

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 13, 1928.

I.—GENERAL; PLANT; MACHINERY.

Sedimentation analysis. III. S. ODÉN and N. WINCKLER (*Tekn. Tidskr.*, 1927, 57, [Kemi 6], 16—18; *Chem. Zentr.*, 1927, ii, 962—963).—The application of sedimentation analysis to the investigation of powdered substances of technical interest is indicated.

A. A. ELDRIDGE.

Condensation of liquids as mists. M. PAILLY (*Rev. gén. Colloid.*, 1928, 5, 745—748).—The formation of mists is a result of the dilution in other gases of the vapour concerned, and the principal force acting on the droplets is surface tension which varies with the composition of the vapour and gas. Tar and oil, which are of high viscosity, are readily precipitated mechanically. Sulphuric acid mists are much more difficult to precipitate, and the problem of designing sulphuric acid chambers of the greatest efficiency can be stated in terms of mist condensation, all designs employing cooling and "shock" methods for the purpose. In the condensation of sulphuric acid mists from concentration plants the Cottrell apparatus has great advantages over coke scrubbers, with or without cooling, but requires somewhat delicate operation owing to the structure of the lead apparatus and the necessity of electrical insulation.

C. IRWIN.

Detection of phosgene in chemical fire extinguishers. E. GLASER and S. FRISCH (*Z. angew. Chem.*, 1928, 41, 263—265).—Most chemical fire extinguishers contain carbon tetrachloride which on thermal decomposition in presence of oxygen produces phosgene. A number of accidents have occurred from this cause, and it has been claimed that the addition of ethylene dibromide reduces the formation of phosgene. The authors have found that by passing a mixture of dry air and carbon tetrachloride vapour through a 30 cm. tube heated at 250° and charged with anhydrous ferric chloride and pumice, phosgene up to 45% of the theoretical yield is obtained. The resultant gases were freed from chlorine and passed into aqueous aniline, and the precipitate of diphenylurea was weighed. The reaction proceeds up to equilibrium and the residual gases freed from phosgene can be further oxidised; methyl bromide, if present, increased the yield of phosgene, but ethylene dibromide reduced it. The composition of the solutions in various fire extinguishers is given, and the use of those containing carbon tetrachloride condemned as dangerous.

C. IRWIN.

Refractories in boiler furnaces. JOHNSON and KING. **Slags from boiler furnaces.** McDOWELL and LEE.—See VIII.

PATENTS.

Furnaces. STEAM PRODUCTION CORP., Asses. of F. J. CURRAN (B.P. 275,177, 11.6.27. U.S., 31.7.26).—

Part of the fuel and air is admitted below a grate through which the mixture passes and burns above it, the upper surface of the grate being provided with upstanding "radiants" or fins which provide a hot surface for promoting combustion. Another part of the fuel and air is admitted to a helical preheating space between the furnace wall and an outer wall, then enters the furnace at an angle to promote whirling some distance above the grate; these entrances also are provided with deflector plates which become hot.

B. M. VENABLES.

Furnace. E. P. STEVENS and R. J. HIMMELRIGHT, Assrs. to AMER. ARCH CO. (U.S.P. 1,656,963, 24.1.28. Appl., 16.11.22).—A section of the furnace roof, normally sealed with the side walls, is provided with means for raising it into position for forward movement along a track extending forward of the furnace. A protective shield is located beneath the section in its forward position.

H. HOLMES.

Furnace. W. M. DUNCAN (U.S.P. 1,657,113, 24.1.28. Appl., 9.1.24).—An inclined arch constituting the front wall of the combustion chamber is built of refractory blocks each comprising a head portion, a body portion provided with a heat-radiating face in the chamber, and oppositely-arranged recesses between the head and body portions. Pairs of rods extend through these recesses and through plates carried by ribs inclined parallel to the arch, and the ribs are suspended by upright rods from transverse horizontal beams in front of the arch.

H. HOLMES.

Annealing furnaces. BRIT. FURNACES, LTD., and E. W. SMITH. From SURFACE COMBUSTION CO. (B.P. 283,767, 22.3.27).—In a heating chamber a combustion chamber is formed by a partition wall along one side and the flames pass out above the partition, over, down the other side of, and underneath a muffle or annealing box to exit ports in the floor of the main chamber which lead to a recuperator. Air from the recuperator and gas from a producer enter through separate ports in the floor of the combustion chamber, and there are also ports through the partition wall so that a portion of the burnt gases from the heating chamber can be mixed with the new fuel. All three sets of ports are controlled by sliding tiles over them. The muffle is off-centre of the heating chamber so that the space round it varies roughly according to the decrease in volume of the gases as they lose their heat.

B. M. VENABLES.

Annealing furnaces. W. G. BEESTON (B.P. 283,725, 31.12.26).—The waste gases are drawn off from one end of the furnace through several flues which lead back underneath the furnace and are individually provided with dampers, so that by regulating the latter the direction of the heating gases in the furnace may be

controlled and the heat uniformly spread. Additional air enters the furnace underneath the inlet for the main combustion gases, one method of effecting this being through a hollow firebridge which has an overhanging lip to project the flames well into the furnace and improve the distribution of heat. B. M. VENABLES.

Recuperative furnace. J. L. HOTT (U.S.P. 1,658,199, 7.2.28. Appl., 14.10.25).—The hot gases from the combustion chamber are passed through pipes arranged within the recuperator chamber, and pressure devices are provided for producing a flow of the air thus heated through a conduit into the combustion chamber. Temperature-controlled means serve to diminish the amount of air supplied to the combustion chamber without varying the flow through the recuperator chamber. H. HOLMES.

Rotary kilns for burning cement, ore, and similar materials. VICKERS, LTD., and L. D. PARKER (B.P. 284,494, 23.3.27).—The kiln has its interior formed of a number of cylindrical portions inclined to the general axis of the kiln joined by cylindrical portions not inclined to, but eccentric to, the axis, so that a longitudinal section is wave-like.

B. M. VENABLES.

Rotary kilns, dryers, etc. E. ALLEN & Co., LTD., and W. J. COLES (B.P. 283,669, 29.10.26).—A rotary kiln is rotated fast enough to cause the material to cascade without using lifting devices, and means are provided to retain sufficient material in the kiln to effect the cascading, one of such means being reduced diameter of the ends.

B. M. VENABLES.

Apparatus for effecting heat transfer. H. F. SMITH, Assr. to GAS RESEARCH Co. (U.S.P. 1,656,836, 17.1.28. Appl., 16.3.22).—A water heater comprises a central, vertical, copper tube which is surrounded by the water under treatment, and up which hot gases are led. To facilitate transfer of heat the tube is provided with a number of copper gauzes which extend across it and are in thermal contact with the inner side.

T. S. WHEELER.

Cooling towers. K. W. BRANCZIK (B.P. 284,018, 21.10.26).—Cooling towers of large diameter or width are constructed in several sections with unoccupied spaces in between so that air has free access to any part of the interior, avoiding dead zones. The spaces are preferably radial with wider outer ends. An arrangement of distributing launders and footways is described.

B. M. VENABLES.

Crushing and separating apparatus. Crushing apparatus. P. L. CROWE (U.S.P. 1,654,638—9, 3.1.28. Appl., 20.10.25).—(A) Fan units are enclosed in a casing mounted on a base. A conduit projects from each side of the casing and a grinding cylinder is arranged concentric with each conduit. (B) Two plates each comprising an upper portion directed downwards and inwards, and a lower portion directed downwards and outwards, are pivoted near their adjacent apices. The upper portions constitute a crushing jaw upon oscillation of the lower portions by a cam between them.

H. HOLMES.

Conical crushing mill. H. ACKERMANN and W. LANGENHEIM (U.S.P. 1,656,895, 24.1.28. Appl., 5.2.27.

Ger., 6.8.25).—A seating element fixed to a shaft carries a conical crushing mantle upon a filling of hard lead or like material. Steps, some of which are undercut, are provided on the seating element. H. HOLMES.

Impact or beater mills. AMME-LUTHER WERKE BRAUNSCHWEIG DER "MIAG" MÜHLENBAU U. IND. A.-G. (B.P. 283,831, 28.10.27. Ger., 31.1.27).—An impact mill of the type where the beaters protrude between spaced bars is also fitted with an easily movable anvil.

B. M. VENABLES.

Grading and separation of mixed materials. F. B. PETRIE, and PETRIE & McNAUGHT, LTD. (B.P. 283,645, 14.10.26).—Material such as towns' refuse is passed over longitudinal grizzly bars where it is pushed by cross-bars and exposed for hand sorting, then through a rotary screen and magnetic separator. Provision is made for removing the non-rolling material such as garbage.

B. M. VENABLES.

Centrifugal extractor. A. R. CURTIS (U.S.P. 1,659,391, 14.2.28. Appl., 23.4.23).—The rotary basket is mounted in a vessel fitted with a lid having means for locking it in closed position. Means are also provided for starting and stopping the motor and for connecting and disconnecting the drive. The starting device for the motor is operable only when the drive is disconnected, and the connexion of the drive is possible only when the lid is closed and locked.

H. HOLMES.

Centrifugal apparatus. WESTERN STATES MACHINE Co., Asses. of E. ROBERTS (B.P. 280,167, 19.7.27. U.S., 5.11.26).—A centrifugal machine suitable, e.g., for the purification of sugar, is provided with means for rotating at a high or low speed, with a brake, and with sprays for washing. The periods of low speed for charging and of stoppage for discharging are under human control, but the periods of accelerating and high speed for separating, of spray washing, of drying, and of deceleration are under automatic (though adjustable) control.

B. M. VENABLES.

Filter for fluids. C. H. CUNO, Assr. to CUNO ENGINEERING CORP. (U.S.P. 1,657,346, 24.1.28. Appl., 19.1.27).—A filtering member is provided with radial filtering passages, and with delivery passages normal to the plane of the latter, and is displaceable angularly to enable cleaning members to be engaged with it.

H. HOLMES.

Rotary filters or strainers. F. W. BRACKETT (B.P. 284,403, 28.10.26).—In a strainer of the rotary-cup type, for large quantities of liquid such as condensing water, comprising a cylindrical rotating strainer with one closed end and with inlet at the open end and outlet through the circumference which is only partly submerged, buckets are provided within the strainer which lift refuse from the outlet side and carry it across to the other side where it is caught by a hood and launder and conveyed away by a separate stream of water. To preserve a good seal between the rim of the open end and the fixed structure of the screen, rollers are placed on the closed end which run over fixed rails and relieve the shaft of canting strain.

B. M. VENABLES.

Vacuum-filter liquid guard. G. S. BACKUS, Assr. to OLIVER CONTINUOUS FILTER Co. (U.S.P. 1,654,717, 3.1.28. Appl., 9.12.26).—An enclosed vacuum and

pressure chamber behind the filter wall is provided with suction pipes for drawing a liquid through the wall and with pipes for supplying a gas or liquid for blasting off the filter cake. A metal plate extends across the filter in front of the blasting pipes to prevent direct impact of the fluid therein on the filter wall. H. HOLMES.

Separation of liquids of different gravity. W. LINNMAN, JUN. (U.S.P. 1,655,191, 3.1.28. Appl., 22.11.26. Ger., 8.6.26).—A tank provided with an inlet encloses a container closed at the top and open to the tank below, and an outlet pipe projects from the container. The upper part of the container communicates with the tank by a passage extending downwards to a level above the inlet end of the pipe. H. HOLMES.

Apparatus for purifying or cooling gases by treatment with a liquid. A. TOMSON (B.P. 283,682, 13.11.26).—The gas passes in one direction through an inner cylindrical casing, where it is sprayed with the liquid and returns through a surrounding annular space which is provided with a helical baffle to remove the mist of liquid. B. M. VENABLES.

Treatment of gases with liquids. M. D. MANN, JUN., Assr. to S. B. HUNT (U.S.P. 1,654,181, 27.12.27. Appl., 14.5.20).—The gases are treated with immiscible liquids in a chamber arranged between inner and outer chambers, through which is passed a temperature-controlling fluid. The liquid aggregate is introduced into the treating chamber near the top, and as it descends is separated into strata, and the gases, introduced near the bottom, bubble through the successive strata. H. HOLMES.

Colorimeter [for liquids]. C. ZEISS, Assec. of L. LORBER (B.P. 280,552, 9.11.27. Hung., 10.11.26).—Light from a common source is divided into two beams which pass through a pair of comparator units and is viewed as a disc divided into halves of colours which are to be equalised. Each comparator unit comprises a fixed outer glass reservoir for liquid, an inner glass reservoir which can be moved (vertically) parallel to the beam of light, and a fixed glass cylinder which extends to the bottom of the inner reservoir when the latter is in its uppermost position. The bottoms of the reservoirs and the ends of the rod are all truly perpendicular to the beam of light. The comparator is suitable for estimating the amount of coloured solute even when the solvent itself is coloured and/or turbid. B. M. VENABLES.

Viscosity tester for oils. F. B. MASON (U.S.P. 1,659,534, 14.2.28. Appl., 16.1.26).—Containers are mounted in a frame, and a receptacle is secured within one end of each container. Means are associated with each receptacle for determining the time required by liquid filling the receptacle to flow therefrom. H. HOLMES.

Flask for laboratory purposes. H. THIENE, P. H. PRAUSNITZ, and G. SCHOTT, Assrs. to JENAER GLASWERK SCHOTT & GEN. (U.S.P. 1,659,383, 14.2.28. Appl., 18.12.26. Ger., 12.12.25).—The lower end of the flask is shaped as a frustum of a cone tapering downwards at an angle of not less than 90° to the slope of the body portion. The lower end of the frustum provides a base perpendicular to the axis of the flask, and the side of the frustum is

flattened to provide a second base oblique to the axis. H. HOLMES.

Process and apparatus for heat transfer. R. W. DAVENPORT, Assr. to CHICAGO PNEUMATIC TOOL CO. (U.S.P. 1,659,681—2, 21.2.28. Appl., [A] 25.9.25, [B] 9.10.25).—See B.P. 258,847; B., 1927, 688.

Temperature-controlling devices for furnace chambers and other apparatus. N. H. FREEMAN (B.P. 284,008, 19.10.26).

Reversing apparatus for gas, air, and waste-gas valves of gas-fired furnaces, e.g., regeneratively heated coke ovens, metallurgical furnaces, etc. KELLNER & FLOTHMAN G.M.B.H. (B.P. 275,206, 13.7.27. Ger., 31.7.26).

Purification of liquids (U.S.P. 1,654,812).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Low-temperature assay [of coal] in the Fischer aluminium retort. HEMPEL (Brennstoff-Chem., 1928, 9, 3—6).—The tar and liquor are collected in a water-cooled receiver the lower end of which is narrow and graduated and carries a tap. The weight of tar and liquor together is noted, and the receiver is then immersed in warm water (about 70°), whereby the tar melts and separates from the water. The latter is run off, the warming and separation are repeated twice, and the remaining tar (containing water not exceeding 0.12% of the original coal) is weighed. The method gives rather lower results for the tar yield than the older method of weighing the water and not the tar. A lignite containing 47.25%, and, after partial drying, 13.52% of water gave by the old method 23.24% and 21.48% of tar, and by the new method 21—47% and 20—63% of tar, respectively. W. T. K. BRAUNHOLTZ.

Comparison of the methods commonly used in Germany and Holland for determining the content of volatile matter of coals. D. J. W. KREULEN (Brennstoff-Chem., 1928, 9, 2—3).—The methods in use in Germany and Holland, which differ mainly in the intensity of the flame heating the crucible, give concordant results provided that air-dried coal is used in both. The use of absolutely dry coal, which is often hygroscopic, may entail errors in weighing in a damp atmosphere. W. T. K. BRAUNHOLTZ.

Graphitic oxide and the Brodie reaction. O. W. STOREY (Amer. Electrochem. Soc., April, 1928. Advance copy. 10 pp.).—The action of the Brodie mixture (fuming nitric acid and potassium chlorate) on Acheson graphite and on samples of lampblack calcined at various temperatures has been examined. Lampblack calcined at 1100—1200° gave a slightly oxidised product after two treatments with the Brodie mixture, but in subsequent treatments the graphitic oxide was destroyed and the whole material went into solution. By calcination at higher temperatures the lampblack became more resistant, but grey or creamy white oxidation products could eventually be obtained. Acheson graphite is still more resistant and yields a pale yellow oxidation product after about 8 treatments with the oxidising mixture. H. J. T. ELLINGHAM.

Determination of asphalt in minerals. E. CASIMIR (Petroleum, 1928, 24, 315—318).—Two methods for the determination have been critically examined; extraction with benzene or chloroform, using the Soxhlet apparatus, was found to give higher results than treatment with a mixture of hydrochloric acid and ether (Prettner method). This was due to conversion of part of the asphalt into an ether-insoluble form by the action of the acid, and also, to a less extent, to loss of sulphur. In both methods the extracted asphalt contains ash which is carried in colloidal solution; chlorine also is present in the asphalt after acid extraction. R. H. GRIFFITH.

Determination of the tar value [of mineral oils]. H. VON DER HEYDEN and K. TYPKE (Chem.-Ztg., 1928, 52, 150).—The method suggested by Marcusson and Bauerschäfer (B., 1926, 427) gives results which, in the majority of cases, differ considerably from those obtained by the specification method, and cannot be regarded as a substitute for the latter. W. J. POWELL.

Constituents of low-temperature tar. E. BÖRNSTEIN (Nature, 1928, 121, 356—357).—A statement of priority involving a correction. A. A. ELDRIDGE.

[Constituents of low-temperature tar.] G. T. MORGAN and D. D. PRATT (Nature, 1928, 121, 357).—An acknowledgment of Börnstein's explanation (cf. previous abstract). A. A. ELDRIDGE.

Separation of the components of petroleum. VI. **Action of glacial acetic acid.** P. F. GORDON and J. MERRY (J.S.C.I., 1927, 46, 429—432 T).—After preliminary tests, crude Persian petroleum (d_{4}^{20} 0.839) was extracted with glacial acetic acid, leaving a residual oil which was filtered to remove asphaltic matter. The acid-soluble portion was fractionally precipitated by the successive addition of small quantities of water. The fractions thus obtained showed a steady increase in sp. gr. of the precipitated oil as water was added of from 0.860—0.962. The acid-insoluble oil was dissolved in ether and fractionally precipitated by successive additions of small quantities of absolute alcohol. The heaviest fractions precipitated first had approximately the same sp. gr., diminishing on further additions of alcohol. After addition of excess of alcohol, a further fraction was precipitated by adding water. This was an oil of a different type from the others, having a high sp. gr. and a terpene-like odour. All the fractions except the last (which had the lowest viscosity) set above 0° and contained paraffin wax. The asphalt insoluble in glacial acetic acid was dissolved in carbon disulphide and decolorised by warming with dry bone-char. On removal of the solvent, a white crystalline wax was obtained, m.p. 67—75°. The remaining liquid contained a viscous, brown, fluorescent liquid. For comparison, crude oil was dissolved in ether, and successive quantities of alcohol were added to give fractions of diminishing sp. gr. A final fraction was precipitated by the addition of water. A study of the constituents of the oils soluble and insoluble in glacial acetic acid shows that the acid-soluble portion contains the lightest components and heaviest oils but no wax, whilst the acid-insoluble oils are of intermediate density and contain all the wax other than that present in the asphaltic portion.

H. S. GARLICK.

Action of sodium-potassium alloy on petroleum. J. B. CONANT and A. H. BLATT (J. Amer. Chem. Soc., 1928, 50, 542—550).—When treated with sodium-potassium alloy in dry ether at the ordinary temperature, certain crude petroleum yield intensely coloured metallic derivatives, which are converted by carbon dioxide into a mixture of salts of carboxylic acids. The following acids, obtained in this manner from the oils mentioned, are described: Pennsylvania crude, m.p. 90—100°, decomp. about 150°; Mid-continent crude, m.p. 120—130°, decomp. about 170°; Mid-continent cracking-coil residue, m.p. 140—150°, decomp. about 185°; California crude, m.p. 125—130°, decomp. about 150°; Columbia crude, m.p. 110—120°, decomp. about 140°. The acids appear to be dibasic, and the first-named acid yielded an imide. They also appear to contain one aromatic nucleus for each carboxyl group. Treatment of the Pennsylvania oil, after complete reaction with sodium-potassium at the ordinary temperature, with the alloy at 250° in presence of carbon dioxide, affords alkali salts of acids, together with a neutral, amorphous product. F. G. WILLSON.

Spontaneous decomposition reactions of berginisation: a reaction common to various petroleum hydrocarbons. E. PYHÄLÄ (Petroleum, 1928, 24, 308—314).—In connexion with attempts to lower the f.p. of still residues from Baku petroleum, a study was made of the behaviour of such substances in a cracking still of special design. A residue from Grosny petroleum was heated slowly to 450°, when a sudden rapid distillation and gas evolution occurred; 35% of the oil was recovered in this distillate, and by raising the temperature above 465° no further product was obtained. Three further experiments were carried out with Tscheleken petroleum, and it was found that reaction could first occur at about 380—400°, but that a second decomposition took place at 410—425° and produced about twice as much distillate as the first. Continued heating, up to 550° produced only negligible quantities of distillate, but the rate of the initial temperature rise materially affected the proportions of benzene, kerosene, etc. formed. 71% of the paraffin and 90% of the asphalt were found to have reacted without giving pitch or coke. Two experiments with an oil from Surachany, containing appreciable amounts of lubricating oil, showed that similar reactions could take place, and high percentages of paraffins and asphalts were decomposed. On account of the general occurrence of this phenomenon and the similarity between the temperatures required and those employed in the Bergius process, it is suggested that reactions of this type are always essentially concerned in the berginisation of coals. R. H. GRIFFITH.

Sulphuric acid test for mineral oils. K. TYPKE (Petroleum, 1928, 24, 314).—It has been proposed to test transformer and similar oils by their behaviour with strong sulphuric acid, the better samples showing less reaction, but the value of this method is now questioned. Apart from lack of information as to the true significance of the test, it is suggested that the presence of limited quantities of substances which are removed by the acid is desirable in order to increase the resistance of the oil to oxidation. R. H. GRIFFITH.

[Physiological] effects of "ethyl gasoline" and its combustion products. R. R. SAYERS, A. C. FIELDNER, W. P. YANT, and B. G. H. THOMAS (U.S. Bur. Mines, 1927, 447 pp.).—Animals exposed for eight months to the combustion products of "ethyl gasoline" showed no distinct storage of lead or symptoms of lead poisoning. The threshold concentration for lead dust is 0.14—1.4 mg./m.³ Vapour concentrations of 0.3—1% (but not of 0.01—0.1%) caused storage of lead and symptoms of poisoning. Skin absorption experiments were also conducted.

CHEMICAL ABSTRACTS.

Free-burning carbonised fuels for the open fire. H. GREGER (J. Fuel Soc. Japan, 1927, 6, 102—110, and J. Akita Min. Col., 1927, No. 3).—See B., 1927, 864.

Graphitic oxide. BROWN and STOREY, also BROWN.—See XI.

PATENTS.

[Sub-heated] coke ovens. N.V. SILICA EN OVENBOUW MIJ. (B.P. 270,765, 9.5.27. Ger., 8.5.26).—To prevent dirt falling into the controlling devices of the gas supply of vertical-flued ovens the gas is fed from a horizontal pipe into the vertical pipe which has at its lower end a removable plug forming a dust box. In the refractory material at the upper end is another duct closed by a plug which permits of inspection from the top to the bottom of the gas-supply ducts and the process of combustion, and cleaning can be carried out without interruption of the working of the ovens.

A. C. MONKHOUSE.

Heating of pulverised fuel. H. A. PROCTOR (B.P. 283,388, 30.4.27).—Pulverised fuel is distilled in horizontal chambers arranged one above the other and communicating with each other at the ends to form a continuous conduit. The fuel is conveyed by rotating paddles along the distillation chambers, which are arranged on one or both sides of a combustion chamber, and are provided with water-, steam-, or air-circulating pipes for regulating the temperatures therein.

A. B. MANNING.

Apparatus for the dry-cooling of coke. SOC. ANON. DES FOURS À COKE SEMET-SOLVAY & PIETTE (B.P. 282,286, 30.5.27. Belg., 5.5.27).—The coke is discharged from the oven into a chamber in which the walls of the coke receptacle are arranged to give a maximum exposed surface of coke. The boiler tubes are situated above and parallel with the coke surfaces. In the lower part of the chamber is a perforated bell for distributing the circulating gases from the fan through the coke, and after imparting their heat to the boiler tubes the gases pass through a chamber for dust removal.

A. C. MONKHOUSE.

Preparation of solid fuel mixtures. J. M. W. KITCHEN (B.P. 282,118, 15.6.26).—Coke or hard coal of relatively large size is mixed with soft bituminous slack, and for fusible coals anthracite dust is also added. A smokeless fuel is obtained by carbonising the mixture in a gas retort or travelling grate heated by the combustion of the carbonisation gases. [Stat. ref. to B.P. 217,236, 18,156 of 1895, and 4487 of 1887.]

A. C. MONKHOUSE.

Production of semi-coke briquettes from bituminous coal. KOKS- U. HALBKOKS-BRIKETTIERUNGS-GES.M.B.H. (B.P. 279,020, 9.3.27. Ger., 14.10.26).—Briquettes are made by mixing fine bituminous coal with a binder consisting of a solution of one or more water soluble, preferably inorganic, substances which do not soften on heating, e.g., a mixture of sulphite waste liquor or cellulose pitch and clay, or coal dust emulsified with sulphite waste liquor. The waste liquor can be replaced by size, water-glass, etc. The briquettes are then dried and carbonised in a shaft furnace.

A. C. MONKHOUSE.

Apparatus for carbonising fuels, particularly coal. R. TORMIN (B.P. 270,277, 19.4.27. Ger., 30.4.26).—Coal is carbonised in the form of briquettes in an apparatus consisting of a number of flat boxes with transverse partitions and corrugated bottoms which, when placed together, form hollow spaces joined together in rows and having the rhombic shape of the desired briquettes. When the juxtaposed boxes are placed upright, these spaces can readily be filled up with coal dust, since the inclined walls of the spaces are steeper than the angle of cohesion of ground coal. After carbonisation in a suitable retort the briquettes can be simultaneously discharged from the apparatus.

A. B. MANNING.

Carbonisation of briquettes and carbonisation plants therefor. MIDLAND COAL PRODUCTS, LTD., and C. INGMAN (B.P. 282,511, 5.10.26).—In the manufacture of briquettes, carbonisation is carried out in a primary retort under normal conditions, and the gas evolved is used in the heating of steam superheaters. The steam is employed for the carbonisation in secondary retorts either by itself or with a small proportion of air. A rich gas and higher yields of coked briquettes and oil are obtained than in the prior process (cf. B.P. 242,783; B., 1926, 37).

A. C. MONKHOUSE.

Distillation of carbonaceous substances. W. E. EVANS. FROM CARLSHÜTTE A.-G. F. EISENGIESSEREI U. MASCHINENBAU (B.P. 283,259, 2.10.26).—Coal, or similar fuel, is carbonised in a vertical retort by the transverse passage through it of a heating medium such as flue gases or steam. The retort is divided into zones by horizontal partitions. The heating medium passes first through the coke in the cooling zone, and then, via a heat exchanger arranged in the outlet chamber of the distillation zone, through the drying zone, or, after having heat supplied to it, is used directly for the distillation. While the heating medium passes through the heater, flue gases from the latter may be added to it if desired.

A. B. MANNING.

Distillation of solid fuels at low temperatures. COMP. DES MINES DE BRUAY (B.P. 272,522 and 282,340, [A, B] 9.6.27. Fr., [A] 14.6.26, [B] 18.12.26).—(A) Briquettes of pulverised coal or other fuel are imbedded in a mass of powdered material, e.g., silica, magnesia, kieselguhr, or metal filings, and carbonised at a low temperature with a carefully regulated rate of heating. A strong coke is produced which burns without smoke. (B) In a modification of the above process the imbedding material itself consists of carbonaceous material, e.g., dust from coal schist or roof coal, waste peat, sawdust,

vegetable waste, etc. The fuel also before briquetting is mixed with powdered charcoal. A. B. MANNING.

Recovery of liquid products from coal. H. NOVÁK and A. BROD (B.P. 283,269, 5.10.26).—Coal or brown coal is heated in an autoclave with an aromatic hydrocarbon boiling above 230°, the temperature being raised gradually from 200° to 500° in such a way that first the low-boiling products and then the high-boiling aromatic hydrocarbons are distilled off and condensed; the residual oils are then cracked above 380° and finally coked at 500°. Liquid homologues of naphthalene, fractions of anthracite or generator tar, or mixtures of these with solid aromatic hydrocarbons form suitable high-boiling liquids for the process. Considerably increased yields of oils are obtained, whilst the pressures necessary are comparatively low (25–30 atm.). A. B. MANNING.

Manufacture of activated charcoal. E. URBAIN (B.P. 257,269, 17.8.26. Fr., 22.8.25).—Carbonaceous materials are heated together with compounds containing oxygen, *e.g.*, metallic or metalloid oxides or hydroxides, at 800–1100°, without the introduction of gases. The oxides or hydroxides, which must be capable of reduction to metal or metalloid under the conditions specified, effect the removal of hydrogenated compounds from the material without reacting substantially with the charcoal. Substances other than oxides or hydroxides, *e.g.*, zinc chloride, which, however, yield oxides when heated to the temperatures used, *e.g.*, by interaction with the moisture present, can be used in the process. If the activating agent is highly volatile the impregnation and heating may be repeated.

