

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCTOBER 14, 1927.

I.—GENERAL; PLANT; MACHINERY.

Recovery of volatile solvents in chemical industries. BODEWIG (Z. ges. Schiess- u. Sprengstoffw., 1927, 22, 167—169).—The volatile solvent recovery plant of the Cheminova Co. of Berlin is described. The method is in essence the same as that of the Brégeat recovery process, and consists in absorbing the solvent vapours in certain cresols which form with them complex compounds. These compounds are easily decomposed on heating, by which means the solvents are obtained in a pure state. The plant consists of three parts in which absorption, decomposition, and rectification, respectively, take place. Absorption of the vapours from the vapour-air mixture is effected by passing the mixture up a tower filled with iron spirals in counter-current to a stream of the cresol absorbent. The cresol-solvent mixture then passes to the decomposer, where it is heated, yielding up the solvent, which passes to the rectification plant. The cresol from the decomposer is then available for use again in the absorber. The application of the process to various industries is outlined. The recovery of ether-alcohol in a Chardonnnet silk plant by this means was over 90% and of acetone in an artificial silk factory was 95—98%.
S. BINNING.

Testing machine for repeated impact. SMITH and WARNOCK.—See X.

PATENTS.

Dry kiln. C. G. ULLIN (U.S.P. 1,637,166, 26.7.27. Appl., 18.8.26).—The kiln is provided with air inlet and outlet conduits spaced apart and with pipe connexions between them, the air being circulated by a blower. Means are provided for moistening the air in the kiln and for separating moisture from the air passing through the pipe connexions, such moisture being delivered through an exhaust opening into the atmosphere.

H. HOLMES.

Retort. K. L. FORD, ASST. to GLASS CONTAINER ASSOC. OF AMERICA (U.S.P. 1,636,768, 26.7.27. Appl., 12.3.26).—A closed vessel is provided with an intake conduit for heating vapour and an exhaust conduit communicating with the vessel at several points remote from the inlet. To ensure uniform heating throughout the vessel the gaseous contents are exhausted from and returned to it under the aspirating action of the inflowing vapour.

H. HOLMES.

Preventing corrosion in condensers. INTERNAT. GENERAL ELECTRIC CO., INC., ASSEES. of ALLGEM. ELEKTRICITÄTS-GES. (E.P. 258,908, 28.9.26. Conv., 28.9.25).—In a condenser for a steam power plant the surfaces in contact with the cooling water are protected

from electrolytic erosion by a coating of "Bakelite," which is preferably applied in the liquid condition and solidified by heating under pressure.
H. HOLMES.

Ball or tube mill. W. KÖPPEN and C. PFEIFFER (U.S.P. 1,636,138, 19.7.27. Appl., 8.12.26. Conv., 8.12.25).—A preliminary grinding chamber communicates with a fine-grinding chamber through an intermediate chamber, the wall between the first two being formed of concentric rings provided with an entirely free peripheral passage for the material to be ground.

H. HOLMES.

Ball mill. W. M. BARKER (U.S.P. 1,636,585, 19.7.27. Appl., 13.10.24).—One end of a rotary grinding drum is provided with a short trunnion mounted in a bearing bracket, the other end having an inlet for the material to be ground. A removable blower casing, surrounding the drum and communicating with it, has separate outlets for the material and for the air.
H. HOLMES.

Pulverising machine. W. K. CRUICKSHANK (U.S.P. 1,633,164, 21.6.27. Appl., 28.9.26).—A pulverising apparatus is fitted with rotary and stationary cutters, and with a heating jacket, being adapted to pulverise sodium sulphate decahydrate and convert it into the octahydrate.
T. S. WHEELER.

Rotary dryer. C. MILLER (U.S.P. 1,637,831, 2.8.27. Appl., 7.3.25).—In an upright cylindrical chamber rotating about its vertical axis, an air entry at the bottom of the chamber wall faces the direction of rotation and an air exit at the top the reverse direction.
C. A. KING.

[Centrifugal] separation of liquids from solids. C. R. ALTENHEIN. From W. KLEIN (E.P. 274,958, 28.4.26).—To enable a screen of coarser mesh and thicker metal to be used, the material to be drained is first graded and the different grades are fed separately, but simultaneously, into the machine so that coarser material impinges directly on the screen to form thereon a porous filtering layer for the finer material. The grades are thrown towards the screen in proper rotation by a feed table with projecting vanes. The draining of the finer material is facilitated when the bulk of the material is in the coarser grades. When a conical screen with slot perforations is used, the length of each slot is arranged oblique to the direction of travel of the material to facilitate the passage of the material over the screen surface. The sludge which falls when the machine is retarded suddenly is caught in a cup provided with draining channels.
H. HOLMES.

Apparatus for the [catalytic] hydrogenation of liquids and semi-liquids. G. R. SCHUELER (E.P. 274,952, 28.4.26).—In a catalytic unit as described in E.P. 273,045 (B., 1927, 690) each disc may be of woven

wire, or of perforated sheet metal, or of a spiral strip of the metal with distance pieces between the convolutions, and may be provided with spacing corrugations, ribs, or projections. A uniform oxidation of the unit is effected by dismantling the discs and suspending them alternately with cathode plates in an electrolytic bath.

H. HOLMES.

Process and apparatus for the continuous beating of viscid liquids. D. G. STEELY (E.P. 274,976, 1.5.26).—The liquid is agitated in a large volume of air with provision for accommodating the continuously increasing bulk of the aerated material passing through the beating chamber to an unrestricted outlet. The chamber is preferably a horizontal cylinder provided with a longitudinal rotary shaft carrying the beating and propelling devices, the shaft bearings being remote from the beating zone, and blades for forcing air through the cylinder are carried by the shaft at the inlet end. A constant head of liquid is introduced between these blades and the beating zone through one or more openings provided with valves. The beating devices are more widely spaced toward the discharge end, *e.g.*, an open helical coil of increasing pitch may be used.

H. HOLMES.

[Reflux] extraction method and apparatus. H. BUEL, Assr. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,636,550, 19.7.27. Appl., 9.7.25).—Extractives injuriously affected by high temperatures are obtained by the use of a reflux extractor operating under reduced pressure. The material is placed in a container to which an exhaust pump is connected through a reflux condenser, and the solvent is vaporised in a vessel connected to the container by a vapour supply duct and a return duct for the solvent and extractive.

H. HOLMES.

Determination of equilibrium vaporisation end-points. C. E. PARSONS and R. STEVENSON, Assrs. to W. P. DEPPE (U.S.P. 1,632,748, 14.6.27. Appl., 6.10.26).—The liquid under examination is completely vaporised by passage down a heated column, the vapour being led into a chamber containing a glass tube coated at one end with platinum black. Means are provided for cooling the coated tip by a current of air, and for observing the temperature at which condensation occurs on it.

T. S. WHEELER.

Apparatus for bringing liquids and gases into intimate contact. P. F. HOLMES, D. M. HENSHAW, and W. C. HOLMES & Co., LTD. (E.P. 275,092, 7.2.27).—Apparatus as described in E.P. 5113 of 1895 and 162,166 (B., 1896, 340; 1921, 422 A) is modified to cause the gases to travel through all the brushes in the same direction to or from the shaft. Still chambers may be located between adjacent compartments, and may serve for the passage of the gases from a point near the shaft in one compartment to a point remote from the shaft in the next compartment, or in the reverse direction.

H. HOLMES.

Production of a catalyst carrier. C. R. DOWNS (U.S.P. 1,636,685, 26.7.27. Appl., 9.12.26).—A carrier for use in the catalytic oxidation of organic compounds in the vapour phase comprises iron particles coated with aluminium.

T. S. WHEELER.

Imparting to drying means a large superficial area. E. SMOLCZYK (U.S.P. 1,633,959, 28.6.27. Appl.,

3.6.26. Conv., 8.6.25).—Phosphorus pentoxide can be deposited on pumice by first impregnating the latter with phosphoric acid.

T. S. WHEELER.

Manufacture of a filter aid. H. S. THATCHER, Assr. to CELITE Co. (U.S.P. 1,632,458, 14.6.27. Appl., 29.7.25).—Diatomaceous earth is ground so that not more than 10% remains on 150-mesh and 15–50% of the particles settle in distilled water at 20° at about 0.36 cm./min.

T. S. WHEELER.

Filters for purifying and sterilising air and gases. M. GRÄFIN VON LINDEN, R. MELBAU, and DEUTS. LUTTFILTER BAUGES. M.B.H. (E.P. 274,942, 23.4.26).—A filtering material composed of or containing copper is coated with a viscous solution of a copper salt such as a mixture of paraffin oil and copper oleate. The solution may be applied to copper plates or sieves, or may be placed with finely-divided copper in a container. The nature and proportions of the oil and salt used may be such that the mixture is destructive to certain bacteria and innocuous to others.

H. HOLMES.

Gas analysis apparatus. K. L. TATE, Assr. to TAYLOR INSTRUMENT COS. (U.S.P. 1,633,352, 21.6.27. Appl., 23.2.26).—A current of a mixture of gases, *e.g.*, flue gas, is drawn through a capillary tube terminating in a chamber with a restricted outlet. The current enters the capillary tube at atmospheric pressure, and leaves the chamber at a constant rate, the restricted outlet being subject to the conditions of maximum flow. Means are provided for compensating for changes in atmospheric pressure, and for determining the pressure in the chamber, which varies as the viscosity of the gas under examination, and hence, in this instance, with its carbon dioxide content.

T. S. WHEELER.

Edge [stream-line] filter. E. A. FORSBERG, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 1,640,698, 30.8.27. Appl., 4.9.24. Conv., 18.9.23).—See E.P. 226,274; B., 1925, 157.

Machine for separating solids from liquids. W. C. LAUGHLIN, Assr. to LAUGHLIN FILTER CORP. (U.S.P. 1,640,707, 30.8.27. Appl., 29.9.23).—See E.P. 235,916; B., 1926, 255.

Method and apparatus for drying or conditioning products composed of different components having different moisture contents. E. C. R. MARKS. From INDUSTRIAL DRYER CORP. (E.P. 275,681, 15.2.26).—See U.S.P. 1,562,764; B., 1926, 81.

Apparatus for analysing [furnace] gases. J. ALSBERG and P. J. RALPH, Assr. to J. ALSBERG (U.S.P. 1,633,881, 28.6.27. Appl., 5.2.24).—See U.S.P. 1,485,757; B., 1924, 496.

[Compression] refrigerating machine. A. A. KUCHER (E.P. 261,709, 13.8.26. Conv., 19.11.25).

Transverse-current cooling tower. M. HAMON (E.P. 263,114, 18.11.26. Conv., 16.12.25).

[Automatic] siphon. N. SWINDIN (E.P. 275,315, 3.12.26).

Apparatus for determining, indicating, and controlling the moisture content of paper and like materials. J. D'A. CLARK (E.P. 275,741, 18.5. and 13.7.26).

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

Peat. II. Distillation under reduced pressure of certain constituents of peat. J. REILLY and (Miss) J. SULLIVAN (Sci. Proc. Roy. Dubl. Soc., 1927, 18, 383—388; cf. B., 1926, 906).—In order to study the decomposition of peat, the authors separated the material into fractions. The peat was first extracted with dilute hydrochloric acid, the amount of extract thus obtained being 5.4% of the dry peat. The residue was separated by extraction with 4% ammonia solution into "insoluble humin" (67%) and "soluble humin" (27.6%). The "humus" precipitated from the "soluble humin" by hydrochloric acid was further fractionated into "alcohol extract of humus" (7%) and "humic acid" (15.5%). Each of these fractions was fractionated under reduced pressure, the products being collected in two receivers—one kept at a constant temperature of 60° and the other in a freezing mixture. The products were coke, 60° distillate, 0° distillate, and gas. Humic acid and hydrochloric acid extract gave very little tar. The highest percentage of coke was obtained from the humic acid, and the lowest from the alcoholic extract of humus. The greatest volume of gas was obtained from the insoluble humin. The gases consisted principally of carbon dioxide, with smaller amounts of carbon monoxide and hydrocarbons. C. W. ROBINSON.

Conditions for success of low-temperature carbonisation. W. E. DAVIES (Chem. Met. Eng., 1927, 34, 480—483).—Mainly in the sphere of economics. The importance of throughput is stressed, and the most promising line of development is considered to be carbonisation at a gas-works in continuous vertical retorts, with a feed so adjusted that low-temperature conditions are approached, the loss in gaseous therms being compensated by steaming. It is also emphasised that avoidance of cracking of the tar and oil produced not only increases the yield of distillate, but results in heat economy. The time required for carbonisation may be almost halved. C. IRWIN.

Apparatus for determining the softening point of paraffins, waxes, pitches, asphalts, etc. H. HERBST (Petroleum, 1927, 23, 1079—1080).—See B., 1927, 244.

Coke for blast-furnace working. GILL.—See X.

PATENTS.

Combination [coke] oven. J. BECKER, ASSR. to KOPPERS Co. (U.S.P. 1,623,460, 5.4.27. Appl., 19.8.25).—Independent channels suitably proportioned for admitting air or gas to the flues of an oven are provided so that the oven can be operated either with preheated gas and preheated air or with cold rich gas and preheated air, with complete utilisation of all the regenerators. S. PEXTON.

Coking still. R. CROSS, ASSR. to GASOLINE PRODUCTS Co. (U.S.P. 1,622,573, 29.3.27. Appl., 12.4.24).—A horizontal cylindrical still rotates around a fixed central shaft carrying a drum. The still is externally heated and the petroleum coke is scraped from the still walls by the rotation of the still around the fixed drum. S. PEXTON.

Wood charcoal briquettes. M. L. Y. DE PANIAGUA (F.P. 615,149, 14.9.25).—Powdered charcoal is mixed with an organic binder, e.g., tar, preferably with the addition of a dichromate, compressed, and carbonised above 350°. A. B. MANNING.

Manufacture of decolorising carbon. L. WICKENDEN and S. A. W. OKELL, ASSRS. to INDUSTRIAL CHEMICAL Co. (U.S.P. 1,634,477—8 and 1,634,480, 5.7.27. Appl., [A] 16.6.21; [B] 18.2.22; [C] 22.3.23).—(A) Carbonised vegetable material, e.g., from waste sodium sulphite liquors, is heated in a rotary kiln at 900°, and then passed against a current of steam and carbon dioxide down an electric shaft-furnace maintained at 900°. (B) The electric furnace contains a lower rotating electrode, through which the gas current is introduced. The material when sufficiently disintegrated is removed by the gas current. (C) Material treated as in (B) is heated with air at 400° in a rotary kiln. T. S. WHEELER.

Electric furnace for revivifying decolorising carbon etc. L. WICKENDEN and W. F. DEMMERLE, ASSRS. to INDUSTRIAL CHEMICAL Co. (U.S.P. 1,634,479, 5.7.27. Appl., 3.7.22).—The furnace comprises a cylinder rotating around a vertical axis, and containing stationary electrodes which also serve as agitators.

T. S. WHEELER.

Production of metallic carbon. C. ROBINSON, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,637,052, 26.7.27. Appl., 1.8.21. Renewed 15.12.25).—A mixture of anthracite coal and cellulose is heated in an electric furnace at 3500° to yield pure elementary carbon in a porous form, which on fusion forms a malleable ductile product of value in the manufacture of electric lamp filaments. T. S. WHEELER.

Production of highly active carbon. I. G. FARBER-IND. A.-G., ASSEES. of S. MÜNCH (G.P. 439,114, 14.3.23. Addn. to G.P. 438,670).—Wood or other cellulosic material impregnated with a solution containing sodium and ammonium sulphates is calcined above 500°, and the product is extracted with water. L. A. COLES.

Production of carbon black. L. J. DALES (U.S.P. 1,633,071, 21.6.27. Appl., 4.4.21. Renewed 27.8.24).—Natural gas is passed through a chamber in which it is heated, the issuing gas then interchanging heat with the entering gas. Any liquid condensate is removed, and the residual gas is burned under the heating chamber to form carbon black. T. S. WHEELER.

Production of carbonaceous material. A. OBERLE (U.S.P. 1,632,845, 21.6.27. Appl., 31.7.24).—Carbonaceous residues from cracking mineral oil are heated at about 540° in a current of steam, first under increased and then under reduced pressure, being finally treated with a mixture of nitric and sulphuric acids.

T. S. WHEELER.

Production of active charcoal from cacao refuse. E. TIEDE (G.P. 440,381, 3.4.25).—The refuse is carbonised at red heat after removal of fat and of theobromine by sublimation. L. A. COLES.

Gaseous fuel [for welding etc.]. J. R. ROSE, ASSR. to CARBO-HYDROGEN Co. OF AMERICA (U.S.P. 1,628,067, 10.5.27. Appl., 31.12.19).—A mixture containing 80—98% H₂ and 20—2% CH₄, together with a

small proportion of propane and butane, is of use as a fuel for welding and cutting metal. S. PEXTON.

Manufacture of gas from coal and like carbonaceous materials. M. W. TRAVERS and F. W. CLARK (E.P. 267,724, 7.4.26. Addn. to E.P. 198,777; B., 1923, 759 A).—In the process of completely gasifying coal in an elongated gas producer, as described in the prior patent, a core of uncarbonised coal, into which the hot gases do not penetrate, is formed. By introducing a mechanical agitator into the distillation chamber, the plastic layer of coal surrounding this core may be broken down. S. PEXTON.

Fuel gas distributing systems for coke ovens. KOPPERS Co., Assees. of J. BECKER (E.P. 252,676, 20.3.26. Conv., 29.5.25).—When fuel gas is supplied to the burners of a coke oven through a horizontal duct at the top of the regenerator walls the fuel gas increases in temperature and velocity towards the centre of the oven. In order to regulate the amounts of gas fed to individual flues, a second gas-distributing flue is built below the sole of the regenerators, and is connected to the upper distributing flue by vertical ducts. The regulating orifices may be placed at the base of these vertical ducts where the temperatures are uniformly low. S. PEXTON.

Gas production from powdered charcoal. C. HILKER (G.P. 439,011, 14.1.23).—The powdered charcoal, mixed with a suitable slag-forming flux, is charged, without admixed air, in at the top of a vertical shaft in such a way that it is distributed over the cross-section of the shaft by centrifugal action, and is gasified in a current of air entering at the bottom of the shaft.

A. B. MANNING.

Water-gas generator for bituminous fuel. O. MISCH (E.P. 255,447, 12.7.26. Conv., 16.7.25).—Bituminous fuel is distilled in an externally heated retort superimposed upon a water-gas generator in which the carbonised fuel is then gasified. During the "blow" period the "blow" gases are burnt in the spiral combustion chamber surrounding the retort, whilst during the "run" part of the water-gas is withdrawn from the lower part of the generator and burnt around the retort, the remainder passing through the distillation retort, and thus assisting the carbonisation. S. PEXTON.

Manufacture of water-gas [from powdered fuel]. I. G. FARBERIND. A.-G., Assees. of F. WINKLER (G.P. 438,843, 26.7.24. Addn. to G.P. 437,970; B., 1927, 548).—The procedure of the main patent is modified by passing the steam downwards through the incandescent material, so that during the steaming period the material is quiescent. The gas produced is then free from dust.

A. B. MANNING.

Purification of [coal] gas. W. H. FULWEILER and C. W. JORDAN, Assrs. to U.G.I. CONTRACTING Co. (U.S.P. 1,632,758, 14.6.27. Appl., 24.6.26).—Sodium carbonate solution which has been used to remove sulphur from coal gas is revived by being stirred with an immersed paddle, under which a stream of minute bubbles of air is introduced. The sulphur separating is removed at the surface.

T. S. WHEELER.

Gas-purifying apparatus [oxide purifier]. T. W. STONE, Assr. to WESTERN GAS CONSTRUCTION Co. (U.S.P.

1,632,251, 14.6.27. Appl., 16.2.22).—The purifying material is packed in upper and lower tiers, valves being provided to enable the gas to be passed in either direction through the tiers, to permit complete utilisation of the purifying material.

T. S. WHEELER.

Gas-separating apparatus. M. M. REYNOLDS (U.S.P. 1,623,636, 5.4.27. Appl., 14.10.25).—Gas to be purified from dust is streamed through a compartment containing inclined deflectors and screened openings. The deflectors, arranged in advance of each screened opening, baffle the gas and serve to deposit part of the dust on the bottom of the chamber, the remainder being removed by the screens.

S. PEXTON.

Separation of carbon dioxide from gas mixtures by means of ammoniacal liquor. BAMAG-MEQUIN A.-G. (G.P. 440,324, 28.8.25).—The gases and ammoniacal liquor pass in the same direction through a series of scrubbers, cooling apparatus being provided between each pair of scrubbers to remove the heat of dissolution and neutralisation of the carbon dioxide.

L. A. COLES.

Apparatus for the distillation of solid bituminous materials. J. DANIELS (G.P. 440,169, 30.6.25).—Coal is carbonised in radial chambers on the outer jacket of a rotary retort; before or during its introduction into the carbonising chamber the coal is compressed, the compression chamber being closed from the supply chamber during the operation by a damper. A dense, homogeneous carbonisation product is obtained.

A. B. MANNING.

Distillation of bituminiferous material. J. J. JAKOWSKY (U.S.P. 1,622,722, 29.3.27. Appl., 11.1.26).—Solid bituminous material is exposed to a heat-radiating surface so that only the upper surface of the mass is heated at the desired temperature. The mass is progressively stirred so as to expose it gradually to surface distillation.

S. PEXTON.

Conversion [cracking] of the components of petroleum and the like into more volatile products. W. C. and F. E. WELLS (U.S.P. 1,635,519, 12.7.27. Appl., 10.8.20).—Petroleum is introduced at the bottom of a still containing molten lead at about 540°, a continuous flow of the higher fractions of the liquid being maintained across the surface of the metal to remove carbonaceous impurities.

T. S. WHEELER.

Production of benzine, kerosene, and higher hydrocarbons from crude petroleum oil emulsions of the water-in-oil type. P. J. DE KADT (Dutch P. 15,167, 7.4.24).—The emulsion is converted by the addition of a suitable agent, e.g., resin soap, into an intermediate stage which cannot be characterised as either a water-in-oil or an oil-in-water emulsion, and, without separation of the water, is then distilled. No frothing or bumping occurs during distillation.

A. B. MANNING.

Production of light hydrocarbon liquids. E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,632,967, 21.6.27. Appl., 4.4.25).—Heavy oil is heated at about 1000° and 9 atm. pressure, the distillate being passed up a column in counter-current to a stream of fresh oil, which is added at about twice the rate at which the distillate is withdrawn.

T. S. WHEELER.

Refining mineral oils etc. with liquid sulphur dioxide. ALLGEM. GES. FÜR CHEM. IND. M.B.H. (F.P. 615,553, 3.5.26. Conv., 5.3.26).—Before the solution containing the sulphur dioxide extract enters the evaporator working under the condenser pressure, it passes through one or more evaporators working under higher pressures, only one of which—that working under the highest pressure—is heated from an external source. Each of the others is heated by acting as a condenser for the evaporator working at the next higher pressure.

A. B. MANNING.

Art of refining oils. C. LEAVER, ASSR. to IMPERIAL OIL, LTD. (U.S.P. 1,635,718, 12.7.27. Appl., 11.3.21. Conv., 13.8.20).—The oil is mixed with partially-spent sulphuric acid in a pipe fitted with baffles and separated from the sludge in a settling tank, being then treated with fresh acid and recovered from the resulting partially spent acid in a similar unit. The same process can be used for the alkaline washing.

T. S. WHEELER.

Prevention of acidity in [transformer] oils. C. J. RODMAN, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,633,956, 28.6.27. Appl., 12.3.25).—The formation of acid in transformer oil is inhibited by coating immersed copper parts with tin.

T. S. WHEELER.

Preparation of an adsorbent for [liquid petroleum] oils. W. D. RIAL and E. W. GARD (U.S.P. 1,634,514, 5.7.27. Appl., 8.9.25).—Adsorbent clay is treated at 190° with pure petroleum distillate containing sulphuric acid, until adsorption of the latter is complete, and is then separated and freed from adherent oil in a centrifuge.

T. S. WHEELER.

Revivification of spent adsorbents for oil treatments. P. W. PRUTZMAN, ASSR. to CONTACT FILTRATION CO. (U.S.P. 1,633,871, 28.6.27. Appl., 2.9.22).—The adsorbents are washed first with light oil, then with a mixture of benzene and alcohol, and finally heated with water.

