

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCTOBER 15, 1926.

I.—GENERAL ; PLANT ; MACHINERY.

Oil-burning equipment for industrial furnaces.

M. H. MAWHINNEY (Trans. Amer. Soc. Steel Treat., 1926, 9, 99—110).—The installation and operation of oil-burning equipment is reviewed. Heating coils should be at the bottom of the tank and the tank connexions of adequate dimensions. The pump must be provided with an air chamber to eliminate pulsations and give a steady oil pressure. The oil viscosity for best atomisation is about 8° Engler. For steady air pressure a turbo-blower is recommended. The burner holes should be flared towards the inside of the wall. The size and location of flues must be such that the products of combustion do not collect in pockets. Baffle-walls should be placed in front of the burners to assist complete combustion. Unsatisfactory heating is generally connected with the size and location of the flues, the size of the combustion space, excessive radiation, or the arrangement of the baffles.

T. H. BURNHAM.

Measurement of surface temperatures. II.

Comparison of various methods. F. W. ADAMS and R. H. KEAN (Ind. Eng. Chem., 1926, 18, 856—857).—A thermo-couple compensated for heat losses (cf. Boyer and Buss, B., 1926, 647) is the most accurate method for determining the temperature of a hot surface (100—150°), being correct to within 1°, and may be applied to the measurement of the temperature of metallic or non-metallic surfaces, either stationary or moving, such as boiler settings, lagging, moving sheets of pulp, etc. For calculating the heat loss from a surface, results accurate to within 10% are obtained if the temperature is determined by a thermo-couple embedded in the surface, a couple soldered to the surface, a couple consisting of wires connecting through the surface, or a couple held under a pad against the surface.

B. W. CLARKE.

Factors influencing sedimentation. C. S. ROBINSON (Ind. Eng. Chem., 1926, 18, 869—870).—A modified form of Stokes' law, viz., $dH/d\theta = ka^2(D-d)z$, where H is the height of the sludge, θ the time of settling, k a constant, a the average dimension of the particles, D the specific gravity of the particles, d the specific gravity of the suspension, and z the relative viscosity of the suspension, is applicable to the settling of fine sludges, as shown by observations with sludges of fine silica particles.

B. W. CLARKE.

Catalysis—an industrial development. H. S. TAYLOR (Ind. Eng. Chem., 1926, 18, 958—960).

Fifty years of development of compressed gases. G. O. CARTER (Ind. Eng. Chem., 1926, 18, 954—956).

PATENTS.

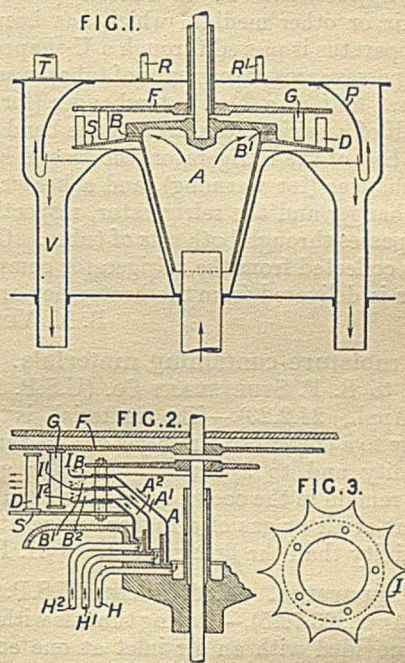
Filters. SEITZ-WERKE G.M.B.H. (E.P. 249,857,

19.3.26. Conv., 25.3.25).—The apparatus comprises a tank containing filter-leaves, which are capable of being removed individually through a side door in the tank. The filtrate is drawn off from the leaves at the same side as the door, from both top and bottom.

B. M. VENABLES.

Atomising and mixing liquids and gases.

E. LUSCHER (E.P. 254,130, 7.8.25).—Referring to the figures, B, B^1, B^2 are outlets from the rotor, A , which



revolves at a high speed. Deflecting plates, G , are attached to a rotor, F , rotating at a different speed, usually in the opposite direction to A . Outer deflecting plates, D , revolve with the rotor, A . In Fig. 2 the rings forming the walls of outlets B, B^1, B^2 are preferably star-shaped, as shown in Fig. 3, the points of the stars being bent alternately upwards and downwards as indicated by arrows, I, I^1, I^2 .

B. M. VENABLES.

Furnaces [for pulverised or liquid fuels]. L. C. HARVEY (E.P. 255,610, 22.6.25).—A preliminary combustion chamber for a U-shaped furnace burning pulverised solid, or liquid, fuel is provided with one or more longitudinal rows of vertical burners projecting a mixture of fuel and insufficient air downwards. One or more horizontal burners are also provided in the front of the furnace. These burners are lit first and are supplied with enough air for complete combustion of the fuel fed to them. The heat thus generated serves to gasify

the fuel from the vertical burners and combustion is completed by air admitted through the walls of the preliminary combustion chamber, also if desired through the ash-pit which is situated at the bottom where the gases turn upwards to the other limb of the U.

B. M. VENABLES.

Apparatus for heating, cooling, or drying materials. V. CANO (U.S.P. 1,592,078, 13.7.26. Appl., 10.9.25).—An elongated casing is divided by a horizontal partition. The articles to be treated are conveyed through the upper compartment, and a source of heat (or cold) is situated in the lower compartment. A circulation of air is maintained by a blower drawing air through the lower compartment and returning it through the upper compartment in the same direction as the goods travel.

B. M. VENABLES.

Drying apparatus. F. G. HATCH (E.P. 255,724, 6.1.26).—Air or other medium which has been used in a drying apparatus is passed through a U-shaped passage in which it passes across five groups of tubes in succession, viz., the first half of a heat interchanger (for cooling), the first half of a refrigerator, around the bend of the U, the second half of the refrigerator, the second half of the heat interchanger (for warming), and a heater. In the heat interchanger and the refrigerator, respectively, the same fluid passes through both sets of tubes. Condensed moisture is collected from the refrigerator compartments and the air is delivered from the heater hot and dry for re-use.

B. M. VENABLES.

Drying moisture-containing materials. K. REDMAN, Assr. to B. F. STURTEVANT Co. (U.S.P. 1,593,598, 27.7.26. Appl., 24.2.23).—The material is subjected successively to a current of air or gas of high temperature and humidity and to a current of cooler gas. It is allowed to remain in the cooler gas, and is then subjected to a current of air or gas of a higher temperature and a lower humidity than the first current.

H. HOLMES.

Drying apparatus. M. M. MINTER (U.S.P. 1,594,316, 27.7.26. Appl., 23.7.25).—Drying tunnels with end doors are each provided with an air inlet at one end and an outlet near the opposite end. A supply flue communicates with the air inlets by individually-controlled dampers. Air is discharged into the flue from the lower portion of a chamber having a fan mounted in its upper portion. An upper intake opening in the side of the fan chamber communicates with the upper portion of a riser and the lower portion of the riser communicates with an intake flue. A normally-closed opening is provided in the upper portion of the riser for communication with the atmosphere.

H. HOLMES.

Apparatus for finely dividing and drying fluid substances. Reducing heavy liquid substances to dry finely-powdered form. (A) J. M. MACLACHLAN, (B) J. C. MACLACHLAN (U.S.P. 1,594,064—5, 27.7.26. Appl., [A] 19.1.22, [B] 2.8.20).—(A) The material to be dried is sprayed centrifugally in the upper part of a chamber. A stream or streams of fluid for disintegrating purposes enters above the spray and passes vertically downwards through it. A drying medium enters the chamber through side inlets. The bulk of the dry

powdered material is removed through an outlet at the bottom, and the drying medium with some suspended powder is exhausted through a higher outlet and passes to a separator (e.g., a cyclone) where further dry material is collected. In (B) constructional details of a similar device are given, steam and air being used as the disintegrating and drying medium respectively.

B. M. VENABLES.

Decolorising, purifying, and adsorbent composition. J. K. STEWART, Assr. to SHELL Co. OF CALIFORNIA (U.S.P. 1,592,543, 13.7.26. Appl., 27.2.24).—Sodium silicate and a solution of an aluminium salt are mixed and the precipitate is dried, ground, and washed to remove the sodium salts. The resulting product contains only silica and alumina and is used as a decolorising agent when ground to 200-mesh and as an adsorbent when ground to 40-mesh. The product is activated by heating to 200°.

E. S. KREIS.

Extraction apparatus. R. P. SOULE, Assr. to COMBUSTION UTILITIES CORP. (U.S.P. 1,594,024, 27.7.26. Appl., 11.6.24).—A vessel closed for operation at pressures above atmospheric is divided into a mixing chamber and a settling chamber. Means are provided for introducing the raw material and the solvent into the mixing chamber, for agitating them while maintaining them at an elevated temperature, and for the separate removal from the settling chamber of the solution and the insoluble residue.

H. HOLMES.

Balanced ball mill. W. M. BARKER (U.S.P. 1,594,990, 3.8.26. Appl., 26.5.26).—The mill comprises a feed chamber, a discharge chamber, and an annular ball-race chamber. The material passes through inlet ports from the feed chamber to the ball-race chamber, is ground by the balls therein, and passes through outlet ports into the discharge chamber.

H. HOLMES.

Cooling hot salt solutions in a vacuum. KALIFORSCHUNGS-ANSTALT G.M.B.H., Asses. of E. RITTER (G.P. 430,482, 18.3.25. Addn. to 413,710; B., 1925, 743).—The addition of water to replace that evaporated is made at such places that incrustations during the crystallisation are avoided.

B. FULLMAN.

Filter-presses. F. J. BISBEE (E.P. 256,898, 19.4.26).—See U.S.P. 1,589,834; B., 1926, 696.

Apparatus for washing and classifying minerals. C. CLOUWEZ (E.P. 245,770, 6.1.26. Conv., 9.1.25).

Cooling towers. J. M. SEYMOUR (E.P. 256,889, 29.3.26).

Removal of dust, fumes, and the like. J. FORGAN-POTTS and V. R. CHADWICK (E.P. 257,032, 23.5.25).

Heat interchanging apparatus. L. CHAVANNE (E.P. 231,186, 249,534, and 249,536, 19.3.25. Conv., 19.3.24).

Steam distillation (U.S.P. 1,594,957).—See II.

Exothermic gaseous catalytic reactions (E.P. 255,963—4).—See VII.

Separating gas mixtures (U.S.P. 1,594,336).—See VII.

Lining pipes etc. (E.P. 255,546).—See XIII.

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

Lignin and oxycellulose theories [of coal formation]. J. MARCUSSON (*Z. angew. Chem.*, 1926, **39**, 898—900; cf. *B.*, 1925, 386).—The lignin content of coniferous wood remains approximately constant (about 20%) during its decay. The alkali-soluble compounds can be resolved into humic acids, insoluble in water, and a water-soluble portion showing the reactions of glycuronic acid and also apparently containing pentoses. The content of these compounds, both soluble and insoluble in water, increases as decay proceeds, the insoluble acids being partly derived from cellulose, since they occur in combination with carbohydrates. The content of volatile acids, in particular acetic acid, and of methyl alcohol increases with advancing decay. Both lignin and cellulose go to form coal. Cellulose is converted, by hydrolysis and oxidation, into oxycellulose, and pectins are also formed. The glycuronic acid residue in oxycellulose is readily converted into humic acids which, in turn, lead to coal.

W. T. K. BRAUNHOLTZ.

Oil obtained by "berginisation" of Lower Silesian coal. M. HEYN and M. DUNKEL (*Brennstoff-Chem.*, 1926, **7**, 20—25, 81—87, 245—250).—A detailed analysis of the oils obtained from unwashed coal dust containing 17.7% of ash, 28.5% of volatile matter (both reckoned on the dry coal), and an exceptionally high proportion of fusain, which is known to yield very little oil on "berginisation." The coal was made into a paste with half its weight of an oil obtained from a previous "berginisation" of similar coal, and the experiment was run continuously for 2½ days at a throughput of 7 kg. of paste per hr. The total oil yield was 36.45% of the raw coal, *i.e.*, 33.09% of oily product and 3.36% of benzine recoverable from the gas. The black, viscous oil contained 29.5% of dust, to remove which the light oils were distilled off up to 230° (during which ammonium carbonate separated out in the receiver), and the residue was dissolved in benzene and filtered warm. After removing the benzene, the light and heavy oils were united and fractionally distilled (under diminished pressure above 220°) up to pitch (9.26% of the raw coal). It is difficult to remove the oil completely from the dust, which, after extraction, contained 40.65% of ash and 10.45% of volatile matter. The oil fractions were extracted by the usual methods for phenols, bases, etc., showing the total distillate to be composed of 75.7% of neutral oils, 12.0% of phenols, 3.5% of bases, and small traces of carboxylic acids, resin, and a sediment. 10.3% of the phenolic compounds was phenol itself, whilst phenol, cresols, and xylenols (b.p. up to 220°) constituted 66.5% of the whole. The basic fraction contained primary (aniline, toluidine, xylidine), secondary (not identified), and tertiary bases (pyridine absent; collidine, quinoline, quinaldine, and probably isoquinoline identified). The "benzine" from the gas was refined with sulphuric acid and fractionally distilled, the bulk distilling below 185° and containing benzene, toluene, xylene (shown by nitration), with a large proportion of their hexahydro-derivatives (shown by dehydrogenation). The light oil fraction

(b.p. up to 180°) of the main product, similarly refined and fractionally distilled, had an analogous composition, the ratio of aromatics to hydroaromatics being, however, greater than in the "benzine." The higher fraction (180—220°) of the oil contained naphthalene and its tetrahydro- and decahydro-derivatives. All the neutral oil fractions were remarkable for their low sulphur content (0.76—1.12%). Attempts to prepare a satisfactory lubricating oil from the higher fractions were unsuccessful. The solid constituents of the neutral oils comprised a small quantity of paraffins and a considerable quantity of aromatic hydrocarbons (mainly phenanthrene). W. T. K. BRAUNHOLTZ.

Combustion of carbonic oxide. W. A. BONE (*Gas J.*, 1926, **174**, 725—731; *Gas World*, 1926, **85**, 4—6, 34—38).—A brief historical survey of the subject is given, followed by a description of recent investigations carried out under the author's direction (cf. *A.*, 1925, ii., 928; 1926, 8, 480).

Nomenclature of wood distillation products. G. BUGGE (*Z. angew. Chem.*, 1926, **39**, 1032—1033).—In agreement with Mallison ("Teer, Pech, Bitumen, u. Asphalt," Halle, 1926), it is proposed to employ for tars and pitches obtained by distillation of natural organic materials names derived both from the raw material and the method of distillation. The name "wood oil" should be restricted to tung oil. The name creosote should only be used with a prefix denoting the origin. S. I. LEVY.

Ammonia recovery [from coal gas] by gypsum. H. W. JACKMAN (*Gas J.*, 1926, **175**, 428—429).—In a method worked out on the laboratory scale sulphuric acid for the recovery of ammonia at gas works is replaced by gypsum. Ordinary 15% ammonia concentrate is prepared and is saturated with carbon dioxide obtained from flue-gases in a packed tower. To this solution gypsum is added in small quantities at a time with stirring, the calcium carbonate filtered off, and the liquor passed through a still to remove the remaining free ammonia. The liquor is then evaporated until ammonium sulphate crystallises. A direct process is also possible by washing the gas with saturated calcium sulphate solution in a packed tower. The calcium carbonate sludge is drawn off from time to time and the liquor finally evaporated to crystallisation. The removal of ammonia from the gas is incomplete and depends chiefly on the temperature and content of volatile ammonia of the scrubbing liquor. The residual ammonia is removed by a scrubber fed with water.

C. IRWIN.

Vacuum or steam distillation of benzol [wash-oil]. O. HILGENSTOCK (*Brennstoff-Chem.*, 1926, **7**, 87—89).—As regards the rate of driving off the benzol, and the steam, wash-oil, and cooling water consumption, distillation of wash-oil with steam is preferable to vacuum distillation, and it is not inferior from the point of view of the efficiency of scrubbing of the gas, the composition of the crude benzol, or the corrosion of the iron apparatus. The cause of the thickening of the wash-oil is not yet established. W. T. K. BRAUNHOLTZ.

Oil-bearing shales in North Carolina. F. C. VILBRANDT (Ind. Eng. Chem., 1926, 18, 793—795).—An examination of the shale in the Deep River Valley coal field leads to an estimate of the presence of one billion tons of oil-bearing shale capable of yielding approximately 27 billion gallons of shale oil (30% of which would be a straight-run motor spirit) and 47,500 tons of ammonium sulphate. The shales are overlying and underlying coal measures which are being worked.