A. B. MANNING.

Production of carbon electrodes. I. SZARVASY (B.P. 284,818, 6.12.26).—The mixture of carbon and tar-like products formed by decomposing gaseous hydrocarbons at high temperatures is coked, preferably in an atmosphere containing hydrogen and methane or other hydrocarbons, at a temperature not exceeding 1700°, and the product worked up into electrodes by being ground, mixed with a binder, moulded, and calcined. The hard carbon deposited on the walls of the decomposition vessel may be mixed in with the coked product or may be worked up separately into electrodes.

A. B. MANNING.

Gas generators. C. W. ANDREWS and W. B. CHAPMAN (B.P. 284,805, 22.11.26).—In a gas generator provided with a beam which rotates over the grate for the purpose of discharging the ash and agitating the fuel bed the ash is collected in an annular trough attached to the beam, the rate of removal of ash from this trough being controlled by an adjustable plough projecting thereinto. A pressure chamber beneath the grate serves to receive the air, steam, etc. fed to the generator. The inner faces of the side walls are formed with ridges which co-operate with the ends of the beam to grind up any large clinkers in the ash as it passes to the trough. The beam and the driving ring to which it is attached may be hollow, in which case means are provided for the passage of cooling water through them. A. B. MANNING.

Working of water-gas and double-gas producers. H. STRACHE (B.P. 269,936, 23.4.27. Austr., 24.4.26).—In operating a gas producer with an alternate "blow"

and "run," instead of removing coke from the producer and thereby increasing the calorific value of the gas by augmenting the proportion of distillation gases therein, the coke is gasified in the producer by prolonging the "blow," the carbon monoxide produced being used for heating or power purposes. The higher temperatures attained increase the efficiency of the process during the "run."

A. B. MANNING.

Production of gaseous fuels. SYNTHETIC AMMONIA & NITRATES, LTD., and K. GORDON (B.P. 282,141, 11.9.26).—Water-gas is produced from finely-divided, high-grade, solid fuel and steam by blowing the mixture, with or without oxygen or air, through an externally heated reaction vessel consisting of an alloy-steel tube or tubes made from "Staybrite," "Era A.T.V.," or "V2A." Using a tube 30 ft. long and $\frac{1}{2}$ in. diam. heated at about 1000°, coke and steam were converted into a water-gas containing 51% H₂, 48% CO, 1% CO₂; the time of contact was 1 sec.

A. C. MONKHOUSE.

Manufacture of producer gas. C. W. ANDREWS (B.P. 260,628, 1.11.26. U.S., 31.10.25).—Producer gas containing carbon monoxide as its main combustible constituent is made by passing air, mixed with steam or metallurgical gases, alternately up and down through a deep fuel bed in such a manner that the upper layer and the lower layer (and ash) of the fuel serve alternately as regenerators; or the hot producer gas may be passed through a separate regenerator, which is used to heat the air for the succeeding "blow."

A. B. MANNING.

Production of volatile hydrocarbons such as gasoline, benzene, or other hydrocarbon oils contained in natural gas, coal gas, water-gas, air, or other gaseous mixtures. M. OKOCHI (B.P. 282,541, 12.11.26).—The gaseous mixture is compressed, cooled, and then suddenly expanded into a tower packed with absorbing material. The dried gases leave the tower and are used for heating purposes. The temperature in the tower is controlled by means of the admission of uncompressed gas. When the absorption material becomes saturated the volatile materials are expelled by a current of hot gas and condensed; the tower is then cooled ready for subsequent absorptions.

A. C. MONKHOUSE.

Distillation of tar and other viscous substances, and apparatus therefor. J. F. CARMICHAEL and J. F. CARMICHAEL & Co., LTD. (B.P. 282,525, 28.10.26).—The material to be distilled is distributed on to an annular or spiral coil or series of coils situated in a chamber. The coils are heated internally and the material flows by gravity over the surface of the coils and is withdrawn at the bottom of the chamber. The vapours evolved pass from the top of the chamber to a condenser.

A. C. MONKHOUSE.

Production of a composition of matter [from pitch]. L. BOLGAR (B.P. 277,291, 2.5.27. Hungary, 13.9.26).—Molten coal-tar pitch or petrol pitch (with, if desired, natural asphalt) is mixed with not more than 5% by wt. of sulphuric acid (*d* 1.53), and heated to 200–250°; the liquid product, after being mixed with filling material, *e.g.*, sand, stone-meal, clay, etc., is cast into the desired shape. H. ROYAL-DAWSON.

Production of gasoline from natural gases.

E. B. BIRD (U.S.P. 1,656,813, 17.1.28. Appl., 19.3.27).—Natural gas is circulated at 400–500° over a catalyst comprising a mixture of copper, nickel, gold, platinum, and palladium to form liquid hydrocarbons which are condensed and separated. T. S. WHEELER.

Breaking of petroleum emulsions. M. DE GROOTE and W. C. ADAMS, Assrs. to W. S. BARNICKEL & Co. (U.S.P. 1,656,622–3, 17.1.28. Appl., 11.8.26).—(A) A tar acid oil is treated with sufficient sulphuric acid to sulphonate only the phenols present, the product is mixed with oleic acid which has been treated with sulphuric acid, and the mixture is treated with this acid to give a product containing a Twitchell reagent. (B) A mixture of a phenol, *e.g.*, β -naphthol, and a fatty acid, *e.g.*, oleic acid, is sulphonated to give a Twitchell reagent. Both products induce rapid separation of petroleum emulsions. T. S. WHEELER.

Apparatus for the conversion of hydrocarbon oils. E. T. HESSLE and W. LÖLGEMANN (U.S.P. 1,658,985, 14.2.28. Appl., 20.3.25).—Oil and gas are introduced into a reaction kettle through opposed nozzles, whereby a spray of oil and gas is produced and brought into contact with a catalyst. C. O. HARVEY.

Control of pyrolytic conversion [of hydrocarbon oils]. J. R. CARRINGER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,656,724, 17.1.28. Appl., 28.5.23).—The oil is heated to a high temperature and passed to a chamber in which cracking is allowed to progress at a slightly lower temperature. To control this lower temperature a desired portion of the oil vapour passing to the chamber is led through a coil cooled by air. T. S. WHEELER.

Cracking of hydrocarbon oils. C. P. DUBBS (B.P. 284,507, 29.4.27).—In a process involving the continuous cracking of hydrocarbon oils by passage through a heating coil (at about 427–482°) to a reaction zone and dephlegmator, the proposed introduction of an incondensable gas to the reaction zone (*cf.* B.P. 249,604; B., 1926, 525) is modified by subjecting the gas to a separate external heating operation (at about 644–816°) and making use of gas other than that produced during the cracking operation. C. O. HARVEY.

Cracking of [hydrocarbon] oil. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,659,398, 14.2.28. Appl., 11.1.26).—Oil is cracked continuously under its self-generated super-atmospheric vapour pressure by passage through a cracking coil to the large end of a conical expansion chamber, wherein the rate of flow of the evolved vapours (and the rate of conversion) is gradually increased as they pass axially along the cone. The vapours, passing out from the apex of the conical chamber, pass through a dephlegmating column, and the condensate therefrom is combined with the feed oil, the remaining vapours being condensed and collected. C. O. HARVEY.

Apparatus for treatment of hydrocarbons. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,659,397, 14.2.28. Appl., 23.7.20. Renewed 2.9.26).—A horizontal cylindrical still and a heating element contained therein are arranged

to rotate in opposite directions. The still is fitted with an inlet for oil, and with outlets for residuum and vapours, the last being refluxed and the resulting condensate being returned to the still. C. O. HARVEY.

Refinery power plant [for hydrocarbon oils]. M. J. TRUMBLE (U.S.P. 1,658,910, 14.2.28. Appl., 1.12.22).—Hot oil is delivered to a regulating device and the expansion of the evolved vapours is used to operate a turbine. A substantially constant pressure is maintained by adding vapour to the hydrocarbon vapours from the oil. C. O. HARVEY.

Burning of oil fuel. J. E. HACKFORD (B.P. 284,777, 4.11.26, 8.1.27, and 18.2.27).—Oil fuel is burnt without atomisation by passing a steady stream of air over a concave surface (a cup or trough) which is covered with a thin film of the oil, the oil being fed to the surface at such a rate as to maintain this film. Means are provided for varying the heat intensity of the flame by adjusting the direction etc. of the air current. In its simplest form the apparatus consists of a cup into which the oil is allowed to drip from a gravity feed. C. O. HARVEY.

Manufacture of refined lubricating and like oils. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (B.P. 263,167, 15.12.26. Holl., 15.12.25).—Oils of low "cloud test" (*i.e.*, comparatively free from paraffin wax) are produced by extraction at a low temperature with liquid sulphur dioxide, which yields an extract free from wax and a residue saturated in sulphur dioxide, from which the wax may be removed by filtration. The sulphur dioxide reduces the viscosity of the mixture to be filtered and the solubility of the wax in the oil; its removal by evaporation is easily effected, and the pressure developed may be used to force the oil through the filters etc. The acid extract containing aromatic hydrocarbons etc. may, if desired, be recombined with the de-waxed residue. C. O. HARVEY.

Preparation of artificial petroleum and de-colorising carbonaceous substances from rubber scrap or vulcanised rubber waste. H. NISHIDA and K. SHIMADA (B.P. 282,565, 22.1.27).—Rubber waste or the oil obtained from its distillation is mixed with a natural bleaching earth, and distilled in an iron retort to 350°. The distillate is fractionated and washed with sulphuric acid and caustic soda. The residue is in the form of a black powder. A. C. MONKHOUSE.

Manufacture of agglomerated carbonaceous material. E. URBAIN, Assr. to URBAIN CORP. (U.S.P. 1,659,931, 21.2.28. Appl., 29.4.24. Fr., 25.6.23. Renewed 20.7.27).—See B.P. 218,242; B., 1925, 435.

Gas-purification process and apparatus. E. H. BIRD, Assr. to KOPPERS Co. (U.S.P. 1,660,741, 28.2.28. Appl., 24.11.22).—See B.P. 207,488; B., 1924, 459.

Production of lighter hydrocarbons. E. S. ANDREWS. From B. ORMONT ASSOCIATES, INC. (B.P. 285,199, 11.12.26).—See U.S.P. 1,608,664; B., 1927, 163.

Dehydration of alcohol for carburants. E. RICARD and H. GUINOT, Assrs. to SOC. RICARD, ALLENET, & C^{IE}. (U.S.P. 1,659,958, 21.2.28. Appl., 27.9.23. Belg., 30.9.22).—See B.P. 204,697; B., 1924, 899.

Smoke washers. W. W. ROBINSON (B.P. 284,919, 7.6.27).

Regeneration of lubricating oils contaminated in thermal engines. L. MIRLESSE and A. I. KOUSNETZOFF (B.P. 269,148, 14.3.27. Fr., 7.4.26).

Viscosity tester for oils (U.S.P. 1,659,534).—See I. **Deodorisation of isopropyl alcohol** (U.S.P. 1,657,505).—See III.

III.—ORGANIC INTERMEDIATES.

Catalytic chlorination of acetic acid to chloroacetic acid. H. BRÜCKNER (Z. angew. Chem., 1928, 41, 226—229).—The velocity of chlorination of acetic acid in presence of iodine, phosphorus, or sulphur increases with the amount of catalyst used, but not in direct proportion. Of the three catalysts iodine gives the best results, but more effective chlorination is obtained with mixtures of three catalysts, chlorination in presence of a mixture of iodine, phosphorus, and phosphorus pentachloride being more rapid than in presence of sulphur, phosphorus pentachloride, and phosphorus. Binary mixtures of catalysts are less effective than the ternary mixtures. Acetic anhydride is chlorinated in the absence of a catalyst, and in the chlorination of acetic acid the presence of chloroacetyl chloride and the mixed anhydride of acetic and chloroacetic acid has been established. The suggested mechanism of the chlorination is represented by a series of equations. Sulphur and phosphorus appear to act by accelerating the formation of acetyl chloride, and iodine by the dispersion of the chlorine through the reaction mixture in consequence of the formation of iodine chloride and iodine trichloride. R. BRIGHTMAN.

Determination of alcohol in solutions containing acetone. J. M. MACOUN (J.S.C.I., 1928, 47, 43—44 T).—About 50 c.c. of the sample are weighed into a 500 c.c. Kjeldahl flask, benzaldehyde is added in excess (3 c.c. for every 1 c.c. of acetone supposed to be present) and 1 g. of solid potassium hydroxide to 10 c.c. of aqueous liquid, and the mixture digested at its b.p. for 30 min., after which 100 c.c. of water are added to the cooled liquid and the mixture is distilled, using a 500 c.c. separating funnel as receiver. The distillate consists of alcohol, water, and excess of benzaldehyde. To not less than 100 c.c. of distillate 50 c.c. of saturated salt solution and enough solid sodium chloride to saturate the whole are added, followed by 75 c.c. of light petroleum, and the benzaldehyde extracted by Thorpe and Holmes' method (B., 1903, 232). The ethereal layer is washed with two successive portions (25 c.c.) of salt solution, and the extract and washings are distilled into a tared flask fitted with a glass stopper. The weight of the distillate (which should not be less than 100 c.c.) and its sp. gr. are ascertained, and from this the percentage of alcohol by wt. is calculated. If the n_D test for indicating methyl, ethyl, or isopropyl alcohol is to be relied on, the trace of benzaldehyde in the distillate must be eliminated, either by an elaboration of the above method, or less satisfactorily by the action of phenylhydrazine hydrochloride on the benzaldehyde before final distillation. D. G. HEWER.

Nephelometric method for determining benzene in alcohol. L. DESVERGNES (Ann. Chim. analyt., 1928,

[ii], 10, 6—11).—The approximate solubility of benzene in water and in alcohol-water mixtures has been determined for several temperatures. The turbidity produced when alcohol-water solutions of benzene are cooled to -15° may be used to determine the percentage of benzene by comparison with the clouds formed by solutions containing known amounts of the hydrocarbon. Should no cloud be produced, distillation gives an initial benzene-rich fraction, in which the benzene concentration may be determined as described. L. M. CLARK.

Determination of anthracene and anthraquinone. H. PIRAK (Z. angew. Chem., 1928, 41, 231—233).—In the original Höchst method the results obtained are too low on account of over-oxidation; the modified method of Rhodes and Sielisch (B., 1926, 941) gives low results through incomplete oxidation, and a slight excess of chromic oxide solution over the amount indicated by them is necessary for accurate results. The time required for the determination may be shortened by diluting the hot sulphuric acid solution of anthraquinone with boiling sulphuric acid. Loss by sublimation may be high at 110° in the original Höchst test (cf. Rhodes and Sielisch, *loc. cit.*) but is reduced by use of the crucibles advocated by Jacobsohn (B., 1926, 734). A lower temperature is, however, preferred. In the procedure recommended, anthracene (1 g.) is dissolved and oxidised as in Sielisch's method, a further 1 c.c. of the chromic acid solution being added when the solution is definitely brown. After cooling and dilution with 400 c.c. of ice-cold water, the anthraquinone is collected on a Jena glass filter-crucible, washed as in Jacobsohn's method, and dried at $95-100^\circ$. The contents are then transferred to a glass beaker and heated with 10 c.c. of sulphuric acid monohydrate at 105° (boiling brine-bath) for 20 min.; acetic acid (25 c.c.), free from traces of reducing impurities and previously heated to 100° , is added. On cooling, the anthraquinone separates as a white lump at the bottom of the beaker. When the contents reach room temperature, the beaker is placed in ice for 20 min. and 200 c.c. of cold water are then added, rise of temperature being avoided by occasionally cooling in ice. The anthraquinone is collected on the original filter, washed as before, and dried at $95-100^\circ$ to constant weight, after which it is sublimed at $180-200^\circ$ (1 hr.) in a drying oven. The door of the oven is not completely closed, and the stopper carrying the thermometer is notched to allow of a slight air-current. The determination is complete in 4—5 hrs., and gives satisfactory results with technically pure anthracene and anthraquinone, and with crude anthraquinone. With mixtures containing less than 50% of anthraquinone the method gives low results for anthraquinone, and consequently high values for anthracene. R. BRIGHTMAN.

n-Butyl alcohol, acetone, and methyl alcohol from maize. TROPSCH.—See XVIII.

PATENTS.

Method of retarding or preventing the corrosion of metal containers containing alcohol. U.S. INDUSTRIAL ALCOHOL Co., Assees. of W. T. SCHREIBER (B.P. 269,135, 1.12.26. U.S., 6.4.26).—Containers (especially those made of tin plate, terne plate, or iron) for commer-

cial alcohols, especially commercial or denatured ethyl alcohol, may be protected against corrosion by dissolving in 100 c.c. of the alcohol 0.01—0.25 g. of soap (*e.g.*, alkali soaps of palmitic, stearic, or oleic acid, etc., or rosin soap) or soap-forming materials (such as the higher fatty acids).
B. FULLMAN.

Deodorisation of isopropyl alcohol. C. S. HUDSON, ASSN. TO STANDARD DEVELOPMENT CO. (U.S.P. 1,657,505, 31.1.28. Appl., 28.6.21).—Calcium hypochlorite and hydrochloric acid are added to isopropyl alcohol, prepared from hydrocarbon material, and the mixture is agitated.
H. ROYAL-DAWSON.

Production of acetic anhydride. C. RUZICKA (B.P. 283,781, 27.5.27).—Sulphur trioxide fumes, produced by direct distillation of the trioxide or by oxidation of sulphur dioxide with air or oxygen (preferably activated or ozonised) in the presence of a catalyst, are conducted into cooled acetic acid, glacial or otherwise, with agitation. The product may be used directly (*e.g.*, in the acetylation of wood cellulose), or distilled for the separation of acetic anhydride.
B. FULLMAN.

Manufacture of acetic acid. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 284,143, 7.7.27. Ger., 7.4.27).—A small quantity of calcium acetate is dissolved in acetic acid and treated with sulphuric acid. Thereafter acetic acid may be continuously produced by dissolving calcium acetate in the paste of acetic acid and calcium sulphate and adding sulphuric acid. Excess of the latter is avoided.
B. FULLMAN.

Manufacture of [crystalline] urea. I. G. FARBEININD. A.-G. (B.P. 266,378, 19.2.27. Ger., 19.2.26).—Needle-shaped crystals of urea are produced by exposing drops of a solution containing between 50% and 90% by wt. to slow cooling in a current of air or other gas.
B. FULLMAN.

Manufacture of acylamides. R. B. TRUSLER, ASSN. TO ROESSLER & HASSLACHER CHEMICAL CO. (U.S.P. 1,656,252, 17.1.28. Appl., 27.12.26).—Primary or secondary amines, which are less basic than ammonia, and react with acid anhydrides to form amides, are heated with amide hydrogen sulphates to yield the corresponding alkylated amide and ammonium hydrogen sulphate. *E.g.*, aniline and formamide hydrogen sulphate give formanilide.
T. S. WHEELER.

Manufacture of aromatic hydrocarbons. I. G. FARBEININD. A.-G. (B.P. 258,608, 17.9.26. Ger., 12.10.25).—Aromatic (especially benzenoid) hydrocarbons are obtained on passing methane, or gases containing it, at 500—800° and under 50—1000 atm. pressure, over catalysts. The latter may be carbonates or other salts of the alkaline-earth metals, including magnesium and beryllium; the hydroxides of the last two, or their oxides (especially beryllium oxide); compounds of selenium, tellurium, or thallium; or active silica or charcoal; alone or mixed.
B. FULLMAN.

Production of ester mixtures. W. CLAASEN (U.S.P. 1,656,883, 17.1.28. Appl., 7.3.27. Austr., 29.1.27).—The process of B.P. 250,910 (B., 1926, 769) is applied to cyclohexanol.
T. S. WHEELER.

Preparation of leuco-quinizarin. I. GUBELMANN and H. R. LEE, ASSNS. TO NEWPORT CO. (U.S.P. 1,655,462,

10.1.28. Appl., 25.1.26).—Chloroquinizarin in acid solution is treated with aluminium powder at room temperature, and the reaction mixture is poured into water.

T. S. WHEELER.

Production of 4-nitro-2-aminophenoxy-ethanol or -propandiol [4-nitro-2-aminophenyl β -hydroxyethyl or $\beta\gamma$ -dihydroxypropyl ether]. CHEM. WORKS FORMERLY SANDOZ [CHEM. FABR. VORM. SANDOZ] (B.P. 263,191, 18.12.26. Ger., 19.12.25).—2 : 4-Dinitrophenyl β -hydroxyethyl and β -dihydroxypropyl ethers are reduced, without hydrolysis of the ether group, to the nitro-amino-ethers by aqueous sodium sulphides, especially by the tetrasulphide, at 60°. 4-Nitro-2-aminophenyl β -hydroxyethyl ether, m.p. 114°, and the $\beta\gamma$ -dihydroxypropyl ether, m.p. 111—112°, are described.

C. HOLLINS.

Concentration of acetic acid. J. DUCLAUX, ASSN. TO FABR. DE SOIE ARTIFICIELLE DE TUBIZE SOC. ANON. (U.S.P. 1,660,418, 28.2.28. Appl., 31.3.24. Ger., 4.4.23).—See B.P. 213,914; B., 1925, 227.

Carbon disulphide (B.P. 282,049).—See VII.

IV.—DYESTUFFS.

Photographic properties of cyanine dyes. BLOCH and HAMER. **Dyes and fogging reactions.** LÜPP-CRAMER.—See XXI.

PATENTS.

Basic dye. J. KERN, ASSN. TO E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,660,167, 21.2.28. Appl., 26.12.22).—A dye suitable for textile printing is obtained by heating to boiling a solution of a basic dye and tannin.
A. R. POWELL.

Production of oil-soluble azo colouring matters. J. P. PENNY, ASSN. TO NAT. ANILINE & CHEMICAL CO., INC. (U.S.P. 1,656,322, 17.1.28. Appl., 2.3.26).—An excess of a diazotised arylamine is added to a phenol, and, after coupling is complete, the mixture is heated to decompose the excess of diazo compound. The mixture of azo dye and the decomposition products of the diazo compound is more soluble in hydrocarbons than the pure dye.
T. S. WHEELER.

Manufacture of monobenzoyldiaminoanthraquinone compounds. W. H. PERKIN and H. M. BUNBURY, ASSNS. TO BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,660,090, 28.2.28. Appl., 4.3.26. U.K., 8.7.25).—See B.P. 255,630; B., 1926, 817.

Manufacture of an azo dye [1-(*o*-chloro)phenyl-3-methyl-5-pyrazoloneazobenzene]. M. SCHMID, ASSN. TO SOC. CHEM. IND. IN BASLE (U.S.P. 1,660,097, 28.2.28. Appl., 19.7.26. Switz., 1.8.25).—See B.P. 256,205; B., 1927, 869.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Preservation of fishing nets by treatment with copper soaps and other substances. W. R. G. ATKINS (J. Marine Biol. Assoc., 1928, 15, 219—235).—For the preservation of cotton and hemp net copper oleate has no advantages over a mixed copper soap containing oleate. Details are given of tests of various preserving agents, and it is concluded that the best results are

obtained by the use of copper soap reinforced with anti-fouling paint or coal tar, petrol being better than paraffin as a diluent. After this treatment, nets, the life of which when untreated is from 2 to 3 months in aquarium water, are sound after 18 months.

W. O. KERMACK.

Action of formaldehyde on wool. S. R. TROTMAN, E. R. TROTMAN, and J. BROWN (J. Soc. Dyers and Col., 1928, 44, 49–52).—The reaction between wool and formaldehyde is incomplete at ordinary temperatures, proceeds more rapidly with rise of temperature, and is complete in 3 hrs. at 75°, when the amount of combined formaldehyde is 0.7% on the weight of dry wool. The combination with wool under these conditions is more stable than that with leather. Formaldehyde has little protective action for wool in a soap scouring bath, a chlorinating bath, or in cold dilute solutions of sodium carbonate or hydroxide. The moisture content of air-dried paraformaldehyde wool is considerably less than that of untreated wool, intermediate values being found for the formaldehyde combination. No change occurs in the nitrogen content as a result of the treatment of wool with either formaldehyde or paraformaldehyde, but the former gives the greater resisting power against the action of cold sodium hydroxide solution. Formaldehyde wool takes up more sodium hydroxide from solution than untreated wool, is resistant to boiling for 1 hr. with 0.5% sulphuric acid, and has a lower shrinkage than untreated wool when washed normally. It has a greatly reduced affinity for neutral dyeing acid dyestuffs (though in the presence of acid normal dyeing is obtained), is resistant to bacterial action, and may be diazotised. These properties are attributed to the displacement of the peptide equilibrium in wool to the enol form, the latter being more resistant to decomposition by acids and alkalis than the keto form. At raised temperatures the enol form tends to revert to the keto form, and formaldehyde wool then behaves similarly to untreated wool.

B. P. RIDGE.

Viscose. IX. G. KITA, R. TOMIHISA, K. AZAMI, and M. FUJIMOTO (J. Soc. Chem. Ind., Japan, 1927, 30, 198–204).—The action of solutions of sodium chloride, zinc sulphate, and copper sulphate, respectively, on viscose has been studied. The alkali is not removed by washing with alcohol the xanthate that has been purified with dilute acetic acid and sodium chloride solution. The purified xanthate is gradually decomposed by drying, and a part of the alkali thus set free seems to enter into combination with the carbon disulphide, producing a compound which is neutral to phenolphthalein. Sodium xanthate is much more stable than ammonium xanthate. When zinc sulphate was added to viscose, 75% of the sodium xanthate was found to be substituted by the zinc salt, a white precipitate of the latter being produced which is difficultly soluble in water but readily soluble in alkali. The amount of carbon disulphide combined as xanthate is decreased by drying, but not by treatment with zinc sulphate. When copper sulphate solution is mixed with purified xanthate, a yellowish-brown precipitate of copper xanthate is obtained, which is stable towards dilute sulphuric acid and hydrochloric acid at ordinary temperatures, but is decomposed at 70°. The amount of copper in copper xanthate was about

75% of the amount of carbon disulphide combined in the form of xanthate.

Y. TOMODA.

Ripening of viscose. J. FRENKEL (Cellulosechem., 1928, 9, 25–26).—The original hypothesis of Cross and Bevan that the cellulose molecule of viscose undergoes polymerisation during the ripening process could not be upheld, and it was replaced by the conception of a progressive hydrolysis of the xanthogenic ester in aqueous alkaline solution which corresponds with the analytical results. There remains the question whether the colloid remaining in dispersion in the fully ripened viscose, represented analytically by the so-called "C 24 xanthate," is a chemically homogeneous ester of lower degree of esterification or is a mixture of the original "C 12 xanthate" with free cellulose hydrate resulting from the hydrolysis of a portion of it. The author considers that the weight of evidence favours the latter conception. This is based on the possibility of washing out with water the soluble portion of a ripened viscose coagulated with ammonium chloride, and leaving a residue of pure cellulose hydrate. Further, it is possible to disperse by the Plauson colloid mill 3 pts. of freshly-made viscose with hydrated cellulose regenerated by the action of acid on 1 pt. of the same viscose, and to obtain with the addition of caustic soda a ripened viscose indistinguishable from one ripened in the usual way.

J. F. BRIGGS.

Cellulose xanthate and viscose. I. Reactions involved in the ripening of viscose. K. ATSUKI. II. Changes of the colloidal nature of viscose with the concentration of free alkali. K. ATSUKI, I. OKAMURA, and T. MATSUDA (J. Fac. Eng., Tokyo, 1927, 17, 135–144, 145–151).—I. During the course of the ripening of viscose, the viscosity and surface tension decrease, reach a minimum, and then increase, whilst the amount of 25% ammonium chloride solution necessary for coagulation decreases rapidly at first, and then very slowly, finally becoming nearly constant. The results indicate that the ripening takes place in two stages; dehydration and disintegration of the disperse phase (a concentrated solution of sodium cellulose xanthate) first occur, until the point of minimum viscosity and surface tension is reached, when the solution is suitable for spinning, after which coagulation of the excessively dehydrated particles commences. Determinations of the combined sodium and sulphur present at various stages of the ripening show that no appreciable hydrolysis occurs until the colloidal dispersion reaches its maximum, but it becomes more pronounced when coagulation sets in, the colloidal instability apparently giving rise to chemical instability.

II. From observations of the changes in specific volume of the disperse phase and the surface tension of viscose with increase of the concentration of free alkali present during ripening, it is deduced that when the alkali concentration is about 6–8%, the disperse phase (a concentrated alkaline solution of sodium cellulose xanthate) shows the highest degree of dispersion, and the colloidal system is most stable. With a lower concentration of free alkali there is an increased tendency to hydrolysis, and the disperse phase contains a larger proportion of water of hydration, whilst with a higher

concentration the particles are excessively dehydrated and tend to coagulate. W. J. POWELL.

