not exceeding  $0.07\%$  per month. J. P. OGILVIE.

**Determination of sulphur dioxide in sugars.** R. H. HURST (Trop. Agric., 1927, 66).—Titration with iodine volumetrically in the usual way gives results much higher than reality, but the following procedure is sufficiently reliable for factory control purposes: 5 g. of the sugar are dissolved directly in an excess (say 20 c.c.) of standard iodine solution to which a few drops of hydrochloric or acetic acid have previously been added, the sulphite being thus converted quantitatively into sulphate, after which the excess iodine is titrated with sodium thiosulphate, the starch indicator being added as late as possible. Results obtained in this way agree very well with those found by the sulphide-stain procedure (cf. B., 1927, 312). J. P. OGILVIE.



**Valuation of raw [beet] sugars in respect of their affinity.** O. SPENGLER and C. BRENDL (Z. Ver. deut. Zuckerind., 1927, 229—242).—A procedure is described for determining the relative value of raw beet sugars for refining by mashing with a sugar liquor saturated 3° below laboratory temperature and centrifuging, the amount of syrup and sugar obtained being noted for the calculation of the centrifuging power of the sample and the yield, respectively, and the sugar being graded according to its colour. The amount of fine grain in a raw sugar is no indication of its centrifuging power, which depends also on the size and regularity of the grain. J. P. OGILVIE.

**Decolorisation of thin-juice [in beet sugar manufacture] by activated carbon.** A. LINSBAUER (Z. Zuckerind. Czechoslov., 1927, 51, 483—490).—On the principle that the presence of sugar decreases the decolorising effect, the application of decolorising carbon to the juices rather than to the thick-juice (syrup) is advocated, it being claimed that only 0.03—0.04% of Carboraffin is required for thin-juice, whereas with evaporator thick-juice double this amount is necessary. J. P. OGILVIE.

**Improvements in the desaccharification of [beet] molasses by the Steffen process.** J. BERGÉ (Sucr. Belge, 1926, 46, 1—10).—Difficulty experienced in washing the cake is now overcome by giving it a preliminary washing with water until it shows signs of cracking, and then pumping through a magma of re-precipitated, purified saccharate with which the washing is finished. Further, the amount of lime used has been reduced mainly by securing a more intimate contact between molasses solution and lime powder, the consumption now being less than 100% of the sugar. Only about 3% of sugar is lost in the final residual liquor. J. P. OGILVIE.

**Peligot's saccharine [ $C_6H_{10}O_5$ ] and its isolation from [beet] molasses.** K. VNUK (Z. Zuckerind. Czechoslov., 1927, 51, 460—466, 467—477).—Glucosaccharine (the lactone of saccharic acid) was prepared by evaporating beet molasses to dryness with sodium sulphate, extracting the residue with acetone, dissolving out the acid, and converting it into its barium salt, this being purified by re-precipitation; a preparation was also made by boiling a solution of invert sugar with lime, and purifying in a similar manner. In consequence of its high dextrorotatory power, saccharine disturbs the double polarisation method of determining sucrose, and its effect is not eliminated by either the ordinary Clerget process or the Andrlik-Staněk modification of making both observations in acid solution. J. P. OGILVIE.

**100° point of the saccharimeter.** O. SPENGLER, C. BRENDL, and J. SCHWIRBLIANSKI (Z. Ver. deut. Zuckerind., 1927, 419—428).—Krais and Traegel (cf. B., 1924, 608) and others having found a normal solution of sucrose to produce values less than 100°, viz., 99.82—99.89°, the authors have tested Jäckel's suggested explanation of the adsorption of air on the surface of finely-divided sugar (cf. B., 1923, 671 A) by dissolving sugar in a flask connected with a very sensitive capillary gauge for indicating any minute increase in volume. On dissolving 17.35 g. of finely-powdered

sugar in 18.5 c.c. of water, and taking into account the contraction arising on dissolution, an increase of volume of 5.76 c.c./100 g., equivalent to 7.0 mg., was found, an amount incapable of affecting the result. Results of other experiments are adduced to prove that this explanation of the error will not suffice. J. P. OGILVIE.