W. N. HOYTE.

Anti-knock motor fuels by cracking shale oil. J. C. MORRELL and G. EGLOFF (Ind. Eng. Chem., 1926, 18, 801—802).—Shale oil from America, Australia, and France when cracked at 120—150 lb. pressure, yielded more than 50% of Navy end point gasoline; this, by chemical analysis and by actual engine tests, was found to possess anti-knock properties. A suitable scheme of refining with sodium plumbite and sulphuric acid is described.

W. N. HOYTE.

Fractionation number [of motor fuels]. W. OSTWALD (Petroleum, 1926, 22, 850—853).—The characteristic number of the b.p. curve of a motor spirit is obtained by drawing a straight line at right angles to the temperature axis such that the two areas enclosed by this line, the b.p. curve, and the uprights at either end of the curve are equal. The temperature indicated at the intersection of this line and the temperature axis is the characteristic number. In order to express concisely the sharpness of fractionation by which a spirit was prepared a straight line is drawn through the b.p. curve, inclined to the temperature axis and cutting the curve in at least two points, such that the two areas (above and below the line respectively) enclosed by the line, the curve, and the uprights) are equal. The inclination of this line to the horizontal is the fractionation number of the spirit, and when this is used in conjunction with the characteristic number calculations of the composition of mixtures are possible and the efficiency of a fractionating column can be stated.

W. N. HOYTE.

Variation of specific gravity of Japanese crude oils. T. IKI (J. Fac. Sci. Tokyo, II, 1926, 1, 53—64).—Tables are given showing the variations in d of crude oils from different geological beds (the range here being 11° to 48° B; d 0.993—0.786), from the same bed, from different depths, when under the influence of volcanic activity, and after exposure to the air. The causes of these variations are discussed.

L. J. SPENCER.

Composition of Mid-continent petroleum. C. F. MABERY (Ind. Eng. Chem., 1926, 18, 814—819; cf. B., 1924, 120).—Details of fractional extraction of crude and lubricating oils with ether and alcohol are given. Two series of compounds were separated, viz., the "D hydrocarbons" (lubricants), and the "H hydrocarbons" (asphaltic oils). Molecular weight determinations showed that the series C_nH_{2n-4} and C_nH_{2n-8} are the best lubricants; those poorer in hydrogen show asphaltic properties. Marked differences were observed between the refractive indices of the D and H series, the former having the higher index. Viscosities

of the fractions were determined in Ostwald tubes and the resistance of films of the oils to stress in a hard Babbitt-metal bearing.

W. N. HOYTE.

Specific heats of oils. A. R. FORTSCH and W. G. WHITMAN (Ind. Eng. Chem., 1926, 18, 795—800).—The apparatus used consisted of a large vacuum flask, the contents of which were rapidly stirred and heated by an immersed resistance coil. The outside wall of the flask was surrounded by an air bath maintained at the average temperature at which the determination was carried out. Radiation corrections were applied. Observations were taken over temperature intervals from 50° to 430° F. (10° to 220°). Fifteen oils from Mid-continent crude oil were studied; their specific gravities ranged from 0.75 to 1.00. It was found that specific heats for engineering purposes could be calculated from the formula $C = (t + 670)(2.10 - d)/2030$, where C = specific heat, t = temperature in ° F., and d = sp. gr. at 60° F. The maximum deviation for any oil studied was 5.9%; the average deviation was 2.3% at 240° F. The equation cannot be applied without further correction to oils within 100° F. of their critical temperature. A review of the results of other workers is given.

W. N. HOYTE.

Cracking of petroleum oils. E. H. LESLIE and E. H. POTTHOFF (Ind. Eng. Chem., 1926, 18, 776—785).—The cracking of various petroleum oils has been studied under controlled conditions on the laboratory scale. The apparatus consisted of a preheater in which the oil, about 3 litres, is rapidly brought to the desired temperature by circulation over electrically heated coils, and from which it is discharged into a bomb surrounded by resistance coils whereby heat losses from the bomb can be made good. Special precautions are taken to avoid skin temperature overheating. Three oils were studied, a gas oil, a heavy fuel oil, and a "thermolised" gas oil, i.e., one already cracked and the spirit removed. The rate of cracking was found to double for a rise in temperature of 12°. At 370° the thermolised gas oil polymerised at a greater rate than it cracked.

The formation of oils of low boiling point was found to be a straight line function of time with the above exception of the thermolised gas oil, and can be formulated as a reaction of the first order. Pressure as such has no effect on the percentage or boiling range of the spirit obtained. Increase of pressure decreased the percentage of unsaturated compounds in the spirit. The fuel oil cracked most easily, the thermolised gas oil the least easily. Removal of the spirit as formed had no effect on the percentage obtained; the spirit, if removed progressively, is, however, somewhat more unsaturated. Under no circumstances was a condition of equilibrium attained between the products of the decomposition of the oil. The spirit from the thermolised gas oil for a given boiling range had the highest specific gravity. Experiments at atmospheric pressure in a special apparatus showed that cracking was an endothermic reaction to the extent of approximately 500 cal. per g. of spirit produced. This emphasises the importance of efficient heat insulation of reaction vessels in full-sized cracking plants.

W. N. HOYTE.

Development of a liquid-phase cracking process. S. J. M. AULD and A. E. DUNSTAN (Ind. Eng. Chem., 1926, 18, 803—807).—The theoretical considerations and practical experiments on which the A.D.H. cracking process is based are outlined. The pressure-solubility relations of the permanent hydrocarbon gases and cracking stock oils were examined in a modified Andrews apparatus in which the oil could be heated to 450° under pressures up to 1000 lb. per sq. in. and could be kept under observation in a silica tube. Examination of the time factor in relation to the extent of cracking was carried out in an autoclave. It was found that the higher the pressure the less unsaturated is the resulting cracked spirit and the less is the formation of carbon. High pressures induced by the adsorption of gas oil in activated charcoal maintained at a suitable temperature produced 70% of superior motor spirit. The formation of carbon in the cracking process is primarily due to the thermal decomposition of olefinic compounds. The reduction to a minimum of the superheating of the oil on the surfaces of the plant is emphasised. Persian gas oil was found to begin to crack at 380°, and the rate of cracking to double, other things being equal, for each 10° rise in temperature. As the result of experiments carried out in electrically heated steel tubes it was decided that the oil should be heated while in narrow tubes and moving with low velocity in order to prevent excessive skin temperature in the heating walls. Three definite zones were established in the plant designed, viz., the preheaters, where the oil is brought to a temperature some 20° below cracking temperature; the thermolyser, where cracking temperature is slightly exceeded; and the reactor, where the oil remains sufficiently long at cracking temperature for the desired amount of cracking to take place. Previous to the formation of coke in the cracking plant the oil passed through a pitch-like, viscous state, and it was possible to draw off progressively this substance as formed, thereby preventing the formation of coke to a large extent. W. N. HOYTE.

Determination of unsaturated content of petroleum products. A. W. FRANCIS (Ind. Eng. Chem., 1926, 18, 821—822).—A method is described in which the oil is shaken with an aqueous mixture of potassium bromide and bromate in slightly acid solution. Bromine is generated slowly and is fixed at the double linkings in the unsaturated compounds present, substitution being reduced to a minimum. Results on synthetic mixtures of pure unsaturated compounds in straight-run motor spirits show very good agreement between observed and calculated figures. W. N. HOYTE.

Desulphurising effects of sodium hypochlorite on naphtha solutions of organic sulphur compounds. A. E. WOOD, A. R. GREENE, and R. W. PROVINE (Ind. Eng. Chem., 1926, 18, 823—826).—The desulphurising effect of sodium hypochlorite on naphtha solutions of ethyl sulphide, *n*-butyl sulphide, phenyl sulphide, ethyl disulphide, *n*-propyl sulphide, ethyl mercaptan, *n*-propyl mercaptan, *iso*amyl mercaptan, carbon disulphide, hydrogen sulphide, thiophen, and elemental sulphur was determined. The effect was found to depend on the actual sulphur compound used,

the amount of available chlorine present, the degree of alkalinity of the hypochlorite solution, and the time and intensity of agitation. Thiophen and elemental sulphur are not attacked by sodium hypochlorite; the aliphatic sulphides vary in the ease with which they are oxidised to sulphones; the disulphides are somewhat less readily attacked than the sulphides, and the mercaptans still less readily. Hydrogen sulphide is oxidised to elemental sulphur, and carbon disulphide is partially removed. The conversion of the sulphides into sulphones is beneficial in refinery practice, as the latter compounds are more easily removed from the naphtha by subsequent treatment, re-distillation, and filtration through an adsorbent material. Better results in general are obtained by using hypochlorite solutions comparatively rich in chlorine and alkali rather than solutions weak in these respects. In designing a suitable hypochlorite treatment for a particular naphtha, a careful study must be made as regards the oxidation products; if these are acidic, then sufficient alkali must be present initially to maintain a final alkaline solution. W. N. HOYTE.

Distillation of lubricating oils under high vacuum. B. T. BROOKS (Ind. Eng. Chem., 1926, 18, 789—793).—A brief review of the previous work on the large scale is given. Details of the work of Schulze on the refinery scale in the United States are given. A vacuum of 5—3 mm. is used, and a wax-free oil is distilled. The distillate is emulsified with just sufficient strong caustic soda solution to combine with the naphthenic acids, and a second high-vacuum distillation then yields a lubricating oil requiring no further treatment. Cuts of very narrow boiling range can be made, and oils with viscosities up to 175 sec. Saybolt at 99° can be obtained. W. N. HOYTE.

[Paraffin] wax crystallisation. F. W. PADGETT, D. G. HEFLEY, and A. HENRIKSEN (Ind. Eng. Chem., 1926, 18, 832—835).—The crystallisation of various waxes from solution in oil was studied by microscopical examination of samples under crossed Nicols. The effect of the viscosity of the solvent, the rate of cooling, and time elapsing after crystallisation was complete, was studied. Three types of crystallisation were detected, viz., very small crystals from residual oils and petroleum, fine needles from slack wax and ordinary paraffin wax, and plate-like crystals produced in small numbers during the initial crystallisation of paraffin wax, and also deposited between the above-mentioned needles on keeping for some time. W. N. HOYTE.

Oxidation of coal at storage temperatures. S. W. PARR and R. T. MILNER (Fuel, 1926, 5, 298—300).—See B., 1925, 195.

Deterioration and spontaneous combustion of coal in storage. S. W. PARR (Fuel, 1926, 5, 301—305).—See B., 1925, 195.

Carbon dioxide as an index of critical oxidation temperature for coal in storage. S. W. PARR and C. C. COONS (Fuel, 1926, 5, 306—308).—See B., 1925, 195.

Use of oxygen in manufacture of water-gas. F. E. VANDAVEER and S. W. PARR (Fuel, 1926, 5, 309—314).—See B., 1925, 979.

Fifty years of gas chemistry. W. H. FULWEILER (Ind. Eng. Chem., 1926, 18, 945—948).

Fifty years of wood distillation. L. F. HAWLEY (Ind. Eng. Chem., 1926, 18, 929—930).

Fifty years in the petroleum industry. F. A. HOWARD (Ind. Eng. Chem., 1926, 18, 936—938).

Oil-burning equipment for industrial furnaces. MAWHINNEY.—See I.

Fluorescence of oils in ultra-violet light. CRONER.—See XII.

PATENTS.

Coal washing. W. C. MENZIES (U.S.P. 1,594,626, 3.8.26. Appl., 23.9.24).—Coal is fed on to an inclined grid which forms the false bottom of a tank. A stream of water is directed on to the coal from the under-side of the grid and takes the lighter clean coal into suspension. Two baffles supported at an adjustable distance above the grid, and extending over the whole width of the tank, direct the flow of the suspension. The upper edge of one of the baffles forms a weir over which the coal and water flow into a separate compartment, where the clean coal is separated. The heavier coal constituents are not lifted sufficiently by the water-stream to carry them over the weir, and they gravitate down the inclined grid, being separately collected at the bottom. S. PEXTON.

Agglomerated combustible products. E. GOUTAL and H. HENNEBUTE (E.P. 247,210, 5.2.26. Conv., 9.2.25).—The agglomerated products resulting from baking blends of low-temperature coke and pyroigneous tars and pitches or their products at a medium temperature (cf. E.P. 231,863, B., 1925, 906) disintegrate when heated to a high temperature. This defect is obviated by incorporating in the blend suitable proportions of hard coals or other combustible materials which do not shrink when heated to high temperatures. The compressed moulded blends are baked at 600—700°, and the product, being dense and highly combustible, is suitable for burning in an open grate or in a suction gas producer, where retention of the original shape of the fuel is advantageous for uniform working. S. PEXTON.

Briquette. H. McC. SPENCER, Assr. to SEYDEL CHEMICAL Co. (U.S.P. 1,590,706, 29.6.26. Appl., 17.1.23).—Dry anthracite culm, coal dust, or the like mixed with 1—10% of phosphoric acid in 50% solution in water is briquetted and dried at 220°. A hard briquette which resists the action of water is obtained. In place of phosphoric acid, a soluble phosphate, *e.g.*, the extract obtained from phosphate rock by treatment with sulphuric acid, may be used. The method may also be applied to briquetting iron sulphide ores.

T. S. WHEELER.

Carbonising coal. INTERNAT. COMBUSTION ENGINEERING CORP., Asses. of W. RUNGE (E.P. 253,878, 19.5.26. Conv., 17.6.25. Addn. to 242,621).—Pulverised coal is allowed to gravitate through a retort up which air preheated to approximately 425° is passing at such a velocity that the coal is buoyantly held within the retort

for 60 sec. The treated coal deprived of its agglutinating power retains its original form and most of its volatile matter. In this form it is mixed with a proportion of untreated coal and carbonised, giving a finely-powdered coke, the content of volatile matter in which can be regulated by varying the carbonising conditions.

S. PEXTON.

Producing coke. M. J. TRUMBLE (E.P. 255,620, 2.7.25).—Carbonaceous material is carbonised in an externally heated retort, at a medium temperature, and superheated steam is passed through the charge until it is free from volatile matter. The retort temperature is then further raised and oil is introduced into the incandescent coke. The oil is cracked to light petroleum and a pitchy residue, which is deposited in the voids of the coke and converts it into a consolidated fuel of high calorific value. By regulating the conditions of cracking the volatile content of the coke can be adjusted to satisfy the special purpose for which the fuel is required.

S. PEXTON.

Carbonising coal. R. L. RODGERS (E.P. 256,038, 15.7.25).—Coal is carbonised at a low temperature in narrow metal chambers, which have double walls. The space between the walls is filled with a fusible salt, *e.g.*, a mixture of 75% of calcium chloride and 25% of sodium chloride. The chambers each have tight-fitting charging and discharging doors, and several are mounted in a direct-fired furnace. The flames do not cause local overheating of the coal charge because of the interposition of the salt bath, which also by reason of its large thermal capacity materially increases the rate of carbonisation in the early stages.

S. PEXTON.

Apparatus for carbonising coal. Carbonising process. C. HAYES (U.S.P. 1,593,448—9, 20.7.26. Appl., 22.5.26. Conv., 16.5.25).—Crushed coal or lignite is treated in a preheater at 205° to expel moisture, and then conveyed to the top of two digesters enclosed by a brick-lined chamber, in which the gases generated are collected. A plunger fits into the top of each digester, the lower part of which is heated to 430°, the upper part to 315°, by means of oil burners arranged outside. Superheated steam is also passed through the charge. When the digester has been completely filled, the plunger is forced down to expel air. When the coal in the lower portion of the digester becomes converted into a mass of softened coke-like material from which most of the volatile products have been removed, but leaving just sufficient to make it ignitable, the bottom of the digester is opened and the softened coke is forced out into a bin underneath where the heat lost during cooling is utilised to generate steam for the plant. The charge in the upper part of the digester descends, and its place is taken by fresh coal from the preheater. The operations are then repeated.

R. B. CLARKE.

Manufacture of calcium carbide, coke, and synthetic products [methyl alcohol]. G. L. E. PATART (F.P. 605,530, 9.10.25).—Coke is prepared by the carbonisation of coal in an oven or retort which communicates directly with a calcium carbide furnace, thus facilitating the introduction of coke into the latter a

the highest temperature reached during its formation. The distillation products from the coal are mixed with carbon monoxide and catalysed under pressure for the production of synthetic methyl alcohol. R. B. CLARKE.