T. S. WHEELER.

Petroleum oil composition. A. A. SOMERVILLE, ASSR. to R. T. VANDERBILT CO., INC. (U.S.P. 1,634,054, 28.6.27. Appl., 19.6.26).—*p*-Aminophenol (0.5–1%) is added to petroleum insulating and lubricating oils to inhibit oxidation at high temperatures.

T. S. WHEELER.

Method of using low-compression fuels. C. F. KETTERING and T. MIDGLEY, JUN., ASSRS. to GENERAL MOTORS CORP. (U.S.P. 1,635,216, 12.7.27. Appl., 3.1.24).—Ethyl iodide is employed as an anti-knock material.

T. S. WHEELER.

Coking retort oven. J. VAN ACKEREN, ASSR. to KOPPERS CO. (U.S.P. 1,633,911, 28.6.27. Appl., 18.1.21).—See E.P. 230,167; B., 1925, 345.

Process of distilling oil in presence of comminuted carbonaceous fuel. W. E. TRENT, ASSR. to TRENT PROCESS CORP. (U.S.P. 1,641,305, 6.9.27. Appl., 4.11.20).—See E.P. 171,075; B., 1922, 701 A.

Process and apparatus for cracking hydrocarbon oils. W. F. FARAGHER and W. A. GRUSE, ASSRS. to GULF REFINING CO. (U.S.P. 1,640,444, 30.8.27. Appl., 15.1.21).—See E.P. 174,085; B., 1923, 301 A.

Self-sealing door for coke ovens. H. SCHWARZ (E.P. 268,313, 3.3.27. Conv., 26.3.26).

Gas analysis apparatus (U.S.P. 1,633,352).—See I.
Asphaltic paving (U.S.P. 1,634,241). **Bituminous concrete** (E.P. 275,364).—See IX.

IV.—DYESTUFFS AND INTERMEDIATES.

Identification of vat dyes. VAIS.—See VI.

PATENTS.

Manufacture of an azo dye. H. WAGNER, ASSR. to GRASELLI DYESTUFF CORP. (U.S.P. 1,635,594, 12.7.27. Appl., 22.12.25. Conv., 11.6.23).—Diazotised or tetrazotised aminoazo or diaminoazo dyes, containing at least one free hydroxyl or carboxyl but no sulphonc group, are coupled with 1 or 2 mols. of a 2:3-hydroxynaphthoic arylamide to give products yielding fast black shades on cotton. *E.g.*, diazotised *p*-phenylenediamine is coupled with β -naphthol, and the diazotised product is coupled with 2:3-hydroxynaphthoic 5-chloro-2-toluidide.

T. S. WHEELER.

Manufacture of mordant [azo] dyes. DURAND & HUGUENIN A.-G. (E.P. 255,086, 8.7.26. Conv. 13.7.25).—*p*-Phenylenediaminesulphonic acid is monoacetylated, diazotised, and coupled with an *o*-hydroxy-acid, and the product is hydrolysed, rediazotised, and coupled with the same or a different *o*-hydroxy-acid. Similar products are obtained from *p*-nitroanilinesulphonic acids, diazotised, coupled, reduced, rediazotised, and coupled. Chrome-mordanted wool is dyed in reddish-orange shades.

C. HOLLINS.

Manufacture of disazo [mordant] dyes. I. G. FARBENIND. A.-G., ASSEES. OF FARBENFABR. VORM. F. BAYER & CO. (E.P. 251,637, 28.4.26. Conv., 28.4.25).—An aminobenzoic acid, containing a second carboxyl group or a sulphonic group or hydroxyl and sulphonic groups, is diazotised and coupled with α -naphthylamine-6- or -7-sulphonic acid, the product rediazotised and coupled with an aromatic *o*-hydroxycarboxylic acid, to give chrome-browns for wool or for printing on cotton, which are fast to chlorine. Examples are 4-amino-phthalic acid \rightarrow α -naphthylamine-6-sulphonic acid \rightarrow *m*-cresotic acid; 3-amino-5-sulphosalicylic acid \rightarrow α -naphthylamine-6-sulphonic acid \rightarrow salicylic acid.

C. HOLLINS.

Anthraquinone dyes and dyeing therewith. BRITISH DYESTUFFS CORP., LTD., A. SHEPHERDSON, and W. W. TATUM (E.P. 275,421, 19.8.26).—2-Halogeno-4-(sulphoarylamino)-1-hydroxyanthraquinones are boiled with aqueous sodium sulphite, filtered from unchanged monosulphonic acid, and evaporated to dryness. The product from 2-chloro-4-(sulpho-*p*-tolylamino)-1-hydroxyanthraquinone dyes wool from an acid bath a red-blue, becoming green by after-chroming.

C. HOLLINS.

Production of anthraquinone. A. E. CRAVER, ASSR. to BARRETT CO. (U.S.P. 1,636,856, 26.7.27. Appl., 5.12.21).—A mixture of anthracene vapour and air (15 pts.) is passed at 475° over a catalyst comprising uranium oxide (89%) and molybdenum oxide (11%), the time of contact being about $\frac{1}{2}$ sec.

T. S. WHEELER.

Manufacture of *N*-dihydro-1:2:1':2'-anthraquinoneazine. L. H. ULICH and R. J. GOODRICH, ASSRS. to NEWPORT CO. (U.S.P. 1,634,473, 5.7.27. Appl., 4.4.25. Cf. U.S.P. 1,580,700; B., 1926, 577).—Dibromo-*N*-dihydro-1:2:1':2'-anthraquinoneazine is heated,

with or without addition of solvents, diluents, or catalysts, with 33% alcoholic potassium hydroxide solution (6 pts.) for 1 hr. T. S. WHEELER.

Manufacture of azine dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 275,301, 29.4.26).—1:8-Naphthasultam-3-sulphonic or -3:6-disulphonic acid, when heated with a primary or secondary arylamine at 110–160° preferably in presence of a salt of the arylamine, exchanges the 3-sulphonic group for an arylamino-group. The products condense with nitrosodimethylaniline etc. (or may be oxidised with *p*-diamines) to form violet azine acid dyes for wool. 1:8-Naphthasultam-3-sulphonic acid gives with aniline 3-anilino-1:8-naphthasultam, m.p. 168–170°. 1:8-Naphthasultam-3:6-disulphonic acid with aniline gives 3-anilino-1:8-naphthasultam-6-sulphonic acid (aniline salt, m.p. 277–278°), and similar compounds are formed from *p*-toluidine and *p*-anisidine. C. HOLLINS.

Manufacture of dyes [isoviolanthrone]. BRITISH ALIZARINE CO., LTD., W. H. DAWSON, and P. BEGHIN (E.P. 275,283, 4.3.26).—*iso*Violanthrone [*isodibenzanthrone*] is obtained by heating 3-halogenobenzanthrones, having a free 2-position, with 8–10 pts. of aniline (or other arylamine) and 4–5 pts. of sodium or potassium hydroxide at 150–185°. Impurities are extracted from the dried product by means of aniline. C. HOLLINS.

Manufacture of thioindigo vat dyes. K. THIESS, C. J. MÜLLER, E. RUNNE, and A. SCHAEFFER, ASSTS. to GRASELLI DYESTUFF CORP. (U.S.P. 1,632,252, 14.6.27. Appl. 22.6.26. Conv., 27.6.25).—3:3':5:5'-Tetramethyl-, 3:3'-dihalogeno-5:5'-dimethyl-, 5:5'-dihalogeno-3:3'-dimethyl-, and 3:3':5:5'-tetrahalogeno-thioindigos can be halogenated to the corresponding 4:4'-dihalogeno-derivatives by treatment with a halogen in presence of a catalyst, e.g., antimony pentachloride, and a diluent, e.g., chlorosulphonic acid. The products give on cotton in the vat reddish violet shades, fast to boiling. The 3:3'-dimethyl compounds are not claimed as new. T. S. WHEELER.

Manufacture of indigoid dyes. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 262,457, 3.12.26. Conv., 4.12.25).—Tetrahalogenated thioindoxyls are condensed with an isatin, a thioisatin, or with halides or α -anils of these. 4:5:6:7-Tetrachlorothioindoxyl with 5:7-dichloroisatin chloride gives a violet vat dye, with 5:7-dichloroisatin a brown, with the *p*-dimethylaminoanil of 9-bromo- $\beta\beta$ -naphthathioisatin a violet. C. HOLLINS.

Manufacture of products [indigosols] for dyeing or printing textile fibres and other materials. DURAND AND HUGUENIN SOC. ANON., Assees. of I. G. FARBENIND. A.-G. (E.P. 267,952, 17.3.27. Conv., 17.3.26. Addn. to E.P. 186,057; B. 1924, 252).—The indigosols of the prior patent are obtainable as ammonium salts by treating a leuco-vat dye with aminosulphonic acid in presence of a tertiary base (pyridine, dimethylaniline) and preferably in suspension in an inert medium (chlorobenzene) at 75–85°. C. HOLLINS.

Production of benzidine and derivatives. R. A. NELSON, ASST. to NATIONAL ANILINE & CHEMICAL CO., INC. (U.S.P. 1,633,123, 21.6.27. Appl. 22.6.20).—

Hydrazobenzene in benzene solution is treated at 10° with concentrated hydrochloric acid to separate benzidine dihydrochloride in satisfactory yield. T. S. WHEELER.

Manufacture of wool and leather dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (E.P. 274,999, 21.6.26).—A halogenated aromatic nitro-compound containing reactive halogen (other than 1-halogeno-2:4-dinitrobenzene; cf. E.P. 13,672 of 1912; B., 1912, 1074) is condensed with 4-aminodiphenylamine or a derivative thereof. The products, which must contain at least two nitro-groups and a sulphonic group, dye wool and leather in fast yellow to brown shades. 4-Nitro-4'-aminodiphenylamine-2-sulphonic acid is condensed with *o*- or *p*-chloronitrobenzene or 4-chloro-3-nitrobenzaldehyde; 2-nitro-4'-aminodiphenylamine-4-sulphonic acid with 1-chloro-2-nitrobenzene-4-sulphonic acid; 4-aminodiphenylamine-2-sulphonic acid with 1-chloro-2:6-dinitrobenzene or its 4-sulphonic acid; 4-aminodiphenylamine with 1-chloro-2:6-dinitrobenzene-4-sulphonic acid, 1-chloro-2:4:6-trinitrobenzene, or 1-chloro-2:4-dinitro-naphthalene. C. HOLLINS.

Manufacture of acid dyes of the rhodamine series. DURAND & HUGUENIN SOC. ANON. (E.P. 251,644, 29.4.26. Conv., 4.5.25).—Acid rhodamines are obtained in a single operation by condensing a substituted *m*-aminophenol with phthalic anhydride in presence of concentrated sulphuric acid, preferably 100%. The dye from *o*-tolyl-*m*-aminophenol is redder, that from *m*-2-xylyl-*m*-aminophenol yellower, than the corresponding dye obtained by separate sulphonation. The process is applicable also to substituted phthalic anhydrides. C. HOLLINS.

Production of hydroxy-compounds [from primary amines]. J. W. C. CRAWFORD and F. G. WILLSON (E.P. 274,960, 28.4.26).—An arylamine is diazotised, and the diazo compound decomposed at a suitable temperature, in presence or absence of catalysts, the reaction mixture being agitated with an inert solvent which removes the phenol as formed. The solvents used are xylene (for phenol, *p*-cresol, *p*-chlorophenol, *m*-xylenol, salicylic acid), chlorobenzene (for *m*-hydroxybenzaldehyde), and anisole (for phenol). C. HOLLINS.

Catalytic methylation of ammonia and organic amines. A. MACKERT (E.P. 275,377, 17.6.26).—Dimethyl ether is passed with ammonia, or a primary or secondary amine, over alumina, zirconia, thoria, or titanium oxide at suitable temperature and pressure. Aniline with 100% excess of dimethyl ether over alumina at 270–310° gives in two treatments pure dimethylaniline; with 500–600% excess only one treatment is necessary. β -Naphthylamine, with 600% excess of dimethyl ether over thoria, at 330–350°, gives dimethyl- β -naphthylamine. A mixture of methyl alcohol and dimethyl ether may be used. C. HOLLINS.

Catalytic oxidation of hydrocarbons. E. C. R. MARKS. From E. I. DU PONT DE NEMOURS & Co. (E.P. 275,321, 3.5.26).—In the catalytic oxidation of hydrocarbons (e.g., naphthalene to phthalic anhydride) the gas mixture is preheated by means of preliminary tubes immersed in a bath of molten conducting substance (e.g., a mixture of sodium and potassium nitrates, or a metal

other than mercury) which also surrounds the catalyst tubes and removes excess heat from the reacting mixture.

C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND A.-G., Assees. of P. NAWIASKY, K. ZAHN, and K. SAURWEIN (U.S.P. 1,633,997, 28.6.27. Appl., 23.11.25. Conv., 13.12.24).—See F.P. 607,135; B., 1927, 647.

Preparation of derivatives from vat dyestuffs and alkyl esters of monochloroacetic acid. B. WYLAM, J. E. G. HARRIS, and J. THOMAS, Assrs. to SCOTTISH DYES, LTD. (U.S.P. 1,640,018, 23.8.27. Appl., 19.8.26. Conv., 5.8.25).—See E.P. 263,898; B., 1927, 183.

Manufacture of leuco-hydroxyanthraquinones. W. CARPMAEL. From I. G. FARBENIND A.-G. (E.P. 274,966, 29.4.26).—See G.P. 436,526; B., 1927, 470.

Manufacture of condensation products and dyes of the benzanthrone series [isodibenzanthrones]. I. G. FARBENIND A.-G., Assees. of A. LÜTTRINGHAUS, H. NERESHEIMER, and H. WOLFF (U.S.P. 1,633,866, 28.6.27. Appl., 16.10.25. Conv., 25.10.24).—See E.P. 255,277; B., 1926, 780.

Preparation of [3]-nitro-[4]-aminobenzoyl- α -benzoic acid and derivatives. NEWPORT CO. (E.P. 265,545, 20.10.26. Conv., 8.2.26).—See U.S.P. 1,614,584; B., 1927, 212.

Manufacture of perylene. C. H. MARSHALK (U.S.P. 1,639,658, 23.8.27. Appl., 13.12.23. Conv., 21.12.22).—See E.P. 208,722; B., 1924, 654.

Reaction product of sulphur chloride and primary arylamines. R. HERZ, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,637,023, 26.7.27. Appl., 31.8.21. Conv., 11.4.14).—See E.P. 17,417 of 1914; B., 1921, 619 A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination and significance of the solubility of artificial silks in alkali. W. WELTZIEN (Papier-Fabr., 1927, 25, Fest-u. Ausland-heft, 66—71).—Although in the determination of α -cellulose in wood-pulps the material is extracted with 17.5% sodium hydroxide, it is to be remembered that this concentration of alkali is selected on account of its relation to the viscose process, and not because it represents a maximum point of solubility of cellulose. Such maximum solubility exists at a concentration of 10% sodium hydroxide, and important differences in the chemical characteristics of the various types of artificial silk can be detected by exhaustive treatment with sodium hydroxide, preferably at 9% concentration. 0.5 g. of chopped artificial silk is treated in a glass-stoppered flask with 30 c.c. of the sodium hydroxide solution for about 3 hrs. at 18—19°. The liquor is filtered off through a Schott's glass filter-crucible (size of pores 3/5—7), and the residue is pressed hard. The latter is stirred up again with 10 c.c. of fresh soda, allowed to remain for 15 min., and again sucked dry. Afterwards it is washed and dried at 110—115°. The maximum solubility is found in all cases with 10% sodium hydroxide, but 9% affords a more highly-differentiated characterisation of the various types. Cuprammonium silk is the most resistant, with a maxi-

mum solubility of 30%; viscose, 40—50%, whilst nitro-silk is almost completely soluble in 10% sodium hydroxide. Finer differentiations between silks of the same type may be made by performing the extraction with sodium hydroxide of different concentrations in the neighbourhood of 10%. The size of the individual filament has an influence on the result, and particular attention must be paid to the temperature.

J. F. BRIGGS.

Digestion of beech wood with nitric acid. H. SUDA and H. SADLER (Papier-Fabr., 1927, 25, Fest-u. Ausland-heft, 93—97).—Trials have shown that the nitric acid process is very suitable for the preparation of cellulose from beech wood. This wood has a short fibre and relatively high density, and the usual method of cutting cross-grain chips gives poor penetration and short-fibred pulp. The best results are obtained by slicing the wood longitudinally into slices 1—2 mm. thick. The wood is steeped in nitric acid of the correct concentration at the ordinary temperature for 2—3 hrs. The acid is then drained off and heated up to the desired digestion temperature separately; it is next returned to the digester, which is immersed in a water-bath maintained at 79°. Digestion is complete in about 1 hr.; the acid is drained off, and the chips which retain their shape are washed with water. Disintegration only takes place during the next process, which consists in a treatment with hot 2% sodium hydroxide solution, by which the incrusting matters are dissolved and the cellulose is liberated. A modification of the process, due to Lifschütz, consists in treating the wood with a mixture of sulphuric and nitric acids, but this results in poorer yields and more highly modified cellulose as the concentration of sulphuric acid is increased. It is noted that in the course of the action of the nitric acid on the wood the temperature in the digester rises above that in the surrounding water-bath, due to an exothermic reaction, which must be controlled. The most favourable conditions are obtained with 15% nitric acid and a water-bath temperature of 79°; the time should not be prolonged beyond 90 min. to preserve yield and quality. The consumption of nitric acid is 28—29%. Cellulose of very high quality may be obtained by an after-treatment with 9% sodium hydroxide in the cold.

J. F. BRIGGS.

Action of strong sodium hydroxide on cellulose. P. WAENIG (Papier-Fabr., 1927, 25, Fest-u. Ausland-heft, 112—115).—If cellulose is saturated with twice its weight of strong caustic soda solution and stored it undergoes an "ageing" process whereby the viscosity of its solutions is progressively diminished and its solubility in sodium hydroxide solutions (so-called β -cellulose) progressively rises. This change in solubility does not go on indefinitely, but comes to an equilibrium after about 30 days at the ordinary temperature with 17.5% sodium hydroxide solution. Starting with a bleached sulphite pulp showing 82.5% of α -cellulose and 4.3% of β -cellulose, total immersion in 17.5% caustic soda produced only insignificant losses of α -cellulose in 115 days, whereas the same pulp, pressed out and allowed to "age," showed α -cellulose reduced to 57% after 30 days. At the same time the β -cellulose was correspondingly increased so that the sum of the α - and

β -celluloses remained practically constant. The difference between total immersion and "ageing" in the pressed condition can also be determined microscopically, the fibre being but little changed by total immersion, whereas the cellulose which had been "aged" broke down under slight pressure to a mass of fibrillae. Results of a similar order have also been recorded with cotton cellulose. The suggestion is that the cellulose fibre is not morphologically homogeneous, and the separation into soluble substance and fibrillae can also be partially effected by mechanical shocks (cf. Cross and Bevan, B., 1919, 7 A). In the case now investigated it would appear that small quantities of atmospheric oxygen play a determining part.

J. F. BRIGGS.

Recovery of solvents. BODEWIG.—See I.

Action of hæmatin on fabric. WOODROFFE and GILBERT.—See XV.

PATENTS.

Treatment of fibre. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,632,802, 21.6.27. Appl., 10.8.26).—Crude sulphite fibre is passed through a series of treating tanks in counter-current to a flow of water through a series of containers underneath the tanks, the water being circulated from each container to the corresponding tank. Sodium hydroxide solution is added to an intermediate tank of the series.

T. S. WHEELER.

Production of retted bagasse fibre. E. C. LAITHROP and T. B. MUNROE, Assrs. to DAHLBERG & Co., Inc. (U.S.P. 1,633,594, 28.6.27. Appl., 20.6.24).—Bagasse can be freed from material binding the fibres together by the action of a ferment of the mushroom type, *e.g.*, *Psilocybe*.

T. S. WHEELER.

Manufacture of textile material for spinning and other purposes. A. G. BLOXAM. From N. V. OCEIROOI MAATSCHAPPIJ "VÉDÉ" (E.P. 274,331, 8.11.26).—Crude fibrous vegetable materials such as straw, grasses, and especially agaves and coconut husks are opened suitable for spinning by successive treatment with a solution of a caustic alkali and a saponifiable substance such as a fatty acid or oil. *E.g.*, coir fibres are boiled for $\frac{1}{2}$ – $1\frac{1}{2}$ hrs. in a 14.5% solution of caustic soda containing 0.25% of copper sulphate and 0.1% of ammonium chloride (on the weight of raw material) as catalysts, and after removal of loose intercellular substances the residual fibrous material is washed with a weak lye, then dipped into molten stearic acid, and dried preferably at about 35°.

A. J. HALL.

Manufacture of textiles. C. DREYFUS, Assr. to CELANESE CORP. OF AMERICA (U.S.P. 1,634,613, 5.7.27. Appl., 22.12.21).—Cotton yarn is rendered resistant to water by impregnation with cellulose acetate.

T. S. WHEELER.

Manufacture of artificial textile threads. ERSTE BÖHMISCHE KUNSTSEIDENFABR. A.-G. (E.P. 258,582, 14.9.26. Conv., 17.9.25).—Hollow threads are produced by incorporating with any of the usual cellulose solutions, either before or during spinning, substances such as oils, fats, waxes, etc. which do not form bubbles of gas or vapour under the conditions of spinning and finishing, are insoluble in the cellulose solutions, and are in such a fine state of division that the spinning process is unaffected by their presence, these substances being

finally removed by suitable solvents. With viscose, substances soluble therein such as soaps from which free fatty acids are produced by the action of the precipitating bath, are also used, the acids being finally dissolved out.

B. P. RIDGE.

Manufacture of cellulose derivatives. H. DREYFUS (E.P. 275,286, 26.3.26).—Cellulose esters, especially acetates, of very high viscosity are submitted to a prolonged ripening or secondary treatment in the manner described in U.S.P. 1,217,722 (B., 1917, 451), but without permitting substantial degradation of the cellulose nucleus, until they become soluble in suitable solvents (*e.g.*, acetone–water or other mixtures characteristic of the stage to which the ripening is prolonged). Solutions of much lower viscosity than were obtainable in the earlier stages of ripening are formed, which yield films and threads of increased strength and toughness.

B. P. RIDGE.

Reducing the viscosity of nitrocellulose and other cellulose esters. S. DE V. SHIPLEY, Assr. to ATLAS POWDER Co. (U.S.P. 1,633,292, 21.6.27. Appl., 14.3.25).—Nitrocellulose is heated at 100° in water containing a buffer to maintain the suspension at pH 3.1–8.5.

T. S. WHEELER.

Process for reducing the viscosity characteristics of cellulose ethers. P. C. SEEL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,635,013, 5.7.27. Appl., 16.12.25; cf. F.P. 447,974; B., 1913, 420).—A solution of a cellulose ether in a mixture of benzene and alcohol is evaporated, the residue, which should have an acidity exceeding pH 5, being kept at 65° for several weeks.

T. S. WHEELER.

Reducing the viscosity of nitrocellulose solutions. E. C. PITMAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,636,319, 19.7.27. Appl., 11.8.21).—A soluble salt of a lower monobasic aliphatic acid, *e.g.*, sodium acetate (0.01–4%), is added to nitrocellulose solution, which is then kept at 25° for several days.

T. S. WHEELER.

Manufacture of cellulose acetate [soluble in acetone]. R. BAYBUTT and E. S. FARROW, JUN., Assrs. to EASTMAN KODAK Co. (U.S.P. 1,635,026, 5.7.27. Appl., 20.7.26).—Cellulose acetate powder soluble in chloroform is treated with 10% nitric acid solution at ordinary temperature for about 20 hrs.

T. S. WHEELER.

Production of fine artificial silk filaments. VEREINIGTE GLANZSTOFF-FABR. A.-G. (G.P. 440,227, 26.7.18. Addn. to G.P. 308,427; B., 1922, 807).—The precipitation bath consists of a saturated ammonium sulphate solution containing a little mineral acid.

L. A. COLES.

Drying alkali-cellulose. P. C. SEEL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,635,012, 5.7.27. Appl., 24.5.21).—Cellulose saturated with alkali solution for subsequent use in the manufacture of ethers is treated with hot air which has been passed over sodium hydroxide to remove carbon dioxide and dried, first with sulphuric acid and then by refrigeration, being finally heated.