**Sucrose crystallisation.** I. A. KUKHARENKO and B. G. SAVINOV (Nauch. Zapiski [Russia], 1927, 4, 139—142).—The formation of "crystallons" is explained according to Kukharenko's theory, whereby crystallisation will not take place unless centres of crystallisation are introduced. CHEMICAL ABSTRACTS.

**Pectins and the mellowness of wines.** SEMICHON.—See XIX.

## XIX.—FOODS.

**Detection of hardened whale oil in lard.** A. GRONOVER and A. BLECHSCHMIDT (Z. Unters. Lebensm., 1927, 53, 250—252).—A consignment of lard from a refinery had been rejected on the grounds of containing tallow or hardened fat by reason of the low difference of m.p. given by the Bömer test. Further samples of lard from the same shipment were taken from the refinery, and samples of the original material as received from America. The original material gave a normal Bömer value of 75.2°, but the refined material gave one value of 71.0°, which strongly suggested adulteration, and two values of 69.2° and 68.1°, which clearly indicated that hardened fat had been added at the refinery. It was subsequently admitted that during the summer hardened fat, and during the winter arachis oil, were added to give the lard a good consistency. The Bömer test is therefore valuable for the detection of both tallow and hardened whale oil in lard.

H. J. DOWDEN.

**Effect of benzoic and cinnamic acids on the rate of development of rancidity in lard.** W. J. HUSA and L. M. HUSA (J. Amer. Pharm. Assoc., 1926, 15, 1071—1074).—In an endeavour to discover the constituents of benzoin responsible for its effect in retarding the rancidity of lard, experiments have been conducted with benzoic and cinnamic acids. The addition of either acid in quantities up to 4% of the lard was not effective in retarding the development of rancidity as measured by the Kreis test. This test cannot be used for the detection of rancidity in benzoinated lard because Siam benzoin itself gives a pronounced red colour in the test.

E. H. SHARPLES.

**Determination of gelatin in ice cream.** R. E. REMINGTON and L. H. MCROBERTS (Ind. Eng. Chem., 1927, 19, 267—269).—Deductions of the percentage of gelatin by determinations of its specific rotation and nitrogen content (Ferris, J. Dairy Sci., 1922, 5, 555, and Smith, B., 1919, 228 A) can only be applied to inspection work if it is permissible to apply average values for rotation and nitrogen content to any grade of gelatin likely to be encountered. From analyses of a number of samples of ice cream gelatins, it appears that the figures 14.84% for nitrogen and 117 for specific rotation (corresponding with 17.53 and 139, respectively, on the dry ash-free basis) may be taken as average values with a maximum error of 3% of the amount of gelatin present. Suggested modifications for the Ferris



method are the use of a smaller sample, weight instead of volume dilution, control of the casein precipitation by means of hæmatoxylin indicator, the use of alum to ensure complete precipitation of the gelatin, and the correction of the nitrogen found for that from the milk which comes through into the filtrate. Ferris' original and the modified method are described in detail.

C. O. HARVEY.

**Micro-determination of caffeine in coffee.** A. C. RÖTTINGER (Z. Unters. Lebensm., 1927, 53, 146—151).—5 g. of finely-ground coffee are introduced into a separating funnel, the open end of which is ground to fit into the neck of a flask containing 100 c.c. of chloroform. After uniting the vessels the contents are agitated for  $\frac{1}{2}$  hr., 20 c.c. of the solution are filtered through a pad of cotton wool into a specially designed pipette, which fits the stop-cock of the separator. The solution is transferred from the pipette into a bulb-shaped vessel which is heated on a water-bath and the chloroform removed by aspirating air. To the residue in the bulb are added a small piece of paraffin wax, a few c.c. of ether, and, after warming, a few c.c. of dilute hydrochloric acid, and the whole is thoroughly shaken. The ether is removed by a current of air, and the acid layer drawn off into a small separating funnel, leaving the paraffin wax adhering to the walls of the bulb. The residue is extracted in this way four times with 0.5% hydrochloric acid, the extracts being collected in the same funnel. The acid solution is then shaken thoroughly with four successive quantities of chloroform, the chloroform layer being drawn off through cotton wool into a distillation flask, from which the chloroform is subsequently evaporated. The crude caffeine which is left is treated with 2 c.c. of concentrated sulphuric acid, potassium sulphate, and copper sulphate, and decomposed by heating for  $\frac{1}{2}$  hr., after which a Kjeldahl distillation is performed. If 0.02*N*-sulphuric acid is used, 1 c.c. of acid corresponds to 0.0969% of caffeine. The apparatus described reduces the total experimental error to about 0.02%.