Synthetic bone char from black-strap syrup. C. B. DAVIS (U.S.P. 1,592,599, 13.7.26. Appl., 12.5.23).—125 lb. of commercial superphosphate are extracted with water, and the solution is mixed with 1 ton of black-strap syrup and hydrated lime added until alkaline. The gelatinous mass which precipitates carries with it the albuminous and gummy matter present in the black-strap. It is filtered off and carbonised at 500° in the absence of air. A synthetic bone char which is very effective in the purification of sugar solutions is obtained. T. S. WHEELER.

Apparatus for making water-gas. R. R. and J. S. McDONNELL (U.S.P. 1,592,464, 13.7.26. Appl., 26.3.21).—The generating chamber has a grate containing passages connected with a pipe through which water is supplied for the purpose of generating and superheating steam. The steam is discharged through an outlet below the grate and passes upwards, through the grate, to the fuel bed. W. T. K. BRAUNHOLTZ.

Gas-producing apparatus. E. L. BROOME, Assr. to GEN. REDUCTION GAS & BY-PRODUCTS Co. (U.S.P. 1,590,279, 29.6.26. Appl., 17.1.21).—An apparatus for gasifying slack, sawdust, or other waste fuel consists of a conical vessel rotating about its axis, which is slightly inclined to the horizontal. The fuel is introduced at the upper and narrow end, and the clinker, which is formed into balls by the rotation, is withdrawn at the lower end by a number of extractors, each of which consists of a tube bent twice at right angles through which the spherical clinker masses pass more readily than the fuel. Air or steam is introduced into the interior of the mass of fuel by a water-cooled tuyère. To improve the admixture of vapour and solid fuel the cylindrical vessel is fitted internally with a baffle perpendicular to its axis, which divides it into a drying and a reaction chamber. T. S. WHEELER.

Manufacture of gas. ANC. ÉTABL. BARBIER, BÉNARD, and TURENNE (SOC. ANON.) (F.P. 605,407, 22.10.25).—Animal, vegetable, or mineral oils are cracked at 600–700° in one chamber of a retort having a second chamber on the same level. In this second chamber the vapours are heated to 900–1000°. They then pass to a second retort at a temperature of 500°, this retort being charged with iron borings. C. IRWIN.

Aeration and gas purification apparatus and methods. Aeration and gas purification processes and apparatus. Gas purification processes and apparatus. Gas and liquid contact apparatus and methods. Purification of gases. Removal of hydrogen sulphide and other noxious constituents from gases. Purification of gases. W. J. MELLERSH-JACKSON. From KOPPERS Co. (E.P. 255,139–255,145, 14.4.25).—(A) In the process of removing hydrogen sulphide from gas by washing with a suspension of iron oxide in a solution of sodium carbonate, the spent liquor is regenerated by bubbling air through it in a

“thionizer.” Difficulty has been experienced through the blockage of the pores in the air bubblers with sediment. This has been overcome by surrounding the aeration tubes with an envelope of pliable porous material, such as cotton, which is free to vibrate; no sediment then adheres to the envelope and the aerator remains free. (B) By maintaining the absorber and thionizer in the wet purification process at a temperature above 29.5° instead of 15.5°, the following advantages result:—The capacity of a given plant is doubled; it is no longer necessary to use freshly-precipitated iron hydroxide, as finely-powdered bog-ore gives excellent results; and the sulphur in the thionizer separates as a relatively heavy sludge rather than as a bulky foam. (C) In the aeration of spent washing liquor secondary reactions such as the formation of sodium thiosulphate occur. Such reactions are greatly retarded if the spent liquor entering the thionizer is neutral or slightly acid and of low concentration. To attain this and yet completely purify the gas two absorbers, each connected with separate compartments of a thionizer, are used. The first absorber, which removes the bulk of the hydrogen sulphide, is fed with a suspension of 1% of iron oxide in a 1% solution of sodium carbonate. Renewals of the iron in this circuit may be made by adding acid salts such as the chloride or sulphate to the spent liquor entering the thionizer. When the sulphur in the thionizer connected to the first absorber becomes excessive the first absorber is connected with a second chamber of the thionizer containing regenerated liquor of 1% alkalinity, and the accumulated sulphur is worked up. In the second absorber a 1% suspension of iron oxide in a 3% solution of sodium carbonate is used, but because of the low concentration of hydrogen sulphide in the gas entering the second absorber this liquor can be regenerated without material loss of sodium carbonate. (D) An improved design of contact material for filling gas absorption apparatus is described. It consists of alternate series of distributors and fillers. The distributors comprise a number of surfaces inclined to the direction of liquor and gas flow, whilst the filler surfaces are parallel to the liquor and gas flows. (E) A wet purification process in which the washing medium is a 1% solution of sodium carbonate charged with 3% of finely divided iron oxide, is described. The hydrogen sulphide absorbed by the sodium carbonate reacts with the iron oxide forming iron sulphide in which form the bulk of the sulphur is removed. The iron sulphide is separated from the liquor and oxidised as a sludge in an aerator, free sulphur and iron oxide being formed. This regenerated mixture is filtered and returned with the clear liquor to the washing system. From time to time some of the oxide after regeneration is withdrawn for the recovery of sulphur, and the oxide returned to the system. (F) Foul gas is washed with a 2% suspension of magnesium oxide in a 0.5% solution of magnesium chloride. The spent liquor is heated and agitated, whereby hydrogen sulphide is evolved. The hydrolysis of the hot magnesium chloride with the production of hydrochloric acid assists the liberation of the hydrogen sulphide. The revived suspension is used for washing more foul gas. (G) When purifying gas with aqueous suspensions

of metallic compounds the regeneration is rendered more efficient if a peptising agent such as glycerin is added to the washing medium in order to prevent coagulation of the solid particles of the reagent.

S. PEXTON.

Separation of hydrocarbons from earthy matter.

J. M. McCLAVE, Assr. to BITUMINOUS SAND Co. (U.S.P. 1,594,625, 3.8.26. Appl., 27.11.22).—The addition of a colloidal clay to a suspension of earthy matter containing hydrocarbons causes the latter to separate and become recoverable.

S. PEXTON.

Treatment of hydrocarbon oils [crude benzene etc.]. H. SCHLAMP (E.P. 255,777, 30.3.26).—The crude benzene recovered from the distillation of gases from coal, lignite, peat etc., or a corresponding product in the purification of petroleum, is, if necessary, freed from phenols by washing with alkali and from pyridine etc. by washing with dilute sulphuric acid, and is then treated with concentrated sulphuric acid. A neutralising medium such as ammonia solution or alkali carbonate is added, and the heat of reaction effects the distillation of the lighter hydrocarbons. Carbon dioxide liberated may be used for the separation of phenols from the soda-phenol lye. On keeping, the reaction mixture separates in layers; to recover the remaining volatile hydrocarbons only the uppermost layer has to be distilled. The operation is carried out in one vessel provided with stirring devices, and the products of neutralisation are of commercial value.

H. MOORE.

Treating carbon and carbon compounds under heat and pressure. F. BERGIUS (U.S.P. 1,592,772, 13.7.26. Appl., 30.8.21. Conv., 23.1.15).—The reagents are heated to within 50° of their reaction temperature before introduction into the pressure chamber, which is heated by circulating within its double walls an inert gas under the same pressure as the reagents, so that the heat-conducting wall need not be built thick to withstand pressure.

E. S. KREIS.

Oil-treating [cracking] apparatus. E. A. DIETERLE (U.S.P. 1,589,631, 22.6.26. Appl., 7.4.23).—Oil is sprayed into the top of a cylindrical shell containing a number of S-shaped flues through which hot gases are passed. The oil first falls on to the cooler portions of the flue and then passes on to the hotter portions, and is thus gradually heated and cracked. Uncracked oil is withdrawn from the bottom of the shell. The apparatus is safe to handle as only a relatively small quantity of oil is under treatment at one time.

T. S. WHEELER.

Oil-treating apparatus. J. W. LEWIS, Assr. to ATLANTIC REFINING Co. (U.S.P. 1,593,905, 27.7.26. Appl., 6.7.22).—A pressure still comprises a vertical still combined with a tubular heat absorption structure. The upper and lower ends of the tubes are connected to the still at different levels, and the connexions may be closed by valves which may be operated simultaneously. Pressure above atmospheric is maintained in the still and tubes. An oil chamber is also connected with the heat-absorption structure by a valved connexion, with

by-pass and a check valve in the by-pass. Pressure above atmospheric is maintained in the oil chamber.

H. MOORE.

Removal of sulphur and sulphur compounds from hydrocarbon-oil distillates. G. L. WENDT, Assr. to STANDARD OIL Co. (U.S.P. 1,594,083, 27.7.26. Appl., 16.7.24).—Distillates containing products within the gasoline and kerosene b.p. range are treated with "doctor" solution, and then distilled at atmospheric pressure and at a temperature not above 205°. The distillation is continued under reduced pressure without raising the temperature.

H. MOORE.

Cracking hydrocarbon oil. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,594,093, 27.7.26. Appl., 21.10.22).—The oil is raised to cracking temperature in a heating zone and then collected in a vaporising zone, where vaporisation takes place under pressure. The vapours pass to a dephlegmating zone, where they are subjected to the refluxing action of the entering raw oil. The uncondensed vapours pass under reduced pressure to a series of fractionating zones, the vapours from the last of which are condensed. Portions of the condensate are returned from each fractionating zone to the preceding one.

H. MOORE.

Steam distillation [of hydrocarbon and like liquids, immiscible with water]. F. A. HOWARD and E. M. CLARK, Assrs. to STANDARD DEVELOPMENT Co. (U.S.P. 1,594,957, 3.8.26. Appl., 8.3.20).—The liquid is passed through a series of stills in which successively heavier fractions are removed. Steam is introduced into the later stills of the series and the vapours withdrawn therefrom, containing at least 50% of steam, are compressed. The compressed vapours are introduced into the stills in which the lighter fractions are removed and the vapours from these stills are removed and condensed.

H. HOLMES.

Conversion of hydrocarbons [into products of lower boiling point]. V. LOUKINSKY and W. ROBIKOFF (F.P. 606,233, 19.2.25).—Gas oil or petroleum is converted into light hydrocarbons by the catalytic action of sodium aluminium chloride, NaAlCl_4 , or of a compound of aluminium chloride, alumina, and silica.

C. IRWIN.

Dehydrating crude oil. R. J. BARRY (U.S.P. 1,593,893, 27.7.26. Appl., 4.12.25).—Emulsions of oil and water are separated by adding small quantities of salt and diatomaceous clay and heating the mixture.

H. MOORE.

Purifying [used lubricating] oil. BRIT. THOMSON-HOUSTON Co., LTD., Asses. of C. G. VAN BRUNT (E.P. 242,272, 26.10.25. Conv., 29.10.24).—Oil containing suspended matter, e.g., used lubricating oil, is treated with feebly basic hydrophilic material, such as sodium silicate, in a vessel provided with mixing devices. Thence the oil passes to a settling tank containing heated water. From this the oil passes by a weir overflow to an evaporator where diluents are removed. The silicate solution, in a state of fine division, forms a sludge with the impurities, and this sludge subsides

into the hot water. The operation is conducted at a temperature of 70—80°, and the purified oil gives up its heat to the incoming oil in a heat exchanger.

H. MOORE.

Making coal briquettes of anthracitic quality from immature carboniferous substances. J. BEAUDEQUIN (U.S.P. 1,597,570, 24.8.26. Appl., 6.6.23. Conv., 7.6.22).—See E.P. 216,691; B., 1924, 702.

Manufacturing [fuel] briquettes. J. BEAUDEQUIN (U.S.P. 1,597,571, 24.8.26. Appl., 29.11.24. Conv., 3.12.23).—See E.P. 225,849; B., 1926, 349.

Powdered fuel or gas burner. A. L. MOND. From H. T. DYER (E.P. 256,686, 12.5.25).

Dry-quenching of coke. WOODALL-DUCKHAM (1920), LTD., and J. W. REBER (E.P. 256,727, 25.5.25).

Lining pipes etc. (E.P. 255,546).—See XIII.

III.—TAR AND TAR PRODUCTS.

Determination of anthracene. K. KOCH (Z. anal. Chem., 1926, 68, 359—362).—See Jacobsohn, B., 1926, 734.

PATENTS.

Distillation [dehydration] of tar and the like. W. B. DAVIDSON, A. C. MICHE, and E. W. MUDDIMAN (E.P. 255,919, 1.4.25).—Frothing, which is a serious difficulty, especially in the distillation of low-temperature tars, may be prevented by the provision of a heated surface above the level of the liquid tar. A tar dehydrator which is heated by means of a horizontal steam coil just below the surface of the tar can be modified by the provision of a further series of closely pitched steam tubes in the vapour space. These are fed with steam at 120—160°, which then passes on to the lower coil. Should frothing occur, the froth is broken, and in addition the cooling of the steam results in the rate of heating of the still being automatically slowed. Crude tar, without preheating, enters the still at a low level on one side, and dehydrated tar is drawn off from the upper part on the other side, the process being continuous. C. IRWIN.

Treating wood tar oil. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,590,156, 22.6.26. Appl., 27.3.24).—Tar oil from the distillation of wood is saturated with ammonia and is heated in an autoclave under a pressure of 7 atm. On distillation a good yield of a light-coloured oil, with a pleasant odour, boiling up to 125°, is obtained. It is of value as a solvent for nitrocellulose. A similar product is formed by treating the tar oil with an aqueous solution of potassium permanganate, potassium dichromate, or alkali hypochlorite or other oxidising agent, and distilling the product. The two processes may be combined. T. S. WHEELER.

Purification of [aromatic] hydrocarbons. C. R. DOWNS, Assr. to BARRETT Co. (U.S.P. 1,590,965, 29.6.26. Appl., 7.7.20; cf. U.S.P. 1,318,633, B., 1920, 9 A).—Crude aromatic hydrocarbons, e.g., benzene, toluene, naphthalene, or the like containing impurities such as

thiophen and carbon disulphide are mixed in the vapour phase with an equal volume of air and passed at 250—550° for 0.25 sec. over a catalyst consisting of an oxide or a mixture of oxides of vanadium, cobalt, molybdenum, copper, cobalt, manganese, or ruthenium. The impurities are oxidised to sulphur dioxide and carbon dioxide, carbon monoxide, etc., whilst only a small proportion of the hydrocarbon itself is oxidised, and of this the greater part is recovered in the form of useful compounds. Thus from benzene maleic acid and benzoquinone are obtained. T. S. WHEELER.

Treatment of low-temperature tar phenols [to prevent darkening under the action of light]. Purification of phenols. (A) ZEICHE M. STINNES, and A. WEINDEL (G.P. 425,666, 20.11.23), (B) ZEICHE M. STINNES, Assees. of A. CORRELL (G.P. 426,344, 29.4.25).—(A) The phenoxide solution from the crude tar distillate is diluted and treated as in G.P. 387,375 (B., 1924, 740) to give a clear liquor. This is then extracted thoroughly with benzene and ether or similar solvents. These together remove the unstable constituents which cause the reddening of the phenols. The extracts are driven off by heating to 180°. They are black solutions, deep red after vacuum distillation. The phenoxide solution is decomposed as usual, and the phenols are distilled *in vacuo*. They form a clear yellow, stable, and pleasant-smelling liquid representing a yield of 68% of the phenols in the tar-oil treated. (B) Crude phenols are suspended in benzene, and stirred with 10% of 86% sulphuric acid at atmospheric temperature or with cooling. The acid is separated and the benzene solution neutralised with ammonia and washed with water. The benzene is then distilled off and the phenols are purified by vacuum distillation. The product is stable to light and without odour. C. IRWIN.

Purification of phenoxide liquors. ZEICHE M. STINNES, Assees. of F. ULRICH (G.P. 426,224, 7.8.24).—Crude phenoxide liquors are subjected to steam distillation under vacuum at 50—60°. Naphthalene, pyridine, and other impurities are driven off without any sensible loss of phenols and the formation of resins is avoided. C. IRWIN.

IV.—DYESTUFFS AND INTERMEDIATES.

See A., Sept., 949, Pyrogallol sulphonephthal-
ein, sulphonegallein, 2:3:4-trihydroxybenzoyl-
benzene-*o*-sulphonic acid, and derivatives (ORN-
DORFF and FUCHS). 954, Constitution of catechin.
Disintegration products of acacatechin (NIEREN-
STEIN). 956, Synthesis of anthocyanins (ROBERTSON
and ROBINSON); Diazotisability of aminothiophens
(STEINKOPF and MÜLLER). 960, Methylisoidigotins
and methylindirubius (WAHL and FAIVRET).