Manufacture of cellulose; alkalinity changes, and the action of sodium sulphide [in the cooking process]. C. KULLGREN (Ing. Vet. Akad., Stockholm, 1927, No. 65, 34 pp.).—The ordinary titrimetric method of studying the changes of alkalinity of the liquor during the cooking process gives results which are too low for the early stages and too high for the later stages of cooking. When sodium hydroxide alone is present an electrical conductivity method may be used, but when carbonate and sulphide are also present, as in black liquor, a reaction velocity method is more suitable. In the latter the reaction velocity is measured by means of the catalytic decomposition of diacetone alcohol by alkali into acetone, a unimolecular reaction in which the velocity is proportional to the hydroxyl-ion concentration. Measurements are carried out in a dilatometer and the constants obtained are approximately proportional to the sodium hydroxide content of the liquor. In cooking with sodium hydroxide alone a cessation or pronounced decrease in the attack of the wood occurs within the temperature range 130–150°, whilst above 160° considerable decomposition again takes place. When sulphide is also present such stagnation is negligible and the alkali content of the liquor decreases uniformly. This process for the manufacture of sulphate cellulose depends on the fact that, although the sulphide itself is a bad decomposition agent, it forms sulphur compounds (lignins containing sulphur) which are more easily attacked by sodium hydroxide than is a wood substance containing no sulphur, the attack taking place rapidly at 140–160°. B. P. RIDGE.

Behaviour of cellulose towards acids. P. E. ALTMANN (Chem.-Ztg., 1928, 52, 150–151).—A short résumé of existing knowledge concerning the qualitative behaviour of cellulose with various mineral and organic acids. A product similar to the artificial parchment prepared by steeping paper in 60% sulphuric acid and washing may be prepared without the use of acid by coating unsized paper with a solution containing 125 g. of sodium salicylate and 200 g. of glycerin in 1.25 litres of water. W. J. POWELL.

Cellulose esters. I. Depolymerisation and mechanical properties of the nitrates. H. OKADA (J. Cellulose Inst., Tokyo, 1927, 3, 285–296).—Having worked out a method for preparing regular nitrocellulose films under standardised conditions, the author has studied the relations between viscosity and mechanical properties. Various types of cellulose in the form of cotton, cotton celluloid tissue paper, bleached sulphite wood pulp, and sulphite pulps specially purified with lime and sodium sulphite were subjected to preliminary treatments calculated to induce depolymerisation while avoiding definite hydrolysis and oxidation. Treatments comprised heating at 145° and 125° with water, dilute alkali, and glycerol; treatment with sodium hydroxide under mercerising conditions; regeneration from alkali cellulose after various ageing periods; regeneration from viscose and in the form of viscose silk; treating nitrocellulose with water under pressure. These modified celluloses were nitrated under identical conditions, the

viscosities of the nitrated products were determined in 1% acetone solution, and films were made 0.10–0.12 mm. thick from the ether-alcohol solutions. Over a wide range of relative viscosities from 126 down to 2.5 the tenacity of the films was almost constant at 15 kg./mm.², and the crumpling resistance was rather less constant at about 200. The influence of depolymerisation of the cellulose, whilst very marked on the viscosity, had a nearly negligible degrading effect on the mechanical properties. Only at a very advanced stage of depolymerisation, with reduction of the viscosity figure below 2, was a very sudden breakdown of the mechanical resistance of the nitrocellulose film recorded, obviously brittle products being thereby produced. J. F. BRIGGS.

Researches on wood chemistry. III. Composition of Tsuga and Momi. Y. UEDA and T. YOSHIDA (J. Cellulose Inst., Tokyo, 1927, 3, 297–298).—Air-dried samples of Japanese pulp woods *Tsuga* (*T. Sieboldii*) and *Momi* (*Abies firma*) on analysis gave: cellulose 48.71%, 45.24%; lignin 21.53%, 25.74%; furfuraldehyde (as pentosans) 4.69%, 6.26%, and resins (as benzene extract) 2.17%, 2.26%, respectively. These woods bear a close similarity in composition to the Kabafuto spruce, but showed an exceptional difficulty in the chlorination process of analysis due to the strong affinity between the cellulose and the lignin in these woods; this has an important significance in the pulping process. Owing to their compact structure these woods, especially *Tsuga*, are only suitable for pulping by chemical methods. J. F. BRIGGS.

Silks and dyestuffs. HOUCK.—See VI. **Nitrocellulose solvents.** BROWN, also BRIDGMAN.—**Nitrocellulose thinners.** DAVIDSON and REID.—See XIII. **Hemicellulose-splitting enzyme.** LÜERS and VOLKAMER.—See XVIII.

PATENTS.

Apparatus for the production of fibre from flax and hemp straw and the leaves of *Phormium tenax*, sisal, abacca, yucca, etc. J. C. W. STANLEY, Assr. to OCEANIA INDUSTRIES, LTD. (U.S.P. 1,657,046, 24.1.28. Appl., 18.4.27).—Apparatus is described for the continuous treatment of textile raw material for the production of spinnable fibres, and comprises conveyors, washing and shaking screens, and tipping tanks for alkaline digestion, bleaching, and oiling operations. After treatment with alkali the washed fibres are conducted through a water flume provided with paddles between and below which are arranged perforated pipes for injecting air under pressure. The material is finally dried in a current of heated ozonised air.

D. J. NORMAN.

Production of sterilised and stable vegetable bristle or fibre. G. AVERSENG (B.P. 284,421, 10.11.26).—Vegetable bristles or fibres obtained from the leaves of palm trees are freed from soluble mineral salts, pectic matter, and fermentable juices by extraction with cold or hot water, optionally under pressure and in the presence or absence of alkalis, and are simultaneously or subsequently treated with a solution of an organic or mineral antiseptic or anticryptogamic agent such as copper sulphate, zinc sulphate, salicylic acid, etc. A

suitable solution contains 2% of copper sulphate on the weight of fibre.

D. J. NORMAN.

Treatment of fibre. A. F. RICHTER and F. A. AUGSBURY, Assrs. to STEBBINS ENGINEERING AND MANUF. Co. (U.S.P. 1,653,416, 20.12.27. Appl., 19.7.26).—The hot high- and low-pressure relief gases from the digester are passed into an accumulator tank, where they are absorbed under pressure by fresh acid liquor. The hot liquor thus obtained is used for the treatment of further quantities of raw material.

D. J. NORMAN.

Treatment of Spanish moss. R. STINSON (U.S.P. 1,656,842, 17.1.28. Appl., 10.6.24).—The moss is submerged in a solution of ferrous or ferric chloride or sulphate until the tannic acid present has reacted with the metal, and is then dried and the husk removed.

T. S. WHEELER.

Extraction of fatty matter from foams formed by the beating, froth-floitation, or other treatment of wool-washing waters. E. MERTENS (B.P. 273,642, 27.10.26. Belg., 1.7.26).—The foam is maintained at 80–90° in a digester, and the soaps, albuminous matter, etc. which collect with the water at the bottom of the digester are withdrawn. Fresh water is added and the operation repeated as many times as may be desirable. The digester is then closed and the temperature increased until a pressure of 2–3 atm. is reached. For a charge of 1000 kg. of foam, containing 20% of neutral fat, 10 hrs. at pressure is sufficient. 80% of the fat initially present is recovered in a homogeneous, anhydrous form. No chemicals are used at any stage of the process.

D. J. NORMAN.

Production of brocaded textiles. H. FLORY (U.S.P. 1,655,414, 10.1.28. Appl., 9.10.25).—Fabric having a cotton back and a pile face of wool is printed with a solution containing sodium hydroxide, sodium sulphate, and gum arabic, and the disintegrated pile fibres are removed from the treated portions.

T. S. WHEELER.

Impregnation or coating of fabrics, paper, or the like web material. E. WEINHEIM (B.P. 255,094, 10.7.26. Ger., 13.7.26).—The material is subjected to a constant and uniform tension both during impregnation and drying, suitable apparatus for which is described. [Stat. ref. B.P. 122,680–1, 4494 of 1915, and 26,180 of 1910.]

D. J. NORMAN.

Preparation of painting grounds for artists, decorators, and craftsmen. C. F. CROSS, VISCOSE DEVELOPMENT Co., LTD., and WINSOR & NEWTON, LTD. (B.P. 284,363, 26.8.26).—Cellulosic textile material is filled and coated with cellulose partly as fibrous cellulose and partly as regenerated cellulose. Thus, unsized paper is attached to a fibrous base either during the manufacture of the paper or subsequently by the use of viscose solution as an adhesive, and this composite material is impregnated with viscose solution containing, e.g., 4% of cellulose. The impregnated sheet is kept until reversion of the viscose is complete, after which it is washed free from soluble salts. Alternatively, paper pulp may be incorporated with viscose solution and the mixture applied to a fabric base in quantity sufficient to fill the fabric and give a surface suitable for painting.

D. J. NORMAN.

Manufacture of cellulose esters of inorganic acids. I. G. FARBENIND. A.-G. (B.P. 279,796, 9.8.27. Ger., 26.10.26).—Cellulose esters of inorganic acids are made by treating alkali-cellulose, in the preparation of which more than 1 mol. of caustic alkali per mol. of $C_6H_{10}O_5$ has been used, with an excess of an inorganic acid chloride. The best yield and the highest degree of esterification are realised when the alkali-cellulose is made by impregnating cotton (100 pts.) for at least 4 hrs. with caustic soda solution of 40% strength at about –10° and pressing the mass to 200–300 pts. By suitably varying the conditions the degree of esterification may be controlled with the production of esters which differ as regards their solubility, conductivity, and mechanical properties. E.g., alkali-cellulose prepared as described above from 100 pts. of cotton and pressed to 300 pts. is introduced in small lots into an ice-cold solution of 160 pts. of phosphorus oxychloride in 900 pts. of benzene. The heat of reaction first raises the temperature of the benzene to the boiling point, after which the reaction proceeds smoothly with the production of a tough magma on the bottom of the vessel. After 12 hrs. the product is separated, washed with alcohol and ether, dried *in vacuo*, and dissolved in water. The colloidal solution thus obtained is purified by dialysis, and the cellulose phosphate precipitated by the addition of methyl alcohol. The yield is 80% on the weight of parent cellulose, and the ester contains 16% PO_4 . By pressing the alkali-cellulose to 400 pts. and using as the esterifying agent 220 pts. of sulphuryl chloride dissolved in 1800 pts. of benzene, cellulose sulphuric acid ester may be prepared as a whitish, non-hygroscopic mass soluble in water and containing 60% SO_4 .

D. J. NORMAN.

Manufacture of esters of the cellulose series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 281,927, 19.4.27).—Cellulose is pretreated with a small proportion (less than 2%) of sulphuric acid in the presence of a diluent, preferably an aliphatic acid, and is then partially esterified with an aliphatic esterifying agent. Esterification is subsequently completed by adding a further quantity of sulphuric acid. Thus, 100 pts. of sulphite-cellulose (94% of α -cellulose) are stirred for 6 hrs. at 20° with 400 pts. of acetic acid (99–100%) and 1 pt. of sulphuric acid. 350 pts. of acetic anhydride (90%) are then added, while cooling and stirring, and the mixture is kept at 20° for 2–4 hrs. until a uniform mass is obtained. Esterification is completed by adding 4–5 pts. of sulphuric acid (dissolved in glacial acetic acid) during 5–10 min. After 15–30 min. at 20–30°, or longer at a lower temperature, a clear viscous solution of cellulose acetate is obtained. This process avoids harmful degradation of the cellulose during esterification.

D. J. NORMAN.

Preparation of a modified cellulose for use in the preparation of cellulose acetate. Soc. LYONNAISE DE SOIE ARTIFICIELLE, and P. A. A. CHEVALET (B.P. 264,181, 10.1.27. Fr., 11.1.26).—The acetylation of cellulose is facilitated by pretreatment of the cellulose with 80–90% formic acid containing sulphuric acid (d 1.83) or anhydrous zinc chloride in the proportion of less than 20% by wt. of the mixture, at room temperature or lower for about 2 hrs. A suitable bath is obtained from sodium formate and sulphuric acid. The cellulose,

after washing etc., is acetylated, sulphuric acid (less than 1% by wt. of the cellulose) being used as catalyst, at room temperature or lower.

B. FULLMAN.

Treatment of cellulose fibres. COURTAULDS, LTD., H. J. HEGAN, and J. H. TAYLOR (B.P. 283,672, 1.11.26).—Cellulose fibres which have been immunised by the action of tannic acid and stannous chloride show a tendering effect on storage. This may be avoided by treating the immunised fibre with a weak alkaline reagent such as soap solution or dilute sodium carbonate solution.

D. J. NORMAN.

Treatment of artificial silk manufactured by the stretch-spinning process. HÖLKENSEIDE G.M.B.H. (B.P. 279,791, 29.7.27. Ger., 26.10.26).—Improved results are obtained by the process of G.P. 397,857, in which the freshly spun threads in hanks or skeins are placed on rotating rollers and subjected to vibrational stretching, if, prior to this treatment, the silk is dried. The silk may be remoistened before or during vibrational stretching. This preliminary drying sets the thread and renders it more resistant, so that a more intensive vibrational stretching treatment is permissible.

D. J. NORMAN.

Treatment of transparent material [celluloid]. C. E. SWETT, Assr. to U.S. ENVELOPE Co (U.S.P. 1,656,303, 17.1.28. Appl., 14.3.23).—Celluloid is coated in succession with alcoholic solutions of tartaric acid and potassium acetate to decrease the reflection of light from the surface, and thus improve the transparency.

T. S. WHEELER.

Cooking of sulphite pulp. G. H. TOMLINSON (U.S.P. 1,657,048, 24.1.28. Appl., 7.12.25).—Wood chips and heated sulphite liquor are introduced into the digester simultaneously and through the same opening, whereby the chips become soaked with liquor and are washed into position.

D. J. NORMAN.

Preparations for waterproofing wallpapers and the like for rendering the same washable. A. SANDERSON & SONS, LTD., and H. A. SIMS (B.P. 284,435, 10.12.26).—The paper is treated with a dilute solution of nitrocellulose. If a slightly glossy surface is desired a small quantity of gum, e.g., 5% of ester gum on the weight of nitrocellulose, may be added. A suitable solution for a matt finish contains 10 lb. of nitrocellulose, 5½ gals. of acetone, 4½ gals. of amyl acetate, 7½ gals. of industrial spirit, 11 gals. of petrol, and 1½ gals. of solvent naphtha.

D. J. NORMAN.

Cooking of vegetable fibre. S. D. WELLS (B.P. 273,663, 25.4.27. U.S., 29.6.26).—See U.S.P. 1,626,171; B., 1927, 438.

Increasing and improving the resistance of spun goods from artificial silk waste. J. A. GRAND (U.S.P. 1,660,123, 28.2.28. Appl., 2.3.27).—See B.P. 253,547; B., 1927, 184.

Apparatus for defibrating cotton seed, the decorticated hulls of cotton seed, or other fibre-bearing seeds, and for segregating and collecting the detached fibres. E. CARSTENSEN DE SEGUNDO (B.P. 285,305, 27.6.27).

Apparatus [bobbin] for the fluid treatment of artificial silk yarns. J. BRANDWOOD (B.P. 261,778, 20.11.26. U.S., 20.11.25).

Papermaking machines. P. OFFENHEIMER, S. BLOCK, and E. OFFENHEIMER (CELLULOSE-FABR. OKRIFTTEL A./M. P. OFFENHEIMER) (B.P. 285,311 and 266,670, [A, B] 15.11.26. Ger., [B], 27.2.26).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing. Silks and dyestuffs. R. C. HOUCK (J. Physical Chem., 1928, 32, 161—186).—The adsorption theory of dyeing applied to the dyeing of silk with acid and basic dyes, together with the adsorption of tannin by silk, has been studied. The adsorption of hydrochloric acid by degummed silk shows a continuous variation of the amount of acid in the fibre with the concentration of acid in the dye bath, and, furthermore, the amount of Orange II taken up by silk increases with increasing hydrogen-ion concentration of the acid bath. The addition of sodium sulphate in this case reduced the amount of dye adsorbed. With Crystal Violet E the most dye was taken up in neutral solution and then the amount decreased with increasing concentration of the sulphuric acid added. More dye was taken up in acetic acid solution than in a sulphuric acid solution of equivalent strength, and the addition of sodium sulphate in this case caused increased adsorption of the dye. All these results are in accord with the view that the dyeing of silk by acid and basic dyes is an adsorption phenomenon. The apparently contradictory results of dyeing practice are due to the presence of bast soap or boiled-off liquor in the bath, this being shown to decrease adsorption of the dyes mentioned above. The amount of tannin taken up by silk increases with increasing concentration of tannin in the bath, and adsorption is quickest in hot solution. The addition of sulphuric or acetic acid furthers adsorption from dilute, hot solutions. The tannin adsorbed at the ordinary temperature, but not that adsorbed at about 95°, is easily removed by washing. Experiments on the precipitation of tannins by the mineral acids are described and the work of Thomas and Foster (B., 1922, 302) is criticised.

L. S. THEOBALD.

Wetting-out agents and other auxiliary products used in dyeing and calico printing.

E. J. RATH (J. Soc. Dyers and Col., 1928, 44, 41—44).—Nekals and Leonils are superior to the Turkey-red oil types of wetting agent because, unlike the latter, they are neither absorbed by the fibre nor affected by the acids, alkalis, and electrolytes used in dyeing processes. Nekals have a high wetting-out power even for greasy materials, and a high emulsifying and solvent action on fat, cotton wax, resin, and natural oils, whilst, by their use, preliminary boiling can be shortened or dispensed with, especially in the case of goods to be dyed, in which a pure white is not essential. Nekal cannot be used with basic dyes since the latter are precipitated by it, but good results are obtained by its use with direct, sulphur, and indanthrene dyes, and indigo. It is also useful in the preparation of sizes and finishes, since the starch solutions are thereby made thinner without loss of adhesive power, wet the yarns evenly, with good penetration, and the size does not dust off. In de-sizing 1—2 g. of dry Nekal BX per litre in conjunction with Biolase reduces the time required by one half. Nekal AEM is of no importance as a wetting

agent, but is an excellent emulsifying agent, saponifiable and unsaponifiable fats, oils, waxes, and resins being emulsified by it without the use of soap. Ramasit I and WD are emulsions of paraffin wax, the former being used for sizing and finishing, whilst the latter, which is more concentrated, is used for waterproofing. Laventine BL is a water-soluble fat solvent, and may be used with any soap for cleaning greasy and dirty materials. The Leonils (similar in chemical constitution to the Nekals) are used in the scouring and dyeing of wool. The Prestabit oils possess excellent fastness to acid, lime, and magnesium sulphate, but (with the exception of the KN preparation) lose their wetting-out property in alkaline solution. Prestabit oil BM is of advantage in grey mercerisation since it imparts excellent wetting-out properties to the sodium hydroxide solutions.

B. P. RIDGE.

Humidity and the fading of dyestuffs on wool.

J. J. HEDGES (J. Soc. Dyers and Col., 1928, 44, 52—54).—Patterns of dyed fabric were faded by means of a mercury arc at known humidities between 25% and 100% and compared with corresponding unexposed samples in a Lovibond tintometer. Percentage fading of the predominant colour was then compared with both % R.H. and % regain (the latter being obtained from a humidity-regain curve at the temperature of the experiments). A much simpler relation exists between fading and regain than between fading and humidity, straight-line graphs being obtained in the case of all the colours examined, and is expressed by $F = aR + b$ where F is the % loss of colour, R the % regain, and a and b are constants; the mechanism of the action of the water is not understood.

B. P. RIDGE.

Dyeing with "mangue" extract. SCHIRM.—See XV.

PATENTS.

Dyeing of fabrics. R. R. ROSS (U.S.P. 1,655,973, 10.1.28. Appl., 15.6.27).—Material to be dyed in a variegated pattern is immersed in water, and the excess of liquid is wrung out. The material while still twisted is dipped at different points in a number of dyes and is finally immersed completely in a dye which blends all the colours.

T. S. WHEELER.

Stripping of dyestuffs or colours from dyed or coloured [cellulose acetate silk] materials.

BRIT. CELANESE, LTD. (B.P. 260,289, 22.10.26. U.S., 24.10.25).—Cellulose materials, particularly those consisting of cellulose ester or ether, and which are dyed by means of S.R.A. or other water-insoluble dyes, are stripped by extraction with a liquid containing a substance which exerts a swelling action on the cellulosic material and also a substance which is capable of dissolving the dyes which are to be stripped. The temperature and duration of the extraction are dependent on the swelling action of the substance used. Satisfactory swelling agents and solvents include aromatic hydrocarbons, *e.g.*, xylene, benzene, and ethylbenzene, acetone, chloro-derivatives of saturated and unsaturated hydrocarbons such as tetrachloroethane, dichloroethylene, etc., and alcohols such as ethyl and methyl alcohols; aqueous solutions of ammonium thiocyanate also serve as swelling agents. When alcohols are used as swelling agents it is pre-

ferable to restrain their action by the addition of benzene.

A. J. HALL.

Colour printing on [cellulose acetate silk] fabrics having a foundation of silk or wool. ÉTABL. PETIT-DIDIER (ANC. MAISON JOLLY-BELIN) (B.P. 270,657, 25.3.27. Fr., 7.5.26. Addn. to B.P. 256,238; B., 1928, 50).—After colouring the cellulose acetate silk by printing, the whites are cleared by passing the fabric through a bath containing 20—25 g. of potassium permanganate per litre, and then through a solution of sodium bisulphite (d 1.116); still more effective clearing is thus attained than that described in B.P. 258,562 (B., 1928, 50).

A. J. HALL.

Weighting of natural silk. R. CLAVEL (B.P. 283,019, 24.3.27. Addn. to B.P. 266,640; B., 1927, 295).—By pretreating the silk with an aqueous solution of an acid or a salt, optionally in conjunction with a protective colloid, weighting baths containing considerably smaller quantities of added acid, or even none at all, may be satisfactorily used. *E.g.*, the material may be first impregnated with a 1% solution of phosphoric acid containing 12 g. of glue per litre and is then treated as in the prior patent. The pretreatment with acid imparts to the silk an increased and more uniform receptivity for the weighting agents.

D. J. NORMAN.

Treatment of fabrics. HEBERLEIN & Co. A.-G. (B.P. 276,352, 19.8.27. Ger., 19.8.26).—In the production of wool-like effects on vegetable fabrics by the action of mercerising agents under conditions which permit shrinkage, improved results are obtained if the fabric is composed of raw fibres, *i.e.*, fibres which have not been freed from their natural incrustants. Mild purification by, *e.g.*, dilute acid or diastase is permissible, but treatment with boiling alkaline solutions must be avoided. The fabric may be bleached before or after mercerisation if the bleaching is conducted at a temperature not substantially exceeding 40° and under carefully controlled conditions.

D. J. NORMAN.

Ornamentation of fabrics made of or containing cellulose esters or ethers.

R. CLAVEL (B.P. 269,128, 11.10.26. U.S., 6.4.26).—Metallic and other ornamental effects are obtained on fabrics composed of or containing cellulose esters or ethers by coating or printing the fabric with a paste containing a thickener, a finely-divided metal, pigment, or like powder, and a solvent or swelling agent for the cellulose ester or ether, and fixing the powder on the fabric by, *e.g.*, rapidly drying the coated material. After drying, the fabric may be dyed, preferably at 80—100°, washed, and stretched, the object of the stretching being to cause fracture of some of the partially or completely dissolved cellulose ester or ether threads to give a metallic or other effect on a white or dyed background. The fabric may, if desired, be finally calendered.

D. J. NORMAN.

[Apparatus for] dyeing, cleaning, and mordanting of tissues, felts, etc., and similar operations. SOC. DES CONDENSEURS DELAS (B.P. 280,489, 24.2.27. Fr., 10.11.26).

Bleaching machines. JACKSON & BROTHER, LTD., and C. TAYLOR (B.P. 285,689, 23.4.27).

Improvement of fibres (B.P. 264,529).—See V.
Colouring of rubber (U.S.P. 1,660,213).—See XIV.

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

Hydrochloric acid absorption, its mechanism and the apparatus necessary. B. WAESER (Chem. Fabr., 1928, 101—102).—Data from literature are given, including relations between gas and solution concentrations, calculations of cooler dimensions, back pressures of absorption towers, and various data for different tower packings. For the preparation of very pure synthetic acid, fused silica only must be used, and stoneware, coke, packing rings, etc. excluded. For joints a mixture of pitch with barium sulphate or clay and asbestos is recommended. C. IRWIN.

Catalytic oxidation of ammonia. VIII. L. ANDRUSSOV (Z. angew. Chem., 1928, 41, 262—263).—Views on the mechanism of the reaction between ammonia and oxygen in presence of platinum as previously published are summarised. Nitroxyl is regarded as the intermediate product. The large production of nitrogen at high temperatures and low velocities is not explicable as due to thermal decomposition of ammonia or reaction between ammonia and nitric oxide. It is produced in an explosion zone in front of the catalyst, which zone disappears with increasing gas velocity. The conditions of reaction with excess of oxygen suggest the presence of a layer of oxygen at the catalyst surface, whereby nitroxyl and nitric oxide are formed in preference to di-imide. In absence of a catalyst nitrogen is the chief product of oxidation of ammonia. C. IRWIN.

Detection of traces of soluble bromides. H. BAINES (J.S.C.I., 1928, 47, 11—13 T).—To about 1 c.c. of the solution to be tested, 5—6 drops of fluorescein solution are added, followed by chlorine water (roughly 0.001*N*-solution of chlorine or sodium hypochlorite in water) added drop by drop with shaking. In the presence of bromide a pink coloration is formed after addition of a few drops of the chlorine solution; excess of chlorine bleaches the solution. By using a blank test the limit of accuracy is about 2×10^{-6} *N* for potassium bromide. An alternative single-solution method may be used. The method may be made roughly quantitative by colorimetric comparison. In the absence of bromides the test may be applied for the detection of iodides. Large quantities of reducing substances are inhibitive. D. G. HEWER.

Condensation of liquids as mists. PAILLY.—See I. **Cyanogen and its compounds in the blast furnace.** HAUFE and VON SCHWARZE. **Steels for synthesis of ammonia.** VANICK and others.—See X. **Assay of basic lead carbonate.** GLAZE.—See XIII.

PATENTS.

Preparation of neutral to weakly acid water-soluble silicic acid compounds. J. A. VON WULFING and A. BUSCH (B.P. 284,450, 11.1.27).—Neutral to weakly acid products, readily soluble in warm water, and obtainable in dry form, are produced by the combination of alkali polysilicates or water-glass solution with casein or albumin substances of acid character (*e.g.*, nucleoalbumins or albuminates) and sufficient alkali hydroxide to form metasilicate. *E.g.*, 100 pts. of casein suspended

in 100 pts. of water are mixed with a concentrated solution of about 4 pts. of sodium metasilicate prepared from water-glass or commercial metasilicate by the addition of sodium hydroxide. On evaporation *in vacuo*, casein sodium metasilicate is obtained. B. FULLMAN.

Activation of silicic acid gel. I. G. FARBENIND. A.-G. (B.P. 263,483, 26.10.26. Ger., 22.12.25).—Silicic acid is first dried until the gel structure is consolidated and then treated in a revolving furnace at 300—600° to obtain a solid product. H. ROYAL-DAWSON.

Manufacture of [colourless] sodium sulphide. B. LAPORTE, LTD. From M. SCHLAUGK, G.M.B.H. (B.P. 284,958, 21.9.27).—Sodium cyanide is added to a hot solution of crude sodium sulphide (*d* 1.22) and the whole is heated to 85—90°, allowed to settle, filtered if necessary, and allowed to crystallise. H. ROYAL-DAWSON.

Treatment of natural alkali salts of secondary and tertiary origin. A. LAMBERT, Assr. to COSMIC ARTS, INC. (U.S.P. 1,660,561, 28.2.28. Appl., 12.6.24).—The solution of sodium chloride, carbonate, and sulphate obtained by leaching natural salt deposits is cooled to 33° and the deposited salt separated from the chloride mother-liquor. A. R. POWELL.

Production of colloidal copper sulphide [for use as an insecticide]. A. R. GRAVER, Assr. to GRASSELLI CHEMICAL Co. (U.S.P. 1,657,430, 24.1.28. Appl., 25.10.26).—Copper sulphide is precipitated in any usual manner in presence of a protective colloid, *e.g.*, the residue from the evaporation of waste lye from the manufacture of sulphite-cellulose. T. S. WHEELER.

Modification of glauconite. A. C. SPENCER, Assr. to PERMUTIT Co. (U.S.P. 1,657,700, 31.1.28. Appl., 26.2.23. Renewed 11.2.26).—Glauconite is baked and treated with an alkali. H. ROYAL-DAWSON.

Apparatus for treating aluminous materials. E. MOLDENKE, Assr. to MOLDENKE PROCESS CORP. (U.S.P. 1,658,457, 7.2.28. Appl., 27.4.23).—A paste obtained by mixing the finely-divided material with sulphuric acid is deposited in a layer on a conveyor belt. Means are provided for caking the paste by heat during its travel on the belt, and for flexing the belt to free the cake therefrom. The discharged cake passes through grinding and heating devices (cf. U.S.P. 1,457,787; B., 1923, 718 A). H. HOLMES.

Reduction of solutions containing titanium, iron, and eventually other compounds. TITAN Co. A/S. (B.P. 279,786, 2.7.27. Norw., 28.10.26).—In the reduction of such solutions by means of iron or its alloys, the efficiency of the process depends on adjusting the concentration of the solution so that it contains 90—130 g. of titanium dioxide per litre, and keeping the temperature below 60°. H. ROYAL-DAWSON.