H. J. DOWDEN.

**Physiology of apples. VI. Correlation in the individual apple between the mineral constituents and other properties.** J. W. BROWN (Ann. Bot., 1927, 41, 127—137; cf. B., 1926, 605).—Analyses of 30 Bramley's Seedling apples from the same orchard showed that green apples contained less dry matter, total ash, and potassium, and more nitrogen than yellow, and red and yellow apples; the  $p_H$  of the juice of green apples was also appreciably lower. High values for the  $p_H$  of the juice were associated with high content of total ash and potassium. Partial correlations between some pairs of constituents were calculated, and significant values showing direct correlation between potassium and phosphates, phosphates and acidity, and phosphates and iron were obtained. Potassium and iron, and potassium and acidity were inversely correlated. No significant correlations between nitrogen and any of the mineral constituents were found.

C. T. GIMINGHAM.

**Pectins of grapes and the mellowness of wines.** L. SEMICHON (Chem. et Ind., 1927, 17, 25—32).—By

the addition of alcohol to a wine acidified with 1% of hydrochloric acid, the pectins and gums may be precipitated in a fair state of purity, the former being separated from the latter by preparation of the pectate of calcium. Grape musts contain pectins but no gums. Wines may contain a mixture of the two, or pectins may be absent, according to the stage of fermentation reached. No relation has been found between the amount of pectins which disappear and the gums formed. The pectins consist of the methyl ester of pectic acid, combined with other organic compounds, and with phosphates of calcium, magnesium, and aluminium. On hydrolysis they yield pentoses. The gums are neutral, show no ester character, and on hydrolysis yield hexoses. The influence of the pectin content of the grape must on the mellowness of the wine is discussed. F. R. ENNOS.

**Detection and determination of nitrogen-bearing chemicals added to animal or vegetable nitrogenous materials.** H. C. MOORE and R. WHITE (Ind. Eng. Chem., 1927, 19, 264—266).—Ammonium sulphate and cyanamide have been found in nitrogenous tankage, and urea might be added. The addition of sodium nitrate would not increase the nitrogen content as determined in the usual way. The silver nitrate test for cyanamide is unsatisfactory for testing tankage directly, but may be carried out on the portion which settles to the bottom of the container when the tankage is mixed with carbon tetrachloride. This portion contains the heavy particles, such as ammonium sulphate, carbamide, etc., and the further addition of an equal volume of turpentine will cause crystals of urea, if present, to appear in partial suspension. Modifications of the ordinary qualitative reactions in general use are recommended for testing tankage. Nitrate nitrogen is determined on 50 c.c. of a filtered 2% aqueous solution of the tankage by reduction and alkaline distillation, the ammonia derived from ammonium salts being separately determined and deducted from the figure obtained. Cyanamide and dicyanodiamide are precipitated by the addition of excess of silver nitrate solution to 50 c.c. of a filtered 2% aqueous solution of the tankage, followed by 20 c.c. of 10% potassium hydroxide solution. Nitrogen is determined in the brown precipitate by the Kjeldahl method. The residue obtained by filtration of a solution of 5 g. of tankage in 250 c.c. of anhydrous alcohol contains the ammonium sulphate, which is determined by extracting with water, bulking to 250 c.c., and distilling 50 c.c. with 150 c.c. of water and excess of magnesium oxide. Urea is determined in 50 c.c. of the original alcoholic solution by adding 50 c.c. of water, boiling off the alcohol, adding 100 c.c. of water, cooling, adding 0.25 g. of urease, and, in  $\frac{1}{2}$  hr., distilling after the addition of 1 g. of paraffin wax and 5 g. of heavy magnesium oxide. A correction for the solubility of ammonium nitrate in alcohol must be made if this salt is present, by performing a separate alkaline distillation. The results obtained are sufficiently accurate for most purposes. The presence of more than 0.5% of free ammonia in nitrogenous tankage points to the addition of ammonium salts.