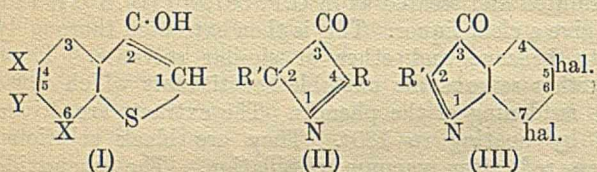
PATENTS.

Manufacture of dyestuffs of the triphenylmethane series. A. G. BLOXAM. From CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 250,785, 29.5.25).—Greenish-blue dyes are made by condensing 1 mol. of *o*-sulphobenzaldehyde with 2 mols. of alkylbenzylarylamines substituted in the benzyl nucleus by halogen and sulpho-groups, and

oxidising the condensation products. The dyes are fast to alkali, and surpass the known similar halogen-free dyes in purity of shade. Examples of suitable alkylbenzylarylamines are the sulphonation products of *o*-, *m*-, and *p*-chlorobenzylethylaniline and 2:4- and 2:6-dichloroethylaniline. They are made by chlorinating the corresponding chlorotoluenes so as to introduce one atom of chlorine into the side chain, and condensing the products with ethylaniline. *m*-Chlorobenzyl chloride, b.p. 210—215°; 2:4-dichlorobenzyl chloride, b.p. 240—243°; and 2:6-dichlorobenzyl chloride, m.p. 39—40°, b.p. 244—245°, are described.

A. DAVIDSON.

Violet vat dyestuffs of the 2-thionaphthen-2-indoleindigo series. R. HERZ and W. BRUNNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,590,685, 29.6.26. Appl., 11.2.26. Conv., 6.11.24).—2-Hydroxythionaphthen derivatives of the general formula (I)



where X is halogen or alkyl and Y is H, halogen, or alkyl, are condensed with a 2-isatin derivative of the general formula (II) where R is an aryl group and R' is halogen or an arylido-group to give dyes formed by doubly linking the 1-carbon of (I) with the 2-carbon of (II). These products dye cotton and wool from an alkaline hyposulphite vat brilliant violet shades fast to light and chlorine surpassing those of known dyes of the same type, and being equal to the best violets of the anthraquinone series in depth and fastness and preferable to them for printing purposes as they are more readily fixed on the fibre and more easily discharged to a pure white. Of especial value are the dyes obtained when X in (I) is halogen, and the isatin derivative has the formula (III). These latter dyes may also be obtained by condensing (I) (X = halogen) with a non-halogenated 2-isatin derivative (R in II = *o*-phenylene) and halogenating the product. The hydroxythionaphthen derivatives (I), all of which are new, are obtained by transforming the corresponding arylamines into the corresponding arylthioglycollic acids and closing the ring either by treatment with an acid condensing agent such as chlorosulphonic acid in the cold, or alternatively converting the arylthioglycollic acid into the corresponding acid chloride and treating this compound with anhydrous aluminium chloride, phosphorus pentoxide, etc. For example, 2:4-dichloroaniline is converted into 2:4-dichlorophenylthioglycollic acid which yields the corresponding chloride with phosphorus trichloride or thionyl chloride, and this with anhydrous aluminium chloride or phosphorus pentoxide gives 4:6-dichloro-2-hydroxythionaphthen, m.p. 140—143°. 4:5:6-Trichloro-2-hydroxythionaphthen, m.p. 150—153°, 4-chloro-2-hydroxy-6-methylthionaphthen, m.p. 106—108°, 6-chloro-2-hydroxy-4-methylthionaphthen, m.p. 120—123°, and 4:6-dichloro-2-hydroxy-5-methylthionaphthen, m.p. 110°, are also described. As an example of dye formation 5:7-dibromoisatin is transformed into

5:7-dibromo-2-isatin chloride by treatment with phosphorus pentachloride in chlorobenzene suspension, and the solution is treated with 4:6-dichloro-2-hydroxythionaphthen in chlorobenzene at 65°, when the corresponding dye separates. It dyes cotton and wool fast blue-violet shades. 4-Chloro-2-hydroxy-6-methylthionaphthen and 5:7-dibromo-2-isatin chloride yield a product which gives fast reddish-violet shades on wool and cotton.

T. S. WHEELER.

Manufacture of azo dyestuffs. A.-G. FÜR ANILIN-FABR. (E.P. 244,782, 16.12.25. Conv., 16.12.24).—Dyes of very good fastness to fulling and light are obtained by coupling tetrazotised chloro-substituted 4:4'-diaminodiphenylmethanes or their homologues with azo-components. The first components are obtained by condensing chloroanilines or chlorotoluidines with formaldehyde and converting the methylene compounds into diaminodiphenylmethanes by the action of a salt of the amine used in presence of acid. Examples are given of wool, direct cotton, and lake dyes prepared from these components.

A. DAVIDSON.

Manufacture of yellow azo dyestuffs. I. G. FARBENIND. A.-G., Assees. of A. DORRER (U.S.P. 1,590,728, 29.6.26. Appl., 4.1.26. Conv., 25.2.24).—A diazotised *p*-chloroanilinesulphonic acid, e.g., *p*-chloroaniline-*m*-sulphonic acid, is coupled with diphenylamine and the product, preferably after conversion into the nitroso-derivative, is nitrated with mixed acid to give various nitro-derivatives, which have a great affinity for animal fibres such as silk, leather, and wool, which they dye in fast yellow shades.

T. S. WHEELER.

Black disazo cotton dye. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of A. L. LASKA and A. ZITSCHER (U.S.P. 1,592,604, 13.7.26. Appl., 2.4.24. Conv., 17.4.23; cf. U.S.P. 1,452,083, B., 1923, 647 A).—Diazotised unsulphonated 4-arylozo-1-naphthylamine derivatives containing basic groups in one or several side chains in the aryl or naphthyl nucleus or both are coupled with an arylide of 2:3-hydroxynaphthoic acid to yield dyes of the general formula, (S_n)Aryl·N₂·X(S_n)·N₂·C₁₀H₅(OH)·CO·NH₂, where S is a group of the type CH₂·NH₂, OCH₂·NH₂, or O·C₆H₄·NH₂, X is the residue of an unsulphonated 1:4-naphthylene compound, and Y is an aryl group. These products when produced on the fibre dye cotton fast black shades, and when mixed with the usual substrata such as barium sulphate or alumina give black colour lakes. As an example, cotton is dipped in an alkaline solution of the β-naphthylide of 2:3-hydroxynaphthoic acid containing Turkey-red oil, and is then immersed in a bath of the diazotised aminoazo-compound from diazotised 4-aminobenzylidimethylamine and α-naphthylamine. A blue-black shade results.

T. S. WHEELER.

Reduction of azo compounds to hydrazo compounds. O. W. BROWN and C. O. HENKE, Assrs. to NEWPORT Co. (U.S.P. 1,589,936, 22.6.26. Appl., 2.11.25).—Aromatic azo- and azoxy-compounds are reduced in good yield to the corresponding hydrazo-compounds by treatment in an organic solvent with hydrogen under pressure in presence of a nickel catalyst. For example,

by treating azobenzene in alcoholic solution at 100° with hydrogen under a pressure of 16.7 atm. in presence of reduced nickel, hydrazobenzene is obtained in satisfactory yield. At 126° and 33.3 atm. aniline is formed in quantitative yield. T. S. WHEELER.

Preparation of condensation products of phenols or phenol ethers and azo-compounds. R. PUMMERER (G.P. 424,863, 4.10.22).—The mixture of the components is heated with acid condensing agents, such as anhydrous zinc chloride, or with free mineral acids, such as hydrogen chloride, or with both. The main products are compounds of undetermined constitution, which are partly soluble and partly insoluble in alkalis. They are either dyes or may be used as intermediates for these.

A. DAVIDSON.

Manufacture of intermediates [monoacylated diamines of the anthraquinone series]. BRIT. DYE-STUFFS CORP., LTD., W. H. PERKIN, and H. M. BUNBURY (E.P. 255,630, 8.7.25).—Benzoic anhydride or an anhydride of a substituted benzoic acid is used as acylating agent. For example, 100 pts. of 1 : 5-diaminoanthraquinone are stirred with 1000 pts. of nitrobenzene at 175–180° for $\frac{1}{2}$ hr. 110 pts. of benzoic anhydride dissolved in 100 pts. of warm nitrobenzene are added, and the whole is stirred at 175° for 15–20 min. The mixture is cooled to 120° and the precipitated monobenzoyl compound filtered off.

A. DAVIDSON.

Making phthalic anhydride. H. D. GIBBS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,591,619, 6.7.26. Appl., 30.10.20).—By passing methyl- or other alkyl-naphthalene mixed with about twice the quantity of air required to furnish the necessary oxygen over vanadium or molybdenum oxide at about 400°, phthalic anhydride is obtained. T. S. WHEELER.

Benzidine flakes. W. L. SPALDING, Assr. to NAT. ANILINE & CHEMICAL Co. (U.S.P. 1,591,688, 6.7.26. Appl., 18.6.23).—A rotating cylinder cooled internally by water is partially immersed in molten benzidine and the layer of solid formed on the cylinder is removed by a scraper. The flakes obtained are more readily handled than the lump form hitherto in use.

T. S. WHEELER.

Purification of anthraquinone. H. F. LEWIS, Assr. to NAT. ANILINE & CHEMICAL Co. (U.S.P. 1,591,712, 6.7.26. Appl., 4.3.20).—Crude anthraquinone from the oxidation of anthracene with chromic-sulphuric acid is extracted with 5–10 pts. of 5% caustic soda solution at 100° for 1 hr., when anthraquinone of good purity remains. Other alkalis, e.g., potassium hydroxide and barium hydroxide, may also be used. T. S. WHEELER.

Method of sublimation [of benzanthrone]. C. A. LYFORD, Assr. to NAT. ANILINE & CHEMICAL Co. (U.S.P. 1,591,715, 6.7.26. Appl., 2.4.20).—Crude benzanthrone is placed on a perforated plate and superheated steam is passed through it. The benzanthrone sublimes in the current of steam at 100–260°, and is obtained in a relatively pure condition. T. S. WHEELER.

Manufacture of 2-hydroxynaphthalene-3-carboxylic acid-*m*-nitroanilide. CHEM. FABR. ROHNER A.-G.

(Swiss P. 111,922, 19.9.24. Addn. to 108,072).—Equimolecular proportions of *m*-nitroaniline and 2-hydroxynaphthalene-3-carboxylic acid are heated in a solution of a tertiary base with phosphorus trichloride. For example, the acid and base are heated at 100° with dimethylaniline until dissolved, phosphorus trichloride is then added slowly, and the mixture stirred for some time longer at 100°. Cold dilute hydrochloric acid is then poured in, the dissolved dimethylaniline hydrochloride removed by filtration, the residue stirred with water, made alkaline with sodium carbonate, and heated until the last traces of dimethylaniline are removed. Technically pure hydroxynaphthoic acid-*m*-nitroanilide is obtained. (Cf. B., 1926, 185.) C. T. GIMMINGHAM.

Separation of ortho-chlorotoluene and para-chlorotoluene. A. R. WAHL, Assr. to SOC. ANON. DES MATIÈRES COLORANTES ET PROD. CHIM. DE ST. DENIS (U.S.P. 1,596,325, 17.8.26. Appl., 23.2.21. Conv., 6.3.20).—See E.P. 159,837; B., 1922, 287 A.

Oxazine dyestuff of the anthraquinone series. R. E. SCHMIDT and B. STEIN, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,596,460, 17.8.26. Appl., 11.2.25. Conv., 18.2.24).—See E.P. 229,668; B. 1925, 875.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Action of light on cotton. H. KAUFFMANN (Textilber., 1926, 7, 617–618; cf. B., 1926, 267).—The production of oxycellulose in cellulose during exposure to light is preceded by the formation of "photocellulose," a substance having similar properties (yellow coloration with caustic soda or phenylhydrazine, and reducing action towards silver salts or Fehling's solution) to oxycellulose. The increased affinity of exposed cotton for methylene blue is not directly due to the presence of photocellulose. Cotton is not deleteriously affected by visible light but only by ultra-violet light such as that emitted by a mercury vapour lamp (wave-length about 350 μ). The action of light on cotton is independent of the presence of oxygen (external or occluded) or moisture, and since there is no accompanying change in weight, the resulting deterioration or formation of photocellulose is not the result of oxidation. Photocellulose has the formula $C_6H_{10}O_5$ and its formation in exposed cotton may be followed by an adaptation of the method previously described using potassium permanganate (*loc. cit.*). In bleaching cotton by exposure to sunlight in meadows, the natural yellow to brown pigment is destroyed by visible light, but the cellulose is simultaneously affected by the ultra-violet light, 1–2% of photocellulose being found in cotton fabric thus exposed for 45 days. The deterioration of cotton by light may be prevented by the presence of suitable light-absorbing pigments, but Anthracene Yellow GC and Chrysoidine have been found unsatisfactory for this purpose. A. J. HALL.

Bacterial decomposition of textile fibres. IV. Action of *B. subtilis* and *B. mesentericus* on cellulose. A. C. THAXSEN and H. J. BUNKER (Biochem. J., 1926, 20, 692–694).—The observations of Trotman and

Sutton (J.S.C.I., 1924, 190 T) that these organisms are capable of decomposing cellulose could not be confirmed.

S. S. ZILVA.

Digestion of spruce wood with dilute nitric acid.

O. ROUTALA and J. SEVON (*Cellulosechem.*, 1926, 7, 113—118).—The oxidation of the lignin with 10% nitric acid sets in with a vigorous evolution of gas at about 75—80° and continues for about 15 hrs. in an open vessel at 95°. As the lignin products do not go into solution the penetration of the acid is slow, and well-digested cellulose is obtained only when uniformly and very finely ground material is used. The penetration is greatly assisted by boiling first with aqueous potassium nitrate and adding later the equivalent quantity of sulphuric acid. The yield and quality of the cellulose, after subsequent boiling out with alkali, are approximately comparable with those obtained by the sulphite process of digestion. The necessary proportion of nitric acid is 35—40% of the weight of the air-dry wood, but the actual consumption of nitric acid is about 30%. The reduction products of the nitric acid comprise nitric oxide, ammonia, and nitrogen; a sensible quantity of hydrocyanic acid is also formed, sufficient to make the process dangerous on a large scale. Nitric oxide and ammonia account for 15% of the total nitrogen consumed, but they are produced in too dilute a condition for practical utilisation. The reaction may be expressed as an oxidation of the side chain of the coniferyl complex of the lignin and the nitration of the aromatic nucleus. The latter process comes to an equilibrium with the production of hydrocyanic acid or nitriles.

J. F. BRIGGS.

Rôle of chemistry in the manufacture of silk.

W. M. SCOTT (*Ind. Eng. Chem.*, 1926, 18, 924—926).

Fifty years' progress in the pulp industry.

J. D. RUE (*Ing. Eng. Chem.*, 1926, 18, 917—919).

See also A., Sept., 942, **Constitution of cellulose** (PRINGSHEIM and others); **Soluble cellulose esters of higher fatty acids** (GAULT and EHRMANN). 983, **Hemicelluloses of beech wood** (O'DWYER).

PATENTS.

Conversion of cellular or fibrous [cellulosic] material. E. TAYLOR and E. F. CHANDLER, ASSRS. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,592—3, 29.6.26. Appl., [A], 29.7.19. [B], 2.12.19).—(A) The patent relates to the formation of products with an unorganised structure, from cellular organic material. Hydrated copper oxide is added to concentrated ammonia to saturation; cotton is then introduced till it ceases to dissolve. The mass is placed in a closed electrolytic cell provided with a copper anode between two copper cathodes, and an exit tube for ammonia gas. On passing a direct current, cellulose, probably as the hydrate, is deposited in colourless flakes of rubber-like consistency on the anode and copper on the cathodes; ammonia escapes at the top and is recovered for further use. The cellulose obtained is pure, easily soluble in cuprammonia solution, and capable of nitration. (B) The cuprammonia solution of cellulose is fed through hop-

pers on to a flexible moving copper belt which passes over pulleys through an electrolytic cell containing dilute sulphuric or nitric acid. Four copper plates serve as cathodes, while the belt itself acts as anode. The cellulose is deposited on one side of the belt, copper being deposited on the cathodes. On leaving the cell the belt passes over pulleys into a washing chamber containing water, then into a drying chamber, where the cellulose shrinks from the belt, next into amyl acetate, which improves the appearance and increases the density. Finally, the finished product is wound on rollers. Means are provided for concentrating the cuprammoniacal solution of cellulose before it is fed to the belt, for collecting the ammonia generated in the cell, and for recovering amyl acetate.