T. S. WHEELER.

Cellulose ester composition. G. L. SCHWARTZ, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,633,683, 28.6.27. Appl., 15.6.23; cf. U.S.P. 1,558,299;

B., 1926, 66).—Glacial acetic acid is heated with coconut or similar oil to give mixed glycerides of acetic and higher fatty acids, which are of value as softeners for nitrocellulose.

T. S. WHEELER.

Fireproofed [cellulose ester] product. C. E. BURKE, Assr. to DU PONT VISCOLOID Co. (U.S.P.) 1,633,067, 21.6.27. Appl., 2.1.26).—Magnesium carbonate trihydrate is of value for fireproofing fabrics.

T. S. WHEELER.

Manufacture of solid cork articles etc. G. SCHELLHAAS (U.S.P. 1,637,877, 2.8.27. Appl., 11.5.26).—A binding medium mixed with the granular substance (*e.g.*, cork) is dried in the open air, the resulting mass being subjected to a pressure of 16,500 lb./sq. in. and heated to 130° without re-expansion on releasing the heat and pressure.

H. ROYAL-DAWSON.

Production of film-like bands from cellulose solutions and similar initial materials. E. CZAPEK and R. WEINGAND (U.S.P. 1,641,322, 6.9.27. Appl., 15.1.26. Conv., 15.10.21).—See E.P. 254,946; B., 1926, 738.

Medium for protection of fibres (E.P. 275,374).—See VI.

Artificial wood (U.S.P. 1,634,346).—See IX.

Antimony pentasulphide (U.S.P. 1,633,754).—See XIII.

Rubber-cellulose ester compositions (E.P. 274,968).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing union materials consisting of cotton and [viscose] artificial silk. W. ALTERHOFF (Textilber., 1927, 8, 795—796).—In dyeing cotton-viscose silk materials, level shades may be obtained if the grey material be scoured for $\frac{1}{2}$ hr. in a boiling bath containing 1 g. of soda ash and 0.5 g. of Monopole soap per litre, and then entered into a boiling liquor containing 20% of Glauber's salt and 1 g. per litre of a soluble oil (*e.g.*, Monopole Brilliant oil or Avirol KM), the dye being added after $\frac{1}{4}$ hr. and dyeing continued for $\frac{3}{4}$ —1 hr. at the boil and $\frac{1}{2}$ hr. in the cooling dye bath. The dyed material is then washed and soured in a warm bath containing acetic or formic acid, a small quantity of a suitable oil being added if a soft handle be desired.

A. J. HALL.

Dyeing wool and silk with azo dyes produced on the fibre. E. T. ORLOFF (Textilber., 1927, 8, 794—795).—Insoluble azo dyes are satisfactorily applied to animal fibres by the following modification of the method used for dyeing cotton. The textile material is impregnated with an acid solution containing the base and a sulphonic acid derivative of the naphthol, and then passed through a 2% solution of sodium nitrite for development of the colour. *E.g.*, 72 g. of β -naphthol are dissolved in 110 g. of sulphuric acid (*d* 1.84) at room temperature, 75 g. of *p*-toluidine added, and the resulting crystalline magma is separated and dissolved in hot water, sodium acetate being then added. Wool or silk is impregnated with the resulting solution, passed through a 2% solution of sodium nitrite, washed with water and also with a solu-

tion of an ammonium salt, and dried, the resulting shade being level and fast.

A. J. HALL.

Influence of tension and conditions of coagulation on the dyeing properties of viscose silk. A. LEHNER and A. JÄGER (Kunstseide, 1927, 9, 219—221, 264—266).—The dyeing and elastic properties of viscose threads are considerably affected by stretching during their coagulation immediately after spinning, and uniform conditions of spinning are essential for the manufacture of viscose silk of a uniform grade. Viscose threads subjected to tension while passing through the usual coagulating bath have less affinity than unstretched threads for direct (Triazole Blue 4B) and vat dyes (Indanthrene Blue BCS), but a greater affinity for basic dyes (Methyl Violet B), this variation of dyeing property being also dependent on the distance from the squirting orifice at which the stretching is applied. After complete conversion of the viscose into cellulose threads, stretching has no appreciable effect. The length of viscose thread immersed in the coagulating bath during the manufacture of viscose silk affects its dyeing properties.

A. J. HALL.

Identification of vat dyes on cotton. M. VAIS (Textilber., 1927, 8, 611—619).—Tables are given of the reactions of about 400 vat dyes towards sodium hyposulphite, cold and hot caustic soda (*d* 1.384), concentrated sulphuric acid, concentrated nitric acid, benzol, and pyridine.

A. J. HALL.

PATENTS.

Dyeing cotton with vat and azo dyes. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 252,384, 20.5.26. Conv., 20.5.25).—Cotton is padded with a coupling component and a leuco-vat dye; the latter is oxidised during coupling with a diazo compound. Leuco-Algol Blue 3G and bisacetoacetylolidine, developed with diazotised 2:5-dichloroaniline or *o*-chloroaniline, give a green; leuco-Algol Blue 3G and 2:3-hydroxynaphthoic 2:5-dimethoxyanilide, developed with diazotised 2:5-dichloroaniline, a brown; leuco-Algol Brilliant Violet R and 2:3-hydroxynaphthoic *m*-nitroanilide, developed with diazotised 4-nitro-*o*-anisidine, a blue-red.

C. HOLLINS.

Process of dyeing silk. E. L. MAUPAI (U.S.P. 1,635,761, 12.7.27. Appl., 1.2.26).—The shades obtained on silk by dyeing with a chromogen, autochrome, or indigosol dye, followed by treatment with a metallic salt, *e.g.*, potassium dichromate, can be rendered fast to boiling soap solution by immediate immersion in a boiling soap solution.

T. S. WHEELER.

Dyeing animal fibres. DURAND & HUGUENIN A.-G. (E.P. 255,501, 20.7.26. Conv., 20.7.25).—Certain acid dyes, including the water-soluble ester salts of vat dyes, which do not yield shades on wool fast to rubbing, give satisfactory results in this respect if dyeing is effected in the presence of fuller's earth, due allowance being made for the alkalinity of this assistant. Improved dyeings are thus obtained with Diamond Black F and indigo, if about 20 g. of fuller's earth per litre of dye liquor are used.

A. J. HALL.

Dyeing animal fibres [with indigosols]. DURAND & HUGUENIN A.-G. (E.P. 267,986, 22.3.27. Conv.,

22.3.26. Addn. to E.P. 255,501; cf. preceding abstract).—The process of the prior patent is modified by adding the fuller's earth to the oxidising bath instead of (or as well as) to the dyeing bath. C. HOLLINS.

Method and apparatus for [spot] dyeing [yarn]. ECLIPSE TEXTILE DEVICES, INC., Assees. of H. E. VAN NESS (E.P. 267,548, 11.3.27. Conv., 11.3.26).—Cross-wound bobbins of yarn are dyed in one or more pre-determined parts (spot dyeing) by utilising centrifugal force to cause dye liquor to pass rapidly through limited portions of the yarn. Each bobbin is rotated about a vertical or horizontal axis at 2000 r.p.m., and dye liquor delivered to the centre of the cone is suitably distributed by a spaced perforated cone closely fitting therein, so that the dye liquor is flung outwards through the yarn in layers or zones. Alternatively, internal cones are dispensed with, the distribution of the dye liquor being effected by suitable delivery of the dye liquor. A. J. HALL.

Dyeing cellulose acetate silk. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (E.P. 275,307, 1.5.26).—An aminonaphthoic acid is used as diazo or coupling component for azo dyes for acetate silk either in substance or on the fibre. Examples are: 4-nitro-*o*-anisidine \rightarrow 2:3-aminonaphthoic acid (red-violet); 2:6-aminonaphthoic acid \rightarrow 2:3-hydroxynaphthoic acid \rightarrow β -naphthol (ruby); 2:3-aminonaphthoic acid \rightarrow β -naphthylamine (brown); 2:6-dimethoxyaniline \rightarrow 2:6-aminonaphthoic acid (yellow). 33 other couplings are mentioned. 1-Amino-2-ethoxy-6-naphthoic acid is prepared from 2:6-hydroxynaphthoic acid by ethylation, nitration, and reduction. C. HOLLINS.

Dyeing cellulose esters and ethers. BRITISH DYESTUFFS CORP., LTD., R. S. HORSFALL, L. G. LAWRIE, and J. HILL (E.P. 275,373, 11.6.26).—Acetate silk etc. is dyed with azo dyes prepared by coupling a diazotised arylaminesulphonarylamide with an arylamine or an unsulphonated derivative thereof. *m*-Aminobenzenesulphonanilide \rightarrow α -naphthylamine gives an orange; *m*-aminobenzenesulphon-*m*-nitroanilide \rightarrow cresidine, an orange-red; α -naphthylamine-5-sulphonanilide \rightarrow cresidine, a deep yellow. The arylamides are obtained from nitro- or acylamino-arylsulphonyl chlorides and arylamines, with subsequent reduction or hydrolysis. C. HOLLINS.

Dyeing viscose filaments and films. W. MENDEL, Assr. to S. A. NEIDICH (U.S.P. 1,633,220—1, 21.6.27. Appl., 23.5.25).—(A) Coagulated viscose is heated in 3% aqueous sodium phosphate solution at 75° for 2 hrs., washed, and dyed in any usual manner. (B) The dye is added to the sodium phosphate solution. T. S. WHEELER.

Manufacture of a medium for the protection of fibrous materials during the chemical treatment thereof. V. HABERKORN (E.P. 275,374, 11.6.26).—A solution of wool waste in aqueous sodium hydroxide is boiled and then made slightly acid. A protective medium is obtained, in solution or in solid form according to the proportions used, which when added to an acid dye-bath ensures level dyeing and prevents attack of the fibre. Dyeing and chroming are also accelerated. C. HOLLINS.

Treatment of fibrous materials and textiles. [Use of wetting-out agents in oiling, sizing, etc.] I. G. FARBENIND. A.-G. (E.P. 246,468, 18.1.26. Conv., 21.1.25).—Aromatic sulphonic acids (or their salts) which have strong wetting-out action, particularly propyl- and butyl-naphthalenesulphonic acids, benzylsulphanilic acid, sulphonated formaldehyde-phenol or formaldehyde-naphthalene condensation products, etc., are added to the liquors used in the oiling, sizing, or similar treatment of textiles etc. C. HOLLINS.

Ornamentation of artificial silk fabrics. CALICO PRINTERS' ASSOC., LTD., and J. R. WHINFIELD (E.P. 275,357, 25.5.26).—Acetate silk is printed with suitably thickened delustring salts (aniline hydrochloride, sodium phenoxide, etc.) and then treated with dilute alkali or acid to decompose the salt, a matt pattern on a lustrous ground being produced. By using slight excess of alkali with an acid delustring agent (phenol) partial hydrolysis results and the matt pattern may be dyed with direct dyes. For lustrous patterns on a matt ground the fabric may be first printed with a suitable resist, *e.g.*, a paste of zinc oxide, and then padded with the delustring agent; dye may be added to the resist paste, or free alkali may be used and the lustrous, partially hydrolysed pattern may be subsequently dyed. C. HOLLINS.

Mercerising or otherwise treating with caustic alkalis textile materials containing viscose artificial silk. SILVER SPRINGS BLEACHING & DYEING CO., LTD., F. E. MASON, and A. J. HALL (E.P. 274,266, 2.7.26).—Cotton materials containing viscose silk which are to be mercerised are manufactured with non-desulphurised silk, since this is more resistant than desulphurised silk to caustic soda. After mercerisation the material is subjected to the usual processes for desulphurising viscose silk. A. J. HALL.

Treatment of cotton or other threads containing cellulose. G. TAGLIANI, Assr. to CHEMICAL WORKS (FORMERLY SANDOZ) (U.S.P. 1,633,617, 28.6.27. Appl., 29.6.25. Conv., 3.7.24: cf. E.P. 233,704; B., 1925, 956).—Cotton which has been substantively dyed is rendered indifferent to the further action of such dyes by treatment first with alcoholic sodium hydroxide solution and then with *p*-toluenesulphonyl chloride dissolved in carbon tetrachloride. T. S. WHEELER.

Manufacture of finished [regenerated cellulose] fabric. J. BENNETT, Assr. to CONSOLIDATED TEXTILE CORP. (U.S.P. 1,633,160, 21.6.27. Appl., 26.8.26).—The lustre of regenerated cellulose fibres can be removed by treatment with sodium hydroxide solution, patterns being obtained by using a resist paste. T. S. WHEELER.

Process of finishing fabrics [viscose]. W. WITTE, Assr. to U.S. FINISHING CO. (U.S.P. 1,633,152, 21.6.27. Appl., 29.6.26).—Viscose silk is delustrated by treatment with alcoholic sodium hydroxide solution, followed by immersion in a solution of *p*-toluenesulphonyl chloride in carbon tetrachloride. The process can be adapted to printing purposes. T. S. WHEELER.

Dyeing. I. G. FARBENIND. A.-G., Assees. of W. HERZBERG and H. OHLENDORF (U.S.P. 1,640,657, 30.8.27. Appl., 4.10.26. Conv., 9.6.25).—See E.P. 253,488; B., 1927, 551.

Anthraquinone dyes and dyeing (E.P. 275,421).—See IV.

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

Alkali chloride industry. J. BILLITER (Z. Elektrochem., 1927, 33, 353–360).—A lecture.

Steels for ammonia synthesis. VANICK.—See X.

Electrolysis of water. SESTINI.—See XI.

Ammonium sulphate and soil acidity. HAGEN.—See XVI.

Determination of sulphuric acid by means of benzidine. HAASE.—See XXIII.

PATENTS.

Production of sulphurous acid for the manufacture of sulphite solutions. A.-G. FÜR ZELLSTOFF- U. PAPIERFABR. (G.P. 440,337, 22.9.25).—Finely-ground pyrites or the like is injected into a combustion chamber wherein it is burnt in a regulated supply of primary or secondary air. L. A. COLES.

Production of hydrated lime. W. K. HUNTER, ASSR. to M. F. NICHOLS (U.S.P. 1,634,424, 5.7.27. Appl., 22.7.25).—Lime is discharged from the kiln without cooling into an excess of water. T. S. WHEELER.

Production of solid ammonium carbonate from its components. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (E.P. 275,459, 20.11.26. Addn. to E.P. 262,408; B., 1927, 652).—Ammonium carbonate produced from gaseous ammonia and carbon dioxide is improved in quality and hardness if the temperature within the lead or aluminium reaction vessel is kept below 60°, preferably 35–45°, either by adjusting the amounts of the reagents so that the heat of formation maintains the desired temperature or by artificial cooling or external heat. The gases may be introduced under pressure, in which case the temperature is maintained at 60–90°. W. G. CAREY.

Purification of alkali cyanide solutions containing sulphides. I. G. FARBENIND. A.-G., Assees. of K. HERR-DEGEN (U.S.P. 1,633,485, 21.6.27. Appl., 27.11.26. Conv., 4.12.25).—Crude cyanide solutions, obtained in the Bucher process, can be freed from sulphides by treatment with bismuth hydroxide. T. S. WHEELER.

Production of potassium nitrate. PREUSSISCHE BERGWERKS- U. HÜTTEN-A.-G., ABTG. SALZ- U. BRAUNKOHLENWERKE (F.P. 616,286, 17.5.26. Conv., 16.6.25).—Double decomposition of a crude potassium salt, e.g., the chloride, and sodium nitrate is effected in the presence of saturated potassium nitrate solution. L. A. COLES.

Production of barium oxide. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (G.P. 440,382, 1.5.23).—A mixture of barium carbonate and carbon packed in containers constructed of paper etc. is heated strongly. L. A. COLES.

Manufacture of precipitated barium carbonate and barium sulphhydrate. J. B. PIERCE, JUN. (U.S.P. 1,634,338, 5.7.27. Appl., 18.11.24).—Barium sulphide solution is treated with pure carbon dioxide at 80° until

one half of the barium present has been precipitated as barium carbonate. The resulting barium hydrogen sulphide solution is concentrated. T. S. WHEELER.

Process of refining barytes. W. J. O'BRIEN (U.S.P. 1,633,347, 21.6.27. Appl., 17.9.25).—Barytes ore is extracted with fuming sulphuric acid, dissolved barium sulphate being precipitated by volatilising sulphur trioxide from the solution. T. S. WHEELER.

[Calcining] treatment of [finely-divided] carbonate materials. A. E. WHITE. From DWIGHT AND LLOYD METALLURGICAL CO. (E.P. 275,463, 26.11.26).—Limestone, dolomite, etc., when in small particles, is calcined by spreading a graded layer, with the larger particles in the upper portion, upon a pervious support and passing highly heated gases through the layer from the top downwards until the carbonate is dissociated, maintaining an excess of heated gases above the layer and removing the gases from below, together with the carbon dioxide as it is formed. The material is separated from the pervious support by a thin layer of previously treated similar material, and may be heated more quickly by admixing solid fuel insufficient for complete calcination. The process may be carried on while the material is being transported bodily through a treatment zone. W. G. CAREY.

Production of sulphites. BRADLEY-McKEEFE CORP., Assees. of L. BRADLEY and E. P. McKEEFE (Can. P. 263,180, 25.8.24).—Dolomitic limestone or lime is converted into calcium and magnesium bisulphites by treatment with sulphur dioxide, and the product is treated with sodium sulphate in the presence of excess sulphur dioxide. L. A. COLES.

Manufacture of hypochlorites. R. B. MACMULLIN, ASSR. to MATHIESON ALKALI WORKS (U.S.P. 1,632,483—5, 14.6.27. Appl., [A, B], 22.8.25; [C], 23.9.25; cf. E.P. 195,366; B., 1924, 173).—(A) Aqueous hypochlorous acid is treated with a tertiary alcohol, e.g., *tert.*-amyl alcohol, the corresponding alkyl hypochlorite which separates being dissolved in a liquid in which water is also soluble, e.g., amyl alcohol, and treated with a limited amount of water and calcium oxide, to form solid calcium hypochlorite with regeneration of the alcohol. The alkyl hypochlorite can also be formed directly by passing chlorine into a solution of sodium hydrogen carbonate in presence of the alcohol. (B) A solution of pure calcium hypochlorite is treated with excess of calcium oxide to precipitate basic calcium hypochlorite. (C) A solution of pure calcium hypochlorite can be obtained by treating a *tert.*-alkyl hypochlorite prepared as in (A) with water and a limited quantity of calcium oxide. T. S. WHEELER.

Production of hyposulphites by the action of amalgams on bisulphite solutions. I. G. FARBENIND. A.-G., Assees. of F. BENCKER (G.P. 440,043, 4.3.21).—Bisulphite solutions are treated with an amalgam and a quantity of free acid equivalent to the amalgam. L. A. COLES.

Production of solid sodium bisulphite or sulphite. CHEM. FABR. KALK G.M.B.H., and H. OEHME (G.P. 440,380, 17.2.25).—Pure sodium bicarbonate, formed by treating sodium carbonate with carbon dioxide liberated

during the process, is treated with sulphur dioxide in the presence of saturated sodium bisulphite solution.

L. A. COLES.

Production of anhydrous sodium bisulphite. N. A. LAURY (U.S.P. 1,638,492, 9.8.27. Appl., 16.8.23).—Crude sodium carbonate is subjected to the action of sulphur dioxide in the presence of a trace of moisture.

H. ROYAL-DAWSON.

Treatment of aluminous ores. R. MOLDENKE (U.S.P. 1,637,451, 2.8.27. Appl., 9.7.25).—Part of the aluminium contained in the ore (alunite) is converted into potash alum, which is separated, and the remainder into an ammonium aluminium alum, both products being then treated with ammonia solution to form aluminium hydroxide.

H. ROYAL-DAWSON.

Production of aluminium sulphate. GEN. CHEMICAL CO., ASSEES. OF R. M. MEIKLEJOHN (Can. P. 263,596, 2.10.25).—A mixture of aluminous material with a quantity of sulphuric acid greater than 1/1.3 of the water content of the mixture is heated above 160°.

L. A. COLES.

Production of aluminium fluoride. E. TEISLER (Can. P. 263,352, 20.5.25).—Finely-divided clay or other material containing silica in addition to alumina is treated with hydrofluoric acid, and the aluminium fluosilicate formed is converted into aluminium fluoride by treatment with material containing aluminium oxide or hydroxide.

L. A. COLES.

Preparation of magnesium chromates. G. KRÄNZLEIN and A. VOSS, ASSRS. TO GRASSELLI DYESTUFF CORP. (U.S.P. 1,632,299, 14.6.27. Appl., 3.6.26. Conv., 22.6.25).—Magnesium chromate can be readily prepared by precipitating magnesium sulphate solution with calcium chromate at the b.p. for 6–8 hrs., collecting the precipitated calcium sulphate, and evaporating the filtrate to separate the magnesium salt. Magnesium dichromate, owing to its great solubility, can be obtained from the interaction of magnesium chloride and sodium dichromate solutions in the cold, the requisite evaporation being carried out *in vacuo*.

T. S. WHEELER.

Production of phosphorus nitride and volatile halides from raw materials. C. G. MINER (U.S.P. 1,634,795–6, 5.7.27. Appl., 25.7.25).—Phosphate rock is treated with sodium chloride at 1300° to give phosphorus pentachloride, which is passed at 500° over aluminium nitride, prepared by heating a mixture of bauxite and carbon at 1500° in an atmosphere containing a hydrocarbon and nitrogen. Phosphorus nitride, of value as a fertiliser, and aluminium chloride are obtained. Silicon nitride reacts similarly to give silicon tetrachloride.

T. S. WHEELER.

Separation of nitrosyl chloride from chlorine. W. DOMINIK (G.P. 440,334, 23.8.25. Conv., 22.12.24).—A mixture of nitrosyl chloride with chlorine is brought in contact with nitric acid of *d* not less than 1.332, at 20–30°, whereby the nitrosyl chloride liquefies without the chlorine entering into solution.

L. A. COLES.

Production of ammonium chloride and alkali sulphate. CONTINENTALE A.-G. FÜR CHEMIE, ASSEES. OF O. GERNGROSS (U.S.P. 1,634,870, 5.7.27. Appl., 22.5.25. Conv., 11.6.24).—See E.P. 235,552; B., 1925, 714.

Manufacture of alkali sulphides. H. FREEMAN, ASSR. TO CANADA CARBIDE CO. (U.S.P. 1,640,314–5, 23.8.27. Appl. [A], 14.2.23, [B], 30.6.24).—See E.P. 211,488; B., 1925, 630.

Manufacture of pure barium carbonate. F. FALCO (U.S.P. 1,640,652, 30.8.27. Appl., 8.9.26. Conv., 27.11.25).—See E.P. 270,559; B., 1927, 522.

Manufacture of lead monoxide by a wet method. J. J. TARDAN (E.P. 265,190, 21.1.27. Conv., 30.1.26).—See F.P. 611,810; B., 1927, 389.

Recovery of oxides of nitrogen in sulphuric acid manufacture. A. M. FAIRLIE (Re-issue 16,709, 23.8.27, of U.S.P. 1,420,477, 20.6.22).—See B., 1922, 630 A.

Pulverising machine (U.S.P. 1,633,164).—See I.

Recovery of copper, zinc, and other metals from solutions containing chlorides and sulphates (E.P. 276,200).—See X.

Electrolysis of water (E.P. 275,785 and G.P. 440,335).—See XI.

Lead compounds of chromic acids (U.S.P. 1,633,948). **Azides** (G.P. 440,568).—See XIII.

VIII.—GLASS; CERAMICS.

Ancient glass. II. B. NEUMANN [with M. RUPPRECHT] (Z. angew. Chem., 1927, 40, 963–967; cf. Neumann and Kotyga, B., 1925, 992).—In continuation of earlier work on ancient glasses analyses are given of two Egyptian samples from Thebes (B.C. 1500), eight mosaic cubes from Salona (200 A.D.), and five glasses from Samarra (850 A.D.). The Egyptian glasses contained 67–62% of silica, 16–20% of alkali oxides, 4–5% of lime, 2–4% of magnesia, 4–1.5% of alumina, and 1–2% of ferrous oxide, and were coloured dark blue with cupric and manganous oxides; it is suggested that these “dolomitic” glasses high in alumina are characteristic of Egyptian work. The Roman glass cubes resembled in composition those already described in the earlier work, but the Mesopotamian samples were of an entirely distinct type, being characterised by a high magnesia and a relatively low alkali content. The high degree of transparency of these glasses combined with the high “dolomite” content (9–11% CaO + MgO) shows that a high temperature must have been used in making them. The Egyptian and Mesopotamian glasses have a softening point about 100° higher than that of the Roman and Rhenish ancient glasses.