C. O. HARVEY.

**Oils in emulsions.** BALDWIN.—See X.



## PATENTS.

**Preservation of fruit.** O. H. HANSEN, Assr. to HANSEN CANNING MACHINERY CORP. (U.S.P. 1,625,207, 19.4.27. Appl., 7.5.24).—Fruit is temporarily sealed in cans, which are then exhausted and filled with steam. Sufficient syrup to cover the fruit is admitted and the submerged fruit completely sterilised, the cans being finally sealed. T. S. WHEELER.

**Removal of residual poisons [lead arsenate] from fruit.** E. M. SEARS (U.S.P. 1,624,074, 12.4.27. Appl., 21.10.26).—Fruit which has been sprayed with lead arsenate can be completely freed from it by immersion in a mixture of a mineral or vegetable oil and a fatty acid, *e.g.*, stearic acid, followed by washing in a dilute alkaline solution and in water. T. S. WHEELER.

**Production of feed stuff.** C. S. MINER and G. STEBRUP, Assrs. to QUAKER OATS CO. (U.S.P. 1,627,921, 10.5.27. Appl., 28.5.21. Renewed 23.3.27).—Roughage, such as seed hulls, straw, and other vegetable products with a high crude-fibre content, is mixed with not more than its own weight of water and with about 8% of lime, and heated at 150° under a steam pressure of about 80 lb./sq. in. for 3 hrs. After allowing the mass to cool without the escape of vapours, it is dried to a moisture content of approximately 12%, and ground. F. R. ENNOS.

**Manufacture of bread, cakes, and other yeast-leavened baked foods.** W. D. STEIN (E.P. 255,469, 14.7.26. Conv., 29.8.25).—The flour is mixed with a monosaccharide, *e.g.*, dextrose or *lævulose*, and sufficient lactic acid to promote the most favourable acidity ( $p_H$  4.8) for the growth of yeast. The dough is then made and baked. F. R. ENNOS.

**Preparation of bakery produce.** C. SCHMITT (G.P. 435,973, 22.3.25).—Addition of fine meal, obtained from cottonseed meal by sifting, raises the albumin content of biscuits, bread, etc. S. S. WOOLF.

**Manufacture of a highly-concentrated substance rich in antirachitic and antixerophilic vitamins from cod-liver oil.** C. FUNK and H. E. DUBIN, Assrs. to H. A. METZ LABORATORIES, INC. (U.S.P. 1,629,074, 17.5.27. Appl., 28.6.23).—Cod-liver oil is heated under reflux with an equal weight of glacial acetic acid for 8 hrs., the acid being concentrated to one tenth its bulk and extracted with benzene. The benzene solution is washed with water and evaporated to dryness, and the residue is heated with alcoholic sodium hydroxide solution. The solution obtained is diluted with water and extracted with benzene. The residue from evaporation of the benzene contains the vitamins of cod-liver oil in a highly-concentrated form. A still more concentrated preparation can be isolated by treatment of this residue with digitonin in the usual way. Finally, 1 kg. of oil yields 0.2 g. of a crystalline product. T. S. WHEELER.

**Manufacture of milk powder.** J. M. SIERRA (U.S.P. 1,609,384, 7.12.26. Appl., 13.3.23. Conv., 27.3.22).—See E.P. 198,496; B., 1923, 738.

**Manufacture of a flour improver.** D. J. BLOCK, Assr. to W. D. STEIN (U.S.P. 1,626,910, 3.5.27. Appl., 29.8.25).—See E.P. 255,469; preceding.

**Treatment of meal, flour, and other milling products with per-compounds.** J. VAN LOON (E.P. 245,801, 11.1.26. Conv., 10.1.25).—See F.P. 609,057; B., 1927, 265.

**Drying chambers for vegetables and like products.** G. SCOTT AND SON (LONDON), LTD., and G. W. RILEY (E.P. 271,143, 16.2.26).