[Production of] coloured sulphur. W. H. KOBÉ, Assr. to TEXAS GULF SULPHUR Co. (U.S.P. 1,655,504, 10.1.28. Appl., 10.10.25).—Molten sulphur is mixed with a sulphur-soluble azo-dye, *e.g.*, malachite-green, or with a substance, *e.g.*, *p*-nitrophenol, which reacts with sulphur to give a coloured product soluble in the molten liquid. T. S. WHEELER.

Treatment of sulphur. J. W. SCHWAB, Assr. to TEXAS GULF SULPHUR Co. (U.S.P. 1,656,504—5, 17.1.28. Appl.,

10.12.26).—The process of U.S.P. 1,646,838 (B., 1927, 907) is modified in that (A) silica gel, or (B) diatomaceous earth is used.

T. S. WHEELER.

Burning or subliming of sulphur. H. F. MERRIAM, Assr. to GEN. CHEMICAL CO. (U.S.P. 1,657,545, 31.1.28. Appl., 6.5.26).—A portion of the molten sulphur is periodically drained off when the impurities present have concentrated to a predetermined value in the burner or sublimer.

H. ROYAL-DAWSON.

Production of carbon disulphide. I. G. FARBENIND. A.-G. (B.P. 282,049, 3.12.27. Ger., 13.12.26. Addn. to B.P. 237,716; B., 1925, 759).—In a modification of the prior patent, sulphur is evaporated and superheated in an apparatus in the heating furnace containing the reaction chamber; or the apparatus may be inside the latter, especially if internal electric heating is used.

B. FULLMAN.

Manufacture of substantially pure hydrogen. C. E. PARSONS, Assr. to METAL RES. CORP. (U.S.P. 1,658,939, 14.2.28. Appl., 2.9.26).—Pyrophoric iron, produced by the action of producer gas on ferrous oxide below 600°, is treated with steam (below 700° to prevent the formation of magnetic iron oxide); the hydrogen is recovered and the ferrous oxide produced is used again in the process.

W. G. CAREY.

Drying of gases containing nitrogen oxides. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 284,839, 1.1.27).—Humid gases resulting from the catalytic combustion of ammonia in air are treated, at between 30° and 100°, with or without pressure, with sulphuric acid of such initial concentration that it becomes diluted (60–65% strength) and capable of absorbing only small amounts of the oxides.

H. ROYAL-DAWSON.

Purification of gases. GEN. ELECTRIC CO., LTD., and C. J. SMITHELLS (B.P. 284,808, 25.11.26).—Gases such as hydrogen or argon are freed from oxidising impurities, particularly oxygen and water, by passing them over metallic chromium at 700–850°, preferably after partial purification by passing them over red-hot copper and phosphorus pentoxide.

W. G. CAREY.

Extraction of carbon dioxide from gaseous mixtures. I. G. FARBENIND. A.-G. (B.P. 284,574, 24.10.27. Addn. to B.P. 271,852; B., 1928, 91).—Gypsum liquors are used, after filtration, in the scrubbing towers, instead of the neutral or acid solutions of the prior process, such liquors being obtained in the manufacture of ammonium sulphate from gypsum, ammonia, and carbon dioxide.

H. ROYAL-DAWSON.

Precipitation of zinc carbonate. N. A. LAURY (B.P. 285,260, 7.3.27).—See U.S.P. 1,640,708; B., 1927, 877.

VIII.—GLASS; CERAMICS.

Relationship between chemical composition and thermal expansion of glasses. S. ENGLISH and W. E. S. TURNER (J. Amer. Ceram. Soc., 1927, 10, 551–560).—Thermal expansion data, obtained by the authors' method (cf. B., 1920, 407 A), on various glasses are reported. For soda-silica and soda-alumina-silica glasses an approximately linear relationship exists between the thermal expansion and chemical composition.

Boric oxide glasses do not follow this linear relationship. Soda and potash exert dominating influences on the thermal expansion of glasses. Thus, the calculation of the constitution of a glass from its batch formula is an unwise procedure when dealing with expansion data. The influence of zinc oxide on expansion is masked by that of the soda in soda-zinc oxide-silica glasses. Considering glasses of the approximate composition $6\text{SiO}_2 \cdot (2-x)\text{Na}_2\text{O} \cdot x\text{RO} \cdot \text{R}_2\text{O}_3$ or RO_2 , the curves connecting thermal expansion with chemical composition for lead oxide and barium oxide glasses are identical and linear, whilst lime, magnesia, and zirconia glasses all show the straight-line relationship. The curve for alumina glasses deviates slightly from the linear. New so-called expansion factors, which give values for the coefficients of expansion in close agreement with those observed, are computed.

A. T. GREEN.

Penetrability of various rays through glass.
VI. **Penetrability of ultra-violet rays through window-glass; influence of iron and manganese.** S. SUGIE (Rep. Imp. Ind. Res. Inst., Osaka, 1927, 8, [8], 1–22).—With soda-lime (but not potash-lime) glass, the effect of ferric iron in lowering the penetrability is greater than that of ferrous iron. The presence of a little chromium with the iron is without effect. The effect of manganese is slight up to, but great above, 1%. The penetrability is not parallel to the depth or mode of coloration by manganese compounds.

CHEMICAL ABSTRACTS.

Solubility of glass constituents. W. MEYER (Chem.-Ztg., 1928, 52, 151).—An ether-alcohol mixture after being kept in a glass vessel for 2 years was found to contain 0.001% of selenium as selenious acid; ethyl acetate, pure ether, methyl alcohol, amyl alcohol, and amyl acetate were also found to have the power of dissolving selenious acid from glass. The selenium is derived from the selenium dioxide or sodium selenate used for decolorising glasses containing iron. Jena glass gave no selenium after a 12 months' test.

W. J. POWELL.

Vitreous enamel slips and their control. W. N. HARRISON (J. Amer. Ceram. Soc., 1927, 10, 970–994).—The problem of reproducing enamel slips of definite consistency under working conditions was studied. Laboratory tests were made on three ground-coatenamels, viz., a high-silica, a high-felspar, and a high-borax enamel. A modification of the Bingham apparatus was used to determine the consistency of the slips, and the effect of various factors on the consistency was studied; these factors were: ageing, varying the concentration of certain solutes in the liquid phase of the slip, adding borax at different stages of grinding, and grinding the frits to different degrees of fineness. In a study of the physico-chemical nature of the slip and its influence on consistency, it was found that both the frit and the clay particles were negatively charged, and that a change in consistency was not necessarily accompanied by a change in p_{H} value. The work was extended to include factory tests, simplified apparatus, consisting of a feed tube 1.3 cm. in diam. and 90 cm. in length and a capillary 0.16 cm. in diam. and 12.0 cm. in length, being evolved for this purpose. The fundamental factors which govern

the consistency of a slip are: kind and quantity of colloidal matter present; the degree of flocculation of this matter; the percentage of water in the slip; and the size of the particles which have to be suspended by the colloidal structure. The practical measures indicated by a consideration of these factors are discussed.

F. SALT.

Ball and china clays. C. W. PARMELEE and T. N. MCVAY (J. Amer. Ceram. Soc., 1927, 10, 598—628).—Thirteen ball clays and seven china clays used in porcelain bodies were examined. Owing to impurities, determinations of the adsorption of malachite-green by ball clays indicate no characteristics of value. With china clays, in general, the greater the adsorption of the dye, the greater is the plasticity of the raw clay and the lower its porosity after a specific heat treatment in the vitrification range. Whilst the experimental electrolytes have no effect on china clay suspensions, ball clays can be divided into four classes according to their behaviour in suspension towards these substances. American ball clays have a greater amount of water of plasticity than English clays. The lowest value for the modulus of rupture of washed unfired clays is given as 284 lb./in.² and the highest as 819 lb./in.² English clays show the higher value for this property. Whilst showing less shrinkage up to cone 12 than American ball clays, the English varieties give indications of overfiring at this heat-treatment. The true sp. gr. tends to increase with increasing mullite content of the fired clay.

A. T. GREEN.

Nebraska clay. A. J. RYMES (J. Amer. Ceram. Soc., 1928, 11, 46—60).—The effect of calcium carbonate on the physical properties of bricks made from ordinary red brick clay was studied. Various amounts of pure calcium carbonate which passed 150-mesh were added to an apparently poor grade of clay, briquettes were made from the mixture, and both fired and unfired specimens were tested. The addition of lime decreased the drying shrinkage and firing contraction at all temperatures. It lowered the crushing strength of mixtures fired at high temperatures, and increased the porosity up to the vitrification point.

F. SALT.

Progress report on investigation of saggar clays.

IV. Elasticity, transverse strength, and plastic flow at 1000°. R. A. HEINDL (J. Amer. Ceram. Soc., 1927, 10, 995—1004).—The data given in Report III (B., 1928, 52) are supplemented by similar data observed at 1000°, together with an investigation of the elastic and plastic properties of the clays and the apparent relation between the rate of plastic deformation and refractoriness (cone equivalent). The range of refractoriness extended from cone 14 to cone 33, but all the clays showed plastic deformation at 1000° with a load producing an outer fibre stress of 30 lb./in.² (approx.). No relation was found between the average plastic deformations and the loads applied. The majority of the clays had a much lower modulus of elasticity at 1000° than at room temperature, though their transverse breaking strengths were greater. The cone equivalents of the clays gave a much better indication of the elastic recovery and plastic flow properties at 1000° than did the chemical analyses. Clays with a cone equivalent

higher than 30 showed little variation in plastic flow; those having an equivalent of 30 and below had greater plastic flow as a group, and the plastic flow was approximately inversely proportional to the cone equivalent. The maximum elongation of the extreme fibre decreased with increase of temperature up to 750°, but at 1000° the deformation at failure was much greater than at 750° and lower temperatures.

F. SALT.

Thermal characteristics of clays. A. E. MACGEE (J. Amer. Ceram. Soc., 1927, 10, 561—568).—The thermal reactions taking place during the firing of clay wares are discussed on the basis of original experimental data, whilst previous work on the subject is reviewed. It is shown that practically all clays undergo an endothermic reaction associated with the evolution of hygroscopic water and the breakdown of the clay molecule between 100° and 650°, which reaches a maximum at 550° and results in the absorption of from 40 to 130 g.-cal./g. of air-dried material. Most clays undergo an exothermic reaction of undefined significance in the neighbourhood of 950° with the evolution of 5—30 g.-cal./g. of air-dried material. Thus, the specific heats of most clays lie between 0.4 and 0.5 over the interval 20° to 1100°. Using such data, the reputed thermal efficiency of kilns is considerably increased.

A. T. GREEN.

Hydrogen-ion concentration and electrical conductivity of clay slips. II. Laboratory study.

A. H. FESSLER and H. M. KRANER (J. Amer. Ceram. Soc., 1927, 10, 592—597; cf. B., 1928, 53).—Attempts are made to define the causes of the variations in the hydrogen-ion concentration, the electrical conductivity, and viscosity of certain clay slips. The influences of temperature, vigour and time of stirring, and the presence of salts on these properties are examined. It is shown that the hydrogen-ion concentration decreases whereas the conductivity increases with rising temperature. With casting slips, vigorous stirring affects the viscosity and conductivity values, but does not influence such determinations on plastic slips. The viscosity and conductivity values are increased by the addition of common salt to a slip, whilst the hydrogen-ion concentration is not affected by this treatment. The influences of sodium silicate, aluminium chloride, and magnesium sulphate are also detailed. It is concluded that viscosity determinations give the best available data for control purposes.

A. T. GREEN.

Effect of potash and soda feldspars in china body.

E. SCHRAMM (J. Amer. Ceram. Soc., 1927, 10, 1005—1006; cf. Geller, B., 1928, 53).—The behaviour of different feldspars in vitreous bodies is discussed. A china body was made up with pure potash feldspar (A), pure soda feldspar (C), and a mixture of the two (B). Plates and briquettes were biscuit-fired to cone 11 and glost-fired to cone 6 (half down). Draw trials were taken with the briquettes at intervals of 20° and the total linear shrinkage, absorption, and bulk sp. gr. were determined thereon. The absorption figures show that an increase in the total percentage of alkali (*i.e.*, increase in the potash:soda ratio) produces earlier vitrification; this is most marked in bodies with the high soda content. Bodies containing 13% and 7% of potash (A and B)

are similar in translucency and firing range, since both overfire together.

F. SALT.

Physical properties of chemical stoneware bodies.

A. E. MACGEE (J. Amer. Ceram. Soc., 1927, 10, 569—579).—Nine commercial chemical stoneware bodies were investigated for crushing, transverse, and impact strength, thermal expansion, resistance to heat shock, and elasticity. In making up the test pieces each body was tempered with 20% of water and fired to cone 8 in commercial kilns on a 9-day firing basis. Thermal data concerning the firing of these bodies is given. The results show that the ordinary semi-vitreous chemical stoneware bodies expand about 0.3% between room temperature and 500°. Great variations in the elasticity and crushing strength values were obtained, the elasticity of a highly vitrified material being distinctly less than that of a porous body. The crushing strength varied from 10,000 to 260,000 lb./in.² It is desirable that stoneware bodies have a high tensile strength and temperature diffusivity together with a low thermal expansion and modulus of elasticity. Such a combination gives increased resistance to heat shock. The mechanism of the impact test together with the interpretation of the results are discussed.

A. T. GREEN.

Use of silicon carbide refractories in boiler furnaces. B. M. JOHNSON and J. A. KING (J. Amer. Ceram. Soc., 1927, 10, 1007—1015).—The chief causes of failure of boiler furnace refractories are slag adhesion, erosion, failure of the structure, and spalling, the importance of each of these factors varying with the type of coal used. Silicon carbide refractories tend to prevent adhesion of clinker. For practical purposes the bonded brick is superior to the recrystallised type. The bricks are installed along the fire line. Silicon carbide bricks are rapidly eroded by coal ash having a high content of iron and consequent low m.p., and also by ash containing medium amounts of iron (8—10%), if it is converted into the molten state by the action of hot flames under pressure. These adverse conditions are met by air-cooling and water-cooling. Air-cooling is carried out either by circulating air behind the walls and then forcing it through the wall into the furnace (Bernitz bricks are used for this purpose), or by circulating it behind the wall and then using it as preheated air for combustion. Silicon carbide is well adapted to air-cooled structures, owing to its high thermal conductivity, great strength under load at high temperatures, and high refractoriness. The use of "water walls," for which silicon carbide has been found satisfactory, is a recent development of boiler furnace design.

F. SALT.

Petrographic study of some slags from boiler furnaces. S. J. McDOWELL and H. C. LEE (J. Amer. Ceram. Soc., 1928, 11, 35—41).—Specimens of slag from two furnaces burning different kinds of coal were examined. The coal ash contained 19.3 and 29.2% Fe₂O₃ and 8.0 and 2.8% CaO. In the cool slag, plagioclase feldspar and iron oxide are the dominant mineral phases. The interface between slag and refractory consists largely of mullite, with iron in solid solution, surrounded by glass. The slag promotes the growth of small mullite crystals in the refractory, and large needles

are formed in the zone of contact between slag and fire-clay refractory. With high-alumina refractories, a thicker, deeper-stained interface is formed as the alumina content increases. Mullite predominates on the outer surface, but becomes mixed with corundum grains as the refractory is approached. Large crystals both of corundum and mullite appear in the stained area. This zone of crystallisation may be conducive to spalling.

F. SALT.

Silica cements. COLE.—See IX.

PATENTS.

Electrical insulators of glass. "OSA" PARTICIPATIONS INDUSTRIELLES SOC. ANON. (F.P. 618,186 and 618,288, [A] 28.6.26, [B] 1.7.26. Ger., [A, B] 1.8.25).—In addition to silica, the glasses contain potassium oxide and sodium oxide and one or more bi- or ter-valent oxides, and, if necessary, boric oxide. Minimum electrical conductivity is obtained when the potash : soda ratio is within the limits 7 : 1 and 3 : 1. The soda may be replaced by iron oxide.

F. SALT.

Lead glasses. "OSA" PARTICIPATIONS INDUSTRIELLES SOC. ANON. (F.P. 618,659, 7.7.26. Ger., 1.8.25).—Calcium oxide (2.5—3%) is added to ordinary glass batches containing silica, soda, potash, and lead, to increase their electrical insulating properties.

F. SALT.

Manufacture of coherent, granular, non-metallic substances (e.g., glass, enamel). A. UHLMANN (B.P. 255,497, 19.7.26. Ger., 18.7.25).—Finely-divided substances are subjected to the action of an electric or magnetic field whereby they are caused to cohere without direct passage of electric current through them.

J. S. G. THOMAS.

Glass vessels. F. MEYER (B.P. 284,888, 26.3.27).—Glass containers for medicines etc. have imparted to them a tension along a definite line causing them to divide along that line after slight scratching with emery. The tension is produced either by bringing the hot glass into contact with a colder body from within (the other end being used for filling), or by outside chilling after filling.

W. G. CAREY.

Continuous kilns. M. BISCH (B.P. 275,616, 29.7.27. Fr., 6.8.26).—In a continuous kiln for pottery etc. the roof slabs are mounted on rollers and rails, so that they can be slid lengthways to expose a chamber for recharging, and a travelling carriage is provided to transfer a roof section from one long set of chambers to another.

B. M. VENABLES.

Ceramic kilns. A. J. COULHON (F.P. 617,468, 12.6.26. Belg., 28.8.25).—An auxiliary kiln is provided outside an ordinary ceramic kiln, and from it hot gases are passed through conduits controlled by dampers into the kiln chambers. Where several kilns are in operation, the waste heat from one may be used to preheat another. The arrangement enables the ware to be dried within the chambers, and it facilitates cooling.

F. SALT.

Combined drying and burning kiln [for ceramic ware]. H. R. STRAIGHT (U.S.P. 1,657,138, 24.1.28. Appl., 14.4.26).—A conveyer passes through a series of drying kilns and a burning kiln in line therewith. A partial load of the plastic ware is delivered to the conveyer

at the outer end of one drying kiln, and the remainder at a point between two of the drying kilns.

H. HOLMES.

Linings for rotary kilns. G. POLYSTIUS (F.P. 618,089, 24.6.26).—Highly refractory materials, such as corundum, zirconium, etc., are mixed with *ciment fondu* and water; blocks etc. formed from the mixture are allowed to set.

F. SALT.

[Preparation of clay for] pottery. C. D. HYTEN (U.S.P. 1,657,997, 31.1.28. Appl., 24.7.24).—Clay is reduced to the consistency of cream, and then combined and ground with a colouring consisting of a ground colour base and a shrinkage-control mixture; after the excess of moisture has been extracted, the plastic mass is ready for use.

H. ROYAL-DAWSON.

Preparation of refractory bodies. VER. F. CHEM. & MET. PROD. (F.P. 618,339, 7.6.26. Ger., 10.6.25).—Magnesia, calcined dolomite, etc., in paste form, are mixed with concentrated colloidal solutions of titanium oxide or titanium salts; products are formed from the mixture, with or without the application of pressure, dried, and fired. The products are denser than ordinary magnesia bricks.

F. SALT.

Apparatus for annealing glassware. BRIT. HARTFORD-FAIRMONT SYND., LTD., Assees. of A. W. RUSSELL (B.P. 279,835, 20.10.27. U.S., 26.10.26).

Production of sheet glass. N. V. MIJ. TOT BEHEER EN EXPLOIT. VAN OCTROOIEEN, Assees. of L. VON REIS (B.P. 280,533, 3.11.27. U.S., 10.11.26).

Cooling of glass plates and sheets. Y. BRANCART (B.P. 282,638, 12.11.27. Belg., 21.12.26).

Annealing furnaces (B.P. 283,725 and 283,767). **Flasks** (U.S.P. 1,659,383).—See I.

IX.—BUILDING MATERIALS.

Progress report on the efflorescence and scumming of mortar materials. H. WILSON (J. Amer. Ceram. Soc., 1928, 11, 1—31).—The subject of soluble salts in clays, clay products, and mortar materials, and of the addition of barium salts as a preventive against scumming, is discussed. A study was made of 21 panels built up of red face bricks all made from the same clay, but with different mortar materials, viz., sand, slaked lime, hydrated lime, and Portland cement. Saturated and dry bricks, sea water and fresh water, and machine-mixed and hand-mixed mortars were all tried, and the effect of adding various percentages of barium carbonate, barium hydroxide, and barium chloride was studied. Both temporary and permanent scums, produced by artificial and natural soaking and drying of the walls over a period of more than a year, were noted. All the mortars could be made to scum if treated with sufficient water for a sufficient length of time. Calcium sulphate from Portland cement produces a bad, early scum; other scum-forming salts are calcium hydroxide from the free lime and hydrolysed calcium aluminates and silicates in Portland cement and from lumps of lime in the lime mortars, and, to a minor degree, sodium chloride from sea water. Calcium sulphate is sufficiently soluble to pass into solution through the pores of the brick and spread uniformly over the surface. Both slaked and

hydrated lime should be free of hard lumps, which cause streaks of calcium hydroxide and carbonate. Heavy rain of short duration is not so injurious as soaking drizzle. Under normal conditions, barium carbonate will prevent calcium sulphate scum from Portland cement; 5% was used successfully in public buildings. It is also suggested that diatomaceous earth might combine with the free lime of the mortar materials to produce insoluble calcium silicate and reduce the temporary scum of calcium hydroxide. Preliminary strength tests on mortar materials showed that barium salts had no deleterious effects.

F. SALT.

Effect of lead oxide on hardening of Portland cement. B. GARRE (Z. anorg. Chem., 1928, 169, 305—308).—Even 0.001% of lead monoxide causes considerable retardation in the setting of Portland cement, but with amounts of oxide up to 0.2% the usual period of rapid hardening sets in some hours after mixing. With cement containing 0.5% or 1.0% of oxide, however, this stage in the setting is not observed, and even after 50 hrs. the cement with 1% of oxide crumbles if placed under water. It seems probable that this effect of the oxide is due to its influencing the colloidal changes which apparently exert a preponderating influence on the hardening of cement, for if the cement is heated at 300° for 1 hr. immediately after being mixed the product is equally hard whether lead oxide is present or not.

R. CUTHILL.

Effect of grain size on the properties of silica cement. S. S. COLE (J. Amer. Ceram. Soc., 1927, 10, 644—650).—The variables causing changes in the properties of silica cements are: chemical composition, clay content, and screen analysis. A "rate of settle" test giving indications of workability is developed. It consists in making a slip of the cement with 50% by wt. of water and placing it in a graduated cylinder. The amounts of settle after 8 hrs. and 24 hrs. are obtained. These data agree with practical observations. Tests, including softening point, linear shrinkage, and rate of settle on commercial and laboratory-prepared cements are reported. It is noted that as the fineness of the mixes increases, the softening point is lowered. There is no apparent relationship between screen analysis and rate of settle, probably due to variations in the clay content of the cement. Slow settling cements, in general, have low softening temperatures.

A. T. GREEN.

Failure to harden of concrete mixtures poor in cement. B. GARRE (Z. anorg. Chem., 1928, 169, 301—304).—Examination of the aggregates used for concrete mixtures which, although made with a good quality cement, had failed to set, revealed the presence of up to 14.1% of acid solubles. As mixtures made with a pure aggregate in the same proportions as those employed for the defective mixtures hardened satisfactorily, it was apparently the acid-solubles which prevented setting, the amounts of these in mixtures containing a large proportion of aggregate actually exceeding the amount of cement.

R. CUTHILL.

Consistency of silicate of soda for curing concrete. R. S. BEIGHTLER (Eng. News-Rec., 1928, 100,

316).—The time at which sodium silicate is added to a freshly made concrete surface affects the spreading power, as dilution occurs to a varying extent with varying moisture in the concrete surface. As a result of several trials it is recommended to use sodium silicate solution of d 1.33—1.35 on the day after laying the concrete, which should be properly protected during the intervening night. This gave a complete seal with the least expenditure of silicate. C. IRWIN.

PATENTS.

Cement-kiln system. D. S. JACOBUS, Assr. to BABCOCK & WILCOX Co. (U.S.P. 1,658,443, 7.2.28. Appl., 25.1.23).—Parallel rotary kilns are connected to a common horizontal flue fitted with draught-producing means at a point beyond the kilns. The connexions between the kilns and the flue are such as to equalise the flow of gases through the kilns irrespective of their differing distances from the source of draught.

H. HOLMES.

Building material. G. KNUDSEN (B.P. 284,576, 23.10.26).—Powdered or granular olivine mineral or magnesium orthosilicate of different origin is mixed with magnesium oxide, talc, and binding material, and is heated in moulds to a temperature (1300—1500°) sufficient for the formation of magnesium orthosilicate from the magnesia and talc, but below the m.p. of the olivine.

W. G. CAREY.

Cementitious composition. J. A. McCORMICK and C. A. CABELL, Assrs. to NAT. LIME ASSOC. (U.S.P. 1,656,984, 24.1.28. Appl., 5.9.24. Renewed 28.5.26).—A quick-setting cement comprises calcium hydroxide, calcium aluminate, aluminium sulphate, and calcium carbonate.

T. S. WHEELER.

Manufacture of cementitious material of cellular structure. G. B. HINTON (U.S.P. 1,657,716, 31.1.28. Appl., 19.9.27).—A pulp consisting of hydraulic cement, water, and a froth-flotation reagent is formed and agitated into a froth.

H. ROYAL-DAWSON.

Manufacture of moulded cement bodies. A. CHATELAIN (U.S.P. 1,657,956, 31.1.28. Appl., 22.5.26).—A mixture of cement, sand, water, and glycerin is formed into shapes upon a glossy surface, after which the surface of the shaped object is treated with a solution of sodium silicate and glycerin before it sets.

H. ROYAL-DAWSON.

Preservation of wood. B. R. V. MALENKOVIĆ (U.S.P. 1,656,863, 17.1.28. Appl., 16.12.25; cf. B., 1925, 208).—An aromatic halogenodinitro-compound, e.g., 4-chloro-1:3-dinitrobenzene, is mixed in alcoholic solution with a non-nitrated primary arylamine (1 mol.), e.g., α -naphthylamine, to yield a double compound of value as a wood preservative. The product in the example given has m.p. about 100°. T. S. WHEELER.

Treatment of walls for heat-insulation purposes etc. [by spraying]. E. H. WENZEL (B.P. 284,849, 15.1.27).

Rotary kilns (B.P. 283,669 and 284,494).—See I. **Washable wallpapers** (B.P. 284,435).—See V. **Compositions from pitch** (B.P. 277,291).—See II.

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

Cyanogen and its compounds in the blast furnace. W. HAUFFE and H. VON SCHWARZE (Arch. Eisenhüttenw., 1927—8, 1, 453—466; Stahl u. Eisen, 1928, 48, 201—203).—Thermodynamical considerations indicate the improbability of any appreciable quantity of cyanogen or of hydrogen cyanide being formed in the blast furnace from the elements or by the reduction of carbon monoxide in the presence of nitrogen. On the other hand, the alkali in the charge should be reduced to free metal; this would immediately combine with excess carbon to form carbide, which takes up nitrogen with the production of alkali cyanide, the greater part of which is volatilised and collects in the upper zones of the furnace and in the flue dust. Secondary reactions may result in the formation of thiocyanate, cyanate, cyanamide, and Prussian blue in the dust chamber. Titanium cyanonitride forms whenever titanium compounds are reduced with excess of carbon in a nitrogen atmosphere, and its formation is accelerated by the presence of manganese sulphide. The volatilisation of potassium cyanide from the blast furnace results in a heat loss of 693 kg.-cal./kg., hence its formation results in a higher coke consumption. Again, it has a highly corrosive action on the walls of the furnace and especially on the tap holes, and appears to be of little value as a reducing or desulphurising agent. A. R. POWELL.

Application of theoretical chemistry to some of the more important processes in the production of steel. H. SCHENCK (Arch. Eisenhüttenw., 1927—8, 1, 483—497; Stahl u. Eisen, 1928, 48, 199—201).—Equations are deduced from the mass-action law to represent the equilibria in the thermal decomposition of carbon dioxide, the dephosphorising of iron, the oxidation of manganese in the converter, and the deoxidation of iron in the converter after the addition of a reducing agent. These equations show that a low temperature and a very basic slag result in the most efficient removal of phosphorus, that the more basic the slag and the lower its content of ferrous oxide the smaller is the amount of manganese oxidised during the period of blow, and that the efficiency of the deoxidation of iron is greater the smaller the solubility of the reducing agent in the molten iron and, except in the case of carbon, the lower the temperature of reduction. A. R. POWELL.

Influence of manganese and the rate of cooling on the separation of ferrite [in steels]. P. SCHAFMEISTER and R. ZOJA (Arch. Eisenhüttenw., 1927—8, 1, 505—510; Stahl u. Eisen, 1928, 48, 312—313).—The carbon content of eutectoidal manganese steels decreases with rising manganese content; thus, with 1% Mn it is 0.83% C, with 2% Mn about 0.77% C, and with 3.1% Mn about 0.71% C. Planimetric measurements of the ferrite areas in steels containing 1—3% Mn with the correct eutectoidal carbon content show that the percentage of ferrite decreases with an increase in the rate of cooling and with an increase in the percentage of manganese, and that the influence of the rate of cooling on the proportion of ferrite is greater the higher the manganese content. For a constant rate of cooling and

manganese content the relation between the proportion of ferrite and the carbon content is a curved line.