A. R. POWELL.

Whiteware bodies developed at the Ohio State University. A. S. WATTS (J. Amer. Ceram. Soc., 1927, 10, 148–149).—The compositions of bodies suitable for statuary, electrical, hotel, and chemical porcelain and compounded mainly from American raw materials, together with glaze formulæ, are given. In bodies containing a maximum of 15% of ball clay, 50% of total clay, and 24% of feldspar, the best colour is produced by the addition of not more than 2.25% of calcium and magnesium carbonates in the proportion of 60% MgCO₃ and 40% CaCO₃.

F. SALT.

Sheet steel cooking ware white enamels economically suitable for use in China. CHI-FANG LAI

(J. Amer. Ceram. Soc., 1927, 10, 150—162).—Two typical ground coats, one high in felspar and cobalt oxide, the other high in quartz and cryolite, and four cover coats were studied. A number of opacifiers were substituted systematically for tin oxide. Test pieces were fired in a simple oil-fired muffle furnace. The effect of varying the composition was more marked in the case of the cover coats than with the ground coats. The cheapest enamels, resembling in quality those made with tin oxide, were obtained with sodium antimonate and calcined alumina. Zirconium oxide increased the resistance to thermal shock and to acids, whilst sodium antimonate increased the impact strength. The best cover coat from the point of view of economy had the refractory portion of base enamel high in felspar, low in quartz, and contained a medium amount of cryolite. Consistently good results were obtained with a coat in which felspar was the only refractory and which contained no cryolite. The gloss was considerably improved, but the opacity impaired, by a high zinc oxide content.

F. SALT.

Effects of composition on the properties of ground-coat enamels for sheet steel. W. N. HARRISON and H. G. WOLFRAM (J. Amer. Ceram. Soc., 1927, 10, 163—179).—Three series of ground coats, 20 in each series, were prepared on three refractory bases, one consisting of 33.5 pts. of felspar and 25 pts. of flint, another of felspar only, and a third of flint only. A fixed addition containing 0.5 pt. of cobalt oxide, 1 pt. of manganese oxide, and 5 pts. each of sodium oxide and boric oxide was also made to each of the enamels. In each case the refractory base and the fixed addition were present in the proportion of 58.5% and 11.5%, respectively, making a total constant portion of 70%. The remaining variable portion (30%) was made up of equal parts of three constituents taken from six fluxes commonly used in enamels. Two standard white cover coats were applied to each of the ground coats, and the enamels were tested for resistance to mechanical and thermal shock. A high felspar content in ground coats produced greatly decreased resistance to mechanical and thermal shock. On the other hand, a high flint content slightly impaired the resistance to thermal shock but greatly improved the resistance to mechanical shock. In the variable portion of the ground coats, sodium oxide produced the most favourable results under the tests. A high boric oxide content was also effective in ground coats containing both felspar and flint. The effect of expansivity on resistance to shock was obscured by other factors.

F. SALT.

Variations in softening points of refractory materials due to rate of heating. S. S. COLE (J. Amer. Ceram. Soc., 1927, 10, 180—184).—Softening point tests were carried out on a number of refractory materials, including fire clay, silica, and silica cements, in a gas-fired furnace. The rate of heating up to cone 28 was varied from 1 to 5 hrs. Long heating did not appreciably lower the softening point of fire clays. With silica bricks the 1½ hr. rate gave the highest results; longer heating reduced the softening point. Results with silica cements were erratic. Variations in softening temperature were reduced by fine grinding and intimate mixing of the materials; the purer the material the less the softening point varied.

F. SALT.

PATENTS.

Manufacture of glass containing barium. RHE-NANIA-KUNHEIM VER. CHEM. FABR. A.-G. (E.P. 259,953, 8.10.26. Conv., 16.10.25).—Barium silicate is employed, in conjunction with the usual batch materials (sand, soda ash, lime-spar), in preparing glasses containing barium. The silicate is obtained from heavy spar by the method described in E.P. 242,996 (cf. B., 1926, 787).

A. COUSEN.

Production of opaque, readily-fusible glass or enamel. PATENT-TREUHAND-GES. F. ELEKTR. GLÜH-LAMPEN M.B.H., Assees. of F. SKAUPY, F. SPÄTE, and H. NACHOD (G.P. 440,298, 5.12.24).—Insoluble compounds of boron or silicon with other metalloids or with acids, such as boron nitride or phosphate, or the corresponding silicon compounds, are added to the glass flux.

L. A. COLES.

Manufacture of an abrasive article. L. H. MILLIGAN and C. H. QUICK, Assrs. to NORTON Co. (U.S.P. 1,635,981, 12.7.27. Appl., 4.9.25).—Granulated alumina is mixed with fusible clay and shaped, the article being then coated with a mixture of ground felspar (82%), plastic ball clay (15%), and lime (3%), and fired.

T. S. WHEELER.

Production of silica articles. BRITISH THOMSON-HOUSTON Co., LTD., Assees. of H. L. WATSON (E.P. 268,780, 29.3.27. Conv., 1.4.26. Cf. E.P. 230,499; B., 1925, 850).—Silica is heated to plasticity by means of imbedded electric resistances around a core of refractory material. The plastic silica is withdrawn through a tubular die of refractory material, the rate of withdrawal determining the nature of the product. Unused silica is excluded from the die, and the product is protected from the heat radiated from the fusion zone.

B. W. CLARKE.

Luminous enamels. P. F. JOOSS and H. E. FICHE-ROULLE (F.P. 610,454, 9.5.25).—The metal surface to be enamelled is coated with a deoxidising layer, then with a mixture of lead-free flux and unground phosphorescent zinc sulphide, with or without a small quantity of radioactive substance. The article is then fired rapidly until the flux just melts. The process is applicable to the enamelling of glass and ceramic products.

A. R. POWELL.

Enamelling sheet iron. C. TOTOT-GIBARU (F.P. 610,645, 5.2.26).—The cleaned sheets are heated at 420° and covered with an enamel containing fluorine, boric acid, zinc oxide, and antimonious oxide in a lead-free flux containing silica, alumina, lime, and alkali. Subsequently a second layer of enamel similar to the first is applied, but containing stannic oxide and pigments such as ferric, manganous, or cobalt oxide in place of the antimony oxide of the first layer. For sanitary ware a second layer similar to the first may be used instead of the coloured layer.

A. R. POWELL.

Eye-protective glass. W. W. COBLENTZ, Assr. to SEC. COMMERCE OF UNITED STATES (U.S.P. 1,637,439, 2.8.27. Appl., 10.6.26).—The glass contains the usual constituents and sufficient cobalt oxide to give a blue colour equivalent to blue 20 + 8 on the Lovibond scale and sufficient cerium oxide to stop ultra-violet rays.

H. ROYAL-DAWSON.

Paving bricks etc. (E.P. 275,104). Enamelling cement (F.P. 610,956).—See IX.

IX.—BUILDING MATERIALS.

Fire-resistant construction. R. E. STRADLING and F. L. BRADY (Dept. Sci. Ind. Res., Building Res., Spec. Rep. No. 8, 1927. 57 pp.).—Metals do not resist the effects of fire owing to the loss in strength which occurs at high temperatures. Timber becomes a good fire resister, provided the wood is of large enough dimensions and free access of air is prevented. Natural crystalline rocks, such as granites, as well as limestones, break down suddenly and completely at high temperatures. Sandstone does not lose so much strength, but is very liable to crack owing to shrinkage. Burnt clay products including terra-cotta are in general good fire resisters, although failure may occur if quartz or silica is present. Lime is likely to cause disintegration in any structure owing to the dehydration of the calcium hydroxide, followed by rehydration and expansion on exposure to air and water. Lime-clinker mortars are more resistant than lime-sand mortars, owing to part of the lime being chemically combined. Similarly, concrete is liable to fail owing to its free lime content. Concrete reinforced near the surface is liable to fail owing to the expansion of the metal with subsequent spalling of the concrete. For a fire-resistant concrete an aggregate which will enter into chemical combination with the free lime is necessary. Pozzolanic materials, *e.g.*, granulated slag, spent shale, baked clay, etc., are best for this purpose, with a fine aggregate composed of red brick or dolerite. Portland blast-furnace cement is very suitable for fire-resistant construction. Concrete of this type has the advantage of increasing in strength upon exposure to water after exposure to fire.

B. W. CLARKE.

Analysis of cements. G. GALLO (Giorn. Chim. Ind. Appl., 1927, 9, 313–317).—From analyses of cements of known composition before and after setting it is concluded that Kriege's method (*cf.* B., 1924, 598) for analysis of cements is inaccurate; an alternative method which is thought to be more satisfactory is described.

R. W. LUNT.

Distinguishing green from old wood. G. FRON (Ann. Falsif., 1927, 20, 386–391).—10 g. of the dried wood, cut into strips 2–3 mm. thick, are boiled with 80 c.c. of water for 10 min. and the hydrogen-ion concentration of the cooled extract is determined colorimetrically with Methyl Red. The method is not suitable for woods such as oak or chestnut rich in tannin, owing to the deep colour of the extract. Green woods and those which have been artificially dried by heat show p_H values between 5.2 and 6.5 according to their nature. The naturally dried woods have higher acidities, the p_H varying between 4.4 and 5.2, and similar values are shown by woods artificially aged by ozonised air.

F. R. ENNOS.

Pigments for mortar. FOSTER.—See XIII.

PATENTS.

Manufacture of paving bricks and other ceramic products. BARTON FOREIGN PATENTS LTD. From G. D. COTTON (E.P. 275,104, 19.2.27).—The bricks are

fired in a continuous kiln which is arranged so that, after passing through a vitrifying zone, the bricks are subjected to a primary devitrifying treatment and then heated again in an annealing zone before reaching the final cooling chamber. The bricks are rendered less brittle, and development of cracks is prevented.

B. W. CLARKE.

Manufacture of artificial marble. G. BIA and J. E. D. DE G. DE BIELIZE (E.P. 275,448, 26.10.26).—A solution containing zinc and magnesium sulphates in definite proportions is treated in a colloid mill with vaseline oil and phenol-formaldehyde condensation products. The viscous colloidal product is made slightly acid with sulphuric acid and added to a mixture of magnesia with water and a suitable filler, such as barium sulphate.

B. W. CLARKE.

Manufacture of bituminous concrete. L. S. VAN WESTRUM (E.P. 275,364, 31.5.26. Addn. to E.P. 269,975; B., 1927, 484).—A cementitious powder, consisting of a mixture of a true cement with stone or slag flour, with or without the addition of oxide of iron, is mixed with a mineral aggregate and with a bituminous soap prepared by saponifying a mixture of bituminous material and a vegetable oil.

B. W. CLARKE.

Asphaltic paving composition. C. N. FORREST, ASST. to BARBER ASPHALT Co. (U.S.P. 1,634,241, 28.6.27. Appl., 22.4.25).—Sodium acetate (1–5%) toughens and stabilises asphaltic paving compositions.

T. S. WHEELER.

Enamelling articles made of fibrous cement. SOC. CO-OP. EMO (F.P. 610,956, 4.1.26. Conv., 19.12.25).—The well-dried article from which all grease has been removed is covered with a thick paste made by mixing stannic oxide, cryolite, and a pigment with powdered flux and water, and, after drying, the article is heated until the flux melts. The flux is made by melting together silica, red lead, borax, and nitre, with subsequent grinding to an impalpable powder.

A. R. POWELL.

Manufacture of artificial wood. T. SURDI (U.S.P. 1,634,346, 5.7.27. Appl., 12.10.25. Conv., 20.12.24).—Vegetable fibres are dried and fireproofed, being then mixed with a binder, and compressed and heated in moulds.

T. S. WHEELER.

Preservation of wood. E. PLANK (G.P. 439,523, 6.11.25).—The wood is placed in a solution of lead nitrate (10%) contained in a pressure vessel fitted with an insulating lining having apertures for electrodes of lead and arsenic. The pressure is increased up to 10 atm. and the solution heated electrically to the b.p. by means of an alternating current. After some time the pressure is released, a protective colloid being obtained from the wood. An induction coil is then inserted into the circuit, and a colloidal solution of lead and arsenic formed in the bath by electrical dispersion. The pressure is finally again increased to 10 atm. and the temperature raised to 100°, a colloidal layer being so produced on the outer surface of the wood and on the walls of the capillaries. This layer, which is very difficult to remove by washing, forms an active protection against destructive agents. The process at the same time artificially ages the wood.

A. B. MANNING.

Material for the manufacture of artificial stone and similar products. R. SCHOENHOEFER (U.S.P. 1,633,132, 21.6.27. Appl., 28.4.24. Conv., 30.9.20).—See E.P. 235,257; B., 1925, 633.

Composition for roofing, road surfacing and other purposes. L. C. LUTYENS and R. O. CHILD (U.S.P. 1,641,637, 6.9.27. Appl., 6.4.25. Conv., 11.4.24).—See E.P. 238,586; B., 1925, 851.

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

High-frequency induction melting. D. F. CAMPBELL (Iron and Steel Inst., Sept., 1927. Advance copy. 9 pp.).—One special advantage in melting metal under high-frequency conditions is the absence of a complicated form of bath necessary in low-frequency melting. Metal may be melted in a crucible supported externally by heat-resisting sand, and, as the heating is not conducted through the crucible walls, maintenance cost of refractory materials is not great. The Ajax-Northrup furnace is described (cf. B., 1927, 683), the method being of particular interest in preparing small quantities of alloy steels, under crucible conditions, for the casting of small parts, *e.g.*, motor-car parts. High-frequency generators of 600 kv.a. are now in regular operation under economic commercial conditions. C. A. KING.

Effect of varying ash in the coke on blast-furnace working. C. S. GILL (Iron and Steel Inst., Sept., 1927. Advance copy. 7 pp.).—Fluctuations in the quality of iron from cast to cast were found to follow closely the varying ash content in coke as supplied to the furnace. Later, when working with coke of regular ash content over a period of five weeks, the make increased by 15.4% accompanied by a reduction in coke consumption of 1.01 cwt. per ton of pig, when using coke containing an average ash content of 9.76% as compared with coke containing 12.15%. The practical working of the furnace also improved considerably. C. A. KING.

Value of an addition of coal dust in the melting of iron in a cupola. P. BARDENHEUER and A. KAISER (Stahl u. Eisen, 1927, 47, 1389—1395).—Considerable economies may be effected in iron foundries by supplying about 10—12% of the fuel required in the cupola in the form of coal dust, which is blown into the furnace through a series of small tuyères situated a short distance above the ordinary air-blast tuyères. Comparative tests on the same furnace with the same iron showed that instead of the ordinary coke consumption of 11% of the charge, only 7% was needed if coal dust equal to 0.7—0.8% of the charge was blown in with compressed air. The melting zone was thus shortened and the preheating zone lengthened with a corresponding decrease in the amount of carbon monoxide in the flue gases without lowering the temperature of the iron leaving the furnace. At the same time the capacity of the furnace per day was increased by 23—30%, the economic utilisation of the fuel by 37%, and the fuel consumption by 30%, the amount of air blast required being reduced by 20% and the sulphur content of the iron decreased. Owing to the smaller fuel consumption and the smaller amount of ash to be slagged, the coke and limestone

consumption were decreased considerably and less slag was produced. Rods cast 30 mm. thick from a furnace fired with coal dust had a bending strength of 46—50 kg./mm.² and a tensile strength of 28—35 kg./mm.²

A. R. POWELL.

Direct production of pure iron. P. LONGMUIR (Amer. Electrochem. Soc., April, 1927, 51. Advance copy. 10 pp.).—A review of the various methods that have been suggested for the production of iron sponge by the low-temperature reduction of iron ore with solid or gaseous reducing agents. The author considers that the ideal method should consist of the following steps: distillation of coal at a low temperature to give a highly reactive coke, gas, and by-products; reduction of the ore, possibly in two stages, by means of producer gas; magnetic separation of the sponge followed by briquetting and melting in electric furnaces; and, finally, regeneration of carbon monoxide from the waste gases by passing them directly from the reduction furnace through a red hot mass of low-temperature coke produced in the first stage of the process.

A. R. POWELL.

Manufacture of steel in "one process" direct from ore. O. SMALLEY and F. HODSON (Amer. Electrochem. Soc., April, 1927, 51. Advance copy. 16 pp.).—In the Pehrson-Prentice process the ore separated from the gangue by means of an electromagnet is mixed with carbon and the mixture charged into a rotating furnace fitted with louvres for the admission of hot gases. Reduction is effected at 950—1000° with a thermal efficiency of 97%. The hot sponge is melted electrically in a gas-tight tilting furnace capable of accurate temperature control to 1800°, and the molten iron is refined and worked into steel as usual. The metal produced compares favourably in mechanical properties with the best crucible steel. Details are given of the mechanical properties of steels produced from New Zealand and Java titaniferous iron sands by the "Carsil" process involving direct reduction of the ore followed by melting and refining in a continuously-operated electric furnace.

A. R. POWELL.

Mechanism of the tempering of steels. T. MATSUSHITA and K. NAGASAWA (Iron and Steel Inst., Sept., 1927. Advance copy. 12 pp.).—Decomposition of martensite during the tempering of steel was followed by measuring the change in electrical resistance and the intensity of magnetisation during the heating. It was indicated that there are two martensites, α decomposing mainly in the interval 100—170° and β decomposing at 170—300°. Complete decomposition of martensite in one specimen was effected by heating at 250° for 2 hrs., and on reheating the specimen in a dilatometer contraction was observed at 300—400°, which leads to the view that the decomposition product of martensite is free carbon, and not cementite, which combines with iron in the range 300—400° to form cementite. Boundary temperatures are proposed for troostite (300°), sorbite (400°), and granular pearlite (550° to Acl point).

C. A. KING.

Non-rusting steels. B. STRAUSS (Z. Elektrochem., 1927, 33, 317—321).—The history of the development of non-rusting steels and their properties and applications are briefly reviewed. Further experiments (cf. B.,

1925, 722) on the electrode potential of chromium steels and chromium-nickel steels in ferrous sulphate solutions are described, measurements being made in the presence of hydrogen, air, or hydrogen peroxide. With steels containing 18–20% Cr and 8% Ni, the metal is passive in the presence of air, unless the carbon content exceeds about 1.2%; in the presence of hydrogen peroxide, even with 4% C, a noble potential was obtained. The effect of the annealing temperature on the magnetic properties of chromium-nickel steels is also dealt with.

H. J. T. ELLINGHAM.

Deterioration of alloy steels in ammonia synthesis. J. S. VANICK (Chem. Met. Eng., 1927, 34, 489–492).—Test specimens in the form of cylinders and containing similar specimens for tensile tests etc. were exposed to a temperature of 500°, a pressure of 100 atm., and an ammonia concentration of 8.3% in a nitrogen-hydrogen mixture for several months. The steels used were first annealed. Little change could be observed in 2 weeks. With longer exposures, fissuring and decarboxisation occurred, the penetration being either uniform or of the "tendril" type. Low-carbon steel deteriorated less rapidly than high-carbon steel, though both were penetrated to the core in 4 months, as were also nickel steel and low-chromium steel. A tungsten-chromium steel suffered much less. Further tests showed that a suitable steel would contain 2.25% Cr and not more than 0.3% C, the high chromium content reducing the action to a uniform penetration. High-chromium steels of the stainless type are most resistant, but are too difficult to work. Vanadium offered no advantage. The protective action of chromium results from the formation of a solid solution of chromium nitride.

C. IRWIN.

Influence of nickel and silicon on an iron-carbon alloy. A. B. EVEREST, T. H. TURNER, and D. HANSON (Iron and Steel Inst., Sept., 1927. Advance copy. 29 pp.).—The influence of nickel and silicon on a simple iron-carbon alloy (American washed iron) was investigated over a range 0–40% Ni, 0–3.6% Si. The original cast alloy was white and unsound, and small additions of nickel rendered this iron first sound, then grey, though four times as much nickel as silicon was required to produce a given degree of graphitisation, which, however, was finer when induced by nickel. Alloys containing 5–18% Ni were hard and martensitic, becoming soft and non-magnetic with austenitic structure when more than 18% Ni was present, and regaining magnetic properties beyond 20% Ni. The presence of increasing quantity of silicon appeared to mask the effect of nickel. The densities of the alloys, whilst high for grey irons, all lay between the normal limits for white and grey irons. Suitable annealing rendered most of the martensitic alloys capable of being machined. All the nickel-silicon alloys were unstable on heating, though to a less degree than ordinary silicon irons, and liability to corrosion was increased with the lower contents of nickel, and retarded when higher additions of nickel produced an austenitic structure.

C. A. KING.

Constitution of silicon-carbon-iron alloys and a new theory of the cast irons. D. HANSON (Iron and Steel Inst., Sept., 1927. Advance copy. 41 pp.).—During investigation into the constitution of ternary alloys of

iron containing 0–2% Si and 0–4% C it was shown that, in ternary alloys, graphite and cementite can occur as stable phases either separately or together. The consequences following the two diagrams representing graphite and carbide, respectively, overlapping instead of remaining always one above the other are discussed, and experimental evidence is advanced for the assumption that the iron-carbide and iron-graphite systems intersect at a point on that boundary of the austenite field representing carbon solubility. The theory is consistent with the stability of iron carbide in the range of ordinary steels, the normally-accepted equilibrium diagrams for these requiring no modification in principle, and accounts for the existence of graphite in stable form in alloys, e.g., cast iron, containing more than 2% C and 0.4% Si with the exception of a short range of temperature in the region of 700°, in which range some carbide can exist where the silicon content is low.

C. A. KING.

Quantitative measurement of the cutting power of cutlery. K. HONDA and K. TAKAHASHI (Iron and Steel Inst., Sept., 1927. Advance copy. 19 pp.).—The cutting power and durability of sharpness of cutlery was determined by reciprocal motions of the cutting edge under a definite load, on a number of superimposed sheets of paper, the number of sheets cut indicating the relative sharpness. Dulling of the edge with repeated use is represented by a logarithmic curve. Steel containing 1.3% C gave the best results for carbon steels, and the addition of 1% Cr (or W) or 0.5% Mo increased both the initial sharpness and the durability of the blades.

C. A. KING.

Testing machine for repeated impact: effects of repeated impact on Lowmoor iron. J. H. SMITH and F. V. WARNOCK (Iron and Steel Inst., Sept., 1927. Advance copy. 33 pp.).—On a machine designed to apply repeatedly stresses of known energy by means of a falling weight results showed that for a given amount of repeatedly applied tensile shock energy, the total energy to rupture a material is constant and momentum is not an important factor. For a given weight the height of fall and the number of shocks to produce rupture are connected by an equation of the form $H = AN^{-x}$ for a portion of the range, after which the value of x alters. The energy required to produce rupture by a single tensile shock is equal to that required by static means, the elongation being 4%, and reduction in area 3% greater than under static conditions. Elongation is more rapid at the beginning and end of a repeated impact test. It was found that for all areas above approximately 1 sq. in. the amount of energy required to rupture was fairly constant, the average value being 2430 in.-lb., but for areas below 1 sq. in. the total energy varies due to losses by distortion of the striking surfaces. The tests showed the serious source of weakness in abrupt changes of section, e.g., a sharp V groove.

C. A. KING.

Corrosion of copper pipes. A. F. DUFTON and F. L. BRADY (Nature, 1927, 120, 367).—Copper water pipes lined with tin may fail by local corrosion when the tin becomes tarnished, since the tarnished lining ultimately becomes electro-positive to copper. The *P.D.* between copper and the lining in water fell from about +0.4 to –0.1 volt.

A. A. ELDRIDGE.

Determination of manganese and magnesium in aluminium alloys. F. MUGRAUER (Chem.-Ztg., 1927, 51, 658).—After precipitation of the iron by zinc oxide, the manganese is determined by titration with potassium permanganate solution. The filtered solution is acidified with hydrochloric acid, concentrated, and treated with excess of ammonium chloride and ammonia. The magnesium is precipitated by addition of sodium phosphate to the hot solution, and the precipitate ignited and weighed as $Mg_2P_2O_7$. F. R. ENNOS.