**Preparation of infusions of coffee or the like.** C. URTIS (E.P. 270,586, 3.12.26).

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

**Separation of the most important opium alkaloids.** F. CHEMNITZ (Pharm. Zentr., 1927, 68, 307—310).—A method for the separation and purification of morphine, codeine, narceine, narcotine, thebaine, papaverine, and meconic acid is described. E. H. SHARPLES.

**Microchemistry of narceine.** M. WAGENAAR (Pharm. Weekblad, 1927, 64, 354—357).—The alkaloid is readily precipitated by addition of potassium nitrite (and other salts of weak acids) to solutions of its salts, in bundles of fine needles, of negative double refraction. The needles are coloured blue or brown by free iodine. The chloride, bromide, and thiocyanate are obtained by addition of the corresponding ammonium or potassium salts to a solution in hydrochloric acid. Addition of platinic chloride produces an amorphous precipitate which soon becomes crystalline, but the crystals are not highly characteristic. S. I. LEVY.

**Poisoning action of mercury alkyls.** A. KLAGES (Z. angew. Chem., 40, 559—561).—The fungicidal and germicidal action of various mercury alkyl halides is tested on fungus spores and on wheat kernels. The poisonous effect increases in the order chloride < bromide < iodide. The effect with these alkyl halides is considerably greater than with the mercurous halides. L. M. CLARK.

**Stability of physostigmine [eserine] solutions.** J. C. KRANTZ, JUN., and F. J. STAMA (J. Amer. Pharm. Assoc., 1927, 16, 412—414).—The red colour which develops in solutions of physostigmine sulphate and salicylate due to the formation by hydrolysis of eseroline, can be prevented for a considerable period by adjusting the hydrogen-ion concentration to  $p_H$  2—3 by means of hydrochloric, boric, sulphuric, or salicylic acid. Water saturated with carbon dioxide, with an atmosphere of the gas above, is the best medium to use for preventing the appearance of this red colour. E. A. LUNT.

**Production of essences.** F. O. SCHOEPPÉ (Riechstoffind., 1926, 92; Chem. Zentr., 1926, II., 3119).—The use of alcohol to render essential oils soluble in water may be avoided by the following method. The oil is mixed with 15—30% of a sulpholeate (Turkey-red oil) containing about 50% of fat, and the mixture is dried with 2% of anhydrous potassium carbonate. No deterioration in quality occurs. E. H. SHARPLES.

**Bergamot.** F. LA FACE (Riechstoffind., 1926, 85—87; Chem. Zentr., 1926, II., 3119).—The following characteristics are given for bergamot oil:  $d_{20}^{25}$  0.880—0.887,  $n_D^{20}$  + 8° to + 24°, linalyl acetate, 35—45%; saponif.



value, 1—3.5; distillation residue in water-bath, 4.5—6.5%; solubility in 90% alcohol, 1 in 0.5 vol.

E. H. SHARPLES.

**[Constituents of] Port Orford cedar wood oil.** F. H. THURBER and L. J. ROLL (Ind. Eng. Chem., 1927, 19, 739—742. Cf. Schorger; B., 1914, 886).—Fractional distillation of Port Orford cedar wood oil yielded 46% of *d*- $\alpha$ -pinene, b.p. 156—156.5°,  $d_{20}^{20}$  0.8584,  $n_D^{20}$  1.4608,  $[\alpha]_D^{20} + 53.01^\circ$ , characterised by the preparation of pinene hydrochloride, m.p. 126—127°, and  $\alpha$ -pinonic acid, m.p. 68°. This sp. rotation is the highest recorded for *d*- $\alpha$ -pinene. The redistilled product gave no trace of the inactive nitrosochloride, the formation of which is characteristic of nearly all samples of *d*- $\alpha$ -pinene obtained from American turpentine oil, and is therefore regarded as a pure *d*- $\alpha$ -pinene isomeric with the ordinary form. Oxidation yielded no trace of pinonic acid, indicating that  $\beta$ -pinene is not a component of the oil. The fractions of b.p. 159—178° and 78—100°/14 mm. (3% of the original oil) contained a small proportion of *d*-limonene, b.p. 170—176° (tetrabromide, m.p. 124°). The next fraction, b.p. 100—122°/14 mm., consisted of *d*-borneol, m.p. 201°,  $[\alpha]_D^{20} + 32.6^\circ$ , which was isolated as the acid phthalate, m.p. 164°, and represented 13.8% of the original oil, and a liquid secondary alcohol (11.5%), of mol. wt. equal to that of borneol, b.p. 104—107°/13 mm. Saponification values before and after acetylation showed that 5.2% of the total secondary alcohol in this fraction was present in the oil as the acetate, and 20.1% as the free alcohol. The fraction of b.p. 122—145°/14 mm. consisted of *d*-cadinene (21%), b.p. 272—274°,  $d_{20}^{20}$  0.9255,  $n_D^{20}$  1.5065,  $[\alpha]_D^{20} + 103.7^\circ$ , from which the dihydrochloride, m.p. 118°, was prepared. The highest fraction, b.p. 145—180°/14 mm.,  $[\alpha]_D^{20} - 10.54^\circ$ , equivalent to 3.9% of the oil, contained *l*-cadinol (cf. Semmler and Jonas, A., 1915, i, 63).