R. B. CLARKE.

Treatment of fibrous or cellular [cellulosic] organic material.

E. TAYLOR, ASSR. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,594, 29.6.26. Appl., 11.3.20. Renewed 17.6.24).—The patent relates to an electrolytic method for obtaining cellulose in a definite form, e.g., rods, tubes, filaments etc., from a solution of cellulose in cuprammonia or zinc chloride. The apparatus consists of three closed tanks arranged vertically one above the other, the top tank containing a solution of cellulose in cuprammonia, and the lower tanks very dilute sulphuric acid. The two lower tanks are connected by four tubes, and acid is pumped from the lowest tank, which is also an electrolytic cell, into the middle tank, in order to maintain a head of acid above the tubes. Four smaller tubes, tapering to fine orifices, emerge from the bottom of the top tank and extend downwards into the tubes covered by acid. The cellulose solution emerging from the orifices meets a downward stream of acid which serves to draw out the cellulose as a thread. In the bottom tank these threads fall on to a moving copper belt, immersed in acid, which acts as the anode of the cell. Above the belt is placed a copper cathode. On electrolysis copper is deposited at the cathode and cellulose on the belt. The precipitated cellulose threads are carried to another belt at right angles, washed, and wound on rollers.

R. B. CLARKE.

Production of colloidal material [cellulose].

E. TAYLOR, E. F. CHANDLER, and T. A. HILL, ASSRS. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,596, 29.6.26. Appl., 8.10.20).—A method for obtaining sheets of cellulose by electrolysis of a cuprammonia solution of cellulose. The apparatus consists of two vulcanite tanks connected by a pipe at the bottom and by a U-shaped pipe near the top. A pump situated in the U-tube circulates a solution of ammonium nitrate from the right-hand to the left-hand tank, both tanks being completely filled. A smaller vulcanite container, having a felt bottom, fits inside the right-hand tank and rests on a non-conducting flange. Copper plate electrodes are arranged at the top and bottom of each tank, the top plates serving as anodes. The anode of the right-hand tank is arranged just above the container, which is charged with a concentrated cuprammonia solution of cellulose. When a current is passed copper is deposited on the cathodes and a pure film of cellulose is left above the felt.

R. B. CLARKE.

Treating silk and cellulose material. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,600, 29.6.26. Appl., 17.6.24).—A mixture of silk and cotton can be separated by treating it with an ammoniacal nickel solution which dissolves the silk only. The cotton is separated by filtration, and the filtrate is electrolysed in the apparatus described in U.S.P. 1,590,594 (*ante*) to recover the silk in a pure form. R. B. CLARKE.

Treatment of organic material [cellulose]. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,601, 29.6.26. Appl., 17.6.24).—A solution of cellulose in cuprammonia contained in a tank is forced out at the bottom through a nozzle by means of gas pressure. As it emerges it is sprayed, by means of a jet of compressed air, and deposited in finely-divided particles on a moving copper belt which forms the anode of an electrolytic cell. A slow stream of dilute acid falling down into the cell in the same direction as the moving belt prevents the particles of solution from coagulating. The belt on reaching the cell travels above a copper cathode at the bottom and the solvent constituents of the cellulose solution are separated and the cellulose is precipitated. On leaving the cell the particles of precipitated cellulose are removed from the belt by means of a water spray and fall to the bottom of a water tank, whence they are removed and dried. The product consists of very finely divided cellulose.

R. B. CLARKE.

Treating organic material [cellulose] and apparatus therefor. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,606, 29.6.26. Appl., 21.6.24).—In a process for converting fibrous cellulose, *e.g.*, old rags etc., into films, the same apparatus, with modifications, is used for dissolving the cellulose and for electrolytically depositing it in a colloidal form. A tank having a porous screen near its base is filled with dilute aqueous ammonia or water and cellulose is placed on the screen; at the top is an outlet tube connected to a cooling worm, an inlet tube for ammonia gas being placed in between. The worm is connected to a cell filled with ammonia solution and having six copper electrodes. The solution is circulated through the apparatus and ammonia gas is passed in under pressure. When an alternating current is applied a solution of cuprammonia is formed which dissolves the cellulose. When a sufficiently high concentration is reached electrodes of antimony-lead are placed in between the copper plates, and on passing a direct current cellulose is deposited on them.

R. B. CLARKE.

Treatment of organic material [cellulose]. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,607, 29.6.26. Appl., 2.7.24).—A film of a cuprammonia solution of cellulose is picked up by a slowly rotating drum around which hot air travels in the same direction, thereby stiffening the film. As it leaves the drum the film passes through dilute acid which increases its strength. It then passes into an electrolytic cell, where the solvent constituents are abstracted. On leaving the cell the material is washed, and to make it non-hygroscopic and suitable for artificial leather it is treated with 30% glycerin and water and dried.

R. B. CLARKE.

Electrolytic cell for deposition of cellulose (U.S.P. 1,590,604 and 1,590,609).—See XI.

Production of artificial leather (U.S.P. 1,590,602).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Technical analysis of Indanthrene dye vats. G. DURST and H. ROTH (*Z. angew. Chem.*, 1926, 39, 900—903).—Simple titration of the vat with mild oxidising agents (*e.g.*, Methylene Blue or ferric ammonium sulphate) does not give the true excess of hyposulphite present, but the total reducing power of the hyposulphite and dye together. By adding formaldehyde to the vat the hyposulphite is stabilised, whilst the dye acids are precipitated, so that, after addition of acetic acid, filtration, and addition of sodium acetate and salicylic acid to the filtrate, the hyposulphite may be determined by titration with ferric ammonium sulphate. The content of dye in the vat may be determined gravimetrically by oxidising the vat with a mixture of ferric ammonium sulphate, sodium acetate, and acetic acid, filtering through a Jena sintered glass crucible, and washing the precipitate thoroughly with acid, alkali, and water. The free sodium hydroxide in the vat is best determined by adding barium chloride (to precipitate sulphites and carbonates) and neutral formaldehyde (to bind the hyposulphite), and then, immediately and without filtering, titrating with 0.1*N*-acetic acid, with phenolphthalein as indicator.

W. T. K. BRAUNHOLTZ.

Influence of additions to the vat in dyeing with indigo. R. HALLER (*Textilber.*, 1926, 7, 612—615).—The process of vatting indigo with a hyposulphite and caustic soda is considerably retarded by the presence of protective colloids, particularly glue, gums, gum tragacanth, and soap (Turkey-red oil has less effect). The stability of the vat is decreased, an unusually large number of submicrons are present, the dispersion of the indigo being less, and the resulting dyeings have unsatisfactory fastness to weathering and soap. The presence of methyl, ethyl, and amyl alcohols or acetone assists the dispersion and stability of indigo vats; vats containing methyl or ethyl alcohol contain a very small number of submicrons. Although the resulting dyeings are deeper, their fastness does not appreciably differ from dyeings obtained from a normal vat. Pyridine, and particularly Tetracarnite (Sandoz), increase considerably the dispersion of indigo vats, and the resulting dyeings have greater depth of shade and fastness than those obtained from normal vats of similar concentration. The presence of nitrobenzene assists the vatting of indigo, the resulting vat being orange-red. Indigo vats containing Aktivin (*cf. B.*, 1926, 484) are orange-yellow, and yield dyeings slightly deeper but of normal fastness. Katanol, and wetting agents such as Nekal A and Curacit, salt, and Glauber's salt affect indigo vats unfavourably. The rates of dialysis of indigo vats containing alcohols or acetone, and protective colloids, were rapid and very slow respectively. A. J. HALL.

Use of potassium salts in printing [textiles]. W. SIEBER (*Textilber.*, 1926, 7, 615—616).—The common

practice of replacing potassium salts by the cheaper corresponding sodium salts in printing processes sometimes gives less satisfactory results. White resists for alizarin shades are much clearer when obtained by means of potassium citrate than with sodium citrate, particularly when the resists are over-printed with Aniline Black. A satisfactory resist for the latter process is prepared by adding about 26.3 kg. of potassium hydroxide (free from sodium hydroxide) to 28 kg. of citric acid (crystalline) and 23 litres of water until the mixture is just slightly alkaline, and then adding 18 kg. of gum arabic or gum senegal. In dyeing with ice colours, *e.g.*, Para Red, the progressive discoloration (brown) which occurs with fabric prepared with a solution of β -naphthol in sodium hydroxide may be avoided by use of potassium hydroxide. The dry prepared fabric may then be stored for several weeks without decomposition or discoloration, and the resulting shade in the case of Para Red is bluer than that similarly obtained with sodium hydroxide. A prepare made with potassium hydroxide is especially suitable for the production of Para Red discharges on Aniline Black. A. J. HALL.

[Preparation of] discharge printing pastes containing caustic soda and having no deleterious action on [copper] printing rollers. W. SIEBER (*Textilber.*, 1925, 6, 829—830; 1926, 7, 244).—Printing pastes suitable for discharging fabric mordanted with tannic acid are prepared by adding caustic soda to a solution of gum arabic or senegal, it being particularly essential to use efficient stirring and cooling so as to avoid coagulation of the gum. For example, 53.5 kg. of caustic soda of 36° B. (*d* 1.334) are added slowly to 20 litres of a 50% solution of gum senegal, the temperature being strictly maintained at 20—22°. A more effective discharge paste is obtained by incorporating 1700 g. of potassium hydroxide in the paste described above. The familiar damage caused to printing rollers by use of similar pastes prepared, with heating or without cooling, with starches and British gum, is due to the adherence of glutinous lumps of the paste to the printing rollers and doctors; it is not due to the separation of crystalline sodium salts. A. J. HALL.

Textile dyeing [during the past fifty years]. W. K. ROBBINS (*Ind. Eng. Chem.*, 1926, 18, 927—929).

PATENTS.

Treatment of textile fibres, yarns, fabrics, or the like for obtaining effects of colour or lustre, or both. N. V. NEDERLANDSCHE KUNSTZIJDEFABRIEK (E.P. 245,407, 7.5.25. Conv., 30.12.24).—See F.P. 597,231; B. 1926, 437.

Multi-chamber yarn dyeing apparatus. P. F. HÖLTZING (E.P. 256,854, 15.1.26).

Yarn-dyeing apparatus. P. F. HÖLTZING (E.P. 257,160, 15.1.26).

Black disazo cotton dye (U.S.P. 1,592,604).—See IV.

Dyeing of leather (E.P. 255,555).—See XV.

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

Catalytic removal of oxygen from gas mixtures containing hydrogen [*e.g.*, ammonia-synthesis gases]. J. A. ALMQUIST and E. D. CRITTENDEN (*Ind. Eng. Chem.*, 1926, 18, 866—867).—Copper prepared by the reduction of copper oxide is more effective than massive copper in promoting the catalytic removal of oxygen as water from, *e.g.*, by-product nitrogen to be used in the synthesis of ammonia. Copper in any form is a more suitable catalyst than platinum.

B. W. CLARKE.

Determination of water-soluble barium in black ash. W. F. MEISTER and T. STEPHENS (*Ind. Eng. Chem.*, 1926, 18, 828—829).—The water-soluble barium in barium black ash may be accurately determined by boiling 0.2 g. of the ash with 125 c.c. of water for 5 min., filtering rapidly through asbestos, washing the residue with 100 c.c. of boiling water, and boiling the filtrate and washings with 25 c.c. of 0.5*N*-sulphuric acid for 5 min. After settling for 1 hr. the precipitated barium sulphate is collected, ignited, and weighed. A rapid volumetric control method may be carried out on 2 g. of ash by treating the filtrate with 50 c.c. of sulphuric acid (23.16 g./litre), boiling until all hydrogen sulphide is expelled, and titrating the excess of acid with sodium hydroxide solution of the same strength, using phenolphthalein as indicator. Addition of a correction of 0.5 c.c. to the volume of sulphuric acid used is necessary, this representing the difference in the end-points for phenolphthalein and methyl-orange; the corrected volume multiplied by 2 gives the percentage of barium sulphide in the sample. J. W. BAKER.

Reagent and standard for borax. L. S. WEATHERBY and H. H. CHESNY (*Ind. Eng. Chem.*, 1926, 18, 820—821).—Borax, either alone or mixed with other salts, is accurately determined by direct titration with 0.1*N*-hydrochloric acid using methyl-orange as an indicator, in the presence either of mannitol, or the much cheaper commercial dextrose (cerelose). The results obtained using lævulose, glycerol, honey, or invert sugar are less satisfactory. Borax in alkaline brine solutions containing sodium carbonate or sodium hydrogen carbonate may also be thus determined if the alkalinity of the borax and other alkalis is first determined separately, and colour standards then prepared corresponding to the calculated end-points of the mixtures. J. W. BAKER.

Chlorination of metals. P. S. BRALLIER (*Trans. Amer. Electrochem. Soc.*, 1926, 49, 65—71).—A description of the preparation of some metal chlorides with special reference to large-scale production. Silicon tetrachloride may be prepared by the action of chlorine at a high temperature on a mixture of 90% silicon and silicon carbide. A vertical shaft furnace is advocated consisting of a water-cooled steel or cast-iron shell, lined with carbon slabs, with a thin layer of dry sand between the carbon and steel. The condenser should be flushed with cooled liquid silicon tetrachloride to prevent choking by ferric or aluminium chloride. Titanium tetrachloride may be prepared similarly using titanium carbonitride containing 70—75% of titanium. The heat of formation

of titanium tetrachloride is roughly determined as 156,000 cal. per g.-mol. Tin tetrachloride may be prepared by the action of chlorine on metallic tin immersed in liquid stannic chloride containing a little sulphur. If the raw material is tin-plate, this may be directly treated with chlorine, but with pig tin the rise of temperature is too great and the above method is better. Antimony trichloride may be prepared by the direct action of chlorine on metallic antimony at about 80–90°, but careful regulation is essential to prevent rise of temperature and fusion of the metal. Zinc chloride can be prepared by bubbling chlorine into molten zinc kept at about 500° in a steel or cast-iron box lined with refractory clay. The crude chloride rises to the top and is tapped off; it contains finely-divided zinc and zinc oxide ("blue powder") in suspension, but a product of 99·8% purity is obtained on redistillation.

W. HUME-ROTHERY.

Fire risk of concentrated hydrogen peroxide solutions. G. AGDE and E. ALBERTI (*Z. angew. Chem.*, 1926, **39**, 1033–1035).—A series of experiments undertaken to elucidate the cause of a fire in a railway wagon loaded with wicker-cased carboys containing 60% hydrogen peroxide, to which stabiliser had been added, showed that used engine-room woollen waste inflamed spontaneously when brought into contact with the solution at ordinary temperatures. Clean waste, soaked with oil, did not ignite under these conditions, but when iron filings were also present, inflammation followed. Sweepings from wooden floors and even fine sand had the same effect as iron filings, causing even fairly thick wood, saturated with the peroxide solution, to ignite. A very small glowing charred patch in wood is sufficient to cause intense combustion when the solution is brought into contact with it.

S. I. LEVY.

Production of iodine in Chile. J. B. FAUST (*Ind. Eng. Chem.*, 1926, **18**, 808–811).—Average Chilean "caliche" contains 0·15% of iodine in the form of sodium iodate. Owing to the repeated use of mother liquors for leaching the caliche, the iodate accumulates until the liquor contains 6–12 g. of iodine per litre. The extraction of iodine is performed by reduction of iodate with sodium bisulphite. Sodium carbonate is prepared by burning 100 pts. of sodium nitrate with 16–20 pts. of coal, and extracting the cooled mass. The settled liquor (*d* 1·12) is saturated with sulphur dioxide prepared by burning sulphur. The iodate mother liquor is mixed with an excess of bisulphite solution in a wooden tank, nearly neutralised with sodium carbonate, and the quantity of mother liquor necessary to complete the reaction added. The iodine is allowed to settle, washed, and hand-pressed, yielding a product containing 75–80% of iodine. This is broken up and heated over direct fire in cement-lined retorts. The vapours are condensed in earthenware pipes, any water being allowed to drain away from the joints. The regular working efficiency of an average plant will not exceed 65–70%. The chief losses occur in weak liquors run away. Large losses are also usual in preparing the bisulphite solution. The amount of mother liquor which can be treated for iodine is limited by the fact

that this treatment means the addition of water to the nitrate cycle and consequently more loss of nitrate in the latter. Other methods which have been suggested for iodine extraction are reduction with sodium thio-sulphate and sulphuric acid, and reduction with sulphur dioxide direct. The latter method would reduce the dilution of the liquors, but the sulphuric acid produced would require to be neutralised. The cost of production of iodine under present conditions is estimated at 50–75 cents per kg. Owing to economic circumstances only 2% of the iodine available is at present extracted.