Preparation of metallic cobalt from cobaltiferous smelter residues. A. BREMHORST (Metall u. Erz, 1927, 24, 7—8; Chem. Zentr., 1927, I, 1518).—Residues containing 0.5—5% Co are roasted alone or with sodium chloride, carbon, or sodium sulphate and carbon, whereby the cobalt is converted into sulphide. The product is melted and cast into tall moulds; it separates into three layers, the lowest of which consists of almost pure cobalt sulphide. Metallic cobalt may be obtained by addition of carbon to the charge. A. R. POWELL.

Influence of variables on the structure of electro-deposited copper. A. K. GRAHAM (Amer. Electrochem. Soc., Sept., 1927, 52. Advance copy. 19 pp.).—Under ordinary conditions of deposition electrolytic copper tends to assume the structure of the base metal even when deposited on smooth, highly polished cathodes. Increase of current density or of temperature results in a coarser deposit with a tendency to "treeing." With a low acidity of the bath the structure consists of fine, broken, twinned crystals, but becomes more conical and columnar with higher acid concentrations. Addition agents, such as aluminium sulphate and gelatin, tend to produce more fibrous deposits, and the effect of the structure of the base metal becomes much less marked. A. R. POWELL.

Enamels for sheet steel. CHI-FANG LAI, also HARRISON and WOLFRAM.—See VIII.

PATENTS.

Production of malleable iron and steel directly out of oxide ore. Production of iron and steel. E. G. T. GUSTAFSSON (E.P. 262,126—7, 26.11.26. Conv., 28.11.25).—(A) The direct reduction of ore in an electric furnace is effected by using a deficiency, amounting to 5—10%, of carbonaceous material in the early part of the process, making up the required reducing agent, together with more ore if desired, towards the end of the reaction. (B) The charge of ore and reducing agent is fed on to the top of the slag layer, which is of sufficient viscosity to sustain the mixture until reduction of the ore is effected before reaching the molten metal below. Heating is concentrated during the smelting process to different layers of the slag bath as required by varying the height of the electrodes. C. A. KING.

Manufacture of malleable iron castings. H. A. SCHWARTZ, Assr. to NAT. MALLEABLE & STEEL CASTINGS Co. (U.S.P. 1,640,674, 30.8.27. Appl., 30.10.24).—In making malleable castings with low silicon content, zirconium is added as a graphitising accelerator. The metal is cast with the maximum quantity of combined carbon, and the casting is annealed to precipitate carbon in finely-divided form. C. A. KING.

Blast-furnace method. F. W. DAVIS, Assr. to S. G. ALLEN (U.S.P. 1,640,485, 30.8.27. Appl., 4.10.24).—In the blast-furnace smelting of iron ore an additional supporter of combustion is introduced into the blast and a reducing agent into the lower end of the shaft.

A. R. POWELL.

Production of pig iron from fine ore by smelting in a blast furnace. C. KILIAN (G.P. 440,099, 15.11.24).—Fine iron ore is mixed with sufficient coke to effect the reduction to metal, and the mixture is charged into the throat of the blast furnace so that as it falls down through the furnace the ore is reduced to metal. Just sufficient coke is supplied in lump form to the furnace to melt the reduced metal, and the gases rich in carbon monoxide are taken from the throat of the furnace and passed back through the tuyères. A. R. POWELL.

Manufacture of cast iron and steel. J. M. LARGE (F.P. 609,138, 12.1.26).—A mixture of iron ore and wood charcoal is heated indirectly in a shaft furnace surrounded by a hollow chamber filled with lumps of lime heated at 1800—2000° by means of burning gases. The molten product is discharged from the bottom of the shaft into a second chamber similarly heated, and the metal is siphoned from under the slag layer into a third chamber similarly heated, in which it is carburised as usual. The iron and steel so obtained are free from sulphur and phosphorus. A. R. POWELL.

Apparatus for utilising low-grade iron ore. H. H. HINDSHAW, Assr. to HINDSHAW ENGINEERING & DEVELOPMENT Co. (U.S.P. 1,633,839, 28.6.27. Appl., 24.1.21).—The crushed ore is mixed with peat or other carbonaceous material, and heated in a rotary kiln out of contact with air, the reduced product being separated magnetically. T. S. WHEELER.

Treatment [colouring] of the surfaces of stainless or rustless steels or irons. W. H. HATFIELD and H. GREEN (E.P. 275,781, 30.7.26).—The surface of stainless steel or iron is coloured by immersion in a solution consisting of water 1000 pts., chromic acid 200 pts., sulphuric acid 400—450 pts., preferably at a temperature of 40—60°. C. A. KING.

Manufacture of alloy steel. C. T. EVANS (U.S.P. 1,639,989, 23.8.27. Appl., 18.4.22).—The steel consists of 3.50—7.75% Cr, 10—20% Ni, 0.4—1.0% C, 0.4—2.5% Mn, 0.5—2.5% Si, the remainder being principally iron. F. G. CROSSE.

[Vanadium-iron] alloy. L. JORDAN and G. W. QUICK, Assrs. to UNITED STATES OF AMERICA (U.S.P. 1,638,855, 16.8.27. Appl., 27.12.26).—An iron alloy contains approximately 2—4% C and 10—15% V. F. G. CROSSE.

[Ferrous] casting having a chromium alloy surface. C. B. JACOBS, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,632,704, 14.6.27. Appl., 22.4.21).—Ferrous castings are coated with a layer of chromium alloy resistant to oxidation by lining the moulds with granulated chromium mixed with a binder, e.g., sodium silicate, and pouring the castings at about 1500°. T. S. WHEELER.

Production of magnetic material. L. ZICKRICK, Assr. to WESTERN ELECTRIC Co., Inc. (U.S.P. 1,632,105,

14.6.27. Appl., 25.3.25).—Magnetic alloys of iron and antimony (2–15%) are claimed, which can be easily reduced to powder. T. S. WHEELER.

Roasting furnace. H. J. HARTLEY, E. J. FOWLER, and D. BAIRD, ASSTS. to NICHOLS COPPER CO. (U.S.P. 1,640,988, 30.8.27. Appl., 21.7.26).—In a rotary shaft furnace containing a number of superimposed hearths, each hearth is constructed in removable sections supported by the furnace wall. C. A. KING.

Smelting furnace. T. LEWIN, ASSR. to W. LEWIN (U.S.P. 1,640,574, 30.8.27. Appl., 6.7.25).—A smelting furnace consists of an elongated cylindrical shell with removable end plates which may be fastened to collars fixed to the cylinder casing. C. A. KING.

Bright-annealing furnace. SIEMENS-SCHUCKERT-WERKE G.M.B.H. (E.P. 262,445, 2.12.26. Conv., 3.12.25).—In a furnace for bright annealing, the space between the furnace body and a protective casing is filled with a gas which will not explode with air or with the protecting gas in the furnace chamber, *e.g.*, a gas of low oxygen content. C. A. KING.

[Flotation] concentration of metalliferous ores. T. R. SIMPSON, ASSR. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,632,419, 14.6.27. Appl., 27.5.26. Conv., 9.6.25).—A mixture of an organic protective colloid, *e.g.*, starch, and an alkaline xanthate is of value as a reagent in the froth flotation of sulphide ores. T. S. WHEELER.

Concentration of ores. R. E. SAYRE, ASSR. to METALS RECOVERY CO. (U.S.P. 1,640,218, 23.8.27. Appl., 23.7.25).—The mineral in the form of a pulp is subjected to a flotation process in the presence of a thiourethane. F. G. CROSSE.

[Ore] flotation process. E. A. HALL and G. R. SANDERS, ASSTS. to SOUTHWESTERN ENGINEERING CORP. (U.S.P. 1,634,615, 5.7.27. Appl., 13.9.26).—The addition of a small quantity of oxalic acid is of advantage in the froth flotation of zinc and lead ores containing carbonaceous and tarry material. T. S. WHEELER.

Controlling the evolution of elemental sulphur [from sulphide ores]. G. N. KIRSEBOM, ASSR. to U.S. SMELTING, REFINING, AND MINING CO. (U.S.P. 1,640,837, 30.8.27. Appl., 16.11.25).—To control the evolution of sulphur from sulphide ores while sintering a quantity of arsenic sufficient to combine with the sulphur is added to the charge. A. R. POWELL.

Method of making [nickel-chromium] alloys. J. H. L. DE BATS, ASSR. to DE BATS METALS CO. (U.S.P. 1,639,330, 16.8.27. Appl., 24.1.24).—A finely-divided mixture of metals of the chromium group is placed on the surface of unmelted nickel, the nickel is melted, and the unfused mixture of other metals is mechanically mixed with the melt. C. A. KING.

Welding [of nickel]. INTERNAT. NICKEL CO., ASSEES. of N. B. PILLING and J. G. SCHOENER (E.P. 259,967, 12.10.26. Conv., 19.10.25).—The welding of nickel or nickel alloys is improved if a metal of good reducing power and having a relatively high b.p. is introduced into the molten weld. For this purpose a nickel or nickel alloy electrode is coated with a composition containing titanium (3 pts.) and calcium (1 pt.). C. A. KING.

Refining lead. P. W. DAVIS (U.S.P. 1,640,486—7, 30.8.27. Appl. [A, B], 11.1.26).—(A) Molten lead alloys are stirred with litharge at a temperature between the m.p. of litharge containing the oxide of the alloying element and the m.p. of pure litharge (about 883°). (B) The metallic product obtained by the above treatment is further refined by treatment with fresh litharge at a temperature above its m.p. Both operations are carried out on relatively small charges of alloy. A. R. POWELL.

Refining and separation of metals. H. HARRIS (E.P. 275,344, 7.5.26).—In the treatment of impure lead containing arsenic, tin, and antimony with molten caustic soda the greater part of the antimony remains insoluble when extracted with water. The smaller soluble portion is separated by treating the solution with a reducing agent, *e.g.*, tin, stannous chloride. C. A. KING.

Recovery of copper, zinc, and other metals from solutions containing chlorides and sulphates. J. Y. JOHNSON. From DUISBURGER KUPFERHÜTTE (E.P. 276,200, 18.11.26).—The solution containing copper, iron, and zinc, together with chlorides and sulphates of the alkali metals (*e.g.*, the solution obtained by leaching the product obtained by the chloridising roasting of sulphide ores of copper and zinc), is treated with zinc hydroxide to precipitate copper and iron hydroxides, and the filtrate is treated with zinc dust to remove lead, silver, and the last traces of copper. The clear liquor is cooled to deposit sodium sulphate crystals, calcium chloride is added to remove the remaining sulphate, and the filtrate is treated with lime to precipitate zinc hydroxide for further use in the process and to regenerate calcium chloride. A. R. POWELL.

Apparatus for determining the composition of copper and zinc alloys [brass]. M. POLANYI and S. VON BOGDÁNDY (E.P. 268,306, 17.2.27. Conv., 24.3.26).—Brass is heated in a silica test-tube enclosed in a larger cylindrical silica vessel closed at its lower end and connected to a vacuum pump at its upper end. Heating is effected by lowering the lower end of the large cylinder into a wire-wound electric furnace. At 800–900° zinc is rapidly volatilised from brass *in vacuo* and the residual copper may be weighed (cf. B., 1927, 413). A. R. POWELL.

Metallurgical process. [Treatment of copper concentrate.] W. E. GREENAWALT (U.S.P. 1,633,091, 21.6.27. Appl., 7.9.26; cf. U.S.P. 1,483,056; B., 1924, 340).—Copper concentrate containing rare metals is roasted, most of the copper being then extracted with dilute acid. The solution is treated with sulphur dioxide to reduce the iron present, and the copper recovered by electrolysis. The residue is smelted with pyrites, thus concentrating the residual copper and rare metals into a matte, which is heated to volatilise the sulphur present. The resulting crude copper is electrolytically refined, the rare metals being recovered from the anode slimes. T. S. WHEELER.

Process of hardening copper. W. H. WEISER (U.S.P. 1,638,076, 9.8.27. Appl., 11.10.23).—Pulverised magnesia is injected into a mass of molten copper.

H. ROYAL-DAWSON.

Compressed compositions for the production of bearings. C. CLAUS (E.P. 275,444, 13.10.26).—Self-

lubricating bearing blocks are made by compressing a mixture of 85 pts. of finely-divided copper, 9 pts. of tin, 2 pts. of zinc, and 4 pts. of graphite under a pressure of 10,000–50,000 lb./sq. in., and then heating the material in a closed container for 10–20 min. at 760–820°.

C. A. KING.

Purifying aluminium and its alloys. METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 265,563, 13.1.27. Conv., 8.2.26).—Calcium or calcium carbide may be removed from aluminium or aluminium-silicon alloys by melting the metal below a bath of cryolite containing dissolved alumina. The resulting castings are stronger and more ductile, and do not exhibit the coarse grey fracture caused by the presence of calcium.

A. R. POWELL.

Producing metal [copper] coatings on articles of aluminium and its alloys. O. SPRENGER PATENT-VERWERTUNG JIROTKA M.B.H., and B. JIROTKA (E.P. 275,828, 27.10.26. Addn. to E.P. 249,971; B., 1926, 496).—The bath recommended in the chief patent may be replaced by the following mixture: 25 g. of copper sulphate crystals, 25 g. of sodium carbonate, 25 g. of sodium hydrogen carbonate, and 10 g. of sodium dichromate in 2.5 litres of water. The dichromate may be omitted, but its use leads to a better deposit in a shorter time.

A. R. POWELL.

Alumino-solder. M. TORII (U.S.P. 1,640,161, 23.8.27. Appl., 7.8.24).—The solder contains 20–70% Zn, 20–70% Sn, 5–35% Pb, 3–50% Cd, 1–5% Mg, and 1–5% of tin phosphide.

F. G. CROSSE.

Protection of magnesium and its alloys. G. MICHEL (E.P. 249,484, 10.2.26. Conv., 20.3.25).—The cleaned metallic surface is painted or sprayed with phenol or a mixture containing phenol or one of its homologues, whereby a tightly adherent film of a reaction product of phenol and magnesium is formed on the metal. This film may be further protected by a coating of varnish or paint.

A. R. POWELL.

Manufacture of refractory metal alloy of high density and high melting point. C. A. LAISE (U.S.P. 1,633,258, 21.6.27. Appl., 8.10.25).—Tungsten oxide is reduced in a current of hydrogen and ammonia, the resulting metal being mixed with boron nitride (0.5%), compressed in a mould, and heated at about 900° *in vacuo*. The porous product is immersed in a bath of molten copper, with which it rapidly alloys.

T. S. WHEELER.

Metal pickling. J. H. GRAVELL (U.S.P. 1,632,833, 21.6.27. Appl., 15.4.26).—Hair, gelatin, or other animal material is destructively distilled, that portion of the distillate which is insoluble in water and soluble in hydrochloric acid being added to metal-pickling baths to reduce action on clean metal surfaces.

T. S. WHEELER.

Production of metals in electric furnaces. E. G. T. GUSTAFSSON (E.P. 252,162, 11.5.26. Conv., 16.5.25).—To protect the refractory bottom of an electric furnace during the interval between tapping and re-charging, a quantity of slag, which may be taken from the previous refining, is introduced into the furnace to act as a protective layer.

C. A. KING.

Cupola furnace. A. POUMAY (U.S.P. 1,640,251, 23.8.27. Appl., 16.4.25. Conv., 18.4.24).—See E.P. 232,630; B., 1926, 132.

Process of rust-proofing [iron]. M. GREEN and H. H. WILLARD, Assrs. to PARKER RUST-PROOF CO. (U.S.P. 1,639,694, 23.8.27. Appl., 10.5.26).—See E.P. 273,168; B., 1927, 658.

Manufacture of sound silicon-iron. V. B. BROWNE (E.P. 276,190, 25.10.26).—See U.S.P. 1,627,269; B., 1927, 448.

Manufacture of alloys containing gold. GENERAL PLATE CO., Assees. of V. D. DAVIGNON (E.P. 257,891, 25.5.26. Conv., 4.9.25).—See U.S.P. 1,557,431; B., 1926, 63.

Process of metallising ores and recovery of metals and by-products. D. M. CRIST (E.P. 275,791, 10.8.26).—See U.S.P. 1,595,454; B., 1926, 984.

Regeneration of metal catalysts contaminated by sulphur. E. A. PRUDHOMME, Assr. to SOC. INTERNAT. DES PROC. PRUDHOMME (U.S.P. 1,640,668, 30.8.27. Appl., 8.5.26).—See E.P. 238,805; B., 1925, 834.

Recovery of copper, zinc, and other metals from solutions containing chlorides and sulphates. K. MATTENKLODT and H. SCHRAMM, Assrs. to DUISBURGER KUPFERHÜTTE (U.S.P. 1,639,610, 16.8.27. Appl., 17.11.26. Conv., 9.4.25).—See E.P. 276,200; preceding.

[Reinforced] lead columns or pipes etc. [for acids]. SOC. BELGE DE L'AZOTE (SOC. ANON.) (E.P. 263,881, 3.1.27. Conv., 4.1.26).

[Electric] bright-annealing furnace [with liquid sealing]. SIEMENS-SCHUCKERTWERKE G.M.B.H. (E.P. 262,462, 3.12.26. Conv., 5.12.25).

Fuel for welding (U.S.P. 1,628,067).—See II.

Luminous enamels (F.P. 610,454). **Enamelling sheet iron** (F.P. 610,645).—See VIII.

Electrodeposition of chromium (G.P. 440,196).—See XI.

XI.—ELECTROTECHNICS.

"Knowles" plant for electrolysis of water. Q. SESTINI (Giorn. Chim. Ind. Appl., 1927, 9, 318–320).—A short description is given of the construction of this cell in which 15% sodium hydroxide is used as electrolyte, and which is said to deliver 99.5% O₂ and 99.8% H₂. Operating characteristics are given for a cell consuming 4500 amp. at 2.5 volts.

R. W. LUNT.

Electrical insulating materials. A. GÜNTHER-SCHULZE (Z. Elektrochem., 1927, 33, 360–369).—A lecture.

High-frequency induction melting. CAMPBELL. **Tempering of steels.** MATSUSHITA and NAGASAWA. **Structure of electrodeposited copper.** GRAHAM.—See X.

PATENTS.

Electric furnace. J. KELLEHER, Assr. to HARPER ELECTRIC FURNACE CORP. (U.S.P. 1,637,486, 2.8.27. Appl., 17.7.25).—A carbonaceous resistor extends longitudinally through a furnace chamber having end and

side walls of refractory silica material, and is surrounded by an inner lining of carbonaceous material forming a resistor chamber. A packing between the inner lining and outer wall is sufficiently thick to prevent entrance of silicon vapour from the furnace chamber walls into the resistor chamber. J. S. G. THOMAS.

Electric resistance furnace. F. KRUPP A.-G. (E.P. 257,896, 19.7.26. Conv., 4.9.25).—In an electric resistance furnace at least one receptacle for receiving articles to be treated rests on a roller system, *e.g.*, through an intermediate member, for which a track is provided in the heating chamber and to which similar tracks for entrance and exit of the receptacle are attached. The heating chamber consists of a chromium-nickel steel or chromium-steel alloy. J. S. G. THOMAS.

Preparation of an electron-emitting cathode. F. HOLBORN, ASSR. to HAZELTINE CORP. (U.S.P. 1,639,698, 23.8.27. Appl., 14.1.26).—An alkaline-earth metal "sputtered" in pyridine is electrically deposited on a conductor, which is then heated to evaporate the pyridine. J. S. G. THOMAS.

Incandescence cathode. H. WADE. From N.V. PHILIPS' GLOEILAMPENFABR. (E.P. 275,449, 27.10.26. Addn. to E.P. 274,981; B., 1927, 754).—The prior patent is modified in that the metal wire wound helically upon a refractory metal core is composed of nickel. J. S. G. THOMAS.

[Filament supports of] electric incandescence lamps. GEN. ELECTRIC CO., LTD., and C. J. SMITHELLS (E.P. 275,735, 14.5.26).—Filament supports, except those parts in contact with the filament, are made of or coated with chromium, manganese, or uranium, but preferably chromium, which serves to decompose any oxidising material, and more especially water vapour, contained in the lamp. J. S. G. THOMAS.

Preparing positive electrodes of storage batteries. A. CELLINO, ASSR. to CENTRAL BATTERY CORP. (U.S.P. 1,640,922, 30.8.27. Appl., 15.6.16. Renewed 8.1.27).—An electrode formed of a mixture of an oxide of lead, manganese peroxide, and ammonium sulphate is treated with sulphuric acid to dehydrate the active material. J. S. G. THOMAS.

Accumulator plate. W. HADDON and J. M. BURNETT (E.P. 275,392, 7.7.26).—Accumulator plates are pasted with a mixture containing sulphur dissolved in a solution of sodium hydroxide and the lower oxides of lead. The plate is, after an interval of 2–3 days, immersed in a mixture of lead peroxide and sulphuric acid or a soluble sulphate and formed in an acid bath. J. S. G. THOMAS.

Accumulators containing lead dioxide, zinc, and sulphuric acid. A. MIOLATI, ASSR. to R. LEPRESTRE (U.S.P. 1,632,307, 14.6.27. Appl., 22.4.24. Conv., 3.5.23).—The efficiency of such accumulators is increased by having the positive plate of lead and lead dioxide, the electrolyte of sulphuric acid, and the negative plate of lead alloyed with 2–3% of zinc, all materials being pure. T. S. WHEELER.

Dry cell. V. YNGVE, ASSR. to NATIONAL CARBON CO., INC. (U.S.P. 1,634,006, 28.6.27. Appl., 12.1.25).—The efficiency of Leclanché dry cells is increased by grinding

together the mixture of manganese dioxide, graphite, and ammonium chloride used, and then adding the electrolyte. T. S. WHEELER.

[Depolariser for] galvanic cells. C. F. BURGESS LABORATORIES, ASSEES. OF B. K. BROWN (E.P. 255,067, 7.7.26. Conv., 7.7.25).—The depolariser used in a galvanic cell of the Leclanché type consists of solid oxides of carbon, *e.g.*, graphitic oxide, prepared in accordance with E.P. 255,068 (B., 1927, 17) mixed, if desired, with manganese dioxide. J. S. G. THOMAS.

Dielectric covering for electric wires, cables, etc. P. ALLMAN, H. N. MORRIS, and L. H. MARLOR (E.P. 275,304, 30.4.26).—Electric wires, cables, etc. are covered by deposition with a rubber-cellulose coating obtained from a combined solution of rubber and cellulose in one or more of the volatile hydrogenated products of aromatic hydrocarbons, or their hydroxy-compounds or isomerides, or esters of these, *e.g.*, cyclohexanone, butyl acetate, with or without non-volatile solvents or softening media. J. S. G. THOMAS.

Electrolytic apparatus [for electrolysis of water]. W. G. ALLAN (E.P. 275,785, 4.8.26).—Apparatus for the electrolysis of water comprises a group of cells, a stand pipe, and a pair of return-flow manifolds communicating with a tank, a tube communicating between each manifold and each cell of a group, a second pair of manifolds connected, respectively, by conduits to the anode and cathode sides of each cell, conduits connecting, respectively, the anode and cathode manifolds with chambers of the tank, and a gas outlet from each chamber of the tank. J. S. G. THOMAS.

Electrolytic diaphragm cells. J. BILLITER, and SIEMENS & HALSKE A.-G. (Austr. P. 105,356, 23.2.25).—In a diaphragm cell excess pressure on the anode side is practically uniformly distributed by filling the cathode chamber with electrolyte over the whole surface of the diaphragm, whilst the difference of level of anolyte and catholyte can be so great that the electrolyte traverses the diaphragm at such a rate that 30%, or even 60%, of the salt is decomposed. J. S. G. THOMAS.

Anode [for electrolysis]. E. C. R. MARKS. From A. P. MUNNING & Co. (E.P. 275,359, 26.5.26).—The crystals, *e.g.*, of nickel, forming the major active portion of an anode are disposed substantially normal to the main path of current travel in an electrolytic bath. J. S. G. THOMAS.

Electrolytic decomposition of water. VEREIN FÜR CHEM. U. MET. PROD. (G.P. 440,335, 31.1.26).—Alkali hydroxide solution produced by the electrolysis of alkali chloride solution by the mercury process is used as electrolyte in the electrolytic decomposition of water. L. A. COLES.