W. J. POWELL.

**Temperature coefficients for specific gravities and rotations of essential oils.** N. SCHOORL (Pharm. Weekblad, 1927, 64, 451—453).—The coefficient for the rotation may be taken as 0.0004, and for the specific gravity as 0.00075; these are approximate average values only, but may be employed to correct readings taken under ordinary conditions.

S. I. LEVY.

**Glass vessels for drugs.** KROEBER.—See VIII.

**Oils in emulsions.** BALDWIN.—See X.

**Caffeine in coffee.** ROTTINGER.—See XIX.

#### PATENTS.

**Production of aqueous solutions of free cyanamide.** A. M. MUCKENFUSS, Assr. to ROESSLER & HASSLACHER CHEMICAL CO. (U.S.P. 1,622,731, 29.3.27. Appl., 14.8.24).—Calcium cyanamide is treated in presence of cold water with sulphur dioxide at such a rate that, while the liquid remains neutral, calcium sulphite is precipitated and free cyanamide is formed. By filtering at intervals and adding fresh calcium cyanamide and sulphur dioxide, solutions containing up to 13.5% of free cyanamide can be obtained.

T. S. WHEELER.

**Process of esterification.** U.S. INDUSTRIAL ALCOHOL CO., Assees. of F. B. ARENTZ (E.P. 246,526, 26.1.26.

Conv., 26.1.25).—A continuous esterification process and plant are described which are applicable in cases where the alcohol or ester or alcohol-ester mixture can form with water an azeotropic mixture, e.g., the preparation of *n*- or *iso*-butyl acetate, amyl formate, acetate, or phthalate, *n*-butyl tartrate or phthalate. The mixture of acid and alcohol is heated under a reflux condenser which allows the vapours of the azeotropic mixture to pass forward to a descending condenser and a separator, from whence alcohol and/or ester is returned to the still and aqueous liquor is removed. If a water layer forms in the still it may be drawn off in order to economise time and fuel. The preparation of *n*-butyl acetate and tartrate is detailed.

C. HOLLINS.

#### Manufacture of polyhydroxy-carbon compounds.

H. ESSEX and A. L. WARD, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,626,398, 26.4.27. Appl., 25.5.26. Cf. U.S.P. 1,594,608; B., 1926, 897).—An olefinic compound, e.g., ethylene or allyl alcohol, is treated with carbon dioxide in presence of sodium hypochlorite solution, and the resulting solution of the corresponding chlorohydrin and sodium carbonate is heated at the b.p. to form the corresponding polyhydroxy-compound.

T. S. WHEELER.

**Manufacture of aliphatic keto- and aldehydo-carboxylic acids.** C. H. BOEHRINGER SOHN CHEM. FABR., Assees. of A. HÄUSSLER (U.S.P. 1,627,091, 3.5.27. Appl., 22.12.25. Conv., 5.3.23).—Esters of hydroxy-aliphatic acids, e.g., ethyl lactate, are passed in the vapour phase with an excess of air, and preferably with steam, at 100—500° over oxygen compounds of metals capable of more than one degree of oxidation, e.g., vanadium pentoxide, lead chromate. A 50—80% conversion into the corresponding keto- or aldehydo-ester, e.g., ethyl pyruvate, is obtained.