A. R. POWELL.

Control of normality in plain carbon steels. F. G. SEFING (Mich. Eng. Exp. Sta. Bull., 1927, [13], 3—25).—Abnormal plain carbon steels (0.15—1.20% C) may be rendered normal by heating at 1093—1107°, regardless of the chemical composition or original micro-structure of the steel, the cementite passing completely into solution.

CHEMICAL ABSTRACTS.

Cementation of ferrous alloys with molybdenum and tantalum, vanadium and cobalt, boron, titanium and zirconium, and uranium. J. LAISSUS (Rev. Mét., 1927, 24, 377—395, 474—484, 591—600, 764—775; 1928, 25, 50—57; cf. B., 1926, 278, 443, 545, 633).—The thickness of the cemented layer produced by heating iron or steel embedded in powdered ferro-alloys of the above metals or in the powdered metals alone increases with the temperature and the time and decreases with a rise in carbon content of the steel. In the case of tantalum and boron the layer consists of solid solution on the inside and a eutectic of iron and an iron-tantalum compound on the outside; with uranium practically the whole of the cemented layer has a eutectiferous structure in which are embedded large crystals of a compound. The structure of the cemented layer of vanadium and chromium in steels is complicated by the presence of complex carbides which cause variations in its thickness. The hardness of the cemented layers after slow cooling decreases in the order: zirconium, titanium, uranium, vanadium, and that of the same layers after quenching from 875° in water in the order: uranium, zirconium, vanadium, titanium, and boron. Cementation with zirconium renders steel practically immune from scaling at 1000°; with chromium the tendency to scale is also very much reduced, but not nearly to the same extent as with zirconium. Rust-resisting coatings are produced only by cementation with tantalum and chromium, whilst with molybdenum, boron, and vanadium the metal is highly resistant to corrosion by 50% hydrochloric acid. Tungsten and cobalt layers resist the action of sulphuric acid (*d* 1.3) but, with the exception of chromium layers, all are violently attacked by nitric acid (*d* 1.15). Cementation with chromium may be effected by electroplating the steel with chromium and heating the metal at 1200° for 10 hrs. whereby a cemented layer of uniform thickness resistant to rusting and to the action of cold dilute nitric acid is produced.

A. R. POWELL.

Deterioration of steels in the synthesis of ammonia. J. S. VANICK, W. W. DE SVESHNIKOFF, and J. G. THOMPSON (U.S. Bur. Stand., Tech. Paper No. 361, 1927, 22, 199—233).—The effects of mixtures of hydrogen and nitrogen containing the equilibrium quantity of ammonia at 500° and 100 atm. pressure on various classes of steels are found to be decarburisation, fissuring, sharp increase in combined nitrogen especially in chromium steels, and porosity in the affected part with consequent lowering of strength and ductility. High-carbon steels are particularly susceptible to decarburisation, even in the presence of 2% Cr, though a steel containing 2.25% Cr and less than 0.3% C is not rapidly

attacked. Addition of tungsten and nickel to such a steel increases the resistance. High-chromium and nickel-chromium steels of the stainless type are most resistant to attack, but the presence of vanadium serves no useful purpose as far as "anticorrosive" properties are concerned. The effects are ascribed to reaction of molecular or activated hydrogen with iron carbide in the straight steels, and to reversible formation and decomposition of iron nitride. In the chromium steels, the tendency to decomposition of the nitride once formed is much less.

L. M. CLARK.

Determination of carbon in pig iron and steel by combustion in oxygen. J. CIOCHINA (Z. anal. Chem., 1927, 72, 435—439).—The determination of carbon in high-carbon steels and pig iron by combustion in a current of oxygen at a high temperature usually gives slightly low results owing to partial dissociation of carbon dioxide to carbon monoxide, which is swept out of the hot zone before it can re-combine with the excess of oxygen. To avoid errors from this cause the gases from the combustion tube should be passed through a narrow silica tube heated at 600°.

A. R. POWELL.

Determination of manganese in iron and steel by the silver nitrate-persulphate method. S. W. LIPIN (Z. anal. Chem., 1927, 72, 401—416).—Comparative tests of the following three modifications of the persulphate method of determining manganese in steel have been made: the method of the American Society for Testing Materials, that of the Verein Deutscher Eisenhüttenleute, and Swoboda's method (B., 1924, 635). The last-named method gives very concordant results with an empirical factor up to a content of 1.6% Mn, but requires the longest time for a determination. The second method gives results which approach most closely to the stoichiometric, whereas the American method is inclined to yield erratic figures. The author recommends that the German method be modified as follows: 0.2 g. of steel is dissolved in 15 c.c. of nitric acid (*d* 1.2); the solution is heated to expel red fumes, treated with 50 c.c. of 0.01*N*-silver nitrate solution and 10 c.c. of a 10% ammonium persulphate solution, heated to 60° during 5 min., cooled in running water, diluted with 50 c.c. of cold water, and, after addition of 3 c.c. of a 1.2% solution of sodium chloride, titrated immediately with arsenite solution until the liquid changes to a pure yellow-green colour. To standardise the arsenite solution, 5 c.c. of 0.03*N*-permanganate are reduced with nitric acid and hydrogen peroxide and the solution so obtained is treated exactly in the same way as the analysis of a steel is carried out.

A. R. POWELL.

Effect of quenching and tempering on the mechanical properties of standard silver. A. L. NORBURY (Inst. Metals, March, 1928. Advance copy. 17 pp.).—Standard silver (92.5% Ag, 7.5% Cu) as ordinarily annealed consists of a solid solution of copper in silver, with small amounts of a copper-rich solid solution. On heating for $\frac{1}{2}$ hr. at 770°, the copper particles go into solution, and the resulting homogeneous solid solution can be retained as such by quenching. These quenched alloys are about 30% softer and 20—30% more ductile than when in the ordinary annealed condition. On tempering these quenched specimens at temperatures

between 200° and 400°, the excess copper is precipitated as copper-rich solid solution, and structures resembling troostite and martensite are obtained. The tempering increases the hardness by 300%, and the tensile strength by 50%, whilst the ductility falls by about 50%; the change is accompanied by a decrease in volume. Quenched specimens may be hardened by cold-work, and then further hardened by tempering. The tarnishing of standard silver in sulphide or other solutions is accelerated by the presence of oxygen, but the quenched and tempered alloys are more resistant than annealed specimens. The removal of coring in cast cupro-nickel alloys by annealing is greatly accelerated by deformation before or during annealing.

W. HUME-ROTHERY.

Constitution of alloys of magnesium and zinc.

R. CHADWICK (Inst. Metals, March, 1928. Advance copy. 14 pp.).—The equilibrium diagram of the system magnesium-zinc has been investigated by thermal and microscopical methods. The m.p. of zinc is lowered by the addition of magnesium and falls to a eutectic at 368°, the eutectic composition being 7.8 at.-% Mg. The liquidus then rises to a maximum at 585° corresponding to the compound $MgZn_3$, and falls to a eutectic at 342° and 69.8 at.-% Mg, from which it rises to the m.p. of magnesium. A second compound $MgZn_5$ is formed at 380° from liquid and $MgZn_3$, but this change is shown only on heating curves, and not on cooling curves; this compound was not observed by Grube (A., 1906, ii, 355). About 0.3 at.-% Mg goes into solid solution in zinc. The compound $MgZn_5$ forms solid solutions to the extent of about 0.4 at.-% at 370°, but these break down on cooling. The compound $MgZn_2$ forms solid solutions within the limits 32.3–43.2 at.-% Mg at 350–400°; on cooling it undergoes, at 357°, a transformation, the nature of which is uncertain. Solid magnesium dissolves about 3.2 at.-% Zn at 480°, but on cooling most of the zinc is precipitated as $MgZn_3$. W. HUME-ROTHERY.

Minute shrinkage cavities in cast alloys of heterogeneous structure.

W. A. COWAN (Inst. Metals, March, 1928. Advance copy. 6 pp.).—The factors affecting the formation of shrinkage cavities are discussed. Tin-base white metals (containing approximately 7.5% Sb and 7.5% Cu) are practically free from shrinkage cavities, but these are present if small amounts of lead are added. The main constituent of these alloys is a solid solution of antimony in tin which solidifies at 237°, but the presence of lead introduces small quantities of a eutectic solidifying at 183°, and if, while still liquid, small drops of this eutectic are completely surrounded by solid, shrinkage cavities must be formed owing to the contraction on freezing. W. HUME-ROTHERY.

Analysis of duralumin and other light alloys.

TOURNAIRE (Ann. Chim. analyt., 1928, [ii], 10, 1–5, 33–37).—Copper is deposited electrolytically from a nitric-sulphuric acid solution of the alloy. For the determination of manganese, the sample is dissolved in a mixture of nitric, sulphuric, and phosphoric acids. Silver sulphate is added and, after oxidation with ammonium persulphate, the resultant permanganate solution is titrated with arsenious acid. The use of hydrofluoric acid in the mixture of solvent acids is deprecated since

it may dissolve manganese from the reaction vessel. Magnesium is weighed as pyrophosphate. The alloy is dissolved in sodium hydroxide solution and the residue is taken up in nitric acid. The nitric acid solution is evaporated to fuming with sulphuric acid. After dilution with water, manganese is precipitated as insoluble oxides by oxidation with ammonium persulphate and, together with silica, is collected on a filter. Citric acid is added to hold up iron, copper, and aluminium, and the magnesium present is precipitated with vigorous agitation as magnesium ammonium phosphate. Occasionally, oxidation of manganese by the method described leaves a trace of permanganate in solution. The use of alcohol to destroy this is not advisable since manganous salts are formed and are precipitated as manganese ammonium phosphate. It is better to determine traces of manganese in the magnesium pyrophosphate by redissolving, oxidising the manganese, and titrating as described above. Silicon is determined by dissolving the alloy in a mixture of hydrochloric, nitric, and sulphuric acids, the residue after evaporation to fuming, dilution, and filtration, being calcined and treated with hydrofluoric-sulphuric acid mixture in the usual way. Metallic silicon which may persist is removed with a mixture of nitric and hydrofluoric acids. Iron is separated from the mixture of oxides insoluble in sodium hydroxide solution by precipitation of the sulphuric acid solution with cupferron, or, alternatively, the acid solution of these oxides is reduced by zinc amalgam and the ferrous iron is titrated with permanganate. For the separation of zinc, the sodium hydroxide solution containing zinc and aluminium is treated with slight excess of sodium sulphide. The precipitate is dissolved in hydrochloric acid, and aluminium is precipitated with ammonia. Zinc sulphide is thrown out from the filtrate with ammonium sulphide and is calcined to oxide. Nickel in the mixture of alkali-insoluble oxides is precipitated as the dimethylglyoxime compound from ammoniacal solution, the other metals present being kept in solution by tartaric acid. L. M. CLARK.

Electrolytic detinning of scrap copper. W. W. STENDER and A. A. IVANOV (Z. anorg. Chem., 1928, 169, 399–404).—Tinned copper may be rapidly and cheaply detinned by using it as the anode in the electrolysis, with an iron cathode, of a 1.2% sodium chloride solution, the completion of the process being indicated by a sharp rise of the *P.D.* between the electrodes. The current density should not exceed 50 amp./m.², and the solution must be kept neutral by the continuous addition of hydrochloric acid. It is, however, somewhat difficult to recover the tin from the solution. This disadvantage is absent if the electrolyte is a 3% hydrochloric acid solution, and the cathode is copper. In this case, copper is separated at the cathode along with hydrogen, and may be melted up for bronze, whilst the solution is readily worked up for a commercial grade of stannous chloride. R. CUTHILL.

PATENTS.

Metallurgical furnace. A. M. Gow (U.S.P. 1,659,869, 21.2.28. Appl., 17.6.21).—Low-pressure air and gas are supplied separately to a reverberatory furnace by means of two adjacent wide conduits and

caused to intermix as they emerge from the conduits by suitably located jets of compressed air. A. R. POWELL.

Rotary melting furnaces. W. BUSS (B.P. 274,451, 5.7.27. Ger., 16.7.26).—The construction of a rotary furnace is such that the longitudinal axis of the drum is at an angle to the horizontal axis of rotation. An internal shoulder is provided close to the axis so that the contents of the furnace do not flow into the hollow trunnion at any point of rotation, the charging hole being situated also in the shoulder. C. A. KING.

Annealing furnace. R. H. SMITH (U.S.P. 1,656,924, 24.1.28. Appl., 14.3.25).—The material is delivered into one end of a horizontal rotary tube mounted in the heating chamber, is conveyed along the tube by a helical rib, and discharged through peripheral openings between the end of the rib and the other closed end of the tube. The openings increase in size in passing circumferentially from the end of the rib in a direction opposed to the rotation. H. HOLMES.

Ore separator. T. W. AINLAY (U.S.P. 1,658,874, 14.2.28. Appl., 5.10.25).—A settling basin, having a retaining lip extending above its lower face at its open side, contains (and extends above and below) a rotatable bowl in order to catch material thrown from the bowl. J. S. G. THOMAS.

Production of iron from iron ores. J. W. HORNSEY and H. E. COLEY (B.P. 284,040, 26.7.26).—Iron ores or oxides are treated in a number of closed chambers arranged in series as, e.g., a series of connected rotary furnaces. In the first the ore is preheated, in the second it is reduced by contact with carbonaceous material and hydrocarbons in the stage of "cracking," whilst in the third chamber cooling of the metallic iron is effected, all the stages being conducted in a substantially neutral or reducing atmosphere. [Stat. ref. to B.P. 215,400 and 11,224 of 1914.] C. A. KING.

Manufacture of malleable cast iron. H. A. SCHWARTZ, Assr. to NAT. MALLEABLE & STEEL CASTINGS Co. (U.S.P. 1,660,398, 28.2.28. Appl., 22.10.26).—Molten cast iron is treated with a rare-earth element which has a high affinity for sulphur and which does not interfere with graphitisation; on cooling, the sulphide of the added element separates first from the mass in the form of spherical inclusions. A. R. POWELL.

Manufacture of articles hardened in their marginal layers by nitrogenation. F. KRUPP A.-G. (B.P. 272,182, 7.5.27. Ger., 2.6.26).—Articles made from steel alloys containing, separately or in combination, 4–8% Si (or Mn, Ni, Cr, Mo, W, V, Ti, or Zr), 0.5–2% Al, and up to 0.7% C are surface-hardened by nitrogenation. C. A. KING.

Zirconium-manganese steel. F. M. BECKET, Assr. to ELECTRO-METALLURGICAL Co. (U.S.P. 1,660,409, 28.2.28. Appl., 9.7.26).—The steel contains 1–2% Mn, less than 1% C, and a substantial proportion of zirconium to render it strong and hard in the air-cooled condition. A. R. POWELL.

Alloy steels for motor valves. R. JARDINE, Assr. to RICH TOOL Co. (U.S.P. 1,660,925, 28.2.28. Appl., 3.1.22).—An alloy containing 1–1.2% C, 13–15% Cr, 5–7% W, 0.8–1% Mo, 2–2.5% Si, 0.4–0.6 Mn,

and a maximum of 0.035% P and 0.035% S, the remainder being principally iron, is claimed.

F. G. CROSSE.

Preparation of an alloy steel for die blocks etc. G. N. HERMAN, Assr. to ALLOY STEEL CORP. (U.S.P. 1,660,790, 28.2.28. Appl., 27.5.26).—A steel alloy containing 0.25–2.25% Cr, 0.20–1.50% Mo, and above 1% Ni is moulded into ingot form, heated to 980–1260° while free from internal strains, forged, and finally annealed. F. G. CROSSE.

Manufacture of a steel alloy. T. A. MOORMANN, Assr. to KINITE CORP. (U.S.P. 1,659,661, 21.2.28. Appl., 11.11.26).—The alloy contains 13% Cr, 1.1% Mo, 0.7% Co, 0.05–0.20% B, and 1.5% C. F. G. CROSSE.

Wet separation of earth, earthy materials, ores, etc. A. C. HOUDIJK (B.P. 284,826, 16.12.26).—The material mixed with water is passed through a horizontal pipe with a branch pipe merging into it from below, the space between the two pipes being open or provided with a suitable screen. The lighter particles are swept upward along the main pipe and the heavier or coarser particles pass downwards into the branch pipe and may be further cleaned by a repetition of the treatment in a series of such pipes. A. R. POWELL.

Manufacture of shaped masses such as briquettes. S. G. S. DICKER, From MALMBRIKETT AKTIEBOLAGET (B.P. 284,418, 8.11.26).—Mixtures of crushed ore, limestone, carbonaceous materials, etc., bonded with tar and/or molasses, together with less than 0.5% of sodium chloride in the form of an aqueous solution, are made into briquettes, which are dried at 150–300° for 1–3 hrs. Such briquettes withstand a temperature of 900° without breaking up, and are therefore very suitable for blast-furnace smelting. A. R. POWELL.

Agglomeration or briquetting of tin ores. METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 275,618, 29.7.27. Ger., 9.8.26).—Fine tin concentrates are mixed with 7–15% of fine, light, tin oxide, such as is obtained by the oxidation of volatilised tin sulphide, and with 4–6% of slaked lime, and the mixture is damped and briquetted in the usual way; during ageing, the briquettes become extremely hard owing to the formation of calcium stannate. A. R. POWELL.

Apparatus for sintering metal ore. P. ANDERSON (B.P. 284,793, 9.11.26).—An apparatus for sintering finely-divided ore concentrates comprises a circular frame carrying a number of sintering pans supported on journals and capable of being rotated about a centrally arranged vacuum chamber. The pans are provided with air pipes extending upwards from the bottom to the level of the journals and connected with the vacuum chamber by means of detachable pipes operating in stuffing boxes. A. R. POWELL.

Reduction of ores. H. WITTEK (B.P. 274,803, 20.11.26. Ger., 26.7.26).—The evolution of heat and reducing gases during the treatment of calcium carbide and nitrogen is utilised directly for reducing ores, e.g., zinc ores, which are mixed with the carbide in the reaction furnace, or reduced in a separate furnace. Calcium cyanamide produced from the mixed batch is

free from residual carbide, and is obtained in a granular form directly available for agricultural use.

C. A. KING.

Treatment [smelting] of ores for the production of metal. D. CROESE (B.P. 284,459, 20.1.27).—The charge of finely-divided ore and fluxes is fed into the top of a vertical shaft-furnace divided into a number of compartments, all of which are heated with suitably disposed gas burners and provided with central double-conical baffles which co-operate with the downwardly tapering throats joining one chamber with the next lower. The first chamber is not heated, but acts as a preheating chamber, and below the lowest chamber is a crucible in which the metal is allowed to separate from the slag.

A. R. POWELL.

Treatment of copper ores. G. W. PRINCE and J. H. ENGLISH, Assrs. to UNITED VERDE EXTENSION MINING Co. (U.S.P. 1,660,817, 28.2.28. Appl., 13.1.26).—Roasted copper ores are leached with water in a vessel through which is passed, at such a rate that the ore pulp is kept in violent agitation, a current of filtered roaster gases containing sulphur dioxide. The upper part of the leaching vat is provided with an electrolytic unit for precipitating the copper as fast as it is dissolved from the ore.

A. R. POWELL.

Manufacture of finely-divided metals from metallic carbonyls. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 284,087, 4.2.27. Addn. to B.P. 269,677; B., 1927, 527).—Metals other than iron can be obtained from their carbonyls by introducing the latter, either singly or mixed together and with or without the addition of iron carbonyl, into a heated chamber in such a way that the decomposition takes place in the hot free space of the vessel. The carbonyls may be introduced in the form of vapour either undiluted or mixed with inert gases; or solid carbonyls may be either fused under compressed carbon monoxide and atomised in the chamber, or dissolved in a suitable solvent (*e.g.*, benzene or another liquid carbonyl) and the solution injected into the chamber. The decomposition may be effected under ordinary, reduced, or increased pressure; the temperature required depends on the nature of the carbonyl, the associated gas (if any), and the pressure employed.

M. E. NOTTAGE.

Production of refractory metals. E. A. LEDERER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,659,205, 14.2.28. Appl., 22.11.24).—The sulphide of the metal is heated *in vacuo* to its temperature of decomposition, and the sulphur volatilised is removed.

H. ROYAL-DAWSON.

Porous metal bodies. A. E. WHITE. From GEN. MOTORS RES. CORP. (B.P. 284,532, 15.6.27).—A compressed mixture of finely-divided metals which do not alloy or only partially alloy together with graphite and a second deoxidising agent is heated; *e.g.*, a mixture including copper, lead, graphite, and phosphor-tin is heated so that metallic oxides present are reduced by phosphorus, an alloy of tin and copper is formed, and lead is melted.

C. A. KING.

Refining of lead bullion containing other metals. G. K. WILLIAMS (B.P. 267,104, 17.2.27. Austral., 6.3.26).—In carrying out the Parkes process for the

removal of copper, gold, and silver from lead, it has been found that a much more efficient separation of these metals is effected if the lead is fed into deep and narrow cylindrical kettles containing a bath of molten desilverised lead below a layer of molten zinc, the lead layer being maintained at a temperature of about 330–340° and the zinc layer at 700–850°. The lead to be desilverised is fed together with about 18 lb./ton of zinc directly into the hot zinc layer, and the purified lead overflows continuously through a long spout leading to the bottom of the kettle and maintained at 400°. By keeping the lead in the lower part well stirred, any crusts that form on the sides are loosened and rise into the conjugate zinc layer. Two or more of the kettles may be used in series, the crusts from the later kettles being used instead of zinc in the feed to the earlier kettles. Operating in this manner it is possible to obtain a final lead containing only traces of gold and copper and less than 0.2 oz./ton of silver, together with a zinc "crust" assaying over 25% Ag and only about 10% Pb. The process is continuous and requires much less zinc than the usual Parkes process.

A. R. POWELL.

Production [melting] of alloys of the platinum metals. E. HAAGN, Assr. to W. C. HERAEUS G.M.B.H. (U.S.P. 1,660,159, 21.2.28. Appl., 9.2.26).—The material to be melted is placed on a small block of lime against an electrode parallel to the surface of the block, and the other electrode is applied diagonally to the opposite side of the metal mixture.

A. R. POWELL.

Preparation of metals [sodium alloys] for chemical uses. C. A. KRAUS and C. C. CALLIS, Assrs. to STANDARD DEVELOPMENT Co. (U.S.P. 1,655,908, 10.1.26. Appl., 5.4.23).—Molten sodium is added to molten lead or tin in an atmosphere of nitrogen, and the resulting alloy is granulated by being dropped down a tower in which it is cooled.

T. S. WHEELER.

Copper refining. A. G. DE GOLYER (U.S.P. 1,660,220, 21.2.28. Appl., 16.4.27).—Molten copper is deoxidised with a carbonaceous material to reduce its oxygen content to a point just above that at which carbon monoxide is retained in the metal; complete deoxidation is then effected by addition of a metallic reducing agent, thus avoiding the presence of adsorbed gases in the refined metal.

A. R. POWELL.

Manufacture of metals and chemicals by means of iodine. A. A. OSSA (U.S.P. 1,660,884, 28.2.28. Appl., 4.2.21. Renewed 14.7.27).—Material containing a heavy metal (*e.g.*, copper) soluble in sulphuric acid is leached with that acid and the solution treated with iodine and sulphur dioxide to precipitate the iodide of the metal. The precipitate is reduced with a more electropositive metal, and the solution of soluble iodide thus formed is treated to recover the iodine which is used again in the process (*cf.* B.P. 215,439; B., 1924, 601).

A. R. POWELL.

Recovery of copper from liquors by precipitation. I. G. FARBENIND. A.-G. (B.P. 276,017, 13.8.27. Ger., 14.8.26).—The precipitation of copper from metallurgical liquors is effected in two or more stages by means of zinc hydroxide. The fresh liquor is treated in the first stage with an amount of precipitant insufficient for the complete precipitation of the copper, so that the

precipitate from this stage is practically free from zinc ; in the second stage (or in the final stage when there are more than two) the precipitant is added in excess, and the resulting zinciferous precipitate is used as the precipitant in the first stage of treating a fresh batch of liquor. Alternatively, when the precipitation is effected in more than two stages, each of the resulting precipitates except the first may be used to effect a partial precipitation of copper from cupriferous liquors of an earlier stage.
M. E. NOTTAGE.

Protection of molten magnesium. H. H. OSBORNE, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,657,693, 31.1.28. Appl., 27.10.22).—Molten magnesium and its alloys are brought into contact with products resulting from the heating of glycerin above the m.p. of the metal.
H. ROYAL-DAWSON.

Treatment of aluminium-bearing ores. R. W. HYDE, Assr. to DWIGHT & LLOYD SINTERING CO., INC. (U.S.P. 1,655,608, 10.1.28. Appl., 22.12.26).—An intimate mixture of finely-divided bauxite, hæmatite, and coal is ignited in a current of air. Combustion of the coal proceeds autogenously throughout the mass, and sufficient heat is generated to dehydrate the bauxite, a portion of which combines with the ferric oxide to form ferric aluminate ; the product melts and forms a cellular cake convenient for further treatment.
T. S. WHEELER.

Aluminium alloys. W. SANDER, Assr. to T. GOLDSCHMIDT A.-G. (U.S.P. 1,656,502, 17.1.28. Appl., 30.11.26. Ger., 21.10.25 ; cf. B.P. 272,706 ; B., 1927, 606).—Alloys of aluminium with zinc, copper, magnesium, etc. are quenched from a glowing heat, left at room temperature for 24 hrs., and then heated at 130° for 24 hrs. to increase their tensile strength.
T. S. WHEELER.

Aluminium alloys. H. SCHORN, Assee. of R. MÜLLER (B.P. 277,701, 19.9.27. Ger., 18.9.26).—Alloys resistant to the action of sea-water and containing 1–6% Mg, 0.05–1% Ti, with remainder aluminium are claimed.
F. G. CROSSE.

Solder for aluminium and its alloys. G. SCHULDIT (U.S.P. 1,661,052, 28.2.28. Appl., 14.6.26. Ger., 15.6.25).—The solder consists of 450 pts. of aluminium, 370 pts. of tin, 180 pts. of zinc, 20 pts. of copper, 15 pts. of silicon, 10 pts. of lithium, and 5 pts. of iron.
F. G. CROSSE.

Production of metallised surfaces on non-metallic bodies. A. I. G. WARREN, and PRECIOUS METAL INDUSTRIES, LTD. (B.P. 284,786, 5.11.26).—The surface of the substance to be metallised is treated with a suitable etching agent which opens the surface pores, e.g., dilute acetic acid for mother-of-pearl, or sulphur in carbon disulphide for vulcanised articles and casein. The surface is then given a smooth coating of a silver or gold compound made into a paste with water, chloroform, benzene, or other volatile liquid, and finally heated in steam under a pressure of 3 atm. so that the metallising substance becomes thoroughly incorporated with the surface of the article. Reduction to metal may be effected by heating in a suitable reducing atmosphere, or by electrolytic processes, and, if necessary, a smooth

finish may be imparted by polishing and plating with the same or another metal.
A. R. POWELL.

Coating metal [with lead alloy]. E. R. MILLRING, Assr. to AMER. MACHINE & FOUNDRY CO. (U.S.P. 1,660,847, 28.2.28. Appl., 19.10.26).—A lead alloy for coating metal is obtained by adding phosphor-lead to molten lead containing a proportion of tin which serves to increase the solubility of the phosphorus in the alloy.
A. R. POWELL.

Removal of incrustation formed on metallic surfaces which are in contact with hot milk. R. SELIGMAN (B.P. 284,778, 4.11.26).—Incrustations, regarded as calcium caseinogenate, which form on metal surfaces used for heating milk, are removed by treatment with a weak (2%) solution of orthophosphoric acid containing casein or gelatin as a protective agent against undue corrosion of the metal.
C. A. KING.

Alloys and their application to telegraphic and telephonic conductors. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 284,789, 6.11. and 17.12.26).—Alloys possessing high electrical resistance and magnetic permeability in fields from 0.001–2 gauss contain 32–42% Ni, 1–4% Si (or Al), 1–4% Cr (W, Mo, V), the remainder being iron preferably within the limits 55–65%. Up to 1% Mn may be present to improve the working properties. Particular compositions of iron alloys are Ni 36%, Cr 3%, Al 3%, and Ni 36%, Cr 2%, Si 1.3%.
C. A. KING.

Electrodeposition of chromium. W. S. EATON (B.P. 284,900, 2.5.27).—An electrolytic bath for the deposition of chromium comprises a 10% solution of chromic acid to every gallon of which is added 1 oz. of a mixture of 94% of sodium hydroxide, 2% of sodium sulphate, 2% of sodium carbonate, and 2% of sodium chloride. The article to be plated with chromium is first cleaned by being made the cathode in an alkaline bath containing an iron or carbon anode. The plating bath is maintained at 43–54°.
A. R. POWELL.

Copper-tin-nickel alloy. R. OZLBERGER (U.S.P. 1,661,083, 28.2.28. Appl., 24.3.27. Austr., 3.4.26).—See B.P. 268,798 ; B., 1927, 560.

Annealing furnaces (B.P. 283,725 and 283,767).
Rotary kilns (B.P. 284,494).—See I. **Metal containers for alcohol** (B.P. 269,135).—See III.