Electrodeposition of chromium. SIEMENS & HALSKE A.-G., ASSEES. OF M. HOSENFELD and H. WALDE (G.P. 440,196, 3.7.25).—In the electrodeposition of chromium from chromic acid baths, the anode consists of one or more lead plates lying on the floor of the cell and covered loosely with powdered chromium, preferably electrolytically produced. A. R. POWELL.

Prevention of oxidation of oil in transformers. J. KUBLER, ASSR. to A.-G. BROWN, BOVERI, & CIE. (U.S.P.

1,635,332, 12.7.27. Appl., 2.9.26. Conv., 24.9.25).—An atmosphere of carbon dioxide is maintained above the surface of the oil by an apparatus working on the Kipp principle. T. S. WHEELER.

Electrochemical production of solid oxides of carbon [graphitic oxide]. C. F. BURGESS LABORATORIES, INC., Assees. of B. K. BROWN, O. W. STOREY, and G. T. COLLINSON (U.S.P. 1,639,980—2, 23.8.27. Appl., 7.7.25).—See E.P. 255,068; B., 1927, 17.

Galvanic cell. C. F. BURGESS LABORATORIES, INC., Assees. of B. K. BROWN (U.S.P. 1,639,983—5, 23.8.27. Appl., 7.7.25).—See E.P. 255,067; preceding.

Decolorising carbon (U.S.P. 1,634,477—80). **Metallic carbon** (U.S.P. 1,637,052). **Acidity in transformer oils** (U.S.P. 1,633,956).—See II.

Preserving wood (G.P. 439,523).—See IX.

Magnetic alloys (U.S.P. 1,632,105).—See X.

XII.—FATS; OILS; WAXES.

Drying of fatty oils. Gas-coagulation of linseed oil. P. SLANSKY (Chem. Umschau, 1927, 34, 148—151).—The author claims priority for the view that the coagulation processes leading to the solidification of drying oil films are necessarily subsequent to purely chemical reactions. The view that drying may occur entirely through colloidal changes (supported by Auer and Wolff) is criticised on theoretical grounds. On keeping linseed oil films of various thickness *in vacuo* for 16 days, under conditions precluding any infiltration of air, negligible changes in viscosity, refractive index, iodine value, and weight are found to occur. From consideration of these results the validity of Auer's "drying *in vacuo*" experiments and the gas-coagulation theory he deduces therefrom is called into question. S. S. WOOLF.

Oil of *Salvia spinosa*. S. BERLINGOZZI and G. DI MASE (Rend. Accad. Sci. Fis. Mat. Napoli, 1926, [iii], 31, 156—157).—See B., 1926, 887.

Yellowing of drying-oil films. MORRELL and MARKS.—See XIII.

PATENTS.

Manufacture of grease. J. MCKEE, Assr. to SUN OIL Co. (U.S.P. 1,637,703, 2.8.27. Appl., 24.8.25).—A mixture of 5—50% of hard coconut oil soap and 95—50% of a paraffin base mineral oil is heated at above 232°, forming a grease of high b.p.

H. ROYAL-DAWSON.

Apparatus for the treatment [to separate kernels and pulp from nuts] of oil-containing fruits [oil-palm]. F. KRUPP GRUSONWERK A.-G. (E.P. 267,092, 25.1.27. Conv., 4.3.26).

Apparatus for hydrogenation (E.P. 274,952).—See I.

Removal of odour from oils (G.P. 439,615).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Yellowing of drying-oil films. R. S. MORRELL and S. MARKS (J. Oil Col. Chem. Assoc., 1927, 10, 186—200).—The changes in colour of various white paint films kept in the dark in a moist atmosphere at ordinary temperatures were observed, using yellow and red Lovibond

tintometer glasses with a freshly-made zinc oxide-poppseed oil paint as standard. Experimental details are quoted, and the extent of yellowing of the films after 1, 2, 3, and 7 months is recorded. The yellowing with raw drying oils was less than with prepared bleached linseed oil, whilst stand oil was superior to raw oil, closely approaching the quality of walnut and poppyseed oil, which showed the least yellowing. White lead, which in general caused greater yellowing than did zinc oxide, was unsatisfactory with the bleached oils. Of the various white pigments tested, anhydrous titanium dioxide gave the best results. The oxidation of β -elaeostearic glyceride is being studied, with the object of isolating the yellowing principle, and a preliminary account of the investigation is given. From the results to date, it is deduced that the property of yellowing in the dark is probably connected with the presence of hydroxyl groupings derived from the peroxide group contiguous to the carboxyl end of the molecule, and may be induced by traces of water or the elements of water in a pigment. S. S. WOOLF.

Pigments for colouring Portland cement mortar. J. E. FOSTER (Chem. Met. Eng., 1927, 34, 487—488).—Various pigments used for colouring cement were tested for tinting value, stability to light, and their effect on the strength of the cement. It was concluded that in no case was a greater addition than 20% of any advantage. Metallic oxides used were iron oxides for yellow, buff, red, brown, and black, chromium oxide for green, and manganese dioxide for black. These all proved permanent to light, and an admixture up to 20% did not reduce strength. Ultramarine was also satisfactory. Carbon black seriously reduced the cement strength. Zinc chromate and cadmium lithopone failed owing to impermanence. The colour of the sand used is not without importance. When possible it should harmonise with the pigment. C. IRWIN.

PATENTS.

Production of [wood] paints and stains. G. W. CARVER (U.S.P. 1,632,365, 14.6.27. Appl., 29.1.25. Cf. U.S.P. 1,541,478; B., 1925, 601).—Clay containing a high percentage of iron and free from alkali is heated with iron turnings and a mixture of hydrochloric and sulphuric acids, the suspension obtained being treated with potassium ferricyanide and nitric acid. The blue pigment formed is of value in the treatment of wood.

T. S. WHEELER.

Production of lead compounds of chromic acids. A. NATHANSOHN (U.S.P. 1,633,948, 28.6.27. Appl., 19.11.24. Conv., 18.9.24).—Lead ores are heated with aqueous alkali or alkaline-earth chloride solutions, the resulting solutions being treated with potassium dichromate or the like to precipitate lead compounds of chromic acids of value as pigments. T. S. WHEELER.

Manufacture of precipitated antimony [penta]-sulphide. R. E. STARK, Assr. to STIBIUM PRODUCTS Co. (U.S.P. 1,633,754, 28.6.27. Appl., 26.12.22; cf. U.S.P. 1,414,836—7; B., 1922, 474 A).—An oxide of antimony, sulphur, and a compound capable of forming a soluble compound containing antimony, *e.g.*, calcium oxide, are heated with water under pressure at 155°, the resulting solution being treated with hydrochloric

acid, with or without addition of antimony pentachloride, to precipitate antimony pentasulphide.

T. S. WHEELER.

Non-inflammable varnish. H. C. P. WEBER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,635,567, 12.7.27. Appl., 5.7.22).—A mixture of carbon tetrachloride and chlorobenzene is added to varnish solutions in place of inflammable solvents. To inhibit formation of acidity on drying the inclusion of a small proportion of aniline or the like is preferred.

T. S. WHEELER.

Phenolic condensation product. A. L. BROWN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,632,113, 14.6.27. Appl., 7.4.22; cf. U.S.P. 1,633,976; following abstract).—The addition of a drier, *e.g.*, "tox tungate" (a mixture of lead and manganese resinate and tungates), to the ingredients employed in the manufacture of cresol-formaldehyde resins is claimed.

T. S. WHEELER.

Manufacture of a flexible composite article. A. L. BROWN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,633,976, 28.6.27. Appl., 24.9.20. Renewed 8.11.26; cf. U.S.P. 1,632,113; preceding abstract).—The addition of a drier, *e.g.*, "tox tungate," and of a polymerisable vegetable oil, *e.g.*, tung oil, to the ingredients employed in the manufacture of phenol-formaldehyde resins gives rise to a product of increased flexibility.

T. S. WHEELER.

Resinous reaction product of urea and formaldehyde. F. LAUTER, Assr. to ROHM & HAAS CO. (U.S.P. 1,633,337, 21.6.27. Appl., 28.7.25).—Formaldehyde is condensed with urea in presence of glycerol, or of a non-aqueous solvent, *e.g.*, benzene, to give resinous products insoluble in water.

T. S. WHEELER.

Manufacture of sulphur-phenol resin. C. ELLIS (U.S.P. 1,636,596, 19.7.27. Appl., 30.12.22).—A phenol (1 mol.), sulphur (more than 3 atoms), and an alkaline catalyst, *e.g.*, sodium hydroxide, are heated at about 170°.

T. S. WHEELER.

Barium carbonate and sulphydrate (U.S.P. 1,634,338).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Ageing of soft rubber goods. R. F. TENER, W. H. SMITH, and W. L. HOLT (U.S. Bur. Standards, Tech. Paper No. 342, 1927, 21, 353—384).—Four different types of vulcanised mixed rubber containing (A) rubber 90, sulphur 10; (B) rubber 100, sulphur 4, diphenylguanidine 0.5, zinc oxide 4, whiting 50; (C) rubber 100, sulphur 6, litharge 10, whiting 50; and (D) rubber 100, hexamine 1, sulphur 6.53, zinc oxide 18.75, gas black 34.85, mineral rubber 10.9, palm oil 6.53, and reclaimed rubber 38.1 pts., respectively, were submitted to ageing tests under various conditions, the progress of the change being followed by tensile tests. The rate of deterioration was greater in oxygen than in nitrogen, air occupying an intermediate position. All the compounds deteriorated more rapidly when exposed to light, but (D) was much less sensitive than (B), which was greatly improved, however, by the introduction of a little red iron oxide or of carbon black. Sunlight caused deterioration even in the absence of air, a hard brittle coating being

formed, particularly on (A) and (B); the coating, which contained more combined sulphur than the inside rubber, was insoluble in acetone and benzene and appeared to protect against further deterioration. Mixing (B) was most resistant to heat, whereas (C) was most sensitive; all four compounds appeared to be most susceptible at a critical temperature between 80° and 90°. The degree of vulcanisation had greatest influence on (A) and least on (B) and (D). For (C) and (D) the rate of ageing was reduced to approximately one half by a fall of 10° from 90° or 80°. The relative order of ageing in accelerated tests was in good agreement with that in tests with exposure away from sunlight, and the statement that 24 hrs. in air at 70° are equivalent to about 6 months of natural ageing is approximately correct; accelerated tests, however, showed no relation to tests with exposure to sunlight. In outdoor tests, (B) showed greatest deterioration in physical properties and greatest increase in acetone extract, whilst (D) was least in these features; (C) was intermediate in both features, whereas with (A) these features showed no interdependence. In the absence of sunlight (B) deteriorated least and showed no increase in acetone extract; (C) and (D) deteriorated similarly, showing corresponding increases in acetone extract, whilst (A) did not show increases in acetone extract corresponding with the physical depreciation.

D. F. TWISS.

Constitution of caoutchouc. R. PUMMERER (Kautschuk, 1927, 233—237).—By prolonged mechanical working, even with exclusion of air, rubber can be so disaggregated that it becomes entirely soluble in ether. Disaggregation can also be effected by dissolving in benzene and re-precipitation, the change being expedited by the presence of basic or acidic substances, *e.g.*, piperidine or acetic acid, in the benzene. The change cannot be a purely chemical one, but is probably to be attributed to a disruption of molecular aggregates. The process of disaggregation is reversible and "sol" rubber isolated from solution may gradually undergo spontaneous conversion into the ether-insoluble "gel" condition; such aggregation is regarded as the result of the grouping of molecules into a colloid-chemical or crystalline arrangement. Cryoscopic measurements in camphor, benzylidenecamphor, benzoic acid, or menthol give results between 1100 and 2500 for the mol. wt. of rubber. Sol and gel rubber in menthol give similar values, viz., 1200—1600 for solutions exceeding 2% in concentration; at 1% or lower, however, the apparent mol. wt. of rubber falls to about 600. Although the osmotic pressure of rubber in benzene indicates a mol. wt. between 30,000 and 50,000, rubber can be dialysed slowly from its benzene solution through a collodion membrane. The results suggest that the molecular nuclei in rubber which are capable of aggregation contain approximately 8 isoprene groups. From their absorption in the ultra-violet, however, neither sol nor gel rubber can have as high a proportion as one pair in 500 of its double linkings in conjugation, or any appreciable proportion of radicals with tervalent carbon. It appears probable, therefore, that the molecular nuclei in rubber involve a cyclic structure.

D. F. TWISS.

Oxidation of rubber. M. LEON and W. N. LISTER

(J.S.C.I., 1927, 46, 220—224 r).—In comparative oxidation experiments with films of fine hard Para, soft Para, pale crêpe, brown crêpe, and smoked sheet rubber on the inside of a flask at 100° containing oxygen, the first-named is found to interact with less oxygen than are the others. Moderate mastication of pale crêpe rubber causes a slight increase in the resistance to oxidation, but the over-masticated rubber oxidises very rapidly, although the final quantity of oxygen absorbed is less. A film of rubber made from latex oxidises less rapidly than pale crêpe rubber, but its resistance is decreased by extraction with water. Removal of the natural resins from smoked sheet or pale crêpe causes a great increase in the rate of oxidation, whereas introduction of such resins or increase in the proportion of natural, protein-rich, insoluble matter increases the stability. Dragon's blood resin, dinitrobenzene, and quinol exert a protective action; benzoic acid, resorcinol, and traces of copper chloride accelerate the oxidation. Petroleum jelly and castor oil, which decrease the permeability of rubber to gases, cause an initial retarding effect and later acceleration.

D. F. TWISS.

Oxidation of rubber from the scientific and technical point of view. F. KIRCHHOF (Kautschuk, 1927, 239—245, 256—261).—A review of the subject covering the influence of natural and ultra-violet light on the phenomena of ageing, and of metallic salts on the depolymerisation and oxidation of rubber; the effect of increased temperature on oxidation in air and in oxygen is also discussed. It is suggested that the natural resins in rubber protect the rubber from oxidation because they are chemically more unsaturated, and so combine preferentially with oxygen.

D. F. TWISS.

Recent röntgenographic researches on rubber and related substances. E. A. HAUSER [with M. HÜNEMÖRDER and P. ROSBAUD] (Kautschuk, 1927, 228—230).—A preliminary account of results obtained. Unstretched frozen rubber can show Debye-Scherrer circles; on stretching, these lose in intensity and point interference develops. All rubber samples, indeed, which are turbid or opalescent show Debye-Scherrer circles. Rubber also can be extended slowly without loss of its isotropic character, whereas on rapid extension it will give rise to interference phenomena. The sol and gel constituents of rubber, separable by ether, also show interference phenomena when stretched, although it is necessary to keep the sol rubber in an atmosphere of carbon dioxide. The interference phenomena with gutta-percha and balata stretched at 35—40° are practically identical, although differences are observed in the results with the unstretched material.

D. F. TWISS.

Structure of gutta-percha and balata. H. MIEDEL (Kautschuk, 1927, 230—232).—The behaviour of gutta-percha when stretched under various conditions is described. The hydrocarbons of rubber and of gutta-percha or balata show approximately the same mol. wt. in camphor, and their light absorption in the ultra-violet region gives no indication of additional ring formation in either. From the greater ease of crystallisation of the hydrocarbon of gutta-percha or balata and its apparent resistance to resolution by fractionation, it is

probable that this hydrocarbon is more homogeneous than caoutchouc in chemical composition.

D. F. TWISS.

PATENTS.

Manufacture of articles from ebonite and like material. A. FRASER, and RISSIK, FRASER & Co., LTD. (E.P. 273,355, 2.2. and 2.11.26).—An ebonite article of "good fracture" is produced from undercured ebonite dust (e.g., with a coefficient of vulcanisation of 25—26), which has been previously warmed at about 130°, by moulding under a pressure upwards of about 500 lb./sq. in. and at a temperature near 220—250° for from 3 to 10 min. The moulding chamber is cooled before the article is removed.

D. F. TWISS.

Accelerating the vulcanisation of rubber. L. B. SEBRELL, ASSR. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,635,193, 12.7.27. Appl., 16.9.26. Cf. U.S.P. 1,604,199; B., 1927, 52).—Vulcanisation is effected in the presence of a 1-mercaptodimethylbenzthiazole or a derivative.

D. F. TWISS.

Production of substances containing rubber and cellulose derivatives. P. ALLMAN, H. N. MORRIS, and L. H. MARLOR (E.P. 274,968, 30.4.26).—Rubber is dissolved in one or more of the volatile hydrogenation products of aromatic hydrocarbons or of hydroxy-, ketonic, or acyloxy-derivatives of such hydrocarbons; non-volatile softening agents may also be present. A solution of a cellulose ester or of celluloid is then introduced in a simple or mixed solvent, e.g., acetone, in which rubber is not soluble.

D. F. TWISS.

Process of rubber vulcanisation. C. O. NORTH, ASSR. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,634,336, 5.7.27. Appl., 27.8.26).—The product formed by the interaction of aniline, acetaldehyde, and heptaldehyde is of value as an accelerator.

T. S. WHEELER.

[Accelerators for] rubber vulcanisation. S. B. MOLONY and Y. NIKAIKO, ASSRS. to C. W. BROWN, W. L. CLAUSE, and E. PITCAIRN (U.S.P. 1,632,617, and 1,632,666—7, 14.6.27. Appl., [A] 12.4.20. Renewed 18.12.26; [B, C], 25.9.26).—(A) The use of aromatic disubstituted dithiocarbamates, e.g., phenyl phenyldithiocarbamate, is claimed. (B) Carbon disulphide (10 pts.), aniline (20 pts.), and ethyl alcohol (5 pts.) when mixed and exposed to a current of air yield unstable *phenylammonium phenyldithiocarbamate*, which is treated with aqueous alcoholic zinc sulphate solution to give *zinc phenyldithiocarbamate*. (C) Phenyldithiocarbimide reacts with methylmercaptan to give *methyl phenyldithiocarbamate*.

T. S. WHEELER.

Vulcanisation of rubber. J. TEPPEMA, ASSR. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,637,790—1, 2.8.27. Appl., [A] 12.2.26; [B] 3.11.26).—Rubber is vulcanised in the presence of (A) a halogen derivative of a mercaptobenzthiazole, or (B) a metallic salt of the compound used in (A), as accelerator.

D. F. TWISS.

Vulcanisation of rubber substances. GRASSELLI CHEMICAL Co., Assees. of I. WILLIAMS and W. B. BURNETT (E.P. 249,113, 8.3.26. Conv., 13.3.25).—The condensation products obtainable from an aldehyde and a nitrogenous base generally have greatest accelerating power

when the molecular proportion of these ingredients lies between 2 : 1 and 7 : 1. Within this range, the most powerful accelerators of vulcanisation are obtained by the condensation of primary amines, *e.g.*, aniline, *n*-butylamine, ethylamine, and *o*-tolylidiguamide, with $\alpha\beta$ -saturated aldehydes, *e.g.*, propaldehyde, *n*-butaldehyde, and heptaldehyde (cf. E.P. 263,853; B., 1927, 757).
D. F. TWISS.

Plastic material. A. R. KEMP, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,638,535, 9.8.27. Appl., 12.7.24).—Material with the thermoplastic properties of gutta-percha is obtained by submitting a mixture of rubber with 8–16% S to a sufficiently high temperature.
D. F. TWISS.

Treatment [purification] of rubber latex. E. HOPKINSON and W. A. GIBBONS, Assr. to NAUGATUCK CHEMICAL Co. (U.S.P. 1,632,759, 14.6.27. Appl., 9.6.25).—Crude rubber latex is mixed with a small quantity of calcium polysulphide, being then easily filterable. The residue is further purified by treatment with a proteolytic enzyme, *e.g.*, trypsin, followed by extraction with acetone.
T. S. WHEELER.

Manufacture of artificial indiarubber. V. VILLA (E.P. 273,029, 1.4.26).—Vegetable or animal oil, *e.g.*, colza oil (75–85 pts.), is subjected to the action of sulphur chloride (6–12 pts.) in a closed vessel with exclusion of air, preferably between 0° and 15°. As catalyst (3–8 pts.) is used a rubber "substitute" produced from oil and sulphur chloride, from which the chlorine has been removed by dissolving in a liquid, *e.g.*, benzene, at a temperature preferably above 40°. It is thus possible to obtain a product containing only a small proportion of sulphur chloride without any previous oxidation or sulphuration of the oil.
D. F. TWISS.

Production of rubber articles. S. G. S. DICKER. From RUBBER LATEX RES. CORP. (E.P. 273,991, 14.2.27).—A jet of latex, containing fibre or fabric, is superficially coagulated by contact with acid, and is then allowed to undergo internal coagulation. The material is then pressed, washed, and dried. With approximately 25% or less of cotton fibre in random arrangement in 75% of rubber, the product has the character of a reinforced rubber material.
D. F. TWISS.

Reclaiming rubber. Mixing a soluble filler with rubber. C. H. CAMPBELL, Assr. to AMERICAN GLUE Co. (U.S.P. 1,640,817—8, 30.8.27. Appl., [A, B] 22.10.26).—(A) The albuminate of a metal is added to old rubber prior to "devulcanisation." (B) In mixing a soluble filler with rubber, with the use of heat, a fine spray of water is applied to the mass until the filler has dissolved and thorough incorporation has been effected. Moisture is subsequently expelled from the mass.
D. F. TWISS.

Method of forming sponge rubber materials [of great length]. E. C. R. MARKS. From FEATHEREDGE RUBBER Co. (E.P. 273,223, 11.4.27).

XV.—LEATHER; GLUE.

Tannin of *Heuchera Americana*, Linn. J. C. and B. L. DE G. PEACOCK (J. Amer. Pharm. Assoc., 1927, 16, 729–737, and Amer. J. Pharm., 1927, 99, 471–482).—The filtered, cold aqueous extract of the powdered

drug was extracted with ethyl acetate and the extracted matter purified by repeatedly re-dissolving in water, filtering, and re-extracting with ethyl acetate. An aqueous solution of the tannin obtained yielded only small extracts when treated successively with light petroleum, chloroform, benzol, and ether. The tannin, recovered from this aqueous solution by extraction with ethyl acetate, differs in appearance and solubility from the phlobaphen previously reported (Amer. J. Pharm., April, 1891), and conforms to the description given in the U.S. Pharmacopœia X for gallotannic acid, though it differs from this substance in some of the reactions described. *E.g.*, it gives precipitates with bromine water and with calcium hydroxide, dissolves readily in isopropyl alcohol, and yields with mineral acids a red insoluble phlobaphen together with traces of a crystalline substance which appears to be present in the drug as such, and not formed by hydrolysis. Under certain conditions of hydration the cold water-soluble tannin of the drug is soluble in ether, and the blue colour given by an ether extract with ferric salts is not due to gallic acid, as was originally supposed. Exhaustive treatment with hide powder of a cold water infusion failed to remove all substances yielding a blue colour with ferric chloride, but the coloration, unlike that due to gallic acid, was transient. The results of successive extraction of the drug with chloroform, ether, ethyl acetate, and acetone are described. Ethyl acetate (though an efficient extractive for aqueous infusions) removes very little tannin or phlobaphen from *Heuchera*, but acetone extracts all forms of tannin. Phlobaphen production shows that the tannin of *Heuchera Americana* is not gallotannic acid.
C. O. HARVEY.

Action of oxalic and hydrochloric acids on vegetable-tanned leathers. D. WOODROFFE (J. Soc. Leather Trades' Chem., 1927, 11, 251–255).—East Indian tanned goatskins were soaked in 1.25% and 4.0% solutions of oxalic acid for 30 min., washed, dried out, and samples tested for strength and stretch. There was only a slight diminution in strength with the stronger solution. The immediate weakening effect of an oxalic acid solution on leather was not serious. Similar tests with solutions of hydrochloric acid showed 35% lower strength by treatment of the leather with the stronger acid, but with the weaker solution of acid it was much lower than that of the leathers treated with the solutions of oxalic acid. Hydrochloric acid has a much more deleterious effect than oxalic acid on vegetable-tanned leathers.
D. WOODROFFE.

Action of hæmatin and hæmatin-ammonia solutions on vegetable-tanned leathers. D. WOODROFFE and A. MEADOWS (J. Soc. Leather Trades' Chem., 1927, 11, 262–264).—Pieces of East Indian tanned sheepskin and sumach-tanned skiver were soaked in 200 c.c. of water containing 1, 4, and 8% of hæmatin, respectively, with and without an addition of ammonia, for 20 min., struck out, dried out, and their tensile strength was determined. The results showed a distinct weakening effect by the hæmatin and ammonia solutions and by the stronger hæmatin solutions. The weakening effect was proportionately greater on skiver and thin leathers, which seems to indicate that it is the grain which

is most affected. Excessive amounts of hæmatin are deleterious on leather, probably owing to their tannin content.