T. S. WHEELER.

**Manufacture of glycols.** CARBIDE & CARBON CHEMICALS CORP., Assees. of G. O. CURME, JUN. (E.P. 264,124, 5.7.26. Conv., 7.1.26).—Chlorohydrins are converted into glycols by hydrolysis with a solution of caustic alkali under such conditions that any appreciable escape of the intermediate olefine oxide is prevented, e.g., by using a vessel completely filled with liquid. The temperature and pressure are regulated so that the conversion of the chlorohydrin into olefine oxide and of the latter into glycol proceed at suitable rates.

B. FULLMAN.

**Manufacture of [inactive] menthol.** RHEINISCHE KAMPFER-FABR. G.M.B.H., Assees. of K. SCHÖLLKOPF (U.S.P. 1,629,002, 17.5.27. Appl., 26.6.26. Conv., 11.3.22).—Thymol is treated with hydrogen at 200° and 5—30 atm., in presence of a nickel, platinum, or palladium catalyst, and the *r*-menthol formed is separated from liquid isomerides by freezing or fractional distillation. These isomerides are heated with sodium at 300° for 20 hrs., and the liquid product is distilled with steam and then fractionated. About 50% is obtained as *r*-menthol. The remainder is mixed with a fresh batch of thymol.

T. S. WHEELER.

**Cyclic production of soluble salts of organic compounds of an acid character.** M. BUCHNER (E.P. 246,142, 12.1.26. Conv., 16.1.25).—Acid organic



compounds, especially fatty acids or their esters (glycerides etc.), are treated with the hydroxides or carbonates of calcium, magnesium, etc., producing insoluble salts which are decomposed by a fluoride (simple or complex) to give the required soluble salt of the acid compound together with a fluorine derivative of the original metal (often insoluble). From the latter is reproduced the fluorine compound used in the reaction, *via* the complex fluoride. The metal compounds required for the production of the insoluble derivatives of the initial organic substance may be used cyclically; *e.g.*, if calcium carbonate be used, the calcium chloride obtained as above is reconverted into carbonate by means of the carbon dioxide liberated and ammonia. B. FULLMAN.

**Compositions containing organic mercury compounds.** E. C. R. MARKS. From E. I. DU PONT DE NEMOURS & Co. (E.P. 269,243, 12.1.26).—A mercury oxide or salt is caused to react with an organic compound (such as a phenol, negatively-substituted phenol, or naphthol, etc.) in the presence of a finely-divided solid material which acts as a diluent or vehicle for the organo-mercury compound formed, which is deposited on its surface. The diluent substance may be either inert (clay, charcoal, barium sulphate, etc.) or such that it reacts with one of the other substances present (hydrated lime, magnesium oxide or carbonate, etc.). *E.g.*, 30 pts. of *o*-chlorophenol are heated with the product formed by the action of 50 pts. of mercuric acetate on 500 pts. of hydrated lime in 2000 pts. of water. The final product contains 6.7% of the calcium salt of hydroxymercurio-*o*-chlorophenol. These substances have application as fungicides etc.

B. FULLMAN.

**Production of 3-bromo-2-hydroxypyridine-5-arsinic acid.** A. BINZ and C. RÄTH (E.P. 263,142, 13.12.26. Conv., 21.12.25).—Bromination of 2-hydroxypyridine-5-arsinic acid (E.P. 250,287; B., 1926, 512) in acetic acid yields 3-bromo-2-hydroxypyridine-5-arsinic acid, which has trypanocidal and spirillicidal properties.

C. HOLLINS.

**Manufacture of complex antimony compounds.** H. HAHN, Assr. to WINTHROP CHEMICAL Co., INC. (U.S.P. 1,628,838, 17.5.27. Appl., 29.3.26. Conv., 6.6.25).—Antimonyl compounds of pyrocatechol and its derivatives are heated in presence of water with sodium malonate or with a neutral salt of a similar carboxylic acid containing in the molecule mobile hydrogen atoms, to yield complex compounds of therapeutic value.