C. IRWIN.

Preparation of aqueous thiocyanic acid. GLUUD, KELLER, and KLEMPF.—See A., Sept., 943.

Ammonia recovery by gypsum. JACKMAN.—See II.

Manufacture of sulphuric acid. METALLBANK U. METALLURGISCHE GES. A.-G. (G.P. 427,541, 4.4.25. Addn. to 370,853; cf. E.P. 184,966, B., 1922, 858 A).—In the plant described in the previous patent there was used in the middle section nitrosylsulphuric acid of lower sulphuric acid content than that used in the front and rear sections. According to the present patent an additional acid cycle is introduced which is independent of the rest of the plant. In this section, consisting of several towers in series, nitrosylsulphuric acid of *d* about 1·71 is used as trickling liquid. The gases are led up these towers in turn. This effects a concentration of the acid, which is taken up to the top of the first tower after leaving the last, thus rendering the process continuous.

R. B. CLARKE.

Manufacture of ammonium phosphates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 255,766, 3.3.26).—Phosphoric anhydride is absorbed from gases containing it by ammonium phosphate solution, ammonia in the quantity required for the desired ammonium phosphate being added before, after, or during the absorption. A hot saturated solution is obtained and the solid ammonium phosphate separates on cooling, the mother liquor being used repeatedly for the absorption.

W. G. CAREY.

Manufacture of diammonium phosphate. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 256,137, 22.2.26).—Phosphoric acid and ammonia are added to a saturated solution of diammonium phosphate while maintaining a ratio of 1 molecular proportion of acid to between 1·5 and 2 molecular proportions of ammonia, these proportions being controlled by titration, hydrogen-ion concentration measurements, or indicators with suitable transition points. The heat of the reaction evaporates the required amount of water, and the solution is cooled and gaseous ammonia is added until the solution contains 2 proportions of ammonia to 1 of acid, when pure diammonium phosphate separates.

W. G. CAREY.

Carrying out exothermic gaseous catalytic reactions [especially the synthesis of ammonia]. SYNTHETIC AMMONIA & NITRATES, LTD., and H. A. HUMPHREY (E.P. 255,963, 4.5.25).—In a reaction of

the kind mentioned in the title a more even distribution of temperature in the catalytic mass is secured by periodic reversal of the gas flow. C. IRWIN.

Carrying out exothermic gaseous catalytic reactions [especially the synthesis of ammonia]. SYNTHETIC AMMONIA & NITRATES, LTD., and R. E. SLADE (E.P. 255,964, 4.5.25).—A catalyst chamber is constructed narrow at the centre and becoming wider towards the inlet and outlet ends, and is fitted with a number of transverse heat-insulating partitions. On commencing the reaction the centre portion is electrically heated. When reaction is established the applied heat is withdrawn, and the zone of maximum temperature gradually travels forward in the direction of the gas flow. When it nears the end of the chamber the gas flow is reversed and it is caused to travel back again. By this method of working a heat interchanger is rendered unnecessary, the catalyst mass itself performing that function. With intense reaction artificial cooling may be necessary, but otherwise the gaseous products of reaction pass away at about 100° only. C. IRWIN.

Method of forming nitrogen-hydrogen mixtures. J. C. CLANCY, Assr. to NITROGEN CORP. (U.S.P. 1,594,815, 3.8.26. Appl., 18.2.22).—In the production of a nitrogen-hydrogen mixture from atmospheric nitrogen, the heat generated by causing the atmospheric oxygen to unite with hydrogen is used to heat a quantity of metal, over which the gaseous mixture is passed to remove the last traces of oxygen. H. ROYAL-DAWSON.

Tricalcium cyanide. R. W. POINDEXTER, JUN., Assr. to CALIFORNIA CYANIDE Co. (U.S.P. 1,592,127, 13.7.26. Appl., 21.11.25).—By combining calcium oxide, water, and hydrogen cyanide in exactly the required proportions, $\text{Ca}_3(\text{OH})_4(\text{CN})_2$ is obtained as a whitish pulverulent compound, dissociating on treatment with water into calcium hydroxide and calcium cyanide, which evolves hydrocyanic acid continuously. The compound is valuable as a source of hydrocyanic acid for fumigating, as it keeps well in the absence of moisture. E. S. KREIS.

Reduction of neutral and acid metal-salt solutions. H. BARDT (U.S.P. 1,592,173, 13.7.26. Appl., 18.11.24. Conv., 7.3.23).—When copper solutions are reduced by treating with cellulosic material, the separation of the metal from the fibrous residue is expensive. According to the present patent the cellulosic material is hydrolysed by heating under pressure with an inorganic acid or salt for several hours, and the resulting clear solution is used for reducing the copper solution. E. S. KREIS.

Prepared metallic chloride and method of making it. H. H. DOW, Assr. to DOW CHEMICAL Co. (U.S.P. 1,592,971, 20.7.26. Appl., 26.9.25).—The patent relates to a method of producing calcium chloride or similar hygroscopic chloride in a form which can be packed without setting and hardening. A cooled rotating drum dipping into molten calcium chloride, corresponding to the composition $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, picks up a thin layer, which is scraped off and conveyed to the top of an inclined rotary cylinder. Here it is mixed with anhydrous

powdered calcium chloride, introduced through a hopper. The material passes down the wall of the cylinder, and travels over a screen at the bottom which removes any powder in excess of that required to coat the flakes. R. B. CLARKE.

Flake magnesium chloride and method of making it. W. R. COLLINGS, Assr. to DOW CHEMICAL Co. (U.S.P. 1,593,440, 20.7.26. Appl., 28.11.21).—Magnesium chloride hexahydrate, containing slightly less than the normal amount of water of crystallisation, is prepared by picking up the molten salt at 169° in a thin layer on a revolving drum, from which it is scraped after cooling. The product, which is non-caking, has a glass-like finish, and is much harder than the normal hexahydrate. R. B. CLARKE.

Producing barium chloride. A. JAHL, Assr. to J. MICHAEL & Co. (U.S.P. 1,593,902, 27.7.26. Appl., 6.3.24. Conv., 5.12.22).—A solution containing barium sulphide and barium hydrosulphide is treated first with solid barium chloride and then with an alkali chloride. C. IRWIN.

Products from barium sulphide. RHENANIA VEREIN CHEM. FABR. A.-G., ZWEIGNIEDERLASSUNG MANNHEIM, and F. RÜSBERG (G.P. 426,925, 20.10.23. Addn. to 423,755, B., 1926, 322).—Decomposition of barium sulphide is carried out by means of suitable acids or similarly acting materials in a solution of barium chloride. A. COUSEN.

Making zinc sulphide. F. G. BREYER and C. W. FARBER, Assrs. to NEW JERSEY ZINC Co. (U.S.P. 1,594,001—2, 27.7.26. Appl., 7.4.25).—(A) A mixture of zinc oxide pigment and sulphur is heated to a temperature at which the formation of zinc sulphide takes place readily, and the product is subjected to a heat treatment under non-oxidising conditions. (B) The reaction is carried out by passing the zinc oxide-sulphur mixture progressively through a long vertical reaction chamber maintained at a suitable temperature to allow the reaction to take place readily. A. R. POWELL.

Treating salt solutions. W. H. BINDER (U.S.P. 1,594,707, 3.8.26. Appl., 5.11.25).—Potassium chloride is added to a saturated solution containing potassium chloride, sodium carbonate, sodium chloride, sodium sulphate, and borax; the liquor is heated to dissolve the potassium chloride, and then added to a dry mixture of the salts mentioned, and again heated, whereby the sulphate, carbonate, and chloride of sodium remain undissolved, and are separated from the liquor. On cooling the liquor potassium chloride separates, and on further cooling, the borax crystallises out. H. ROYAL-DAWSON.

Manufacture of alkali metabisulphites or bisulphites. F. LEROY (F.P. 605,115, 29.10.25. Conv., 26.10.25).—A continuous process in which a suspension of a normal sulphite in a saturated solution, obtained by neutralising a solution of bisulphite with solid alkali carbonate or bicarbonate, is saturated with sulphur dioxide. The carbon dioxide generated is recovered. R. B. CLARKE.

Manufacture of arsenic pentoxide. P. ASKENASY and E. ELÖD (E.P. 255,522, 21.4.25).—150 pts. of arsenious oxide and 150 pts. of 60% nitric acid are introduced into a closed vessel having a stirrer and a jacket. A small quantity of arsenic pentoxide or other catalyst may be added. Oxygen at 20 atm. is then forced in and the mixture stirred for 12—18 hrs. at 70—90°. The nitric acid remains nearly unchanged and can be distilled off, the arsenic acid solution being afterwards converted into calcium arsenate, if desired. Arsenious sulphide may be used in place of the oxide.

C. IRWIN.

Apparatus for manufacture of carbon disulphide. H. SCHULZ (G.P. 407,656, 1.4.23).—Sulphur and charcoal are heated in a chamber with internal heating, as, e.g., by means of vertical heating resistances or pipes through which generator gas or the like is passed. The resistances are provided with protective coverings of suitable material, preferably metal, and these are fixed in tubes of firebrick or the like. The walls of the furnace are of refractory material, with inner and outer metal coverings, and arrangements are made for heating and vaporisation of the sulphur in suitable spaces in the furnace walls. The heating is so arranged in relation to the furnace walls that the latter are more strongly heated in places where the sulphur deposits than elsewhere.

C. T. GIMINGHAM.

Preparation of concentrated solutions of hydrogen peroxide. E. DE HAËN A.-G. (G.P. 428,707, 10.4.24).—In the preparation of concentrated hydrogen peroxide solutions from phosphoric acid and barium peroxide, barium carbonate is added to the latter in increasing amounts as the reaction proceeds, until at the conclusion the barium peroxide may even be completely displaced. Other neutral carbonates, as those of calcium, strontium, or magnesium, or the bicarbonates of the alkalis, may be used in place of barium carbonate.

A. COUSEN.

Separating gas mixtures, more especially air or other difficultly liquefiable gas mixture. R. F. and R. K. E. MEWES (U.S.P. 1,594,336, 27.7.26. Appl., 12.3.24. Conv., 16.2.22).—In the case of a rectifying column fractionating liquid air under high pressure, a surplus of liquid nitrogen is produced at the top and a surplus of oxygen evaporated at the bottom. The descending liquid nitrogen is evaporated and the ascending oxygen gas liquefied, within the column.

C. IRWIN.

Gas purification [removal of carbon monoxide from ammonia-synthesis gases] by ammoniacal cuprous solutions. J. D. DELY, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,597,345, 24.8.26. Appl., 16.8.23).—See E.P. 220,651; B., 1925, 957.

Removal of calcium and magnesium from rock-salt brine. F. A. FREETH and L. A. MUNRO, Assrs. to SOLVAY PROCESS CO. (U.S.P. 1,597,370, 24.8.26. Appl., 21.8.20. Conv., 15.2.19).—See E.P. 149,707; B., 1920, 689 A.

Cooling hot salt solutions (G.P. 430,482).—See I.

Manufacture of calcium carbide etc. (F.P. 605,530).—See II.

VIII.—GLASS; CERAMICS.

Properties of fused quartz and other forms of silicon dioxide. H. L. WATSON (J. Amer. Ceram. Soc., 1926, 9, 512—534).—A compilation of data on the physical properties of the crystalline modifications of silica, the physical and chemical properties of vitreous silica, and a short account of the cryptocrystalline and hydrated amorphous forms of the oxide. A. COUSEN.

Hydrogen-ion measurements on clay slips. D. W. RANDOLPH and A. L. DONNENWIRTH (J. Amer. Ceram. Soc., 1926, 9, 541—547).—For determination of the p_H value of clay slips (clay 1, water 12) an electrometer of calomel and hydrogen electrodes was used. No change in the p_H value was noted when ball clays and kaolins were aged for 2—3 weeks. A definite relationship was found between the dry strength of a clay and the p_H value of the slip from which it was obtained. The relationship between viscosity and p_H value for any particular clay depended largely on the electrolyte used. The effect of a change of p_H of a ball clay on the plasticity (as measured by Bingham's method) was also determined.

A. COUSEN.

Physical properties of ceramic bodies. F. SINGER (Z. Elektrochem., 1926, 32, 382—395).—Ceramic products are arranged according to their common nomenclature in seven groups. Considered solely from the point of view of chemical composition, these groups are shown to vary from two-component to multi-component systems. All the three- and four-component systems are based on the two-component system $Al_2O_3-SiO_2$; e.g., in combination with alkalis as porcelain and earthenware; with alkalis and lime as earthenware; and with alkalis and iron oxide as stoneware. Unlike fusions, sintered ceramic bodies are characterised by incomplete physical and chemical homogeneity in the liquid phase; they contain, in addition to a fused glassy matrix, undissolved crystallites of raw material and newly-formed crystals. The latter determine the characteristics of the product. The results of mechanical and thermal tests on the various types of ceramic products are given in tables. Photomicrographs are reproduced to illustrate the importance of the effect of mullite crystals, their arrangement and distribution, upon the physical properties of ceramic bodies. The modern aim is to produce the maximum number of minute mullite crystals in close, felty formation embedded in a homogeneous glass.

F. SALT.

Deformation of ceramic masses on drying. O. KRAUSE (Kolloid-Z., 1926, 39, 263—264).—Ceramic masses of high plasticity, especially those rich in kaolinite, show on drying a twisting in a direction opposite to that in which they were worked on the potter's wheel. In some cases this torsion manifests itself by the formation of characteristic cracks during firing.

N. H. HARTSHORNE.

Methods of testing and physical properties of wet-process electrical porcelain. L. NAVIAS (J. Amer. Ceram. Soc., 1926, 9, 501—510).—In the determination of compressive strength of electrical porcelain it is suggested that the ultimate failure should be recorded

as it is more definite and constant than the initial failure. The height of the specimen is an important variable, and a cylindrical specimen, 1 sq. in. in area and $1\frac{1}{2}$ in. high, is recommended. A test-piece of similar area is recommended for the transverse strength. As the area of minimum cross-section increases the value obtained for tensile strength increases rapidly, and the diameter of the test-piece should be given, together with the determined values. A convenient apparatus for determining tensile strength is described.

C. A. KING.

Electrical resistance of refractory materials. J. B. FERGUSON (Canad. Chem. Met., 1926, 10, 131—136).

—Refractory materials are regarded as crystalline aggregates cemented together by a glassy bond. The results of previous work on the electrical properties of glasses and crystals are correlated on the basis of the Rasch-Henrichsen formula: $\log \rho = A/T + B$, in which ρ is the resistivity, T is the temperature (absolute scale), and A and B are constants peculiar to the substance. By plotting $\log \rho$ against $1/T$ a straight-line relationship where the rule is obeyed is obtained. Changes of slope are observed in certain critical zones. With Royal Berlin porcelain a slight change of slope occurs at about 1070° . Talc follows the rule fairly closely. Slight changes of slope are observed with sillimanite and magnesite. With kaolin and flint fireclay changes occur at the temperatures at which chemical changes take place. The curve obtained with a firebrick suggested continuous changes in the material; silica bricks followed the rule to a high temperature, at which the resistivity decreased suddenly, indicating that actual melting had taken place; with magnesite bricks the rule held over the whole temperature interval. For ordinary commercial refractories the plotted results may be expected to fall on a series of straight lines, changes of slope occurring at the melting interval of the matrix, at the inversion temperatures of the crystals present, and at the point where chemical reaction takes place.

F. SALT.

Firing terra-cotta in an open kiln. O. E. MATHIASSEN (J. Amer. Ceram. Soc., 1926, 9, 548—550).—Terra-cotta was successfully fired in open oil-fired kilns of the regular rectangular type, but having special flues and combustion chamber. Details of the flues and combustion chamber and method of working are given.

A. COUSEN.

Fifty years of glass-making. A. SILVERMAN (Ind. Eng. Chem., 1926, 18, 896—899).

A half-century of progress in the glass industry. G. W. MOREY (Ind. Eng. Chem., 1926, 18, 943—945).

Influence of chemistry on ceramics [during the past fifty years]. R. C. PURDY (Ind. Eng. Chem., 1926, 18, 952—953).

Production from raw clay of material stable towards water. BUDNIKOV.—See IX.

Inside frosting of electric lamps. PIPKIN.—See XI.

PATENTS.