XI.—ELECTROTECHNICS.

Electrochemical production of graphitic oxide. B. K. BROWN and O. W. STOREY (Amer. Electrochem. Soc., April, 1928. Advance copy. 12 pp.).—Graphitic oxide may be obtained by the anodic oxidation of graphitised carbon electrodes in a neutral or acid electrolyte with an oxidising anion. Dilute nitric acid, with or without the addition of an oxidising salt such as potassium chlorate, is preferred. Impregnation of the electrode with paraffin wax prevents undue disintegration. By using a current density not greater than 10 amp./ft.², and keeping the temperature at 22–25°, a product containing 9–11% of available oxygen is obtained at a current efficiency of about 20%. Graphite scrap can be used as raw material if it is powdered, mixed with molten paraffin wax, and moulded into

electrodes under high pressure (350 kg./cm.²), but since such electrodes tend to swell up and disintegrate with slight rise in temperature, rosin is preferred as a binder; it also has the advantage of being dissolved or destroyed during the electrolysis, so that the resulting graphitic oxide can be used in a dry cell mix without previous washing with an organic solvent. The available oxygen in graphitic oxide can be determined by means of ferrous sulphate; the results are somewhat lower than by the combustion method.

H. J. T. ELLINGHAM.

Graphitic oxide as depolariser in the Leclanché cell. B. K. BROWN (Amer. Electrochem. Soc., April, 1928. Advance copy. 6 pp.).—Graphitic oxide may be used as a depolariser to supplant or supplement the manganese dioxide used in dry cells. Graphitic oxide itself being practically a non-conductor of electricity requires mixing with graphite, but the oxide prepared electrochemically consists of particles with a core of unchanged graphite, and may only require milling in order to make it suitable for use. The preparation of an effective depolarising mix is described. Cells made with a graphitic oxide depolariser have a notably lower, open circuit *E.M.F.* than manganese dioxide cells, and the voltage during discharge is also somewhat lower. But the available oxygen of the graphitic oxide appears to be more completely utilised, and gassing and evolution of ammonia are greatly reduced. The "shelf life" is comparable with manganese dioxide cells, and the weight is less for given volume of depolariser. During discharge the graphitic oxide is reduced to graphite or other form of carbon, so that the conductivity of the mass increases: hence the cells maintain a high short-circuit amperage during discharge. Such cells may be advantageous for certain kinds of service.

H. J. T. ELLINGHAM.

Graphitic oxide. STOREY.—See II. **Conductivity of clay slips.** FESSLER and KRANER.—See VIII. **—Detinning of scrap copper.** STENDER and IVANOV.—See X.

PATENTS.

[Magnetic] separation of material. F. B. MACLAREN, Assr. to C. H. BRASELTON (U.S.P. 1,657,405, 24.1.28. Appl., 15.9.22).—A mixture of metallic and non-metallic materials to be separated is allowed to fall through a long vertical gap between groups of fixed poles of electromagnets energised by alternating current. The magnetic field is such that the metallic constituents are diverted toward one group of poles more than the non-metallic constituents.

H. HOLMES.

Electrical method and apparatus for identifying chemical elements and compounds and biological organisms. M. E. SIMON and J. A. O'CONNOR (B.P. 283,650, 15.10.26).—Substances having different characteristics are distinguished by being brought successively into active relationship with an electrical control circuit, which is caused to operate an electrical response circuit, e.g., an electron emission relay circuit.

J. S. G. THOMAS.

Electrolytic apparatus. H. J. YEAGER (U.S.P. 1,658,872, 14.2.28. Appl., 16.1.25).—An electrolytic cell comprises a container with electrolyte and electrodes,

an electromagnet having a substantially U-shaped core comprising two pole-pieces, and means for producing a magnetic field between them so that motion of the electrolyte is produced.

J. S. G. THOMAS.

Charging of fluids and other substances with ultra-violet rays. E. L. CUNNINGHAM and H. F. MACBETH (U.S.P. 1,659,085, 14.2.28. Appl., 17.1.25).—A fluorescent substance is dissolved in a liquid which is then vaporised and subjected to the action of the rays.

H. ROYAL-DAWSON.

Galvanic cell. A. HEIL (B.P. 262,814, 10.12.26. Ger., 10.12.25).—A narrow, vertical, slidable zinc electrode, immersed only in the upper part of the electrolyte, extends at right angles on either side of a series of parallel positive electrode plates. A chromium alloy is used for the anode and the non-rusting V2A steel for all connexions.

J. S. G. THOMAS.

Dry cell. W. F. HENDRY and V. YNGVE, Assrs. to NAT. CARBON Co., INC. (U.S.P. 1,656,066, 10.1.28. Appl., 23.1.24).—Impalpable graphite for use as a depolariser in dry cells is treated with 10% hydrochloric acid and then with 4% sodium hydroxide solution to yield a product with a reduced ash content.

T. S. WHEELER.

Selenium cell. J. NEALE (B.P. 284,942, 6.8.27).—A non-conducting, heat-resisting, preferably transparent material, e.g., acid glass, is impressed with a number of lines of a solution known as silver-G and containing gold and/or platinum, the lines preferably forming interdigitated grids or combs. After firing the plate at about 525° the process may be repeated a number of times; the plate is finally heated to about 200°, and the interstices between the grids or combs are thereby coated with selenium, and the whole is annealed.

J. S. G. THOMAS.

Preparation of an electron-emitting material. T. P. THOMAS, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,659,175, 14.2.28. Appl., 20.6.22).—A thoriated filament to which phosphorus has been applied is heated, and the phosphorus maintained in intimate contact with the filament.

J. S. G. THOMAS.

Glow-discharge valve. F. SCHRÖTER, Assr. to AMER. TELEGRAPH & TELEPHONE Co. (U.S.P. 1,656,957, 24.1.28. Appl., 30.3.21. Ger., 23.1.18).—The presence of hydrogen (1–3%) in the argon filling of a cold cathode discharge tube decreases considerably the potential necessary to initiate discharge.

T. S. WHEELER.

Electric vacuum valve tube. F. SCHRÖTER, Assr. to AMER. TELEPHONE & TELEGRAPH Co. (U.S.P. 1,656,956, 24.1.28. Appl., 30.3.21. Ger., 29.1.24; cf. B., 1920, 183 A).—A device for protecting circuits against excess voltage comprises a discharge tube containing a small quantity of an inert gas, e.g., argon, and electrodes of an alkali or alkaline-earth metal.

T. S. WHEELER.

Carbon electrodes (B.P. 284,818).—See II. **Glass insulators** (F.P. 618,186 and 618,288). **Coherent granular substances** (B.P. 255,497).—See VIII. **Magnetic alloys** (B.P. 284,789). **Electrodeposition of chromium** (B.P. 284,900).—See X.

XII.—FATS; OILS; WAXES.

Determination of neutral fat in soaps. L. F. HOYT (Oil Fat Ind., 1927, 4, 357—359).—The sample (10—15 g.) is dissolved in hot 94% alcohol (150—200 c.c.) and the solution filtered. Alcoholic potassium hydroxide (0.5*N*) is added to the neutral solution, and the liquid boiled in a reflux apparatus for 30 min., a blank determination also being performed. The liquid is titrated hot with 0.5*N*-acid. Neutral fat= $[c.c. 0.5N\text{-alkali} \times 28.05/\text{saponif. value}] \times (100/\text{wt. of sample})$. For soaps of unknown fat composition the neutralisation value (mg. KOH per g.) of the separated fatty acids, multiplied by 0.97, is approx. the saponif. value of the neutral fat from which they were derived. CHEMICAL ABSTRACTS.

Titre of New Zealand mutton tallow. A. M. WRIGHT and I. THOMPSON (J.S.C.I., 1928, 47, 13—14 T).—Summaries of analyses for titre of several thousands of tallow from the North and South Islands of New Zealand show that there is a gradual increase in titre as the stock becomes more mature and older. In general, a peak is reached followed by a slight but definite fall, the reason for which is obscure, but the fall is more or less coincidental with onset of winter. A definite relationship between titre and latitude from which the stock came is observable, particularly seen when the seasonal average over seven years is compared with the mean latitudes. The lower the latitude the lower was the average titre, due probably to climatic variations. There are, however, many anomalies and contradictory points which render any one explanation insufficient. D. G. HEWER.

Maturing process of polymerised and oxidised linseed oils. P. SLANSKY and L. KÖHLER (Chem. Umschau, 1928, 35, 41—44).—From a critical examination of the variation in viscosity with storing of samples of linseed oil, oxidised or polymerised to varying extents, it is concluded that the highly oxidised or polymerised samples exhibit a maturing phenomenon. An unblown oil retained its original viscosity of 4.2", measured on an Oswald capillary viscosimeter, after storing for three months; a sample blown for 6.5 days at 50° gave values of 1' 16.3" before storing, 2' 18.5" after one month, and 7' 11.2" after three months, whilst a sample blown for 9 days had viscosity of 9' 22.2", and gelatinised completely within three months. Comparable results were obtained for linseed oil containing 1% of cobalt linoleate, and for linseed oil polymerised at 290—295° for periods varying up to 18 hrs. The maturing process was followed with the aid of the ultra-microscope. No corresponding variations in constants such as iodine value and refractive index were observed, and it is concluded that the variation in these characteristics with the degree of oxidation or polymerisation is due to chemical rather than colloidal phenomena. E. HOLMES.

Determination of organically-combined sulphuric acid in sulphonated oils. K. H. BAUER (Chem. Umschau, 1928, 35, 25—26).—Sulphonic acid groups in sulphonated oils can be accurately determined by heating the oil under reflux for 1 hr. with a known amount of *N*-sulphuric acid and back-titrating with standard alkali the total sulphuric acid present. Allow-

ance has to be made for the amount of acid added and for the original alkalinity of the oil (alkali-refining being customary). This is contrary to the views of Herbig, who advocates decomposing the sulphonate with hydrochloric acid in place of sulphuric acid, but results obtained by the two means are shown to agree. Full details of the above method and of the determination of moisture and acid value are quoted. S. S. WOOLF.

Test for refined olive oils in virgin olive oils. A. BAUD and COURTOIS (Ann. Chim. analyt., 1928, [ii], 10, 11—14, and J. Pharm. Chim., 1928, [viii], 7, 215—218).—See B., 1928, 210.

Vitamin-A in cod-liver oil extract. MUNSSELL and BLACK.—See XX.

PATENTS.

Manufacture of a cleansing compound. W. O. NANCE, Assr. to DELETE CO., INC. (U.S.P. 1,657,893, 31.1.28. Appl., 9.11.27).—A solution of 7 pts. of caustic alkali in 3 pts. of water is gradually added to a mixture of 60 pts. of pale, clean, mineral oil and 15 pts. of fatty acid, agitated and heated to a temperature exceeding 100°. The oil is emulsified with the progressively saponified fatty acid and 15 pts. of a mixture of suitable solvents are added. The temperature is reduced, but is maintained sufficiently high to permit the completion of the saponification and emulsification. The mass on cooling produces a substantially stable non-aqueous detergent paste readily soluble in dry-cleaning liquids. S. S. WOOLF.

Extraction of fats etc. SOC. ANON. DES ATELIERS ARMAND & DÉCUNE (F.P. 621,328, 2.1.26).—Fats etc. are extracted by means of trichloroethylene, benzene, etc. in a closed circuit, charging and discharging being mechanical. S. S. WOOLF.

Preparation of concentrate of cod-liver oil rich in vitamin-A and other active principles. K. KAWAI (B.P. 283,265, 5.10.26).—A concentrate of cod-liver oil rich in clinically effective ingredients is obtained by saponifying the oil to the extent of 50—75% of its saponifiable content by means of caustic soda, milk of lime, etc. Vitamin-A and other unidentified active principles are absorbed into the remaining unsaponified oil as the reaction proceeds. S. S. WOOLF.

Extraction of castor oil. I. G. FARBENIND. A.-G., Assces. of J. BEHRENS (B.P. 265,212, 27.1.27. Ger., 27.1.26).—Castor oil is extracted from the seed by means of chlorinated hydrocarbons, e.g., trichloroethylene, carbon tetrachloride, methylene chloride, etc., alone or mixed with other solvents, e.g., mixed with a large proportion of benzene. B. FULLMAN.

Vulcanised or sulphurised oil compositions. A. DE WAELE (B.P. 284,415, 3.11.26).—Elastic masses of vulcanised oil compositions are obtained by subjecting esters of higher unsaturated fatty acids, in which a hydrogen atom of the fatty acid residue has been substituted by an electronegative radical less electronegative than the hydroxyl group, to the action of vulcanising agents such as sulphur chloride, sulphur thiocyanate, dithiocyanogen, etc., which react energetically with

glycerides of unsaturated hydroxy-fatty acids; the reaction is thus moderated. In particular, there are used esters of oxy- or hydroxy-unsaturated fatty acids in which the hydrogen of the hydroxyl group is substituted by acyl groups, *e.g.*, the chlorides, acetates, formates, and carbonates, mixed with esters of other fatty acids. In practice, there are used acetylated castor oil or oxidised or blown oils. Additions of oils, fats, waxes, fillers, diluents, accelerators, or retarding agents may be made. B. FULLMAN.

Extraction of oil from oily waste, oil filters, etc. P. AUZY (F.P. 619,627, 9.12.25).—The material to be extracted is raised to a temperature of 100–110° by dry steam in a slowly rotating centrifuge. After ½ hr. the steam supply is discontinued, any condensed water being run off, and the centrifuge is accelerated to 360–400 r.p.m. The extracted oil collects in a suitably disposed sump. S. S. WOOLF.

Viscosity tester for oils (U.S.P. 1,659,534).—See I. **Fatty matter from wool washing** (B.P. 273,642).—See V. **Edible fat** (B.P. 284,368 and U.S.P. 1,656,474).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Rapid testing apparatus for paint. A. V. BLOM (Chem. Fabr., 1928, 102–103).—Two parallel chains are mounted on pairs of cog-wheels 2 m. apart so as to carry between them 50 test sheets 10 × 20 cm. The chains travel at 20 cm./min. and carry the sheets through three heating chambers and one freezing box. Sprays are fitted between the chambers, one of which can be filled with corrosive gases if desired. The strips are also submitted to the action of ultra-violet light and that from a "Sollux" lamp. A typical time of treatment is 5 days, and it is claimed that with this apparatus natural weathering conditions are closely imitated. C. IRWIN.

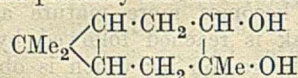
Pigments. I. [Basic carbonate- and basic sulphate-white lead.] H. WOLFF (Farben-Ztg., 1928, 33, 1343–1346).—The chemical similarity of basic carbonate- and basic sulphate-white leads is reflected in parallelism in their properties. Comparisons of oil absorptions, drying times of paints containing these pigments, mechanical properties of films, resistance to weathering, etc. support this contention in general; exceptions are indicated in questions of opacity, retention of gloss, and tendency to support rusting (the greater tendency of the carbonate in this connexion being tentatively attributed to carbon dioxide derived from the pigment rather than to impurities therein). S. S. WOOLF.

Action of pigments in metal primers. E. J. PROBECK and H. W. BATTLE (Ind. Eng. Chem., 1928, 20, 197).—The ratio of pigment to resin in nitrocellulose primers is of considerable importance from the durability point of view. Whilst a low concentration of pigment—with regard to resin etc.—is held to improve adhesion, 16 months' exposure tests showed that relatively high concentration of pigment produced much greater durability of the whole lacquer system. The compositions of the nitrocellulose primers and surfacers used are discussed. S. S. WOOLF.

Rapid assay of basic lead carbonate. F. W. GLAZE (Chemist-Analyst, 1927, 16, [2], 19–20).—The assay consists of the determination of insoluble impurities, lead as chromate, and carbon dioxide alkalimetrically.

CHEMICAL ABSTRACTS.

Constituents of Indian turpentine from *Pinus longifolia*, Roxb. IV. P. P. PILLAY and J. L. SIMONSEN (J.C.S., 1928, 359–364).—Oxidation of *d*- Δ^3 -carene in acetic acid solution with hydrogen peroxide at 40–60° for 148 hrs. followed by digestion of the non-acidic portion of the product with aqueous alcoholic potassium hydroxide solution, yields *d*-carene- β -glycol, m.p. 90–91°, b.p. 147–150°/18 mm. (+ H₂O, m.p. 75°), together with an isomeric substance, b.p. 155–157°/28 mm., α_D –0.69° (in chloroform), which, when heated with 5% sulphuric acid for 48 hrs., yields *l*-carene oxide (below) and *p*-cymene. The β -glycol by treatment with phthalic anhydride at 110° yields a *hydrogen phthalate*, m.p. 191–192°, and therefore contains only one secondary alcohol group and probably has the structure



although it is not identical with the glycol of this structure obtained by oxidation of *d*- Δ^3 -carene with potassium permanganate in alkaline solution (Simonsen, B., 1920, 581 A). Oxidation with chromic and acetic acids causes it to undergo complete degradation, no trace of the hydroxyketone being detected. When treated with dilute sulphuric acid, it is converted into *p*-cymene and *l*-carene oxide, b.p. 152°/99 mm., d_{20}^{20} 0.961, n_D^{20} 1.4740, which is not quite pure but contains a small quantity of a ketone isolated as its *semicarbazone*, m.p. 193–193.5°. When the oxide is heated with phthalic anhydride at 160° for 6 hrs. it yields *d*-carene- β -glycol hydrogen phthalate in good yield. When *l*-carene oxide is oxidised with potassium permanganate in acetone at 0°, a complex mixture of acids is produced from which a small quantity of a keto-acid, C₉H₁₃O₃, was isolated as its *semicarbazone*, m.p. 165–166°. Oxidation of *d*- Δ^3 -carene with sodium hypochlorite solution does not proceed smoothly, the products isolated being *hydroxychlorocarene*, b.p. 90–93°/10 mm., d_{20}^{20} 1.0123, n_D^{20} 1.4992, and impure dichlorodihydroxymethylcyclopropylcyclohexane, b.p. 110–113°/10 mm., which is probably a mixture of isomerides.

J. W. BAKER.

Consistency of lacquers. C. D. BOGIN and C. W. SIMMS (Ind. Eng. Chem., 1928, 20, 190–196).—The application of brushing and spraying lacquers is materially affected by consistency. This is evidenced by variation in brushing time or spraying pressure, orange-peel effects, blushing, and general appearance. Factors influencing consistency are concentration of nitrocellulose, composition of solvent mixture, nature of pigment, variation in batches of commercial nitrocellulose, and ageing of lacquer in the presence or absence of impurities. Each of these factors is discussed and data are adduced. Consistency is measured by means of an efflux pipette, glycerol being taken as standard. An empirical relationship is derived connecting consistency with concentration of nitrocellulose.

S. S. WOOLF.

Thinners for nitrocellulose lacquers. J. G. DAVIDSON and E. W. REID (Ind. Eng. Chem., 1928, 20, 199—200).—The requirements of lacquer thinners are:—good solvent properties and freedom from any hydrolysable, noxious, or “blush”-inducing components. “Blush”-resisting solvents require relatively high b.p., but must not unduly retard the setting of the lacquer film. The qualities of amyl acetate, Cellosolve (ethylene glycol monoethyl ether), Cellosolve acetate, and other modern commercial solvents are discussed in relation to their use in thinners. Empirical relationships for deducing the solvent efficiency of various mixtures are described. S. S. WOOLF.

Solvent structure and solvent action. B. K. BROWN (Ind. Eng. Chem., 1928, 20, 183).—The desirability of a solvent for use in cellulose lacquers cannot be computed from its nature alone; thus, it is found that “two-type” solvents (*e.g.*, diacetone alcohol, ethyl ether of ethylene glycol) that will dissolve nitrocellulose and resins separately, will not, without addition of further solvents, give clear lacquers when the solutions are mixed. S. S. WOOLF.

Constants of nitrocellulose solvents. J. A. BRIDGMAN (Ind. Eng. Chem., 1928, 20, 184—187).—The bearing of the following properties of various high-boiling solvents on their use in nitrocellulose lacquers is indicated:—evaporation rate, viscosity of lacquers made therefrom, stability (as measured by rate of hydrolysis in presence of a limited amount of alkali and by development of acidity in presence of small amounts of organic acid), solubility in water, and temperature depression due to evaporation. S. S. WOOLF.

Explosive properties of lacquer-solvent vapours. E. G. RICHARDSON and C. R. SUTTON (Ind. Eng. Chem., 1928, 20, 187—190).—The explosive limits of mixtures of single and mixed solvent and diluent vapours with air, as determined in suitable apparatus (details of which are furnished), are tabulated. It was found that the medium- and high-boiling alcohols and esters are not capable of forming explosive mixtures at ordinary temperatures, but the introduction of even moderate proportions of hydrocarbon diluents renders the air-mixture explosive. In two standard-type spray booths tested, ventilation was found to be adequate to keep vapour concentration much below explosive limits. S. S. WOOLF.

“Crystallising” varnishes and “crackle” varnishes. F. KOLKE (Farben-Ztg., 1928, 33, 1281—1283).—The nature of “crystallising” varnishes and “crackle” varnishes is indicated, the former depending on the drying peculiarities of tung oil and the microscopically wrinkled surface produced under specified conditions, whilst the latter (which are generally nitrocellulose lacquers) owe their characteristic behaviour to low content of non-volatile matter, freedom from plasticiser, and contrast in elasticity with the undercoat. The undercoat is made necessary by the obviously poor adhesion of the “crackle” lacquer, and is preferably not dry when the latter is applied. S. S. WOOLF.

Lacquer formulation, with special reference to the use of cumar. A. ROGERS and C. BANTA (Ind.

Eng. Chem., 1928, 20, 198).—A brief account of the formulation and production of a typical brushing lacquer is given. The advantages of synthetic resins, *e.g.*, cumar, over natural resins in this connexion are stressed, but the use of cumar or ester gum alone as the resin component of a lacquer is not recommended. A combination of the two or of either with damar or other natural resin gives satisfactory results.

S. S. WOOLF.

Camphor oils. VAUBEL and NEDELSCHIEFF.—See XX.

PATENTS.

Production of red lead paints and the like coating compositions. A. FRANKE (B.P. 264,492, 10.1.27. Ger., 15.1.26).—Red lead in which the size of the particles does not exceed 4—5 μ , produced from lead monoxide obtained by a process such as that described in B.P. 189,132 (B., 1924, 343), is used in the manufacture of paints and similar compositions. L. A. COLES.

Marine coating [paint] and the like. G. W. TARR, Assr. to BLUINE Co. (U.S.P. 1,657,438, 24.1.28. Appl., 3.8.27).—A mixture of a metallic pigment, *e.g.*, ferric oxide, a waterproof binder, *e.g.*, tar, and powdered urea is claimed as an anti-fouling paint for ships.

T. S. WHEELER.

Production of white lead. C. H. BRASELTON (U.S.P. 1,655,723, 10.1.28. Appl., 1.4.19).—Powdered lead is treated in a rotating cylinder containing quartz pebbles with carbon dioxide, air, and superheated steam.

T. S. WHEELER.

Production of varnishes etc. C. E. PECZENIK and F. SCHAMBERGER (B.P. 283,233, 8.7.26).—A viscous varnish-body is produced by heating the juice of the *Anacardium* (*e.g.*, the juice expressed from the shells) in the presence of a hardening agent, *e.g.*, borates, peroxides, sulphur-bearing compounds, etc. The thickening action is assisted by the presence of an aldehyde (*e.g.*, formaldehyde) and anti-corrosive substances (*e.g.*, phosphates, chromates). The product is thinned with the usual varnish solvents and the hardening agent is removed, *e.g.*, by filtration.

S. S. WOOLF.

Manufacture of primings, varnishes, and paint vehicles. W. T. BRANSCOMBE and R. C. L. EVELEIGH (B.P. 283,998, 24.7.26).—In modification of B.P. 266,401 (B., 1927, 333), a priming material contains as a pore-filler a fatty oil which has been treated with a small quantity of sulphur or sulphur chloride. Thinners and resins, driers, varnish, etc. may be added.

B. FULLMAN.

Manufacture of a colour-binding means. F. SICHEL KOMMANDIT-GES. (B.P. 270,652, 15.3.27. Ger., 10.5.26).—Size-colours are rendered more resistant to rubbing by the addition of oxycellulose or hydrocellulose.

L. A. COLES.

Composition of matter [resin]. M. E. DELANEY and L. T. RICHARDSON, Assrs. to CUTLER-HAMMER MANUF. Co. (U.S.P. 1,655,942, 10.1.28. Appl., 25.7.21).—Naphthalene and sulphur monochloride are heated in presence of a catalyst, *e.g.*, tin or aluminium metal or ferrous sulphide, to yield according to temperature and time of heating a more or less insoluble and infusible resin. T. S. WHEELER.

Production of resinous bodies from formaldehyde and colophony. A. AMANN, Assr. to CHEM. FABR. K. ALBERT G.M.B.H. (U.S.P. 1,658,828, 14.2.28. Appl., 7.8.24).—Colophony is heated with aqueous formaldehyde at a temperature above its m.p. but below that at which foaming occurs. B. FULLMAN.

Preparation of a new type of shellac substitutes. J. SCHEIBER and W. NOACK (U.S.P. 1,660,094, 28.2.28. Appl., 15.4.26. Ger., 29.5.25).—See B.P. 252,715; B., 1927, 148.

Painting grounds (B.P. 284,363).—See V. **Vulcanised oils** (B.P. 284,415).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Coagulation phenomena in *Hevea* [rubber] latex. V. Alcohol, alum, and sodium chloride. VI. Further observations on β -mixture. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Med. Proefstat. Rubber, Buitenzorg, 1927, [23], 497—526, and [24], 527—536; Arch. Rubbercultuur, 1927, 11, [11]).—V. The coagulant effect of alcohol on *Hevea* latex is probably in the main due to dehydrating influence and not to solvent action on the rubber resins or precipitation of the latex proteins. Undiluted latex (1 vol.) is coagulated rapidly by 96% alcohol ($\frac{1}{2}$ vol.), but with latex previously diluted with an equal volume of water more alcohol is required, and with a dilution of 1:9 coagulation with 96% alcohol is impossible. The additional presence of salts, however, facilitates coagulation by alcohol. Sodium chloride itself can coagulate undiluted latex, but with a dilution of 1:9 it is no longer completely effective, although alum is still active even at much greater dilution of latex. With dilute latex the range of concentrations of alum effecting coagulation is narrow, a zone of stability being gained at higher concentrations similar to that already observed with higher proportions of acids; with sodium chloride it is not possible to attain this higher zone of non-coagulation. Alcohol and alum both effect coalescence in latex which has been diluted with water (1:9), heated, and then flocculated by acid; their effect resembles that of thymol or β -naphthol and is distinct in character from that of coalase.

VI. The treatment involved in the production of β -mixture, viz., heating latex diluted with water (1:9) and acidifying, causes a separation of certain serum solids; these, however, are probably without appreciable influence on the coagulation phenomena and do not accelerate vulcanisation. The coagulum obtained from β -mixture by inoculation with fresh latex yields a distinct amount of "coalase" to alkaline water; this possibility favours the view that the coalescence of β -mixture by fresh latex is due to a catalytically active substance or enzyme. The velocity of coalescence is considerably influenced by mechanical factors such as the depth of the layer of flocks.

D. F. TWISS.

Vulcanisation tests of guayule rubber. D. SPENCE and C. E. BOONE (U.S. Bur. Standards, Tech. Paper No. 353, 1927, 22, 8 pp.).—The guayule shrub and its contained rubber are prone to deterioration after the tree is removed from the ground. When steps are

taken to prevent this depreciation the rubber, after being freed from resins by acetone, gives results comparable with those obtainable with plantation *Hevea* rubber and can to a large proportion replace this without appreciable decrease of the tensile product. The permanent set results, however, are high relative to plantation rubber, but the guayule rubber appears to possess a better dispersing action on compounding ingredients. D. F. TWISS.

Influence of "mineral rubber" on the strength of vulcanised rubber. W. ESCH (Kautschuk, 1928, 4, 31—35).—"Mineral rubber" is a mixture of gilsonite with petroleum-distillation residues; it accelerates slightly the vulcanisation of rubber. With careful selection of an organic accelerator and adjustment of its proportion and of the conditions of vulcanisation, the presence of mineral rubber up to 20% (relative to natural rubber) may result in superior tensile qualities. It also facilitates the operations of mixing and extruding or calendering. D. F. TWISS.

Cream from latex. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Med. Proefstat. Rubber, Buitenzorg, 1927, [21], 371—396; Arch. Rubbercultuur, 1927, 11, [8]).—Ammonia-preserved latex when kept for several months gives a cream containing from 70% to 76% of rubber by wt.; after dilution to 35% rubber content this, unlike ordinary latex, resists coagulation by an equal volume of 96% alcohol, but the further addition of a little sodium chloride causes the mixture to coagulate. Acetic acid effects satisfactory coagulation of the cream only after dilution. Ordinary latex, the viscosity of which has been reduced by dilution or by the addition of a small proportion of aqueous ammonia, can be separated, by centrifuging, into two portions containing 3—5% and 55—60% of rubber, respectively. The concentrate from latex containing ammonia may be kept indefinitely. If purified further by re-dispersion of this cream in water and again centrifuging, the rubber obtained is more plastic, vulcanises slowly, and has a tendency to become sticky. D. F. TWISS.