D. WOODROFFE.

Action of hæmatin and iron salts on leather and fabric. D. WOODROFFE and D. B. GILBERT (J. Soc. Leather Trades' Chem., 1927, 11, 259—262).—Pieces of fabric and chrome-tanned goatskin, when treated with solutions of hæmatin, alone and mixed with ferrous sulphate and with ammonia, respectively, dried out, and tested for strength and stretch, showed no definite weakening effect by 1% or 2% solutions of hæmatin.

D. WOODROFFE.

Report of the committee on the revision of the official method for sampling tanning materials. (J. Amer. Leather Chem. Assoc., 1927, 22, 452—458).

Report of the committee on a provisional method for the separation of pyrogallol and pyrocatechol tannins for 1926—1927 (J. Amer. Leather Chem. Assoc., 1927, 22, 470—471).

Tannery waste disposal. W. HOWALT and E. S. CAVETT (Proc. Amer. Soc. Civil Eng., 1927, I, 1675—1712).

PATENTS.

Treatment of skins, e.g., hare skins. DELAHAYE, RACHET, & CIE. (F.P. 596,198, 12.7.24).—After the skins have been bated they are placed for 24 hrs. in a solution of sodium carbonate, bicarbonate, borate, chromate, dichromate, hypochlorite, hyposulphite, or any organic salt, or potassium hydroxide (d 1.075), then immersed in a sodium sulphide solution, unhaired, washed, and immersed in a second solution of sodium sulphide and excess sodium hydroxide until the mercuric sulphide stains have disappeared, after which the pelts can be tanned.

D. WOODROFFE.

Depilating and preserving hides and skins. E. BOHON, E. MAILLIARD, and P. MAILLIARD (F.P. [A] 590,901, 27.2.24; [B] 606,214, 17.2.25; [C] 612,583, 6.7.25).—(A) Soap is heated, phenol and caustic soda or other alkali or alkaline-earth hydroxide are added, and the diluted mixture is boiled. By immersing the skins in the liquor the hair is quickly loosened. (B) The phenol may be replaced by its derivatives or homologues, e.g., trichresol, and the caustic soda by other bases or by a salt having an alkaline reaction. (C) The skins are dipped in a solution of equal parts of resorcinol and a zinc salt, e.g., zinc sulphate, which preserves them against putrefaction and coagulates their albumin.

D. WOODROFFE.

Soaking hides. E. BOHON, E. MAILLIARD, and P. MAILLIARD (F.P. 612,409, 30.6.25).—The hides are soaked in a solution of starch, lime, calcium phenoxide, and sodium hydroxide which is effective in half the time required by other means.

D. WOODROFFE.

[Manufacture of] glue. W. R. LONG (U.S.P. 1,633,840, 28.6.27. Appl., 24.9.24).—Maize starch is heated under pressure with dilute hydrochloric acid, the product being neutralised with sodium carbonate and mixed with finely-ground sodium hydroxide and granulated borax.

T. S. WHEELER.

Treatment of leather. M. C. LAMB, Assr. to ROHM & HAAS Co., INC. (U.S.P. 1,640,706, 30.8.27. Appl., 6.3.26).—See E.P. 255,555; B., 1926, 839.

XVI.—AGRICULTURE.

Correlation of nutritive value with dry matter content of pastures. E. J. SHEEHY (Sci. Proc. Roy. Dublin Soc., 1927, 18, 389—398).—The composition and nutritive value of two pastures of differing reputation were studied by removing yard square turves of each and replanting them together with the successive layers of their accompanying soil and subsoil in adjacent pits. Similar plots were also laid down in which both turves were broken up and sown with the same seed mixture. A third plot was afterwards included, obtained from the locality in which the other turves were laid down. The herbage of the good pasture contained a higher percentage of dry matter than that from the poorer pasture. Although there were great differences in botanical composition, the chemical composition of the dry matter differed but slightly. The digestibilities, determined with guinea pigs, were similar. The dry matter content of the herbage may be correlated with botanical composition.

G. W. ROBINSON.

Soluble silicate content of soils. W. R. G. ATKINS (Sci. Proc. Roy. Dublin Soc., 1927, 18, 433—436).—Results are obtained for soluble silica in aqueous extracts of soils, using a colorimetric molybdate method, details of which are given. The amounts obtained vary from 18 to 124 pts./million. The results are too few in number for any general conclusions to be drawn.

G. W. ROBINSON.

Manuring trials with town sewage. W. ZIELSTORFF and A. KELLER (Z. Pflanz. Düng., 1927, B 6, 350—365).—Irrigation with sewage water containing the effluent from cellulose factories, which, as previously reported (B., 1926, 764), caused injury to crops, showed no after-effects in the second season. Town effluent containing little or no cellulose effluent improved the yield of mustard and oats. Effluents harmful to other crops did not injure grass land, but, on the other hand, did not increase the yield as did the ordinary sewage effluent alone. Oats treated with a mixed effluent adjusted to contain 75 g./litre of residue on evaporation showed a decreased yield of grain and a somewhat smaller amount of straw.

A. G. POLLARD.

[Plant] stimulants. E. HASELHOFF and W. ELBERT (Landw. Versuchs-Stat., 1927, 106, 285—322; cf. *ibid.*, 1922, 100, 59).—An exhaustive series of experiments on the effect upon subsequent growth and yield of treating seeds of many different plants with solutions of manganese and magnesium salts and of certain proprietary materials leads to the conclusion that no case can be made out for any general stimulating action of such solutions upon plant growth. The results obtained by Popoff could not be confirmed. C. T. GIMMINGHAM.

Practical value of soil experiments. HELLMERS (Z. Pflanz. Düng., 1927, B 6, 366—373).—The necessity of more than one type of experiment in the characterisation of a soil and its fertiliser needs is pointed out. In particular, the value of a study of soil and subsoil maps in conjunction with the determination of fertiliser requirement (Neubauer) and of the buffer capacity is emphasised.

A. G. POLLARD.

Ammonium sulphate and soil acidity. G. HAGEN (Z. Pflanz. Düng., 1927, B 6, 337—349).—Evidence is

advanced that the continued use of ammonium sulphate tends to increase soil acidity. The acid condition of many German soils is, however, not attributable to the excessive use of ammonium sulphate, nor can its ordinary use with other fertilisers be counted a source of danger to crops, except on soils already acid. The commonly accepted relative fertiliser values of ammonium sulphate and sodium nitrate, viz., 94:100, depend on trials in which soil reaction has not been considered. Under comparable conditions in this respect, the ratio should be more favourable to ammonium sulphate.

A. G. POLLARD.

Granulated cyanamide and dicyanodiamide. G. LEFORT DES YLOUSES (*Chim. et Ind.*, 1927, 18, 216).—Conclusions adverse to "granulated cyanamide" based on cultures in pots are criticised as unfair. Dicyanodiamide which is present in this particular fertiliser is not directly poisonous to plants, but in certain proportions may paralyse the nitrifying bacteria. General experience has shown that the altered granules containing a large proportion of dicyanodiamide are as efficacious as a source of nitrogen as other forms of cyanamide, and cases are quoted in which cyanamide containing 80% of dicyanodiamide gave a better yield with potatoes than nitrate of lime. The explanation is that the hard granules break down too slowly for a poisonous concentration of dicyanodiamide to be reached on the soil.

C. IRWIN.

Determination of ammonia in fertilisers and soils by a cold method. A. DEMOLON (*Ann. Falsif.*, 1927, 20, 412—413).—A rapid current of air, freed from ammonia by sulphuric acid, is passed for 2½ hrs. through the material (0.5—2 g. of fertiliser or 25 g. of soil) mixed with a solution of sodium carbonate or soap lye, with the addition of alcohol if frothing occurs, and thence into standard sulphuric acid. The ammonia is determined by titrating the excess of sulphuric acid. F. R. ENNOS.

PATENTS.

Preserving seeds from attack by pests. SACHARIN-FABR. A.-G., and A. KLAGES (E.P. 274,974, 1.5.26).—The seed is treated with a complex mercury salt of an aromatic base, or with a mixture of salts which, by interaction with water, produces the complex mercury salt. For example, 2 pts. of mercury aniline hydrochloride are finely ground with 1 pt. of sodium chloride, the mixture being made up by addition of sodium sulphate and a dyestuff to contain a desired amount of mercury. The product is readily soluble in water.

C. T. GIMINGHAM.

Treatment of lime-nitrogen [calcium cyanamide]. J. M. A. STILLESSEN (U.S.P. 1,633,200, 21.6.27. Appl., 8.8.19. Renewed 9.11.26).—Crude calcium cyanamide is treated with water and carbon dioxide under pressure at 135° to form carbamide and calcium carbonate.

T. S. WHEELER.

Treatment of phosphate rock. H. BLUMENBERG, JUN., ASS. to STOCKHOLDERS SYNDICATE (U.S.P. 1,638,677, 9.8.27. Appl., 23.6.25).—Ground phosphate rock is mixed with ammonium sulphate and heated in a closed retort at below 300°, to form ammonium phosphates and calcium sulphate.

H. ROYAL-DAWSON.

Decomposition of crude phosphates. RHEINANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assees. of F. RÜSBERG (G.P. 440,001, 22.6.23).—Dry, non-caking phosphatic fertilisers are produced directly by treating crude phosphates with a mixture of nitric acid of, e.g., 50% strength and concentrated sulphuric acid in the presence of more than one equivalent of an alkali or ammonium sulphate, calculated on the weight of calcium nitrate formed during the decomposition.

L. A. COLES.

Conversion of fertilisers (U.S.P. 1,632,312).—See XVIII.

XVII.—SUGARS; STARCHES; GUMS.

PATENTS.

Preservation of cane juice. A. K. BALLS (U.S.P. 1,634,348, 5.7.27. Appl., 30.7.26).—Cane juice for subsequent use in growing yeast is inoculated with lactic acid bacilli, and kept at 55° until formation of acid ceases.

T. S. WHEELER.

Production of glucose [dextrose] from materials containing cellulose. L. MEILER and H. SCHOLLER (U.S.P. 1,641,771, 6.9.27. Appl., 10.9.24. Conv., 26.3.21).—See G.P. 407,412; B., 1925, 518.

Recovery of hydrochloric acid in glucose manufacture (U.S.P. 1,633,877).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Constitution and determination of pectins and gums in grape wines and musts. L. SEMICHON and M. FLANZY (*Ann. Falsif.*, 1927, 20, 395—399).—The pectins of wines and musts consist of a methyl ester of pectic acid combined with glycerophosphates of calcium, magnesium, and a little aluminium. The proportion of glycerophosphate is practically constant in the freshly precipitated pectins. For the determination of pectins and gums, 100 c.c. of the wine or must, acidified with 1 c.c. of hydrochloric acid, are treated with alcohol so that the mixture contains 80% of alcohol by vol. After being kept for about 24 hrs., the precipitate consisting of the pectins and gums is collected, washed with 80% alcohol till free from acid, dried at 90°, and weighed. It is redissolved in cold water, 20 c.c. of 0.1N-potassium hydroxide free from carbonate are added, and the whole is heated under a reflux for 1 hr., cooled, and acidified with acetic acid. After adding 10% calcium chloride solution, the whole is again refluxed for ¼ hr., the precipitate of calcium pectate collected, washed with water, dried at 90—95°, and weighed. The calcium content of the precipitate has been found practically constant in all determinations, viz., 6.46%, giving a minimum mol. wt. for pectic acid of 581. The gums are precipitated from the filtrate by means of alcohol, filtered, washed with alcohol, dried at 90—95°, and weighed. The proportion of pectin may be obtained by difference, or may be expressed by the weight of calcium pectate obtained.

F. R. ENNOS.

PATENTS.

Manufacture and propagation of yeast. DISTILLERS Co., LTD., and E. A. MEYER (E.P. 275,328—9, [A, B] 4.5.26).—(A) During the so-called continuous fermentation process, degeneration of the yeast is prevented by a frequent change of feeding material which

renders possible the control at different stages of the fermentation of the ratio between proteins and carbohydrates and the content of vitamins. (b) The medium in which the yeast propagation takes place is sterilised by the addition of a halogen, preferably chlorine, or of materials capable of liberating a halogen.

C. RANKEN.

Stopping or arresting alcoholic fermentation. H. BOULARD (F.P. 615,014, 23.4.26).—Fermenting liquor is heated to a point above the lethal, but below the usual sterilising temperature, *e.g.*, to 43°, the process being repeated if necessary.

L. A. COLES.

Ageing of alcoholic liquors etc. SIEMENS & HALSKE, A.-G., H. ENGELHARDT, and K. ENGELHARDT (E.P. 275,454, 16.11.26. Conv., 21.10.26).—Alcoholic beverages and fruit juices are matured by the addition of ozone and the products of oxidation affecting the flavour removed by the addition to the ozonised liquid of powdered substances of density not exceeding 4, *e.g.*, burnt magnesia.

C. RANKEN.

Conversion of chemical fertilisers to yeast and other organic substances. F. C. RAETH (U.S.P. 1,632,312, 14.6.27. Appl., 25.1.24).—A nutrient solution suitable for growing yeast contains dextrose, ammonium and magnesium sulphates, ammonium phosphate, potassium dihydrogen phosphate, sodium chloride, calcium carbonate, and traces of ferrous and manganese sulphates. Continued aeration of the solution is necessary.

T. S. WHEELER.

Recovery of hydrochloric acid [in the manufacture of dextrose from wood waste]. H. TERRISSE and M. LÉVY (U.S.P. 1,633,877, 28.6.27. Appl., 15.3.21. Conv., 25.3.20; cf. E.P. 154,170; B., 1921, 484 A).—The mixture of lignin, dextrin, dextrose, and hydrochloric acid, obtained in the preparation of dextrose from wood, is introduced at the top of a cylindrical vessel containing a number of superposed hollow shelves, the passage of the material through the apparatus being directed by rotating rakes. The shelves are maintained at a temperature of 50°, and a current of air heated at the same temperature is passed through the cylinder in counter-current to the material. Vaporised hydrochloric acid is condensed from the air current, which is then returned to the cylinder.

T. S. WHEELER.

Preservation of cane juice (U.S.P. 1,634,348).—See XVII.

Malted milk (U.S.P. 1,636,563).—See XIX.

XIX.—FOODS.

Determination of the freshness of milk. G. INICHOFF (Z. Unters. Lebensm., 1927, 53, 435—449).—Morres' method of judging the freshness of milk has been critically examined. The method consists in titrating the acidity of 40 c.c. of milk with 0.1N-caustic soda and the "degree of freshness" by adding to an equal volume of milk the exact volume of 0.1N-sulphuric acid to cause the casein to separate. The sum of the two titers is called the "coagulation value," and should not be less than 26. Observations on a large number of samples over a period of two years showed variations within wide limits. Using samples of 100 c.c. each in the place of the 40 c.c., "coagulation values" between

56.8 and 95.0 were obtained. Values below 50.0 were rare, 22% of the tests being above 70.0 and 92% between 50 and 80. The fixing of a definite value for normal milk is therefore difficult, but any milk giving a value below 50 should be regarded with suspicion. Addition of water to milk lowers both its acidity and "freshness," the "coagulation value" being correspondingly reduced. Preservatives in most cases appreciably increase the "coagulation value," sodium fluoride being an exception, whilst salicylic and benzoic acids have no influence. Heating or freezing milk produces a lowering of both acidity and "freshness." In view of the wide variations amongst different classes of milk and the powerful influence of a number of factors, the method is not considered suitable for the examination of fresh milk.

H. J. DOWDEN.

Significance of solids-not-fat for the detection of watered milk. A. GRONOVER and F. TÜRK (Z. Unters. Lebensm., 1927, 53, 520—524).—Teichert's observation that milk containing less than 8% of solids-not-fat must have been diluted with water is only true within certain limits. Amongst 650 samples collected at the time of milking, 14% contained 7—8.25% of solids-not-fat. It is shown that determination of the lowering of f.p. is a much more reliable means of detecting the addition of water, especially as in many cases the ordinary determinations of total solids, fat content, etc. give no indication of the addition. Measurement of the refractive index in such cases affords additional evidence.

H. J. DOWDEN.

Determination of chlorine in milk. F. MACH and W. LEPPER (Z. Unters. Lebensm., 1927, 53, 454—458).—Filippo and Adriani's observation (cf. B., 1926, 847) that the ashing process leads to a loss of chlorine has been confirmed. Direct titration by Drost's method (cf. B., 1923, 994 A; 1925, 822) involves errors owing to the bulky precipitate, and the end-point of the Volhard titration is not sharp. The relative merits of several methods for the removal of proteins before titrating the chloride have been investigated. To 50 c.c. of milk, diluted to 400 c.c., 50 c.c. of nitric acid were added, the mixture being filtered after shaking. Determination of chlorine by the Volhard method in 100 c.c. of the clear filtrate gave a moderately sharp end-point in the presence of ether. Weiss' method, using as precipitant 25 c.c. of 20% aluminium sulphate solution and 20 c.c. of 8% caustic soda solution, gave a granular precipitate and a sharp end-point without the addition of ether. Satisfactory results were obtained by using tannic acid; 50 c.c. of milk diluted to 400 c.c. are shaken in succession with 5 c.c. of 10% tannic acid, 10 c.c. of 10% oxidised ferrous sulphate solution, saturated sodium carbonate solution to alkalinity, 1 c.c. of 3% hydrogen peroxide, and acetic acid in excess. The filtrate was clear and the end-point very sharp. The three methods gave concordant results, which were higher than those obtained by ashing processes. Application of the methods to milk powders gave similar results. Subsequent investigations showed that phosphotungstic acid is the simplest precipitant. To 50 c.c. of milk, diluted to about 400 c.c., are added 40 c.c. of phosphotungstic acid solution (50 g. with 500 c.c. of nitric acid, *d* 1.4, diluted to 1 litre). After shaking and filtering, 100 c.c. of the filtrate are

titrated by the Volhard method [in the presence of ether. H. J. DOWDEN.

Simplified chlorine determination in milk. G. SCHULZE (Milch. Zentr., 1927, 56, 157—160).—A method based on that of Drost (cf. B., 1923, 994 A), has been developed, which makes use of the so-called Martius-Lüttke's 0.1*N*-silver nitrate solution, consisting of 17.5 g. of silver nitrate, 900 c.c. of nitric acid diluted to 1 litre, and containing an iron indicator. 10 c.c. of milk are vigorously shaken with 5 c.c. of the silver nitrate solution, the protein and silver chloride being precipitated in fine particles. The excess silver is titrated with 0.1*N*-ammonium thiocyanate solution, with vigorous shaking after each addition. The end-point is best detected if the precipitate is allowed to settle. The results are in fair agreement with those obtained by the ashing process. H. J. DOWDEN.

Securing true average samples of milk. K. WEBER (Z. Unters. Lebensm., 1927, 53, 449—454).—The fat contents have been determined in numerous samples of milk withdrawn from the vessel before and after stirring, by dipping the ladle to the bottom of the can, and after pouring several times from one vessel to another. The results show that a thorough stirring is sufficient for sampling purposes. H. J. DOWDEN.

Methylene Blue (reductase test) in milk grading. M. GRIMES, H. S. B. BARRETT, and J. REILLY (Sci. Proc. Roy. Dublin Soc., 1927, 18, 437—441).—The decolorisation of Methylene Blue by reductase from bacteria has been proposed as the basis of a test for grading milk. It was found that samples of Methylene Blue of different origin were all suitable for the test, and that stock solutions were stable over a period of nine months. Variations between duplicates and triplicates were increased with longer reduction times. When the reduction time was less than 2 hrs., variations in the ratio of Methylene Blue to milk did not alter the result obtained. G. W. ROBINSON.

Simplified molecular constant applied to milks from the Somme. G. JORET and E. RADET (Ann. Falsif., 1927, 20, 341—353, 403—411).—Analyses are given of genuine milks from various herds fed in different parts of the Somme valley, together with their simplified molecular constants (C.M.S.). For 196 milks from individual cows the C.M.S. varies between 69.2 and 82.8, with an average value of 73.9. Only 5 fall below 70, which figure, with a margin of one unit below, is thus confirmed as the lower limit for genuine milks. Mixtures of milks from different cows often show lower C.M.S. values than those calculated from their constituent milks. The C.M.S. is influenced by the nature of the food, replacement of pasturage by oil cake or bran food tending to lower it, by the amount of food, the age of the cow, the stage of lactation, and the hour of milking. F. R. ENNOS.

Testing the refractive index and lactose content of milk from individual herds. G. SCHULZE (Z. Unters. Lebensm., 1927, 53, 509—520).—The variations in the composition of the milk from 5 herds of cows have been observed at regular intervals over a period of one year, determinations being made of the density, fat content, solids-not-fat, lactose, chlorine, and refractive index.

In March and April, when grazing commenced, there was a perceptible fall in the lactose content of the milk, which was restored in winter when artificial feeding was resumed. The milk from cows which had been artificially fed throughout the year did not show any great variation in lactose content. Graphical representation of the results showed a distinct parallelism between the lactose content and the refraction of the calcium chloride serum. H. J. DOWDEN.

Determination of small amounts of benzoic acid in milk, butter, margarine, meat, and eggs. J. GROSSFELD (Z. Unters. Lebensm., 1927, 53, 467—483).—The determination of benzoic acid involves three processes, viz., procuring an aqueous solution of the acid, extracting this solution by means of a solvent, determination of the extracted material. To secure a satisfactory aqueous solution avoidance of emulsions by removal of proteins is essential, the most suitable methods being steam distillation or precipitation with 15% solution of potassium ferrocyanide and 30% solution of zinc sulphate. For the extraction from the aqueous solution, benzene was found to be a satisfactory solvent. When, however, the concentration of benzoic acid is less than 5 mg./100 c.c., the partition coefficient is not constant and Berthelot's law no longer applicable. By shaking equal volumes of benzene and an aqueous solution of sodium benzoate acidified with 2 c.c. of sulphuric acid per 100 c.c. of benzoate solution, 80% of the benzoic acid is extracted by the benzene. For the determination of the extracted acid, weighing or titration with alkali are only applicable for relatively large quantities of material. For very low concentrations the colorimetric methods of Jones (cf. B., 1925, 824) are preferable. The red coloration produced by the Mohler reaction as modified by Grossfeld (cf. A., 1916, ii, 158) is compared with a standard iron solution mixed with thiocyanate solution. The details of procedure with the various foodstuffs are given. H. J. DOWDEN.

Microscopical determination of husk content of cocoa powders. R. TURNAU (Z. Unters. Lebensm., 1927, 53, 483—486).—The number of scleridæ in pure cocoa powders, cacao husks, and commercial cocoas have been determined by a modification of Griebel and Sonntag's method (cf. B., 1926, 605). The dry, fat-free substance (0.5 g.) in 10 c.c. of 0.1*N*-caustic soda and 90 c.c. of water is gently boiled for 1 hr. with 2.5 c.c. of 30% hydrogen peroxide. After cooling, a further 10 c.c. of 0.1*N*-caustic soda are added and the mixture is again boiled for $\frac{1}{2}$ hr. After allowing the sediment to settle the staining and counting are performed as described by Griebel and Sonntag (*loc. cit.*). The cacao husks were ground to pass 0.15-mm. mesh, the coarse residue being dried for 2 hrs., reground, and sieved. The powdered material was then defatted by extraction with ether for 6 hrs., followed by refluxing with trichloroethylene (100 c.c. per 10 g. of material). The results were in good agreement with those obtained by Griebel and Sonntag, but appreciably lower than those of Plücker (cf. B., 1926, 105). One of the commercial powders showed a number of scleridæ greater than 400 per mg. of fat-free substance, which Griebel and Sonntag regarded as the highest permissible number for unadulterated cocoa. H. J. DOWDEN.

Analysis of conserves containing apple and fruit (determination of the apple-fruit ratio). C. F. MUTTELET (Ann. Falsif., 1927, 20, 391—394).—The citric acid content of fruit juices used in mixed fruit and apple conserves is practically constant for each type of fruit, the minimum value for black currants being 3%, for gooseberries 2%, and for raspberries and strawberries 1%. Apple juice contains no citric acid. In the absence of added citric acid, the fruit juice content in a conserve may be calculated from the citric acid contents of the conserve and of the fruit juice. The apple content is obtained by deducting that of the sugar and of the fruit juice from 100.