Fining glass. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY ET CIREY (E.P.

250,536, 5.2.26. Conv., 10.4.25).—Glass is fined by causing it to flow from the lower region of the melting chamber, as a thin sheet, into the first portion of the fining chamber, which possesses a raised hearth; the glass being subjected completely to an elevated temperature therein. The second part of the fining chamber is deeper and at a lower temperature than the first; and the glass flows down a slope to the bottom of this zone, finally passing out from below into the pouring zone. Modifications of the above: (1) the fining zone has a second portion without slope; (2) the connecting channels between the melting and fining zones are heated electrically; (3) suitable heating and cooling arrangements are provided to maintain the desired temperatures in the various zones.

A. COUSEN.

[Alloy for] glass-melting container. J. BRYCE (U.S.P. 1,592,336, 13.7.26. Appl., 30.1.23).—An alloy containing Fe (73—53%), Cr (25—45%), Si, C etc., is used to construct containers for the manufacture of glass; the alloy is completely resistant at all the temperatures employed to the action of the ingredients forming the batch.

T. S. WHEELER.

Treating glass. C. A. KRAUS (U.S.P. 1,592,429, 13.7.26. Appl., 5.6.24).—A glass surface is treated with silver nitrate solution and heated at 160° for 10 min. It is then cooled, washed, and reheated at 220° for 20—60 min. Some of the sodium ions present in the surface of the glass are replaced by silver ions, and although the presence of the silver ions is normally invisible, yet on causing liquid to condense on the glass, e.g., by breathing on it, the effect is apparent. The method is suitable for trade-marking glass articles, such as lenses.

T. S. WHEELER.

Melting of quartz glass and other highly refractory materials. DEUTSCH-ENGLISCHE QUARZSCHMELZES G.M.B.H., and Z. VON HIRSCHBERG (G. P. 428,654, 28.2.24).—As a fuel, the vapour of a liquid hydrocarbon such as benzene, benzine, petroleum, etc., or mixtures of these, is mixed with oxygen under pressure. Such a fuel is more convenient than acetylene and allows of better observation of the material.

A. COUSEN.

Manufacture of pottery. R. G. VARCOE (E.P. 255,607, 16.6.25).—As a source of silica and alumina in a cheap, rapidly mixed form for potter's clay, china clay waste or tailings is dried and powdered. Approximately 76% of this is incorporated with 14% of ball clay, 5% of china clay, $2\frac{1}{2}\%$ of flint, and $2\frac{1}{2}\%$ of china stone.

A. COUSEN.

Manufacture of porcelain bodies which can be easily fused and cast. H. BECKER, SEN. (G.P. 430,387, 1.4.25).—Fluorspar is smelted with glass waste, or a similar material, containing a small quantity of alumina which decreases the volatilisation of silicon fluoride and, consequently, damage to the furnace walls; the quantity of fluorspar should be at least 20% of the total. If a metal oxide be added to the melt a metallic lustre is imparted to the surface of the finished product.

R. B. CLARKE.

Fire- and acid-proof plastic substances [refractories]. A. WOLFSHOLZ (E.P. 256,790, 24.8.25).—See F.P. 602,475; B., 1926, 632.

IX.—BUILDING MATERIALS.

Action of sodium and magnesium sulphates on Portland cement. G. R. SHELTON (Ind. Eng. Chem., 1926, 18, 854—856).—A suspension of hydrated commercial Portland cement in water contained particles of gel, hexagonal plates of hydrated tricalcium aluminate, crystals of hydrated lime, and sulphoaluminate crystals. On treatment with sodium and magnesium sulphate solutions, the crystals of lime and tricalcium aluminate rapidly disappeared, leaving amorphous masses of gel which subsequently became granular. The sulphoaluminate crystals remained unaltered and were probably formed from the gypsum present in the cement. A white Portland cement containing tricalcium aluminate, tricalcium silicate, and β -dicalcium silicate yielded different results, no sulphoaluminate being found while the crystalline grains in the suspension persisted considerably longer. In both cases the disintegration was more rapid than with pure hydrated tricalcium aluminate (cf. B., 1926, 91), probably owing to the presence of silicates. The action of sulphate solutions on cement clinker was also determined.

B. W. CLARKE.

Action of sulphate water on concrete. D. G. MILLS (Public Roads, 1925, 6, 174—179, 183; Chem. Abstr., 1926, 20, 2056).—Cylinders of high-alumina cement and of standard cement cured in steam at 100° were unchanged, whereas cylinders of standard cement cured in water vapour at lower temperatures deteriorated, after immersion for 1 year in a lake water containing 2.34—4.72% of salts, chiefly magnesium and sodium sulphates.

A. A. ELDRIDGE.

Effect of calcium chloride on strength of concrete. A. S. LEVENS (Eng. News-Rec., 1926, 97, 214—215).—The addition of 2% of calcium chloride slightly increases the strength of concrete, higher percentages resulting in a decrease of strength. The texture of the concrete is also improved, but the shrinkage is increased considerably, which renders the use of calcium chloride dangerous in concrete structures. The addition of calcium chloride, however, enables a high strength to be developed when curing in dry air.

B. W. CLARKE.

Production from raw clay of material stable towards water. P. P. BUDNIKOV (Kolloid-Z., 1926, 39, 269—275).—With the object of producing a cheap building material experiments have been carried out on the stabilising action towards water of various substances on raw clay. The addition of alkalis up to about 5% increases the susceptibility to attack by water, larger additions make the clay immune, whilst with still larger quantities the clay is again attacked by water. Phosphoric acid (3—6%), free or in the form of a mixture of tricalcium phosphate, superphosphate, or phosphorite and sulphuric acid, renders the clay quite resistant to attack. It also reduces the shrinkage on drying, particularly if 10% of asbestos be added. It has no marked effect on the tensile strength except when added as calcium phosphate and sulphuric acid, when it increases it by 50%. Unfortunately, the action of phosphoric acid depends on the composition of the clay; e.g., in a series of clays having a silica content ranging from 5.5 to

42.5%, only those with 27.0% and under were stabilised completely by the above amount, viz., 3—6%. Experiments designed to explain the action of phosphoric acid were inconclusive. It probably depends on the colloidal nature of the clay and the coagulative action of the acid.

N. H. HARTSHORNE.

Portland cement industry [during the past fifty years]. R. K. MEADE (Ind. Eng. Chem., 1926, 18, 910—913).

PATENTS.

Burning material [cement] in rotary kilns. M. VOGEL-JORGENSEN (E.P. 255,569, 4.5.25).—Slurry, e.g., cement slurry, is dried in the kiln in the usual way to form nodules, which are ground before reaching the decarbonating zone by means of grinding members in the interior of the kiln or in a grinding chamber communicating with the kiln and arranged to prevent the leakage of air into the kiln. The grinding process facilitates the expulsion of carbon dioxide from the material and increases the capacity of the kiln.

B. W. CLARKE.

Improving the properties of articles formed of fibrous materials with cement binding agents. W. H. KOBBE, Assr. to TEXAS GULF SULPHUR Co. (U.S.P. 1,594,417, 3.8.26. Appl., 22.5.24).—The articles, e.g., asbestos-cement products, are impregnated wholly or in part with sulphur. (Cf. B., 1926, 668.)

H. ROYAL-DAWSON.

Cement manufacture. E. G. WEEKS, Assr. to MERZ & McLELLAN (U.S.P. 1,596,956, 24.8.26. Appl., 7.2.22. Conv., 17.3.21).—See E.P. 181,811; B., 1922, 635 A.

Cold bituminous paving composition. O. H. BERGER (E.P. 257,114, 28.9.25).

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

Elimination of sulphur from spathic iron ore by roasting. J. RUHRMANN (Stahl u. Eisen, 1926, 46, 1118—1119).—In order to test on a large scale the results obtained by Weyel (cf. B., 1925, 762) on a laboratory scale several tons of a spathic iron ore were roasted in a cylindrical Siegerland furnace and the sulphur content of the product determined at different heights in the furnace. In the upper three-fifths of the furnace 31% of the total sulphur was removed and in the lower two-fifths a further 21%. Passing steam through the furnace did not have any appreciable effect, but quenching the red hot charge in water reduced the sulphur content by 24—34%. Prolonged leaching of the roasted ore with water removed 60% of the sulphur content, but only a small fraction of the manganese, so that it seems that the greater part of the sulphur remaining after roasting is in the form of ferrous sulphate.

A. R. POWELL.

Factors influencing the rate of pickling of sheet iron. J. E. HANSEN and G. S. LINDSEY (J. Amer. Ceram. Soc., 1926, 9, 481—492).—A freshly made sulphuric acid (6%) pickling bath was found to clean iron more quickly than one in which ferrous sulphate was

present, and there was no evidence that the addition of a portion of old pickling solution improved the rate, contrary to general practical experience. Ferric sulphate accelerated the rate of pickling, but was converted quickly into the ferrous salt with a retarding effect. Alteration in concentration of acid influenced the speed of reaction, but not to the same degree as variation in temperature of the solution, and the addition of hydrochloric acid or sodium chloride to the solution retarded the process. Contrary to theoretical views, increased concentration of ferrous chloride in a 11.45% hydrochloric acid bath at 30° accelerated pickling. Annealed iron lost 250 to 400% more in weight during the early stages of treatment than unannealed iron, and the process was not so easily controlled. Contact of iron with a containing basket of Monel metal increases the pickling rate by reason of the electrochemical effect.

C. A. KING.

Is the direct change from austenite to troostite possible? K. HONDA (Iron and Steel Inst., Aug., 1926. Advance proof, 4 pp.).—From a consideration of the crystal structure of austenite and martensite and the mechanism of the A₁ transformation it is concluded that troostite is invariably the decomposition product of martensite, and not a product formed by direct decomposition of austenite. The direct change from austenite to troostite is not possible. The carbon in solution in austenite and martensite is present as carbon atoms, and not as cementite molecules.

M. COOK.

Spheroidising of cementite. B. STOUGHTON and R. D. BILLINGER (Ind. Eng. Chem., 1926, 18, 785—788).—The A₁ points of six specimens of steel, viz., file steel (hypereutectoid) carbon 1.4%, eutectoid carbon 0.80%, and hypereutectoid, carbon 0.45%, were determined, and the steels heated for varying periods in the neighbourhood of these points, both below and above. Brinell hardness tests taken before and after treatment showed a considerable softening in all cases (even below the A₁ point). Spheroidisation took place on each occasion, and may be accomplished within a temperature range extending from about 30° below the critical point to 70° above this temperature. Contrary to Honda and Saito (B., 1921, 12 A), spheroidisation of lamellar pearlite can take place below the A₁ point. The micrographic results appear to indicate that there is actual spheroidisation of pearlitic cementite and not simply a breaking up of grain boundaries. Spheroidisation is associated with the softest and weakest, but most ductile state of a steel.

A. COULTHARD.

Carburisation and decarburisation of iron, and surface decarburisation of steel. A. JOHANSSON and R. VON SETH (Iron and Steel Inst., Aug., 1926. Advance proof, 58 pp.).—The equilibria of the reaction $3\text{Fe} + 2\text{CO} \rightleftharpoons \text{Fe}_3\text{C} + \text{CO}_2$ has been determined between 700° and 1100° by following analytically the variations in carbon content of thin milled cuttings of pure carbon steel which had been heated in a current of carbon dioxide and carbon monoxide of constant composition. Below A₁ (720°) a bivariant equilibrium was found with ferrite and cementite as solid phases. Above 720° two bivariant equilibria occurred, given by the lines ferrite-austenite

and cementite-austenite and between them an infinite number of equilibria for varying content of carbon in the austenite. At 700° the equilibria coincide closely with the experimental equilibria of Schenck (B., 1926, 633), but his extrapolated figures for higher temperatures were not corroborated by direct determination. Decarburisation of steel cuttings in a current of hydrogen was apparent at 600°, increasing rapidly above 650° to a maximum at 950°. The carbon content (0.58%) of a steel maintained at 1050° decreased to 0.35% in 8 hrs. 0.21% in 16 hrs., and 0.13% in 24 hrs. Manganese, nickel, tungsten, and chromium-nickel steels showed about the same tendency to decarburise as pure carbon steels, but chromium steels were more resistant and silicon steels considerably less. In pure nitrogen at 1050° no decarburisation was found other than that associated with gaseous and solid inclusions in the steel. The results of the surface decarburisation of steel between 650° and 1100° in a mixture of carbon dioxide and carbon monoxide, and also in air, are discussed.

C. A. KING.

Treatment of steel with ferro-carbon-titanium. G. F. COMSTOCK (Iron and Steel Inst., Aug. 1, 1926. Advance proof, 9 pp.).—An account of the practical use of ferro-carbon-titanium for deoxidising steel. The alloy as used contains about 17% Ti and 7.5% C, and although of lower deoxidising capacity than 50% ferro-silicon, the stronger affinity of titanium for oxygen enables it to be used as a final addition after silicon or other deoxidisers. In effervescing steels over-oxidation may be corrected, leaving a cleaner steel with less segregation than when aluminium is added. In this connexion the fluxing power of titanium oxide on the slag inclusions in steel is a marked advantage.

C. A. KING.

Initial temperature and mass effects in quenching [of steels]. H. J. FRENCH and O. Z. KLOPSCH (Trans. Amer. Soc. Steel Treat., 1926, 9, 33—74).—The rate of cooling at the centres of spheres, rounds, and plates of high-carbon and alloy steels was determined on quenching from 875° in water, 5% sodium hydroxide solution, oils, and air. The best single measure of the hardening power was found to be the cooling velocity at 720°.

T. H. BURNHAM.

Characteristics of cooling curves [for steel]. H. J. FRENCH and O. Z. KLOPSCH (Tech. Papers, U.S. Bur. Standards, 1926, 20, [313], 365—385).—From time-temperature cooling curves taken at the centre of variously shaped steel samples, quenched in different media, the following equation is derived: $T - x = y(W/S)^n$, where T is the cooling time of the centre, S the surface area of the specimen, W its volume, y a time constant, n a constant depending on the nature of the cooling medium, and x the lag factor (time required for the centre to cool through 2% of the cooling range). The last-named varies with the surface per unit volume.

A. R. POWELL.

Physical investigation into the cause of temper-brittleness [of steels]. J. H. ANDREW and H. A. DICKIE (Iron and Steel Inst., Aug., 1926. Advance proof, 38 pp.).—In steels susceptible to temper-brittleness, a moderate rate of cooling (2° or 3° per min.) from the

tempering temperature causes a considerable fall in specific volume and in hardness, accompanied by comparatively small variations in impact value. The magnitude of the physical change is proportional to the degree of brittleness which may be produced by very slow rates of cooling. In steels of high susceptibility to brittleness, such as nickel-chromium steels, the change in specific volume and hardness is very distinct, but in nickel and chromium steels which are not so susceptible, the change is small. In nickel steel the magnitude of the change increases with the phosphorus content. As an explanation of the phenomenon of temper-brittleness, it is suggested that at higher tempering temperatures, ferrite may dissolve appreciable amounts of carbide (either iron carbide or a double carbide of iron and chromium) which is retained in solid solution by quenching. On cooling slowly from the tempering temperature the carbide is deposited from solution at the grain boundaries to form a brittle network throughout the mass. Certain special elements such as nickel, manganese, chromium, and phosphorus seem to increase the solubility of carbide in ferrite at temperatures near the A₁ range and to cause it to separate again at lower temperatures on slow cooling, whereas small quantities of molybdenum eliminate brittleness by causing the retention of carbide in solid solution irrespective of the rate of cooling.

M. Cook.

Mechanical properties of four heat-treated spring steels. G. A. HAWKINS, D. HANSON, and (Miss) G. W. FORD (Iron and Steel Inst., Aug., 1926. Advance proof, 26 pp.).—Results of tests on 0.6% C, 0.8% C, silico-manganese and chrome-vanadium spring steels showed that the quenching temperature of 0.6% C steel was high, but that elongation and reduction of area were lower than for other steels of similar hardness. At a hardness (Brinell) figure of 450 the fatigue limit was as high as ± 47 tons per sq. in. as compared with ± 43.5 for chrome-vanadium, and ± 50 for water-quenched silico-manganese steels. Izod values were lower than for the alloy steels. The 0.8% C steel could not be hardened properly in oil even by quenching from 950°. Tempering at 550° reduced the hardness, tensile strength, and fatigue limit and increased ductility. A more uniform material was produced by quenching from 900° than from 830° though without marked differences in mechanical properties. When tempered above 400° the decrease in hardness of silico-manganese steel was more rapid than for the other steels, and water-quenching gave high values for the limit of proportionality for tempering temperatures of 500°, 550°, and 600°, the results being generally slightly superior to those obtained by oil-quenching. Except when tempered at 600°, chrome-vanadium steel possessed lower values of fatigue limits than silico-manganese steel treated similarly, but the Izod values were superior to those of all the other steels. Chrome-vanadium steel tempered at 600° (Brinell no. 400) combined a high Izod value and good reduction in area, with a fatigue limit of ± 42.5 tons per sq. in. C. A. KING.