Tackiness in crude rubber. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Med. Proefstat. Rubber, Buitenzorg, 1927, [25], 557—572; Arch. Rubbercultuur, 1927, 11, [12]).—Rubber which develops tackiness after treatment with mineral alkali gradually becomes sticky, weak, and opaque, and collapses to a doughy mass; tackiness caused by hydrochloric acid or by traces of copper is succeeded by the formation of a brittle resinous coating which gradually deepens until the rubber has been converted into a vitreous mass; tackiness induced by sunlight consists in a weakening of the rubber, which, however, does not proceed further if the exposure to light is discontinued. Rubber from ammoniated latex does not exhibit tackiness of the type induced by sodium hydroxide. The changes involved in the development of tackiness are at present obscure. D. F. TWISS.

Aldol- α -naphthylamine [as an anti-oxidant for rubber]. H. KLOPSTOCK (Kautschuk, 1928, 4, 40).—Artificial ageing in a Geer oven indicates that a sample of aldol- α -naphthylamine of German origin is quite as effective an anti-oxidant as "Age-rite" for vulcanised rubber. D. F. TWISS.

Aldol- α -naphthylamine. W. ESCH (Kautschuk, 1928, 4, 40).—The experimental methods in the preceding work are adversely criticised, and doubt is expressed as to the justification of the conclusion drawn.

D. F. TWISS.

Latex contaminated with copper compounds as a source of danger of fire. J. G. FOL and W. DE VISSER (Bull. Rubber Growers' Assoc., 1928, 10, 125—127).—The acceleration of the oxidation of rubber on latex-treated cloth by traces of copper compounds constitutes a distinct fire-risk which, however, can be avoided by ensuring that the ammonia-preserved latex is kept out of contact with any machinery or utensils made of copper alloys.

D. F. TWISS.

Application of the quartz-lamp in rubber laboratories. F. KIRCHHOF (Kautschuk, 1928, 4, 24—27).—The light from a quartz-mercury lamp, passed through a uviol filter so as to include only the ultra-violet rays, causes a characteristic fluorescence in the visible or invisible spectrum with many of the materials used in the rubber industry, and can be used for their identification or grading. Zinc oxide, lithopone, oils, waxes, starch, and natural resins (including rubber resins) can be usefully examined in this way.

D. F. TWISS.

PATENTS.

Production of rubber mixings. K.D.P., LTD., Assees. of METALBANK & METALLURGISCHE GES. A.-G. (B.P. 262,487, 7.12.26. Ger., 7.12.25).—Compounding ingredients, such as barium sulphate, lithopone, silicic acid, calcium oxalate, and barium carbonate, are incorporated in a very fine state of division in rubber by introducing into rubber latex chemically equivalent amounts of soluble materials adapted to form the desired dispersed solid by precipitation. The vulcanised products have high tearing resistance, and it is possible in this way to obtain white or coloured rubber with mechanical properties equal to those of rubber containing carbon black.

D. F. TWISS.

Colouring of aqueous dispersions of rubber. M. C. TEAGUE, ASSR. to AMER. RUBBER CO. (U.S.P. 1,660,213, 21.2.28. Appl., 31.3.25).—Aqueous dispersions of rubber are mixed with an emulsion formed by dispersing in water a solution of a water-insoluble dye in a suitable solvent, e.g., sulphonated castor oil.

D. F. TWISS.

Direct production of rubber goods from rubber dispersions. DUNLOP RUBBER CO., P. KLEIN, and A. HEALEY (B.P. 284,736, 4.8.26).—By incorporating in rubber dispersions, e.g., latex, a proportion of protective colloids in excess of that normally employed for preserving purposes, the mechanical condition of the deposits obtained by electrophoresis is improved. Soft soap is suitable for the purpose.

D. F. TWISS.

Manufacture of rubber and rubber articles. DUNLOP RUBBER CO., D. F. TWISS, and F. THOMAS (B.P. 284,829, 20.12.26).—Vulcanised rubber scrap is comminuted, sulphur and an anti-oxidant are then introduced evenly, and the mixture is finally heated under pressure.

D. F. TWISS.

Manufacture of rubber articles. DUNLOP RUBBER CO., and G. W. TROBRIDGE (B.P. 285,113, 14.10.26).—

Rubber articles, produced by electrophoretic deposition on an anodic former, after being dried, are heated, with or without vulcanisation, in a suitably engraved or embossed mould.

D. F. TWISS.

Manufacture of sponge rubber. R. J. NOAR (B.P. 284,938, 19.7.27).—Sponge rubber articles are produced by forming on the surface of the shaped raw mass of mixing a vulcanised skin; this can be effected by the external application of a vulcanising agent active at low temperature. The mass is then heated to cause the internal generation of gas and concurrent vulcanisation throughout the mass. The preformed skin confines the swelling mass and aids the retention of shape.

D. F. TWISS.

Buoyant oil-proof india-rubber. E. YOSHIOKA (B.P. 284,912, 19.5.27).—An oil-resistant india-rubber having d 0.9 (approx.) is produced by mixing raw rubber with factice, sulphur, alkaline soap, and diphenylguanidine, the rubber content of the product generally being at least 80%.

D. F. TWISS.

Vulcanisation of rubber. Preparation of a rubber-vulcanisation accelerator. W. SCOTT, ASSR. to RUBBER SERVICE LABORATORIES CO. (U.S.P. 1,656,834 and 1,656,891, 17.1.28. Appl., [A] 6.5.27, [B] 14.12.25).—A mercapto-compound, e.g., mercaptobenzthiazole, is heated with an organic base (1 mol.), e.g., di-*o*-tolylguanidine, and the product is condensed with an aldehyde (1 mol.), e.g., crotonaldehyde, to yield an accelerator.

T. S. WHEELER.

Artificial petroleum etc. from rubber waste (B.P. 282,565).—See II.

XV.—LEATHER; GLUE.

The "mangue" and its tannin. E. SCHIRM (Bol. Soc. Chim. São Paulo, 1928, 1, 2—7).—The leaves of the "mangue" (*Conocarpus racemosus*, Linn., or *Laguncularia racemosa*, Gärtner), which is a tropical shrub growing in salt marshes on the east coast of S. America and the west coast of Africa, yield a tanning liquid on extraction with hot water. The tannin is probably an ellagotannin. In conjunction with other materials, a hot infusion of the leaves is used for tanning leather, lightening the red colour due to mangrove and quebracho extracts; alone it yields a soft leather of greenish-yellow colour. It may replace sumach in chrome-tanning. By leaching the leaves with hot water or bisulphite solution, and evaporating under reduced pressure, extracts are prepared which are completely soluble in hot or cold water, respectively. The natural liquid extract as manufactured contains 50% of solid matter and 28% of tannin. Owing to the presence of salts the solid extract is hygroscopic. The maximum yield is 5—6% of the weight of fresh leaves in winter. Cotton is dyed in a fast khaki shade by treatment with 20% of the liquid extract, followed by 2—2.5% of potassium dichromate and 2—2.5% of copper sulphate in a second bath. "Mangue" can also be used with direct colours and as a mordant. Wool is dyed directly in a similar shade.

R. K. CALLOW.

Fat-liquoring [of leather]. H. B. MERRILL (Ind. Eng. Chem., 1928, 20, 181—2).—Samples of one-bath chrome-tanned calfskin were fat-liquored with a sul-

phoned neatsfoot oil, dried, split into five layers, and the penetration in each split was determined. The results showed that the oil penetrated more from the grain than from the flesh side, and that the middle split contained natural skin-fat only. No change occurred in the distribution of the fat during the drying out. The amount of oil absorbed increased in direct proportion to the quantity present in the fat-liquor, decreased as the fat-liquor was diluted, and increased with the time up to 4 hrs. and was then constant. Alterations in the p_H value of either the liquor or the leather had little effect on the quantity of oil taken up, but increasing p_H value favoured the penetration of the oil into the skin.

D. WOODROFFE.

PATENTS.

Treatment of hides or skins. H. DODGE (U.S.P. 1,659,520, 14.2.28. Appl., 14.5.27).—Puered or bated skins or hides are agitated with a mixture of alum, salt, and water, then formaldehyde and potassium nitrate are added, and the agitation is continued.

D. WOODROFFE.

Preparation of leather. A. M. KINNEY, Assr. to STANDARD OIL Co. (U.S.P. 1,655,868, 10.1.28. Appl., 23.3.25).—A method of fat-liquoring tanned hides comprises immersing them in an aqueous emulsion of a mineral oil containing the sodium salts of the sulphonic acids obtained by treating mineral oils with sulphuric acid, and a sodium rosin soap.

T. S. WHEELER.

Production of glue from casein. G. LANZENDORFER, Assr. to MARSTIN ADHESIVE Co. (U.S.P. 1,659,271, 14.2.28. Appl., 4.12.24).—Casein is mixed with a liquid containing a phenol, alcohol, water, and at least sufficient alkali to neutralise the acidity of the glue, and heated at 60° until a liquid glue is produced.

B. FULLMAN.

Tanning and dyeing of furs and leather. V. ZETTLITZ and A. PFEIFER (B.P. 281,592, 16.12.26. Ger., 30.11.26).—See U.S.P. 1,649,502; B., 1928, 25.

XVI.—AGRICULTURE.

Influence of reaction on the action of ammonium salts on growth of *Zea mays*. W. MEVIUS (Z. Pflanz. Ding., 1928, 10A, 208—218).—The toxic action of ammonium salts on maize grown in water culture is attributed not to the physiological acidity of these salts, but rather to the harmful influence of the ammonium radicle.

H. J. G. HINES.

Influence of various potash salts on the yield and starch content of potatoes. O. NOLTE and R. LEONHARDT (Mitt. Deut. Landw.-Ges., 1927, 689; Bied. Zentr., 1928, 57, 68—69).—Confirming earlier observations that potassium salts other than sulphates tend to reduce the starch content of potatoes if applied late in the season, "Patent Kali" (mixed sulphates of potassium and magnesium) produced a higher yield of tubers of higher starch content than "40% potash salts" in field trials.

A. G. POLLARD.

Influence of nitrate concentration on the development of barley and the absorption of nitrate from nutrient solution. M. S. MILLER (Mem. Leningrad Agric. Inst., 1926, 3, 57—114).—The absorption of nitrate by barley is largely influenced by the reaction.

Absorption is maximal at the time of heading and blooming, decreasing during ripening and at the end of the vegetation period. The concentration of nitrate indirectly influences the transpiration of moisture.

CHEMICAL ABSTRACTS.

Effect of aluminium sulphate on rhododendrons and other acid-soil plants. F. V. COVILLE (Smithsonian Rep., 1926, 369—382).—Rhododendron, *Franklinia*, and *Vaccinium* plants which were stagnant on rich soil were stimulated to normal growth by treatment with aluminium sulphate solution. Hydrangea flowers were pink when grown on neutral, and blue on treated, soil.

CHEMICAL ABSTRACTS.

Efficacy of mixtures of natural and soluble phosphates, as measured by a bacteriological method, and the effect on the higher plants. G. TRUFFAUT and N. BEZSSONOFF (Compt. rend., 1928, 186, 522—524).—The amount of available phosphate in a mixture of natural and soluble phosphates can be measured by the growth of nitrogen bacteria in a suitable medium containing a known weight of the phosphates. Using this method, verified by experiments on potatoes, maize, etc., it is found that the addition of up to 66% of soluble phosphate to natural phosphate increases its assimilability, but that further addition is useless and even harmful.

B. W. ANDERSON.

Important nitrogen losses during the fermentation and humification of highly nitrogenous plants.

I. Lucerne. J. ZOLCINSKI. **II. Red clover.** J. ZOLCINSKI and A. MUSIEROWICZ (Rocz. Nauk Poln. i Lesnych, 1927, 17, 349—377, 377—396; Bied. Zentr., 1928, 57, 64—67).—I. During the humification of lucerne (in flower) as much as 40% of its total nitrogen is lost in 10 days and 60% in 100 days. Of this loss, approximately one half is as ammonia and the balance as elementary nitrogen. The loss is minimised by leaving undisturbed and avoiding destruction of the covering layer of fungus mycelium. The considerable losses of nitrogen may to a large extent be attributed to physico-chemical processes. It is suggested that plants of moderate nitrogen content build up protein matter differing in constitution from that of legumes, and characterised by the presence of $\cdot NH_2$ and $\cdot OH$ groups in the *meta*-position (*i.e.*, the least oxidisable position), and contain also more aliphatic groupings which are quite unoxidisable. There is practically no loss of nitrogen during the humification of plant tissue of moderate nitrogen content.

II. Red clover loses 28% of its total nitrogen during humification, largely as free nitrogen. The formation of nitrite or nitrate during the process was not observed. The carbon:nitrogen ratio of the fermenting mass approaches that of soil humus. The total phosphorus in the humified material does not vary. Temperature and time govern the extent of the humification process. The combination between chalk and humic acids is accelerated by a rise of temperature. Leguminous plants serve as an excellent source of humus substances. Sufficient lime supplies are necessary to complete the humification process, which is very largely a physical and chemical as well as a biological one.

A. G. POLLARD.

Relationship between the sodium chloride content of soils and plant growth. TACKE (Abhandl. des Naturwiss. Vereins, 1927, 26, 503—527; Bied. Zentr., 1928, 57, 56—57).—The sodium chloride content of subsoils is more constant than that of surface soils liable to flooding with saline river water. Variations in plant growth are more closely correlated with the salt content of subsoils than of surface soils. In general, the salt content and natural flora of soils are closely related, and variations in the former bring about periodic changes in the nature of the latter. A. G. POLLARD.

Lime, phosphate, and nitrogen requirements of arable soils. F. ROSSBACH (Forts. Landw., 1927, 2, 450; Bied. Zentr., 1928, 57, 57—59).—Methods for the determination of nutrient requirements of soils are compared. Extracts of soils made with 10% hydrochloric acid indicate the total as distinct from assimilable nutrient contents; but the ratio of (Fe, Al, Ca, Mg): PO_4 is indicative of the relative solubility of the soil phosphate. A 1% citric acid extract of soil gives a measure of the root-soluble nutrients. In the case of phosphates the fertiliser requirement is measured by the ratio of this value to the "total" phosphate value above. The Neubauer process, in spite of careful management, fails to determine the root-soluble phosphate in soil. Satisfactory values for potash are obtained in all but extreme and abnormal types of soils. With further development of the method promising results for the nitrogen requirement of soils are indicated. A. G. POLLARD.

Manurial action of calcium silicate. E. BLANCK, F. GIESECKE, and H. KEESE (J. Landw., 1928, 75, 325—336).—Silicates were without injurious action on growing plants. In fertiliser trials potassium silicate adequately replaced potassium sulphate, and calcium silicate replaced calcium sulphate, lime, or chalk without markedly affecting crop yields. Soluble silica in the form of potassium silicate had no specific effect on plant growth. A. G. POLLARD.

Potash fertiliser action of a mica waste. E. BLANCK, F. GIESECKE, and H. KEESE (J. Landw., 1928, 75, 337—342).—Waste material from the preparation of lithium contained potash as a relatively insoluble silicate. Although exhibiting a definite fertiliser effect, its efficiency was very small compared with soluble potassium silicate. A. G. POLLARD.

Absorption of aluminium and iron by soil. N. D. PRJANISCHNIKOV, JUN., and E. K. LUKOVNIKOV (Z. Pflanz. Düng., 1928, 10A, 232—237).—By determination of the amount of exchangeable calcium replaced from a podsol-loam by solutions of hydrochloric acid, aluminium chloride, and ferric chloride it is found that the adsorption-energy value, e (Gedroitz), for hydrogen ion is not constant but increases with increasing p_{H} . The values of e for aluminium and iron will form the subject of a further communication. H. J. G. HINES.

Variation of phosphorus content in South African vegetation. J. P. VAN ZIJL (J. S. African Chem. Inst., 1928, 11, 3—11).—The phosphoric acid content of South African grass decreases rapidly with age, and is greater in spring than in summer. A table is given showing the variation in the phosphoric acid

content of several samples of grass of different ages and at the various seasons of the year, as well as the variations in the different parts of the same plant. A. R. POWELL.

The Neubauer method [for determining root-soluble nutrients in soils]. F. MAJEWSKI (Polish Agric. and Forest Ann., 1927, 17, 35; Bied. Zentr., 1928, 57, 60—61).—The Neubauer method does not compare favourably with that of Lemmermann (relative solubility) or with field trials, results obtained being, in general, the same as those arising from direct chemical analysis. Wheat is a more suitable plant than rye for use with the seedling method as it is more sensitive to changes in potash and phosphate concentrations in the soil. A. G. POLLARD.

Determination of nutrient content of a soil by pot experiment. H. WIESSMANN (Z. Pflanz. Düng., 1928, 10A, 206—208).—Pots containing 1500 g. of soil mixed with 5700 g. of glass sand are given a full manuring and are planted with oats. Pots with 7200 g. of sand alone are treated similarly, and by comparison of the two yields a correction is obtained for the physical influence of the soil. By omitting phosphoric acid from the full manuring it is possible to calculate the amount of assimilable phosphate originally present in the soil. H. J. G. HINES.

Determination of soil reaction by means of Merck's universal indicator. H. WIESSMANN and K. STEINFATT (Forts. Landw., 1927, 2, 488; Bied. Zentr., 1928, 57, 49—50).—Values of p_{H} obtained by the Merck universal indicator agree closely with those determined by Sørensen buffer solutions and by electrometric methods, although the limits of differentiation are much narrower in the latter processes. A. G. POLLARD.

Determination of the phosphate requirement of soils. E. BLANCK (J. Landw., 1928, 75, 343—352).—Comparison is made of methods for determining phosphate requirements of soils involving the "relative solubility" of soil phosphates, the Neubauer seedling method, and field trials. The first-named process yields results most nearly in agreement with plant-culture experiments, the values being more regular, definite, and more quickly obtained than those by the Neubauer method. A. G. POLLARD.

Determination of reaction of peat soils. T. ARND and W. HOFFMANN (Z. Pflanz. Düng., 1928, 10A, 219—232).—A critical examination of the methods in use for the determination of the p_{H} of mineral soils showed that they could be applied to peat soils with but little modification. A method of procedure to be adopted with the quinhydrone electrode is recommended, which can be used either with water or potassium chloride suspensions. H. J. G. HINES.

Deterioration of strophanthus seed. PICKERING.—See XX.

PATENTS.

Drying of agricultural produce. H. WADE. From A. J. MASON (B.P. 283,014, 9.3.27).—The crop in the form of a mat is passed on a perforated conveyor through a long chamber or drying oven, in which the space on

the upper side of the mat is divided into two compartments. Drying gases heated to about 150° pass downwards from one compartment through the crop mat at a point near the outlet end of the chamber where the mat is in a highly dried condition, thence for a substantial distance along the chamber in a direction opposite to that of the movement of the mat, and finally upwards through the moist portion of the mat at the inlet end of the chamber to the second compartment, whence they escape to the atmosphere. The dried crop mat is then thoroughly disintegrated mechanically.

F. R. ENNOS.

Production of manure from spent molasses waste. E. BAUER and L. SZOLD (EISLER & SZOLD) (B.P. 280,219, 3.11.27. Austr., 5.11.26).—Spent molasses waste is concentrated to $d_{1.26}^{20} = 1.32$, cooled below 60°, and intimately mixed with aqueous lime sludge or with carbonated lime. The pasty mass is dried at 100–105° to a moisture content of 7–8%, and broken up to a fine powder which is non-hygroscopic and should contain about 30% of lime.

F. R. ENNOS.

Manufacture of chemical manures. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., and H. BRENEK (B.P. 265,197, 25.1.27. Ger., 1.2.26. Addn. to B.P. 242,512; B., 1926, 71).—The process of heating mineral phosphates, silicates, and alkali carbonates or alkali-containing rocks, *e.g.*, leucite or phonolite, is carried out in the presence of steam, a reducing substance, *e.g.*, coal, being added if the phosphate contains alkaline-earth sulphates. Fuels rich in hydrogen may be used to supply the steam.

W. G. CAREY.

Manufacture of manure. E. W. M. HAMMEL (B.P. 284,741, 27.9.26).—Substances (*e.g.*, bone meal) containing or capable of yielding albuminoids or peptones by the action of an inorganic acid other than sulphuric acid are treated with hydrochloric acid, a potassium fertiliser is added, the whole concentrated to a rubber-like consistency, neutralised with ammonia, and dried at 100–120° *in vacuo*.

H. ROYAL-DAWSON.

Manufacture of fertiliser material. K. R. LINDFORS, Assr. to MICHIGAN SUGAR Co. (U.S.P. 1,657,128, 24.1.28. Appl., 12.2.26).—Hot dehydrated peat is mixed with hot distillery waste-liquor, and the product is kept for 48 hrs. and then mixed with phosphate rock.

T. S. WHEELER.

Insecticide. R. P. SOULE, Assr. to COMBUSTION UTILITIES CORP. (U.S.P. 1,655,573, 10.1.28. Appl., 23.6.24).—The use of the heterocyclic nitrogen bases present in low-temperature tar is claimed.

T. S. WHEELER.

Copper sulphide as insecticide (U.S.P. 1,657,430).—See VII. **Reduction of ores** (B.P. 274,803).—See X.

XVII.—SUGARS; STARCHES; GUMS.

Clarification of juice in the cane sugar industry [in white sugar manufacture in Java; presence of glucose in cane molasses]. H. I. WATERMAN (Chim. et Ind., 1927, 18, 758–767).—De Haan's carbonatation process, in which lime and carbon dioxide are added simultaneously to the juice at 55°, shows an economy in lime amounting to 40% compared with the ordinary procedure, besides which filtration is

more easily effected and less sugar is left in the press-cakes. Similarly, in Harloff's hot-sulphitation process the juice at 70–80° is treated simultaneously with lime and sulphur dioxide, after which it is heated to 100° and allowed to subside without filtration. Glucose has been determined in the molasses from different countries by measuring the reducing power of the residue left after fermentation; Javan molasses (sulphitation) gave 6.2%; Cuban (defecation) 9.9%; and Egyptian (sulphitation) 6.1%.

J. P. OGILVIE.

Relation between alkalinity and electrical conductivity of carbonatation juices. V. STANĚK and K. SANDERA (Z. Zuckerind. Czechoslov., 1927, 52, 209–213).—Determinations of the electrical conductivity carried out during the operation of carbonatation lead to the conclusion that the former is in direct proportion to the latter, and that the effect of the varying composition of the beet, or of the diffusion water, does not generally exceed the experimental error of 0.01–0.02% CaO. In principle, therefore, it should be possible to use electrical conductivity as a means of measuring the termination of the first carbonatation.

J. P. OGILVIE.

Influence of over-saturation [in the carbonatation process] on the precipitation of some acids in presence of magnesia and alkalis. R. KARGL (Z. Zuckerind. Czechoslov., 1928, 52, 253–259).—Precipitation of the vegetable acids present in beet juices during carbonatation after having added lime containing magnesia depends on several factors, but especially on the alkalinity of the carbonated juice. The presence of alkalis has an adverse effect, lowering the efficiency of the clarification and causing over-saturation. Magnesia exerts no influence so long as its amount does not exceed that of the lime used.

J. P. OGILVIE.

PATENTS.

Preliminary treatment of raw sugar, after-product sugar, etc. before refining. RAFFINERIE TIRLEMONTAISE SOC. ANON. (B.P. 276,611, 2.11.26. Ger., 26.8.26).—The sugars are placed in store rooms the temperature and moisture content of which are so controlled that the syrup surrounding the sugar crystals attains and maintains a low limit of purity, *viz.*, 60 pts. of sugar to 100 pts. of total solids, below which it is not economical to attempt further extraction of sugar from the syrup. The whole is then centrifuged at a force 1600–6000 times the weight of the mass treated, and the separated sugar crystals are passed to the refinery whilst the syrup is used as molasses.

F. R. ENNOS.

Manufacture of grape sugar [dextrose]. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 1,658,998, 14.2.28. Appl., 6.8.24).—A starch-converted dextrose solution is crystallised to flocculate crystalloidal impurities. The crystallised material is melted, and recrystallised with agitation.

B. FULLMAN.

Manufacture of laundry starch. L. O. GILL, Assr. to A. E. STALEY MANUF. Co. (U.S.P. 1,656,190, 17.1.28. Appl., 25.4.23).—A starch solution which remains fluid at room temperature is obtained by mixing in solution two starches, one of which has been more

vigorously treated with sulphuric acid than the other, but neither of which has been entirely converted.

T. S. WHEELER.

Crystallisation mixer for treating masecucites in sugar works etc. L. F. GARDNER. From SCHNEIDER & Cie. (B.P. 284,954, 8.9.27).

Centrifugal apparatus (B.P. 280,167).—See I. **Manure from molasses waste** (B.P. 280,219).—See XVI.

XVIII.—FERMENTATION INDUSTRIES.

Production of *n*-butyl alcohol, acetone, and methyl alcohol from maize. H. TROPSCH (Brennstoff-Chem., 1928, 9, 1—2).—In two American works a total of 635 tons of inferior maize is daily converted into about 55 tons of *n*-butyl alcohol, 27 tons of acetone, and 9 tons of ethyl alcohol, whilst at one of the works 13 tons of methyl alcohol are simultaneously synthesised from the fermentation gases. The maize, containing about 8% of starch, is mixed with water at 71°, sterilised with steam under pressure, and fermented by means of the micro-organism *Clostridium acetobutylicum*. The fermentation requires about 2½ days, and its progress is followed by determining the acidity of the mixture and the gas evolution. The mixture of alcohols and acetone is concentrated and fractionally distilled. The mixture of carbon dioxide (60%) and hydrogen (40%) liberated during fermentation is washed with water under pressure, to bring the proportion to 25% and 75%, respectively, and is then compressed to 300 atm., and passed over a catalyst comprising 97—75% of zinc (or magnesium) oxide with 3—25% of iron hydroxide. From the resultant mixture of 68% of methyl alcohol and 32% of water pure methyl alcohol is obtained by rectification. W. T. K. BRAUNHOLTZ.

Presence of glycuronic acid in wines made from diseased or rot-infested grapes. D. CHOUCHEK (Compt. rend., 1928, 186, 520—522).—Considerable amounts of glycuronic acid are present in musts and wines from rot- and mould-infested grapes. The acid may be isolated by means of its osazone or by precipitation with ammoniacal lead subacetate. Musts and wines from healthy fruit contain only traces of this acid, and thus the amount detected in a wine gives some indication of the care with which the grapes have been selected. Glycuronic acid in a wine falsifies the determination of dextrose and of several organic acids.

B. W. ANDERSON.

Hemicellulose-splitting enzyme (cytase) of malts. H. LÜERS and W. VOLKAMER (Woch. Brau., 1928, 45, 83—87, 95—99).—The action of aqueous extract of green malt upon xylan prepared from elder pith by the methods of Salkowski and E. Schmidt are studied. The xylan was dissolved in alkali and dialysed till neutral. The temperature and acidity optima are 45° and p_H 5.0, respectively. The rate of action increases with increasing amounts of enzyme, but not in direct proportion, the xylose produced having an inhibiting effect. Under the most favourable conditions about 70—75% of the xylan is hydrolysed in 48 hrs. The enzyme is destroyed by heating in solution for 15 min. at 60°,

but not in presence of xylan. The xylose was identified by its rotatory power and osazone, and was also obtained from xylan prepared from barley. Xylose, but not xylan, was found to be present in finished malt. It was found possible to concentrate the enzyme by adsorption on alumina (optimum p_H 5.0) and removal by phosphate solution (p_H 8.3). Since by twice repeating this procedure the cytase strength of a preparation was increased 21 times, while the diastatic power of the same preparation only increased sixfold, the authors conclude that the two enzymes are distinct.

F. E. DAY.

Extension of malt analysis by a flavouring-test. H. KROFF (Woch. Brau., 1928, 45, 87—88).—If 200 c.c. of the laboratory extract are carefully evaporated to 30 c.c., and a drop of the syrup is applied by a glass rod to the tongue, marked differences of flavour will be noted. The flavour is much affected by malting conditions; e.g., a bitter flavour results from early and slow withering of the rootlets due to excessive aeration. To obtain the best malt, which under the conditions of the test gives a honey-like flavour, the rootlets should be unwithered at the completion of modification, and kilning should be sufficiently rapid to dry the rootlets before their fluids can be reabsorbed by the grain. For this the author considers two 12-hr. periods on a two-floor kiln as suitable.

F. E. DAY.

Yeast as fodder. STICH. **Fermentation of bread.** ELION.—See XIX.

PATENT.

Apparatus for determining the alcohol in a liquid. B. STEIN (U.S.P. 1,658,950, 14.2.28. Appl., 15.4.27).—The apparatus consists of a water-bath (with thermometer), into the air space of which extends a receptacle for the liquid under test, carrying a thermometer and condenser. The percentage of alcohol may be determined when the columns of both thermometers become stationary.

B. FULLMAN.

XIX.—FOODS.