T. S. WHEELER.

**Medicinal product.** H. C. FULLER (U.S.P. 1,627,963, 10.5.27. Appl., 30.1.20).—Glycerol and derivatives, *e.g.*, glycerylchlorohydrin, can be employed in place of ethyl alcohol as solvents and preservatives in the preparation of medicinal mixtures. T. S. WHEELER.

**5-Ethyl-5-*n*-hexylbarbituric acid.** A. W. DOX, Assr. to PARKE DAVIS & Co. (U.S.P. 1,624,546, 12.4.27. Appl., 15.12.24).—5-Ethyl-5-*n*-hexylbarbituric acid, m.p. 126° (cf. A., 1924, i, 1037), is claimed as a new substance of therapeutic value as a hypnotic. T. S. WHEELER.

**Production of algin compounds.** B. F. ERDAHL (U.S.P. 1,625,301, 19.4.27. Appl., 20.7.26. Cf. Kunig; B., 1926, 784).—A colloidal solution of sodium alginate is added to dilute sulphuric acid, and after 24 hrs.

the precipitate is washed, dried, and added to a solution obtained by the hydrolysis of beeswax with excess of ammonia solution. The product is a thick syrup, of value as a waterproofing agent for fibrous materials, *e.g.*, paper. The coated material is heated at 60°.

T. S. WHEELER.

**Antidiabetic substance.** F. S. MACY (U.S.P. 1,626,044, 26.4.27. Appl., 8.5.26).—Ground pancreas tissue is macerated with water, to which ammonium sulphate is then added to precipitate proteins. The liquid is filtered and treated with ammonia, the precipitate obtained being dried and mixed with fresh beef muscle juice.

T. S. WHEELER.

**Manufacture of the pure phosphorus-containing nuclear substance of milk casein.** S. POSTERNAK, Assr. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,631,887, 7.6.27. Appl., 11.1.24. Conv., 25.1.23).—See E.P. 221,716; B., 1924, 966.

**Preparation of products of organic origin.** H. L. P. TIVAL (U.S.P. 1,630,985, 31.5.27. Appl., 24.4.22. Conv., 26.4.21).—See E.P. 179,164; B., 1923, 518 A.

**Manufacture of physiologically-active substances from ovaries, corpus luteum, and placenta.** M. HARTMANN, Assr. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,625,360, 19.4.27. Appl., 5.8.26. Conv., 8.5.23).—See E.P. 226,372; B., 1925, 149.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Colour photography and the "lignose screen."** (Phot. Ind., 1927, 439–443).—The new lignose screen-unit process of colour photography on film is described in detail. The screen-film is prepared by the "dusting-on" process, using particles dyed in three colours having the following transmissions: red,  $\lambda$ 5800 to  $\lambda$ 7500 Å. with a maximum at about  $\lambda$ 6400 Å.; green,  $\lambda$ 5000 to  $\lambda$ 6000 Å. with a maximum at about  $\lambda$ 5500 Å.; blue,  $\lambda$ 4000 to  $\lambda$ 5200 Å., with a maximum at about  $\lambda$ 4700 Å. The size of the screen elements ranges from about 0.005 mm. to 0.012 mm., the mean size being about 0.009 mm., *i.e.*, 10,000–12,000 elements per mm.<sup>2</sup> The proportion of coloured particles is 100 green, 70 to 80 red, and 80 to 90 blue. After the particles have been allowed to settle on the film coated with adhesive, the excess is removed and those adhering are rolled so as to spread them into contact at the edges. No filling agent is needed. The surface is finally varnished to protect the screen from the action of the developer and the reversing bath. The finished film transmits 16–17% of the incident light. The film is treated to prevent any possibility of separation of the emulsion from the screen, and the screen is not affected by dilute alcohol. A fine-grained, panchromatic, contrasty emulsion of speed 14 Scheiner is used, but in daylight the exposure of the screen film under a suitable filter is 40–75 times that required for a black-and-white reproduction on an emulsion of speed 14 Scheiner. Conditions for correct colour rendering, and the applications of the film are discussed. It can be used in all conditions in which ordinary screen-plates are employed, and in addition it possesses all the advantages of films, including their use in cinematography.

W. CLARK.