F. R. ENNOS.

"Mikrobin" [sodium *p*-chlorobenzoate as food preservative]. C. VON DER HEIDE and R. FÖLLEN (Z. Unters. Lebensm., 1927, 53, 487—509).—Methods for the detection and determination of *p*-chlorobenzoic acid, its solubility in water, alcohol, and fruit juices, and its fungicidal efficiency have been investigated. Mohler's reaction as modified by von der Heide and Jakob (cf. A., 1910, ii, 359) is used to distinguish benzoic acid from the *p*-chloro-acid. The alkaline extract is evaporated to dryness and nitrated at 130—140° with a few drops of sulphuric acid and nitric acid. After cooling, excess of ammonia solution is added, followed by 10% solution of sodium sulphide until a reddish-brown colour appears. With *p*-chlorobenzoic acid the colour deepens on boiling, but with benzoic acid it vanishes. With hydroxylamine, nitrated benzoic acid gives a red coloration, *p*-chlorobenzoic acid no colour. For the determination of *p*-chlorobenzoic acid in foods, the acid is separated by steam distillation or by ether extraction, and chlorine determined in the distillate or extract by evaporating to dryness, treating with sodium peroxide, gently igniting, and precipitating as silver chloride. Ether extraction gives slightly low results, and is recommended for food analysis. The solubility of *p*-chlorobenzoic acid in water-alcohol mixtures varies from 0.078 g./litre for water to 31.3 g./litre for pure alcohol. The relative efficiencies of benzoic, salicylic, and *p*-chlorobenzoic acids in arresting the growth of micro-organisms have been investigated. Against yeast fermentation *p*-chlorobenzoic acid was most effective, against moulds benzoic acid, whilst against mother of vinegar benzoic and salicylic acids were both more potent than *p*-chlorobenzoic acid, but the tests in this case were not under anaerobic conditions. The statement of Neissen and Tillmans that mikrobin may be used in foodstuffs, provided it is declared, is strongly opposed, since the addition to wines of mikrobin is already prohibited.

H. J. DOWDEN.

Nutritive value of pastures. SHEEHY.—See XVI.

Determination of water. CANTZLER and ROTH-SCHILD.—See XX.

PATENTS.

Manufacture of yeast foam malted milk. C. B. HILL and M. H. GIVENS, Assrs. to NORTHWESTERN YEAST CO. (U.S.P. 1,636,563, 19.7.27. Appl., 12.3.26. Cf. U.S.P. 1,589,853; B., 1926, 719).—Yeast is grown on a nutritive medium of cereal flour treated with a malt cereal, after which the mixture is mashed, neutralised if necessary, and heated at 88—97° for 30—60 min. to stop yeast growth and fermentation, and also to gelatinise

the starch, dissipate alcohol formed, and sterilise the product. Milk is then added, the whole heated at 70° for at least 30 min, and dried to a powder if desired.

F. R. ENNOS.

Manufacture of processed cheese. E. E. ELDRIDGE, Assr. to PABST CORP. (U.S.P. 1,634,410, 5.7.27. Appl., 19.7.24).—Neutralised whey is concentrated to a paste and mixed with ground cheese (9 pts.), the mixture being then pasteurised.

T. S. WHEELER.

[Protectively] treating food products. W. W. WILLISON, Assr. to THERMOKEPT CORP. (U.S.P. 1,637,068, 26.7.27. Appl., 2.2.24).—The selected food product, while subjected to a high vacuum, is completely immersed in a suitable liquid, *e.g.*, water or brine, below 38°, and the vacuum maintained until the occluded gases are removed and the mass becomes quiescent. The vacuum is then broken, thus allowing the liquid to penetrate and surround the cell structure of the food product, and the whole is heated at 100° to hydrolyse the starch, which then acts as a protective seal.

F. R. ENNOS.

Extraction of volatile odorous constituents of oils, especially edible oils. N. C. FURBO (G.P. 439,615, 14.5.24).—The oil is treated with a counter-current of gas or steam in apparatus comprising a number of superposed, rotating, concave plates. The oil is fed on to each plate near the centre, and flows over it in a thin film towards the outer lip, whence it is conveyed to the next plate.

L. A. COLES.

Manufacture of edible substances. P. N. WOO (E.P. 269,576, 14.4.27. Conv., 15.4.26).—See U.S.P. 1,602,958; B., 1927, 26.

Beating of viscid liquids [*e.g.*, eggs] (E.P. 274,976).—See I.

Active charcoal from cacao refuse (G.P. 440,381).—See II.

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

Determination of water in organic substances by means of calcium carbide. A. CANTZLER and S. ROTH-SCHILD (Z. Unters. Lebensm., 1927, 53, 425—435).—About 3 g. of the substance are weighed in a flask in which is a small beaker containing about 5 g. of calcium carbide. The flask is fitted with inlet and outlet tubes which are closed during the weighing. The inlet tube is then connected to a series of soda-lime and drying tubes, the outlet tube being fitted to an aspirating device. After overturning the beaker and mixing the calcium carbide with the substance under examination, the flask is heated for 1 hr. on a toluene bath at 60—100° according to the nature of the material. Residual acetylene is removed by a current of dry air free from carbon dioxide, and, after cooling, the loss of weight is determined. Theoretically, 1 g. of acetylene is equivalent to 1.385 g. of water, but blank experiments using sand moistened with a known weight of water showed that this factor is too low, and that 1.443 gives more accurate results, the disparity being attributed to the fact that part of the water is absorbed by the lime formed. The results with the higher factor are in good agreement with those obtained by vacuum desicca-

tion. For such materials as spices and seasoned food-stuffs the method has the advantage that there is no risk of volatilising the ethereal oils. The use of aluminium carbide in place of calcium carbide and determination of the methane evolved was found to be unsatisfactory.

H. J. DOWDEN.

Determination of total citric acid in solution of magnesium citrate. J. L. MAYER (J. Amer. Pharm. Assoc., 1927, 16, 719–722).—The method given in the U.S. Pharmacopœia X for the examination of "Solution of magnesium citrate" is criticised, and a modification is described in which the titration of free citric acid is carried out in a platinum dish, the residual solution being evaporated and ashed prior to titrating for combined citric acid. Magnesium may be determined gravimetrically in the final residual solution, but it is claimed that determinations made directly on a solution of the citrate are just as accurate.

C. O. HARVEY.

Determination of nicotine in tobacco. L. FRANK (Chem.-Ztg., 1927, 51, 658).—10 g. of finely-divided air-dried tobacco are shaken for $\frac{1}{4}$ hr. with 10 c.c. of 20% potassium hydroxide solution, 100 c.c. of ether, and 100 c.c. of light petroleum in a graduated Röhrig separator having a side tube and tap. After allowing to settle for several hours, 100 c.c. of the clear liquid are drawn off, filtered through ignited sodium sulphate, the filter washed with dry ether, and the solvent distilled from the filtrate. The greenish residue is extracted with cold water, collected on a filter, and the colourless filtrate titrated with 0.1N-hydrochloric acid, using Congo Red as indicator. Test results were in satisfactory agreement with those obtained by the standard methods.

F. R. ENNOS.

Solubilities of drugs in glycerol. J. A. ROBORGH (Diss., Amsterdam, 1927, 1–45).—The solubilities in 86.5% and 98.2% glycerol of the following substances have been determined: ethyl acetate, stearic acid, anæsthesine, sodium arsenate, ammonium carbonate, guaiacol, guaiacol carbonate, codeine hydrochloride, calcium and sodium hypophosphites, novocaine, phenacetin, phenol, sodium pyrophosphate, atropine sulphate, tannin, quinine tannate, theobromine, zinc valerate, vanillin.

CHEMICAL ABSTRACTS.

Theoretical structure of the correction factor as applied in the menthol assay of peppermint oil. Assay of oil of rosemary. S. MENDELSON (J. Amer. Pharm. Assoc., 1927, 16, 726–729).—The method of calculation of the menthol content of peppermint oil from figures obtained by saponification of the acetylated oil as given in the U.S. Pharmacopœia X is explained. Modification of Power and Kleber's original formula (cf. B., 1895, 594) is necessary owing to the presence of up to 10% of menthol esters in the oil. The same correction factor is applied in the assay of oil of rosemary, though in this case the figure sought is the % of total borneol. The official method assumes that menthol and borneol are the only acetyltable constituents, and that they are the only alcohols and their acetates the only esters present. As none of these assumptions is strictly correct, Parry's method (involving a determination of combined alcohols by saponification and of total

alcohols in the saponified oil) is recommended, the necessary correction for the reduction in weight during saponification being derived from the amount of alkali absorbed.

C. O. HARVEY.

Determination of benzoic acid. GROSSFELD. "Mikrobin" as preservative. VON DER HEIDE and FÖLLEN.—See XIX.

PATENTS.

Manufacture of formates. L'Air Liquide, Soc. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (E.P. 246,168, 16.1.26. Conv., 17.1.25).—Formates are prepared by the action of carbon monoxide or gases containing it (especially gases rich in it separated by partial liquefaction of water-gas etc. containing it together with hydrogen) on caustic alkali solutions or on suspensions of alkaline-earth hydroxides in liquids or in alkali carbonate solutions, at pressures above 300 atm. and at a temperature about 400°. By the use of concentrated solutions, e.g., those containing 400 g. of sodium hydroxide per litre, solutions of formates containing 700 g. per litre are formed.

B. FULLMAN.

Production of acetaldehyde. A. E. CRAVER, Assr. to BARRETT Co. (U.S.P. 1,636,952, 26.7.27. Appl., 13.3.22).—A mixture of ethyl alcohol and air (14 pts.) is passed at about 300° over a catalyst comprising vanadium oxide, with or without addition of molybdenum oxide, the time of contact being about 0.4 sec.

T. S. WHEELER.

Manufacture of alkali xanthates. W. HIRSCHKIND, Assr. to GREAT WESTERN ELECTRO CHEMICAL Co. (U.S.P. 1,636,229, 19.7.27. Appl., 3.11.25; cf. U.S.P. 1,507,089; B., 1924, 908).—The addition of a small quantity of a chlorohydrocarbon, e.g., carbon tetrachloride, to the mixture of carbon disulphide, alcohol, and alkali hydroxide employed in the preparation of alkali xanthates, enables the reaction to be carried out at a lower temperature (10°) with increased yield.

T. S. WHEELER.

Production of maleic acid. A. E. CRAVER, Assr. to BARRETT Co. (U.S.P. 1,636,857, 26.7.27. Appl., 23.5.23).—A mixture of benzene vapour and air (13 pts.) is passed at about 500° over a catalyst containing vanadium oxide (70%) and molybdenum oxide (30%), the time of contact being about 0.3 sec.

T. S. WHEELER.

Manufacture of lactic acid esters. CANADIAN ELECTRO PRODUCTS Co., LTD., Assees. of H. W. MATHESON and K. G. BLAIE (E.P. 257,907, 11.8.26. Conv., 3.9.25).—Acetaldehydecyanohydrin (lactonitrile) is treated with aqueous alcoholic hydrogen chloride containing not more than 1 mol. of water and a small excess of hydrogen chloride. Ethyl lactate, b.p. 148–150°, is obtained in 90% yield by passing hydrogen chloride into a mixture of ethyl alcohol, lactonitrile, and 35.5% hydrochloric acid at 30–35° until 1.01 mol. of hydrogen chloride is present, and heating for 3 hrs. at 80°. Alcoholic hydrogen chloride may be used instead of the gas.

C. HOLLINS.

Manufacture of esters of α -hydroxy-acids. CANADIAN ELECTRO PRODUCTS Co., LTD., Assees. of H. W. MATHESON and K. G. BLAIE (E.P. 264,143, 18.12.26. Conv., 11.1.26. Cf. E.P. 257,907; preceding abstract).—

The process of the prior patent is applied generally to the conversion of cyanohydrins of aldehydes and ketones (excluding acetaldehyde) into esters of α -hydroxy-acids. Examples are ethyl furylglycollate, ethyl α -hydroxy-*n*-valerate, ethyl α -hydroxyisobutyrate, and ethyl mandelate.

C. HOLLINS.

Manufacture of hexamethylenetetramine. C. B. CARTER, Assr. to S. KARPEN & BROS. (U.S.P. 1,635,707, 12.7.27. Appl., 9.2.24; cf. U.S.P. 1,566,820—2; B., 1926, 217).—Methylene chloride is heated at 100° under pressure with a large excess of ammonia solution saturated with hexamethylenetetramine and ammonium chloride. The resulting liquor is cooled, treated with ammonia, and used again, the mixture of hexamethylenetetramine and ammonium chloride precipitating being separated into its components in any suitable manner.

T. S. WHEELER.

Manufacture of furan. W. C. WILSON, Assr. to QUAKER OATS Co. (U.S.P. 1,636,030, 19.7.27. Appl., 28.11.24).—Furfuroic acid if heated at 250° partly sublimes and the remainder decomposes to furan and carbon dioxide. The acid is separated by cooling the vapours to 40°, carbon dioxide being removed with sodium hydroxide solution, and furan condensed by cooling with brine.

T. S. WHEELER.

Barbituric acid derivative. E. H. VOLWILER, Assr. to ABBOTT LABORATORIES (U.S.P. 1,636,201, 19.7.27. Appl., 16.7.26).—5-*n*-Butylbarbituric acid is treated with allyl bromide in presence of concentrated sodium hydroxide solution in the cold, to give 5-*n*-butyl-5-allylbarbituric acid, m.p. 128°.

T. S. WHEELER.

Manufacture of alkoxyamino-[8-amino-6-alkoxy]-quinolines. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 275,277, 4.2.26).—8-Nitro- or 8-azo derivatives of 6-alkoxyquinolines are reduced, or 8-amino-6-hydroxyquinoline is alkylated, or 8-acylamino- or 8-arylideneamino-6-hydroxyquinolines are alkylated and hydrolysed, the process of E.P. 267,457 (B., 1927, 379) being excluded. 8-Amino-6-methoxyquinoline, m.p. 41°, is obtained by reducing the nitro-compound with iron and acetic acid. 8-Amino-6-hydroxyquinoline, m.p. 177°, obtained from the 8-nitro-compound, m.p. 239—240° (decomp.), prepared by Skraup's reaction or by demethylation of the methyl ether, is converted into the formyl derivative, ethylated with sodium ethoxide and ethyl iodide, and hydrolysed to give 8-amino-6-ethoxyquinoline, m.p. 60°, b.p. 144—145°/1 mm. 8-Amino-6-hydroxyquinoline, treated with methyl sulphate and alkali, gives 8-amino-6-methoxyquinoline, b.p. 145—147°/2 mm.

C. HOLLINS.

Manufacture of preparation which emits β -rays. M. WRESCHNER and L. F. LOEB (E.P. 265,600, 3.2.27. Conv., 8.2.26. Addn. to E.P. 248,765; B., 1926, 768).—The process of the prior patent is modified in that the ferric salt there used is replaced by an aluminium salt.

B. FULLMAN.

Manufacture of betaine hydrochloride. D. K. TRESSLER, Assr. to LAROWE CONSTRUCTION Co. (U.S.P. 1,634,221, 28.6.27. Appl., 13.7.25).—The process described in E.P. 265,831 (B., 1927, 423) is modified in that in place of hydrochloric acid, a mixture of sulphuric acid and calcium chloride is added to the solution under treatment.

T. S. WHEELER.

Manufacture of a glucoside of *Adonis Vernalis*, L. F. HOFFMANN-LA ROCHE & Co., A.-G. (E.P. 265,941, 20.1.27. Conv., 13.2.26).—The aqueous or alcoholic extract of *herba adonidis vernalis* is treated directly, or after treatment with lead acetate, with an adsorbing agent such as animal charcoal. The latter is extracted with chloroform, and the chloroform solution concentrated and poured into light petroleum. The cardio-active glucoside separates as a thick oil, which, on dissolution in water and evaporation *in vacuo*, yields a brown powder.

B. FULLMAN.

Preparation of stable colloidal silver solutions. W. TRAXL (U.S.P. 1,633,466, 21.6.27. Appl., 6.3.24. Conv., 13.3.23).—A mixture of silver oxide and hydroxide (1%) is added to an oil or fat at 250°, the solution obtained being filtered. The filtrate is treated with hydrogen, and the process is repeated several times. The product has therapeutic value.

T. S. WHEELER.

Stable and sterilisable solutions containing organic phosphorus and complex auro-compounds. I. G. FARBENIND. A.-G., Assees. of L. BENDA (U.S.P. 1,635,169, 12.7.27. Appl., 18.6.26. Conv., 22.6.25).—A dialkylaminoarylphosphinous acid, e.g., *p*-dimethylaminotolylphosphinous acid, is dissolved as the sodium salt in a sodium aurothiosulphate solution containing sodium thiosulphate, the resulting solution being sterilised with steam and employed in the treatment of tuberculosis.

T. S. WHEELER.

Manufacture of synthetic camphor by means of a liquid catalyst. L. and E. DARRASSE, and L. DUPONT (U.S.P. 1,641,579, 6.9.27. Appl., 1.6.21. Conv., 9.6.20).—See E.P. 164,357; B., 1922, 610 A.

Manufacture of borneol esters. H. BLUM, Assr. to SOC. ALSACIENNE DE PROD. CHIM. (U.S.P. 1,640,639, 30.8.27. Appl., 18.3.25. Conv., 28.3.24).—See E.P. 231,468; B., 1925, 900.

Manufacture of betaine hydrochloride. D. K. TRESSLER, Assr. to LAROWE CONSTRUCTION Co. (U.S.P. 1,634,221, 28.6.27. Appl., 2.7.24).—See E.P. 265,831; B., 1927, 423.

Manufacture of diarylguanidines. C. J. T. CRONSHAW and W. J. S. NAUNTON, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,639,724—5, 23.8.27. Appl., [A] 28.6.26, [B] 2.3.27. Conv., [A, B] 4.7.25).—See E.P. 255,220; B., 1926, 769.

Manufacture of metallo-organic [alkyl] compounds. C. A. KRAUS and C. C. CALLIS, Assrs. to STANDARD DEVELOPMENT Co. (U.S.P. 1,639,947, 23.8.27. Appl., 13.4.23).—See E.P. 214,221; B., 1925, 827.

Heavy-metal mercaptosulphonic [acid] compounds. CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING), Assees. of A. FELDT, W. SCHOELLER, and E. BORGWARDT (U.S.P. 1,633,626, 28.6.27. Appl., 10.12.25. Conv., 15.12.24).—See E.P. 266,824; B., 1927, 348.

Catalyst carrier (U.S.P. 1,636,685).—See I.

Hydroxy-compounds from amines (E.P. 274,960). **Catalytic methylation of ammonia and amines** (E.P. 275,377). **Catalytic oxidation of hydrocarbons** (E.P. 275,321).—See IV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Production of coloured photographic films.

L. F. DOUGLASS (U.S.P. 1,632,278, 14.6.27. Appl., 16.10.19).—Prints of corresponding red and green colour selection negatives are produced on a positive film, comprising a transparent carrier sensitised on one side, by exposure from opposite sides, the outer red image being mordanted after development by treatment in a solution containing, amongst other ingredients, potassium dichromate, ferric alum, oxalic acid, and potassium ferricyanide. The image is dyed red and treated with alkali to remove any blue colour deposited on it. The green inner image is then treated by prolonged immersion in the mordanting bath, and the blue colour deposited on it is converted into green in a bath containing chromic acid and potassium ferricyanide.

T. S. WHEELER.

Manufacture of kinematograph colour films.

J. E. THORNTON (E.P. 275,331, 5.5.26).

XXII.—EXPLOSIVES; MATCHES.

[Continuous] nitroglycerin manufacture.

A. SCHMID (Z. Ges. Schiess- u. Sprengstoffw., 1927, 22, 169—173, 201—206).—It is proposed to nitrate glycerin continuously in a nitrator containing a series of water-cooled coils, the inner one having its turns so close together that it forms a partition between the centre of the nitrator, which contains a stirrer, and the remaining coils. The nitrator is filled with an emulsion of nitroglycerin and waste acid. Glycerin enters at the top of the nitrator and mixed acid at the bottom; the latter mixes with the emulsion and is forced by the action of the stirrer to the bottom of the nitrator and then up through the coils. Working details of the temperature and acid composition are given. The emulsion of nitroglycerin and spent acid flows from the nitrator into a separator, where it first passes through perforated plates, most of the nitroglycerin being thereby separated, and then over sloping corrugated plates from which nitroglycerin passes off at the top and the waste acid lower down. There is no after-separation. Washing takes place in an earthenware tower which contains perforated plates. It is claimed that this process reduces risk, decreases capital expenditure and maintenance charges, and gives an increased yield of nitroglycerin with smaller consumption of nitric acid.

S. BINNING.

Recovery of solvents. BODEWIG.—See I.

PATENTS.

Production of finely-crystalline lead or other heavy-metal azides.

A.-G. LIGNOSE (G.P. 440,568, 15.9.21).—Solutions of sodium azide and a soluble lead salt, *e.g.*, lead acetate, flow simultaneously into a funnel-shaped receiver from containers with the outlets regulated to deliver equivalent volumes of the two solutions.

L. A. COLES.

Manufacture of match-striking compositions.

H. W. ROBINSON (U.S.P. 1,640,224, 23.8.27. Appl., 18.2.26. Conv., 4.2.26).—See E.P. 265,378; B., 1927, 269.

Manufacture of nitrated cellulose from wood pulp. V. PLANCHON (U.S.P. 1,641,292, 6.9.27. Appl., 27.2.25. Conv., 29.2.24).—See E.P. 230,092; B., 1925, 902.

XXIII.—SANITATION; WATER PURIFICATION.

Zeolite water treatment in a large central-heating plant.

A. H. WHITE, J. H. WALKER, E. P. PARTRIDGE, and L. F. COLLINS (J. Amer. Water Works Assoc., 1927, 18, 219—249).—Preliminary experiments indicated that the high caustic alkalinity in boilers resulting from the use of a zeolite system with water high in bicarbonate could be safely reduced by partial neutralisation of the bicarbonate in the softened water by addition of sulphuric acid with subsequent removal of the carbon dioxide set free by the passage of steam. In the case of a large central-heating plant satisfactory results were obtained when about 50% of the bicarbonate in the zeolite-treated water was neutralised by sulphuric acid and the water then passed through a deaerating heater. After such treatment water had p_H 6.4 on entering the deaerator and 8.5 on leaving the deaerator and entering the boiler. Inspection of the boiler after eight months of operation revealed clean tubes and no serious corrosion. For more complete treatment an auxiliary feed of phosphoric acid instead of the addition of further quantities of sulphuric acid appears feasible, and would avoid excessive lowering of the p_H of the water.

W. T. LOCKETT.

Determination of sulphuric acid in water analysis by means of benzidine.

L. W. HAASE (Chem.-Ztg., 1927, 51, 637—638).—The precipitation of benzidine sulphate is not affected by the presence of salts of calcium, ferrous iron, or manganese, or by small quantities of chlorides, nitrites, or nitrates, provided the liquid is faintly acid. Magnesium and ferric iron tend to prevent the precipitation, but the effect of the latter may be counteracted by boiling with a few c.c. of 1% hydroxylamine hydrochloride. The determination of sulphate by precipitation of the benzidine salt and titration of the base liberated after hydrolysis gives results in good agreement with the gravimetric barium sulphate method for concentrations of SO_3 of 100 mg./litre and over; for smaller concentrations very low results are obtained, particularly when the SO_3 falls below 60 mg./litre. In order to render the method applicable to drinking water, and waters in which the SO_3 content is lower than 100 mg./litre, concentration to about one quarter bulk with addition of 5—10 c.c. of 1% hydroxylamine hydrochloride is necessary before precipitation.

F. R. ENNOS.

Manuring trials with town sewage. ZIELSTORFF and KELLER.—See XVI.

PATENTS.

Purification of waste water.

T. FRANZ (G.P. 430,669, 22.1.24).—Waste water containing clay or earthy impurities in suspension is mixed with water containing less than $\frac{1}{2}\%$ of cutting oil, with or without the addition of small quantities of substances which by colloid-chemical or physical action can facilitate separation of the impurities.

L. A. COLES.

Purifying air etc. (E.P. 274,942).—See I.