Wheat and flour studies. XII. Factors influencing the viscosity of flour-water suspensions. II. Effect of hydrogen-ion concentration during extraction or digestion period. A. H. JOHNSON and B. L. HERRINGTON (Cereal Chem., 1928, 5, 14—44).—Using the procedure of Gortner, maximum viscosities were obtained by extracting at p_H 4.34—5.07, when the extraction was carried out at 25°. With extraction at 40°, the maximum viscosity was obtained at p_H 4.90—5.44. The proportion of the total nitrogen extracted was about 50% in the case of patent and first-clear flours, and about 35% in the case of second-clear. E.g., a patent flour with distilled water at 25° gave an extract of p_H 5.75, viscosity 155° (MacMichael), and 31.9% of the total nitrogen was extracted. The maximum viscosity (260° MacMichael) was obtained at p_H 4.77, 51.5% of the nitrogen being extracted. The corresponding second-clear flour with distilled water gave p_H 6.20, viscosity 76° (MacMichael), and total nitrogen extracted 37.4%; at p_H 4.39 the values obtained were 188° (MacMichael) and 32.4%. Comparison of flour before and after bleaching with chlorine showed that

the same maximum viscosity was obtained. The increased viscosity observed in water-extracted, bleached flours is not due to oxidation of protein. The decrease of viscosity on extracting at successively lower p_H values frequently showed a marked irregularity at about p_H 8, attributed to the increase of water-imbibing capacity of the protein before dissolution by the hydroxyl ions more than balancing the effect of decreasing extraction of electrolytes. The extraction of electrolytes was separately investigated, using a resistivity method, and it is suggested that an important factor is the liberation of phosphates by phytase. The very much lower viscosities given by flours digested but not extracted at various p_H values may be due in part to this. The results of such experiments were parallel to those with extracted flours. Small quantities of salt depressed the viscosity. From the values observed for the solubility of the proteins, the isoelectric points for those of patent, first-clear, and durum flours at 25° are between p_H 6.2 and 6.5, and for second-clear between p_H 5.5 and 6.0. At 40° the isoelectric points are slightly more to the alkaline side.

F. E. DAY.

Wheat and flour studies. XIII. Relation between the fusibility of flour ash and its mineral constituents. A. H. JOHNSON and S. G. SCOTT (Cereal Chem., 1928, 5, 56—64).—Flour ashes, burned off at 585° for 16 hrs., were classified as fluffy, partially fused, and completely fused. The three classes showed little difference in lime or magnesia content, but the fluffy ash contained about 33% K_2O and 48% P_2O_5 , and the completely fused ash about 26% K_2O and 54.5% P_2O_5 , the partially fused ash being intermediate. Three flours giving the three types of ash, when incinerated for 16 hrs. at various temperatures, gave substantially the same ash contents at all temperatures from 485° to 700°, though at 485° the fusible ash was black and fluffy. At about 700° the normally fluffy ash fused, and at high temperatures all three lost weight slightly. The phosphorus in flour ash incinerated at 585° exists mostly as pyrophosphate; in fluffy ashes a small part is present as orthophosphate, in fused ashes a little metaphosphate is present.

F. E. DAY.

Refractive indices of aqueous and alcoholic extracts of flour. D. W. KENT-JONES and A. J. AMOS (Cereal Chem., 1928, 5, 45—56).—No relation was found between the refractive index of an aqueous flour extract and its soluble extract, gassing power, or ash content, nor between the protein precipitated and the change of refractive index on heating such an extract. The increase of refractive index over that of the solvent is chiefly due to gliadin in 70% ethyl alcohol extracts, and though other substances have a slight influence on the refractive index, this has no relation to the gassing quality of the flour. The refractive index of methyl alcohol extracts appears to give no useful information, and is but slightly affected by the glutenin. The equation to the concentration-refractive index curve for gliadin in 70% alcohol at 20° is $\mu_s = 1.3634 + 0.0018C$, where μ_s is the refractive index of gliadin solution at 20° and C is the concentration of gliadin in g./100 c.c.

F. E. DAY.

Viscosity of flour suspensions. G. VAN DER LEE (Cereal Chem., 1928, 5, 10—13).—A theoretical discussion of the inapplicability of Einstein's equation for the viscosity of colloidal solutions to the viscosity of flour suspensions as measured in the MacMichael viscosimeter.

F. E. DAY.

Aid in the determination of flour strength. F. L. ENGLEADOW (Cereal Chem., 1928, 5, 1—9).—Flat portions of dough, 2.5 in. in diam. and 0.2 in. thick, prepared and rolled under standard conditions from the unknown and a standard flour, are lightly clamped over the open ends of two tubes, of 0.8 in. in diam. The tubes communicate with a vessel from which the air can be exhausted. As this occurs, the discs of dough become distended inwards, and finally the weaker bursts. By repeating the observations it is possible to observe fine differences of strength. Thus, in mixtures of Yeoman and Little Joss flours, 10% variations in the proportions could readily be distinguished. The method, though only comparative and not universally applicable, gave results which compared well with the baking tests, and requires a much smaller sample.

F. E. DAY.

Relation of water-absorbing capacity of flour to protein content, baking quality, and loaf-weight. C. E. MANGELS (Cereal Chem., 1928, 5, 75—77).—The correlation coefficients between water-absorbing capacity and protein content, loaf volume, and loaf weight are given for the years 1923—1926, calculated on the average of the flours from 200—300 samples of hard red spring wheat milled experimentally at the North Dakota Experimental Station. As regards protein and loaf volume, the correlation with water absorption is low, but a coefficient of correlation above 0.50 is found between water absorption and loaf weight for each year studied.

F. E. DAY.

Improvements in the fermentation of bread produced by (the addition of) yeast nutrients. L. ELION (Z. angew. Chem., 1928, 41, 230—231).—The fermentation of dough, as measured by the amount of carbon dioxide evolved, is increased by the addition to the mixing of yeast (1 g.) and flour (50 g.) of small amounts (0.1 g.) of yeast nutrients such as carbamide, peptone, asparagine, and ammonium chloride, tartrate, lactate, or phosphate. Ammonium phosphate gave the most marked increase, pronounced increase being also observed with the lactate. When the proportion of yeast in the mixing was increased to 3 g., the increase in fermentation produced by addition of the nutrients was less pronounced, and a deficiency of sugar was observed. With yeast extract in place of ammonium phosphate fermentation was increased, but sugar deficiency occurred even with 1 g. of yeast, although in the absence of the extract sufficient sugar was always present. Addition of "super-rapidase" produced a remarkable increase in fermentation, and this product appears to act as a sugar producer, by virtue of its diastatic properties, in addition to functioning as a yeast nutrient.

R. BRIGHTMAN.

Determination of chlorine in bleached flour. A. SEIDENBERG (J. Assoc. Off. Agric. Chem., 1928, 11, 132—135).—The flour was thoroughly extracted by

the ordered addition of definite quantities first of 70% and then of 95% alcohol by vol. followed by ethyl ether and light petroleum to avoid the formation of emulsions. The mixed solvents, after separation from the flour, were washed with water, evaporated with alcoholic soda prepared from chlorine-free sodium, and the residue was charred and extracted with dilute nitric acid. The chloride was then determined in the acid solution by means of potassium thiocyanate. Duplicate determinations agreed within 6—7 pts. per million. In all cases the amount of chlorine found was less than that used in bleaching.

F. R. ENNOS.

Determination of field corn in canned mixtures of field and sweet corn. J. L. HEID (J. Assoc. Off. Agric. Chem., 1928, 11, 136—138).—After removal of the liquor and debris by flotation, the kernels are hardened with alcohol and a representative sample of about 100 kernel fragments is taken for examination. The kernels are then cut through, and a portion removed from the centre of each is tested separately with iodine stain on a white tile. A dense brown cloud disseminates from the sweet corn which contains dextrin, whilst the field corn gives the blue-black starch-iodide colour, the proportion of each kind of corn being determined by counting the number of dextrin and non-dextrin containing particles.

F. R. ENNOS.

Feeding trials with milch cows using ammonium acetate as a substitute for protein foods in agricultural practice. H. BAREISS (J. Landw., 1928, 75, 265—324).—In agreement with the work of Pasch it was found possible to replace 25% of the digestible protein in a ration with ammonium acetate with no ill-effect on the cow. Normal live-weight increases in the animals occurred. There was a slight decrease in milk yield, but corresponding increase in the fat content. The utilisation of ammonium acetate by the animal takes place in conjunction with the amides present in the other food material.

A. G. POLLARD.

Large-scale production of yeast as fodder. E. G. STICH (Chem.-Ztg., 1928, 52, 149—150, 170—171).—The possibility of producing yeast economically from molasses as a substitute for other imported food-stuffs is discussed. In order that the price (calculated on the protein content) should be sufficiently low, 100 kg. of sugar must yield 76—80 kg. of dry yeast containing 45% of digestible protein, and this yield is obtainable only by sufficient aeration during the fermentation process. Tables showing the power requirements for this aeration and the costs of production are given, and several alternative factory processes are outlined.

W. J. POWELL.

Volumetric method for determination of proteins in milk. [Mrs.] A. JONESCO-MATIU and C. V. BORDEIANU (Bul. Soc. Chim. România, 1927, 9, 76—85).—A rapid method for the determination of proteins in milk by precipitation in a graduated tube with a 5% solution of mercuric chloride in acetone and measurement of the volume of the precipitate is described. To the mercuric chloride solution (7.5 c.c.) 2.5 c.c. of milk are added drop-wise from a fine pipette, the solution

is well shaken to dissolve out all fats from the precipitate and ultimately left to settle for 24 hrs. at 18°. The volume of the precipitate multiplied by a factor (which varies slightly from tube to tube, being 1.02 in the apparatus used) gives directly the number of grams of protein in 100 c.c. of the sample. The value so obtained never differs from that determined by the Kjeldahl method by more than 2%. The value is also exact for diluted milk containing up to 30% of added water, and when the volume of precipitated protein is less than 2.8 c.c. fraudulent dilution may be assumed. By simultaneous determinations of the density, fat content, and protein content of milk samples it is possible to detect the fraudulent dilution of milk, separation of the cream, or cream separation and dilution combined.

J. W. BAKER.

The sand-Gooch method for analysis of butter. L. C. MITCHELL (J. Assoc. Off. Agric. Chem., 1928, 11, 138; cf. B., 1926, 605).—90-Mesh crystalline alumina (R.R. Alundum, Norton Co., Worcester, Mass., blue label) forms a satisfactory and easily obtainable substitute for the white river sand originally used in this method.

F. R. ENNOS.

Cause of "fishiness" in dairy products. W. L. DAVIES and A. T. R. MATTICK (Nature, 1928, 121, 324).—Butter-fat olein easily absorbs oxygen yielding a labile peroxide, the reaction being strongly catalysed by salts of heavy metals, especially copper. The nitrogenous base portion of lecithin is thereupon oxidised with the production of volatile bases (chiefly trimethylamine) and their fatty acid salts. Appreciable amounts of copper were present in all fishy dairy products. In the absence of metallic contamination the absorption of oxygen and development of rancidity would be slow.

A. A. ELDRIDGE.

Detection and determination of added moisture in sausage. P. A. SIGLER (J. Assoc. Off. Agric. Chem., 1928, 11, 112—125).—The water content of the various kinds of meat used in sausage when calculated on the fat- and ash-free material is nearly constant at about 79%. The ratio of protein (nitrogen content $\times 6.25$) to that of water, calculated on the original meat, is approximately 1 : 4. The percentage of added moisture in sausage may consequently be determined with sufficient accuracy for practical purposes by deducting from the total moisture the quantity of water naturally derived from the ingredient meats, as found by multiplying the protein content by 4.0.

F. R. ENNOS.

PATENTS.

Production of a food product from vegetables containing starch. L. E. JONES, Assee. of H. M. HEIMERDINGER (B.P. 260,277, 21.10.26. U.S., 21.10.25).—Whole vegetables, e.g., potatoes, are boiled with water until the skins are in a filmy condition, and the cooked pulp containing all the nutrient materials is freed from the skins etc. by pressing with a continuous supply of water through perforations in a rigid wall. The mixture of pulp and water is then dried by spraying it in and across a current of air heated at 260—315°, thus yielding

white granules containing 5—8% of water and a high proportion of yeast food substances. F. R. ENNOS.

Production of bread and the like baked goods. L. E. JONES, Assee. of H. M. HEIMERDINGER (B.P. 260,278, 21.10.26. U.S., 21.10.25).—Cereal flour is mixed with the flour-like product obtained as in B.P. 260,277 (preceding) in proportions varying from 0.5—35 pts. of the latter to 100 pts. of the former, and the resulting mixture is leavened, converted into dough, and baked.

F. R. ENNOS.

Manufacture of an edible fat. H. A. NEWTON (B.P. 284,368, 25.9.26).—A vegetable fat is heated with finely-divided onions.

F. R. ENNOS.

Edible fat composition. H. E. DUBIN, Assr. to H. A. METZ LABORATORIES, INC. (U.S.P. 1,656,474, 17.1.28. Appl., 10.10.25).—The composition described in U.S.P. 1,624,164 (B., 1927, 530) is modified by the addition of a fat-soluble vitamin concentrate. T. S. WHEELER.

Separation of liquids. J. ALSBERG, Assr. to B. HELLER & Co. (U.S.P. 1,656,716, 17.1.28. Appl., 31.10.25).—Oily constituents of value as food flavours are separated from the distillate obtained by heating maple or birch wood by saturating it with sodium chloride.

T. S. WHEELER.

Manufacture of an easily-soluble cocoa powder. H. BOLLMANN (U.S.P. 1,660,541, 28.2.28. Appl., 15.12.25. Ger., 30.10.25).—See B.P. 262,239; B., 1927, 171.

Method and apparatus for tempering chocolate. NAT. EQUIPMENT Co., Assees. of A. L. BAUSMAN (B.P. 271,066, 4.5.27. U.S., 12.5.26).

Manufacture of a food composition [from albumin and sugar]. A. LEO (B.P. 282,923, 18.10.26).

Continuous production of butter. C. G. T. SALENUS (B.P. 284,423, 19.11.26).

Treatment of fresh fruits and vegetables [by spraying]. H. G. ZELLNER (B.P. 284,408, 29.10.26).

Cleaning of milk-stained metal (B.P. 284,778).—See X.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Assay of a so-called cod-liver oil extract for vitamin-A content and calcifying properties compared to cod-liver oil. H. E. MUNSELL and H. BLACK (J. Amer. Pharm. Assoc., 1928, 17, 139—144).—A commercial product described as an alcohol-soluble extract of cod-liver oil has been tested biologically for its content of vitamin-A and -D. It is indicated that 100 mg. of the extract contain not more than one fifth as much vitamin-A as is present in 1 mg. of cod-liver oil, and that the vitamin-D content is not greater than that of cod-liver oil.

E. H. SHARPLES.

Properties and technical application of camphor oils. W. VAUBEL and N. NEDELSCHIEFF (Farben-Ztg., 1928, 33, 1406—1409).—The physical and chemical constants of three commercial light camphor oils have been determined and are compared with those of turpentine oil. The limiting values for the three oils and

those for turpentine oil are d^{18} 0.8589—0.8756, 0.8630, n_D^{20} 1.4672—1.4788, 1.4700, α —63.25° to —80.4°, +36.45°, boiling range 165—184°, 157—170°, evaporation residue 1.09—2.6, 1.66, bromine value 99.91—160.93, 231.6, flash point 41—43°, 33°, time of evaporation at 20—23°, 10—12 hrs., 10—11 hrs., acid value 0.26—1.6, —. Colour reactions and the drying periods of mixtures of the oils with both linseed oil and varnish show that the camphor oils are quite suitable for use as thinners and solvents in the paint and varnish industry. They have a pleasant odour, and are free from dangerous physiological action after application.

E. H. SHARPLES.

Absolute essence of *Salvia sclarea*. Y. VOLMAR and A. JERMSTAD (Compt. rend., 1928, 186, 517—519).—From the absolute essence of *S. sclarea*, m.p. 35—36°, n_D^{20} 1.5038, d 0.9826, α_D^{20} +4° 12' (alcoholic solution) were isolated 42% of a crystalline substance *sclareol*, 28% of a sesquiterpene alcohol, $C_{15}H_{26}O$, b.p. 169—178°/1 mm., d^{15} 0.9841, n_D^{20} 1.5138, α_D^{20} +24° 47', 21.8% of volatile constituents (chiefly linalyl acetate and linalool with traces of an unsaturated ester, $C_7H_{12}O_2$), and a small amount of acetic acid and of a free unsaturated acid.

B. W. ANDERSON.

Causes of deterioration of strophanthus seed during storage. E. PICKERING (J. Amer. Pharm. Assoc., 1928, 17, 121—123).—An examination of the causes of deterioration of strophanthus seeds suggested by published work. The seeds should be protected from light, moisture, heat, and inoculation with mould spores.

E. H. SHARPLES.

Standardisation and stabilisation of *Nux vomica*, *Gelsemium*, and *Veratrum*, and the hydrogen-ion concentration factor. IV. E. E. SWANSON and C. C. HARGREAVES (J. Amer. Pharm. Assoc., 1928, 17, 23—27).—Fluid extract of *Nux vomica* suffers no loss in activity, either chemical or physiological, over a period of 10 years, and its therapeutic value is not affected by changes in hydrogen-ion concentration. Chemical and physiological examination of eleven samples of fluid extract of *Gelsemium* prepared in the years 1917—1927 indicated varying degrees of activity and also showed that the two methods do not correlate, the chemical method being unreliable. Changes in hydrogen-ion concentration do not influence the stability of this extract. The chemical method for the assay of fluid extract of *Veratrum* is also unreliable, and the biological method should be employed. This extract seems to require a definite hydrogen-ion concentration to control its deterioration and stabilisation.

E. H. SHARPLES.

Synthetic drug analysis. IX. **Determination of acetylsalicylic acid (aspirin), phenylcinchoninic acid (cinchophen), and caffeine in admixture.** W. O. EMERY (J. Amer. Pharm. Assoc., 1928, 17, 18—22).—In a mixture of aspirin, cinchophen, and caffeine, caffeine is isolated by extraction with chloroform of a solution of the mixture in aqueous sodium carbonate and evaporation of the extract. The residual alkaline solution is treated with iodised potassium iodide solution (Wagner's reagent), which converts the aspirin into

di-iodophenylene oxide, $C_6H_2I_2O$, which is collected on a filter and weighed. The filtrate, on acidification, yields a precipitate of cinchophen periodide ($C_{16}H_{11}NO_2$), HI , I_2 , and iodine. The cinchophen can be isolated and weighed by extracting the precipitate with an ether-chloroform mixture and treating with sulphurous acid. In the absence of caffeine, cinchophen may be determined, either alone or admixed with aspirin, by conversion into the periodide in acetic acid solution and titration of the excess of iodine in the filtrate from the precipitate. Representative determinations of many control and commercial samples by the above methods give very satisfactory results. E. H. SHARPLES.

Indian turpentine. PILLAY and SIMONSON.—See XIII.

PATENTS,

Manufacture of phenolic bases of the alkaloid series. E. MERCK CHEM. FABR., ASSEES. of M. OBERLIN (G.P. 444,587, 18.7.25).—Alkaloids containing alkoxy groups are dealkylated by heating at 120 – 200° with aluminium chloride alone or in a suitable solvent, methylimino-groups being under these conditions unattacked. Papaveraldine in 1 hr. at 140 – 150° gives in 84% yield papaveraldoline (sulphate, $C_{32}H_{22}O_{10}N_2 \cdot H_2SO_4 \cdot 6H_2O$), and at 170 – 175° papaveroline (sulphate, $C_{32}H_{26}O_8N_2 \cdot H_2SO_4 \cdot 10H_2O$); bromopapaveroline (picrate, $+3H_2O$) is similarly prepared. Quinine gives apoquinine, hydroquinine gives hydrocupreine. Laudanosoline (sulphate, $C_{34}H_{38}O_8N_2 \cdot H_2SO_4 \cdot 4H_2O$) and cotarnoline (sulphate, $C_{20}H_{22}O_6N_2 \cdot H_2SO_4 \cdot 2H_2O$) are obtained from laudanosine and cotarnine, respectively. C. HOLLINS.

Pharmaceutical product. F. MÜLLER, Assr. to CHEM. WORKS (FORMERLY SANDOZ) (U.S.P. 1,655,795, 10.1.28. Appl., 18.5.27. Switz., 3.6.26).—*iso*Butylallylbarbituric acid (1 mol.) and 4-dimethylamino-1-phenyl-2:3-dimethyl-5-pyrazolone (1 mol.) combine in methyl alcoholic solution to a compound of therapeutic value. T. S. WHEELER.

Emulsions to be used as shaving creams, anti-septic ointments, or liniments. T. D. KELLY (B.P. 283,711, 13.12.26).

Silicic acid compounds (B.P. 284,450).—See VII. **Glass vessels** (B.P. 284,888).—See VIII. **Cod-liver oil rich in vitamin-A** (B.P. 283,265).—See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Optical and photographic properties of a series of typical cyanine dyes. O. BLOCH and F. M. HAMER (Phot. J., 1928, 68, 21–34).—The chemical constitution, optical absorption, and photographic sensitising properties of the following cyanine dyes are described: *pseudocyanine*, *isocyanine*, *cyanine*, Sensitol Red (2:2'-carbocyanine), dicyanine (of the type 2:4'-carbocyanine), kryptocyanine (4:4'-carbocyanine), thiocyanine, thio-carbocyanine, thiopseudocyanine, thioisocyanine, indo-carbocyanine, oxacarbocyanine, erythroapocyanine, xanthroapocyanine, and indopseudocyanine. In the series

pseudocyanine, *isocyanine*, and *cyanine*, and in the carbocyanines, as the 2:2'-linking is replaced by the 2:4', and that by the 4:4'-linking, there is a displacement of the absorption maxima towards the red. The shift towards the red corresponds with the lengthening in the carbon chain which connects the two nitrogen atoms. Also the lengthening of the chain which joins the nuclei gives a similar shift towards the red. The shift is also apparent in the case of the pair thiopseudocyanine and thioisocyanine, and in the replacement of the methenyl group by a three-carbon chain in the case of thiocyanine and thiocarbocyanine. With the exception of thiocyanine, every type of cyanine dyestuff, under the conditions described, behaves as a photographic sensitiser. Absorption curves were obtained in the presence of alcohol, water, and gelatin; the nature of the medium has a marked effect on the absorption. Addition of potassium iodide to an aqueous alcoholic solution of dicyanine completely changes the character of its absorption curve. The results throw little light on the relation between absorption and sensitisation. The shift between the maxima of absorption and of sensitisation is towards the red, and this shift becomes greater as the dye sensitises further towards the red. The properties of 1:1'-diethylpseudocyanine iodide, 1:1'-diethyl-3:3:3':3'-tetramethylindocarbocyanine iodide, and 2:2'-diethyloxacarbocyanine iodide (cf. Hamer, A., 1928, 76) are described. W. CLARK.

Action of dyes in fogging reactions. LÜPPO-CRAMER (Z. wiss. Phot., 1928, 25, 121–128).—The author has previously shown (Camera [Lucerne], 1924, 3, No. 1) that the fogging action of acids, peroxide, and ozone is favoured by the presence of bromide ions, but that the type of plate used has a marked influence on the results. It is now confirmed that the fogging action of acidified bromide solutions varies markedly with the type of plate, and that the reaction does not take place with orthochromatic plates. The inhibition of the action is due to the dye present. Dyes similarly prevent the fogging action of hydrogen peroxide, phenosafranine, erythrosin, and rhodamine-B being effective. The preventative action decreases in the order of the dyes given. This order is probably bound up with the fact that phenosafranine is least easily washed out of the plate by bathing in water. Desensitisers are not specific in preventing fogging by peroxide, so that the observation that they do prevent fogging can no longer be used as a support for the luminescence theory of fogging by peroxide. Phenosafranine has a specific action in destroying the latent image in presence of acidified bromide. One and the same acid bromide solution can produce a marked fog, and on the same plate can destroy the latent image in presence of phenosafranine. W. CLARK.

Photochemical studies. X. Photographic fog. A. REYCHLER (Bull. Soc. chim. Belg., 1928, 37, 33–39).—The greyish "fog" produced when a photographic plate is developed and then fixed with sodium thio-sulphate, without previous exposure to light, is due to a number of black granules. Its appearance may be prevented by immersing the unexposed plate for a few

minutes in a solution of an oxidising agent, 0.1% permanganate, 0.2% chromic acid, or 0.01% ferricyanide, and then treating with acidified sulphite. The negative obtained by subsequent exposure is very clear and brilliant. Mercuric chloride may be used for the same purpose, but in very dilute solution, 0.0025%. Immersion should last only a few seconds and no reducing bath is required. Ordinary fog may also be prevented by preliminary treatment of the plate. The oxidising agents generally used are, at the same time, acid and astringent, so, in order to investigate the cause of the clarifying action, the effect of acids alone and of oxidising agents alone has been investigated. Treatment with acid alone, and subsequent exposure, has a very marked clarifying action on the negative. This effect diminishes with increasing dilution until a concentration is reached at which it is negligible. Such a solution, however, can become an excellent clarifying agent if there is added to it an amount of oxidising agent so small that, if alone, it also would be quite inactive. Anthraquinone- β -sulphonic acid has a marked effect even at very high dilutions, viz., 0.0004*N*, because, besides being an acid, it also becomes a strong oxidising agent on exposure to light. If the plates treated with acidified permanganate or chromic acid are dried before exposure the sulphite bath should be omitted in the preliminary treatment. Sodium thiosulphate is also a very effective clarifying agent. The plate may be immersed in a 30% solution for a few seconds, washed, and exposed to the air for a few minutes. If developed and fixed before exposure a denser fog is formed than without treatment, but after exposure a brilliant negative is obtained. The cause of this action is discussed.

M. S. BURR.

PATENTS.

Photographic plates. SOC. ANON. UNION PHOTOGRAPHIQUE INDUSTRIELLE (ÉTABL. LUMIÈRE ET JOUGLA RÉUNIS) (F.P. 619,395, 1.12.25).—The back of the plate is coated with a layer of cellulose acetate containing a plasticiser and a black aniline dye or lamp-black. After evaporation of the solvent an easily strippable coating remains.

W. CLARK.

Manufacture of films for colour photography. SOC. DU FILM EN COULEURS KELLER-DORIAN (B.P. 261,363, 3.11.26. Fr., 16.11.25).—In a reticulated film for colour photography the width of each reticulation is from 10–40% greater than the width of the image of the colour-selecting screen which it projects on the sensitive surface, for a given aperture of the projecting lens.

W. CLARK.

Films for colour cinematography. R. BERTHON (B.P. 274,837, 28.6.27. Fr., 20.7.26).—A reticulated film for colour cinematography which does not require to be treated with a liquid consists of a support of gelatin, celluloid, cellophane, etc., on one face of which is fixed a layer of celluloid or other plastic material carrying colour-selective, refractive, microscopic elements in relief. The other face of the base is provided with a layer pervious to gases and containing a diazo compound or a mixture of a diazo compound with one or more

phenols, which forms a dark-coloured substance under the action of ammonia etc. when it has not been affected by light (Kalle process).

W. CLARK.

Film images resembling drawings. O. BORNHAUSER (G.P. 445,079, 3.10.26).—Contrasty negatives obtained in the normal way are copied on a film carrying a chromated layer which is coated, before or after developing in water, with a protective layer impervious to water and dyed in an aqueous dye solution. A silvered chromate-containing positive film may be used, development being with a normal developer. The film appears as an outline drawing, consisting of a practically colourless layer of chromium hydroxide, with thin lines and free areas of celluloid.

W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Explosive properties of lacquer-solvent vapours. RICHARDSON and SUTTON.—See XIII.

XXIII.—SANITATION; WATER PURIFICATION.

Correlation between differential tests for colon bacteria and sanitary quality of water. I. M. LEWIS and E. E. PITTMAN (J. Amer. Water Works' Assoc., 1928, 19, 78–92).—Examination by various methods of colon bacteria isolated from waters of different sanitary quality showed the superiority of the "citrate test"; a combination of indole production and the methyl-red reaction is suggested as the best indicator of sanitary quality of waters.

C. JEPSON.

PATENTS.

Preparation of mouth washes and dentifrices. F. BRÄUNLICH (B.P. 259,942, 28.9.26. Czechoslov., 14.10.25).—To tooth pastes or mouth washes are added sulphonated fats or sulphonated fatty acids, or their alkali or ammonium salts, in quantity sufficient to dissolve any admixed volatile oil or other water-insoluble perfume etc. (e.g., 10 pts. of alkali sulphate to 90 pts. of paste).

B. FULLMAN.

Centrifugal apparatus for the purification of liquids [water]. F. JENSEN, Assr. to B. E. HILL (U.S.P. 1,654,812, 3.1.28. Appl., 21.11.24).—Centrifugal separation of objectionable matter from water to be used for the production of raw ice is effected by cutting the water rapidly while whirling it in opposite directions alternately.

H. HOLMES.

Stabilisation of ferruginous water. C. PEETERS (U.S.P. 1,656,232, 17.1.28. Appl., 8.9.25. Belg., 9.9.24).—The separation of iron from water containing it is inhibited by the addition of a polyhydric alcohol, e.g., glycerol, or of an aldehyde, e.g., dextrose.

T. S. WHEELER.

Water-softening apparatus for use with steam generators or other plant requiring softened water. J. MILLS & Co. [ENGINEERS], LTD., and W. H. PORTER (B.P. 285,629, 20.1.27).

Grading of mixed materials (B.P. 283,645).—See I. **Glauconite** (U.S.P. 1,657,700).—See VII.