Effect of phosphorus on the endurance limit of low-carbon steels. F. F. McINTOSH (Min. and Met., 1926, 7, 332—333).—Although phosphorus is regarded as an impurity in steel, and is generally considered to

exert a deleterious influence on the properties of the metal, a consideration of the results obtained by Stead, Campbell, and others shows that within limits, the strength and fatigue-resisting properties of steel improve as the phosphorus content increases, without causing a proportional loss in ductility. This has been confirmed by tensile, hardness, and fatigue tests on basic open-hearth steel, containing 0.10% C and phosphorus between 0.01 and 0.10%, which show increasing strength, hardness, and ability to withstand fatigue in direct proportion to the phosphorus content. For each increment of 0.01% P the strength increases by 850—1000 lb. per sq. in., and the endurance limit increases 820 lb. per sq. in. Phosphorus up to 0.085% in steels containing 0.09—0.11% C does not manifest itself in any peculiarity of microstructure nor do the carbonising qualities of the steels show any effect traceable to phosphorus.

M. Cook.

Testing of hardened steel. A. LUNDGREN (Iron and Steel Inst., Aug., 1926. Advance proof, 37 pp.).—Methods of testing the mechanical properties of hardened tool steel which are more rapid than practical cutting tests, include a form of bending test, from which the limit of elasticity, the limit of proportionality, and the ultimate strength are derived, the Charpy impact test, and the Rockwell hardness test. Carbon steels containing 1.3, 1.0, 0.9, and 0.6% C have been used in the investigation. The two steels containing 1.3 and 1.0% C were subjected, prior to hardening, to various annealing treatments to obtain various structural forms which have been correlated with mechanical properties, determined after hardening and tempering. The effect of quenching temperature and tempering temperature is considered in detail. With very rapid cooling in benzene after tempering at 300° a much lower bending strength is obtained than by cooling in air after tempering at the same temperature.

M. Cook.

Electrochemical potentials of carbon and chromium steels. C. BENEDICKS and R. SUNDBERG (Iron and Steel Inst., Aug., 1926. Advance proof, 39 pp.).—The electrochemical potentials of some carbon and chromium steels have been determined in neutral oxygen-free 0.82*N*-ferrous sulphate solution (potential = E_H) and in the same solution oxidised with hydrogen peroxide (potential = E_0). The value of E_H was found to be higher (more negative) than that of E_0 . For pure iron in *N*-ferrous sulphate solution $E_H = -0.708$ volt, and $E_0 = -0.62$ volt. Increasing carbon up to 0.9% lowered the value of E_H , whilst E_0 was increased. For higher carbon contents, the effects were reversed. Quenching lowered the E_H values, but raised the E_0 values up to 0.9% C. The difference between E_H and E_0 tends to vanish for high carbon contents. Differential aeration will therefore only have a slight effect on the corrosion of a hardened high-carbon steel. E_H rises with the addition of chromium up to 8%, and then falls showing a marked minimum at 13—14% Cr, in unquenched specimens, but rises steadily in quenched specimens. The value of E_H in quenched steels containing 13% Cr is much lower with a high carbon content. In unquenched specimens E_0 is unchanged by the addition of chromium, but may be lowered to +0.31 volt

in quenched specimens. Stainless steel immersed in ferrous sulphate was found to be light-sensitive, and it is suggested that light influences the rusting of iron.

C. J. SMITHELLS.

Anomalies in heat conduction as investigated in spherical steel specimens, with some determinations of thermal (and electrical) conductivity in iron and carbon steels. C. BENEDICKS, H. BÄCKSTRÖM, and P. SEDERHOLM (Iron and Steel Inst., Aug., 1926. Advance proof, 46 pp.).—Measurements of the temperature gradient in different radial directions in hollow spherical specimens of steel showed variations in the ratio of 1 to 8.5 in some cases. The existence of these variations in thermal conductivity was confirmed by thermoscopic observations using a mercury copper iodide indicator, and by direct measurements of thermal distribution. Similar irregularities were found in electrical resistance, though on a much smaller scale, the ratio not exceeding 1 to 1.05. From this it is deduced that the accepted proportionality between thermal and electrical conductivities does not always exist. The irregularities are explained by assuming that thermal conductivity depends on two phenomena, (i) purely thermal, (ii) thermo-electric. The theory previously suggested by Benedicks (Ann. Physik, 1918, [iv], 55, 1—80) is developed. A method for the determination of the thermal conductivity (λ) is described. A close relation between thermal and electrical resistivities was found for some carbon steels, but not that corresponding with the Wiedemann-Franz law. The thermal resistivity of carbon steel is given by the equation

$$1/\lambda = 4.4 + 8.7 \Sigma C,$$

where ΣC is the carbon value in weight per cent. of added elements. This gives for pure iron the value $\lambda = 0.227$ cal. per cm. sec. deg. The elements are placed in the following order with respect to their effect (increasing) in lowering thermal conductivity in steel: nickel, manganese, hardening carbon, aluminium, silicon. Cementite carbon exerts only a slight influence.

C. J. SMITHELLS.

Chemical composition of tool steels. J. P. GILL and M. A. FROST (Trans. Amer. Soc. Steel Treat., 1926, 9, 75—98).—The analyses of seventy-five tool steels are critically considered. In carbon tool steel the sulphur and phosphorus should not exceed 0.035%. Non-deformable oil-hardening tool steels are improved by the addition of 0.5% of chromium and tungsten. For finishing cuts steels containing over 1% of carbon and 0.5% of tungsten are recommended. Uniform hardening is assisted by the addition of 0.5% of chromium. For hot-working dies semi-high-speed steels are satisfactory, but should not be hardened in water. Cold-drawing dies are generally made of steel containing 1.5—2.5% C and 11—15% Cr. The effect of additions of molybdenum, uranium, and cobalt to standard high-speed steels (18% W, 4% Cr, 1% V) are discussed and also the rôle of the principal impurities. The authors consider that efforts to standardise tool steel specifications would be detrimental.

T. H. BURNHAM.

Cutting power of high-speed steel tools and methods of testing. F. RAPATZ (Stahl u. Eisen, 1926, 49, 1109—1116).—The cutting power of tungsten steels increases with the hardening temperature, slowly between 1000° and 1170° and then rapidly to a maximum at about 1300°. The best cutting properties are obtained when the microstructure consists entirely of evenly oriented, fairly large polyhedral grains containing regularly distributed, small inclusions of the ledeburite eutectic, *i.e.*, when the maximum amount of carbide possible is retained in solid solution. With plain 14—18% W steels this structure is obtained by heating for 10 min. at 1250° or for 1—3 min. at 1300°. The longer period of heating at 1250°, although it gives slightly the more satisfactory structure, has the disadvantage that it is difficult to prevent serious oxidation taking place; on the other hand, care must be taken not to exceed 3 min. at 1300° or to allow the temperature to rise more than 20° above this temperature, otherwise the metal will be ruined owing to fusion and redistribution of the eutectic accompanied by abnormal growth of the polyhedral grains. Alloys containing cobalt or vanadium in addition to tungsten may safely be heated to a higher temperature as these elements appear to raise the m.p. of the eutectic. All high-speed tool steels are rendered harder and tougher by subsequent tempering at 580°, whereby the austenite is converted into martensite and internal strains are relieved. As the tensile strength of the alloy increases its life as a cutting tool is reduced very rapidly; an increase in the speed of cutting also reduces the life of the tool but the reduction is not proportional to the cutting speed. The composition of a steel is no indication of its behaviour as a cutting tool; the controlling factors in determining the life of a tool are hardness combined with toughness and suitable microstructure, as well as homogeneity and freedom from slag inclusions.

A. R. POWELL.

Constitution of iron-silicon alloys. G. PFRAGMÉN (Iron and Steel Inst., Aug., 1926. Advance proof, 8 pp.; cf. B., 1925, 285).—X-Ray and microscopical examinations show that three intermediary phases occur in the system; two of them which are of variable composition approximately correspond in composition to the compounds FeSi and FeSi₂. These two phases give rise to three eutectics, namely FeSi- α -phase, FeSi-FeSi₂, and FeSi₂-Si, which contain about 21.2, 48.2, and 61% Si respectively. The hypothesis of Oberhoffer that in the presence of sufficient silicon (more than 3.5% is found to be necessary) the α -range in iron disappears and the α - and δ -ranges unite, has been confirmed. The view of Murakami that the third phase corresponds in composition to the formula Fe₃Si₂ and is formed at 1000° by reaction in the solid state is also confirmed.

M. COOK.

Alloys of iron and chromium. E. C. BAIN (Trans. Amer. Soc. Steel Treat., 1926, 9, 9—32).—Two series of alloys of iron with chromium up to 35% and containing 0.1—0.26% and 0.26—0.50% of carbon respectively were produced from Armco iron and low-carbon ferrochrome, and subjected to metallographic examination after quenching from seven different temperatures between 900° and 1450°. Quenched taper-heated bars

were also used as corroborative evidence of structural changes effected by temperature. The first group of alloys, containing 3—14% Cr, was martensitic if quenched from above 900°, but at the top end of the range, e.g. above 12% Cr, with 0.35% C, δ -iron solid solution was preserved unchanged at room temperature on quenching from 1420°. The α - γ transition point was raised and the γ - δ change lowered with increasing chromium content. In group II with 14—25% Cr γ -iron is not formed on heating except as influenced by the carbon content, the effect of which is marked. High-carbon alloys of the second group were martensitic on quenching from 950—1000°, more austenite being retained and the δ -iron transformation raised to 1350—1400°. Alloys in group III, containing 25—35% Cr, showed no transformation unless the carbon was high, the δ -iron and α -iron solid solutions being continuous. Charts are given showing the temperature of development of the various constituents as affected by the chromium and carbon contents, also the hardness of the high-carbon series in relation to the quenching temperature. Up to 0.5% of carbon was soluble in δ -iron, but on reheating the excess carbon was precipitated within the grains. The austenite in the low-carbon chromium alloys required a high temperature for decomposition but was not resistant to cold work.

T. H. BURNHAM.

Effect of nitrogen on chromium and some iron-chromium alloys. F. ADCOCK (Iron and Steel Inst., Aug., 1926. Advance proof, 10 pp.).—Nitrogen was passed over the surface of molten pure iron, chromium, and iron-chromium alloys in a high-frequency induction furnace, and the metals were examined in respect of microstructure and hardness. Nitrogen was absorbed rapidly by chromium up to the extent of 3.9%, but only a small quantity of nitrogen (0.02%) was retained by iron. With alloys of iron and chromium the amount of nitrogen retained increased with the chromium content. In alloys containing about 12% Cr the presence of nitrogen produces a martensitic structure resembling that of iron-carbon alloys, and the hardness may be modified by heat treatment from 115 to 315 Brinell. With higher proportions of chromium (20—60%) alloys containing nitrogen usually present a two-phase structure, one constituent being of sorbitic or pearlitic type, absent in the alloy free from nitrogen, or in a similar alloy containing 0.05% C. Penetration of nitrogen in these alloys was especially rapid in the vicinity of crystal grain boundaries.

C. A. KING.

Thermal methods for assaying gold and platinum. J. A. PINTO (Rev. Fac. Cien. Quim., 1926, 4, 95—140).—A critical review of methods for assaying gold and platinum. A detailed account is given of the methods used by the author, for which the original paper should be consulted.

G. W. ROBINSON.

Action of hydrogen on hot solid copper. C. S. SMITH and C. R. HAYWARD (Inst. Metals, Sept., 1926. Advance copy, 20 pp.).—The brittle state assumed by copper when it is heated in hydrogen (gassed), and which is considered to be due to steam formed from the hydrogen and the oxygen of the cuprous oxide, has been studied in the case of cast copper. Lengths of copper

wire, 4 in. by 0.073 in. diam., containing 0.03 to 0.04% of oxygen, were heated in hydrogen at various temperatures up to 1050° in an electric furnace. The products showed severe embrittlement at 700—800° followed by a marked recovery in strength and ductility at higher temperatures. The recovery is considered to be due to a sintering at higher temperatures which causes the cracks or voids formed during the gassing to close up. To avoid the passing of copper into this brittle state it should not be annealed above 400° in a reducing atmosphere (heating for 30 min. at 550° is sufficient to make the metal quite brittle). The penetration of hydrogen into cast copper was studied on cylinders of the metal, 1.25 in. in diam. and 1.75 in. long. After heating and cooling the expansion was measured and the depth of penetration found from an etched section. The value of $100e/2p$, where e = total expansion and p = the penetration (both in inches), is a measure of the loss of density and is termed the "per cent. linear unsoundness." For copper of low oxygen content (0.03%) the penetration rises rapidly and more or less uniformly with the temperature, whilst the "unsoundness" reaches a maximum about 800° and does not vary above 850°. For metal, however, of high oxygen content (above 0.07%) the penetration is abnormal, rises to a maximum at about 800°, and then rapidly falls off. This maximum is less pronounced as the quantity of oxygen decreases. It is suggested that the sintering has closed up the voids and that further penetration can only take place by ordinary diffusion. That it only takes place with high oxygen content is due to the larger amounts of steam evolved, which strain the metal and induce recrystallisation which always precedes sintering. Photomicrographs are given which support this theory. Copper which has been forged after casting does not show abnormal penetration values. The time and penetration curve for low-oxygen copper is almost a straight line, that for high-oxygen copper approaches a parabola. Gassed copper, which is usually considered incapable of restoration unless re-melted, can be converted into metal of good mechanical and electrical properties by rolling to about half the thickness of the gassed material. The ductility is far in excess of that of the original copper, whilst the electrical conductivity of wire of 0.08 in. (after deoxidation and annealing) was 101.1% on the Matthiessen scale. This treatment could be applied to copper bars before rolling into rods. A. COULTHARD.

Season-cracking in arsenical copper tubes. A. PINKERTON and W. H. TAIT (Inst. Metals, Sept., 1926. Advance copy, 6 pp.).—Hollow sunk arsenical copper tubes are liable to season-cracking when reductions of sectional area exceed 21%, but sufficient data have not been obtained to fix definitely a safe working limit. Annealing at 240° renders the tubes immune from cracking in mercurous nitrate without affecting the hardness. Arsenic-free copper tubes having average internal stresses of the same order as arsenical copper tubes are not liable to season-cracking. M. COOK.

Direct production of brass from mixed ores. A. W. GUERTLER (Metall. u. Erz, 1926, 23, 325; Chem. Zentr., 1926, II., 824).—By melting sulphide ores of

lead and zinc with metallic copper in excess, copper matte and a mixture of metallic lead, copper, and zinc are formed, which separate, in the liquid state, into a layer rich in lead with little zinc, lead, and a copper-zinc layer with little lead. The last-named is melted with copper and yields a serviceable brass.

C. T. GIMMINGHAM.

Working of brass at high temperatures. K. HANSER (*Z. Metallk.*, 1926, 18, 247—255).—Diagrams have been constructed on the equilibrium diagram of copper-zinc alloys containing more than 50% Cu showing lines of equal compressibility (under static and dynamic pressure), hardness, tensile strength, elongation, and reduction of area. The reduction in height of β -brass under a dynamic blow remains fairly constant up to 500°, then increases rapidly with rise of temperature, whereas with α -brass a slow but steady increase takes place throughout the temperature range. Under the conditions of the ordinary compressibility test the maximum brittleness is shown by alloys containing 8—20% Zn between 200° and 500°, but no sign of fracture occurs until the height has been reduced by 50—55%. The hardness curves are similar to those of compressibility, but the impact test figures are lowest between 300° and 500° for all alloys with more than 3% Zn. The tensile strength of all the brasses falls fairly regularly with rising temperature, but the reduction in area of α -brass decreases to a minimum between 300° and 500° according to the composition, then increases to its original value above 700°, whereas the greatest reduction in area with β -brass occurs just above 500° and the minimum at the ordinary temperature. These results show that the reduction in area is the best indication of the most suitable temperature for working brass. Further tests on brass containing 67% and 60% Cu show that, when rolled above 500°, the metal is stronger the more rapid the passage through the rolls.

A. R. POWELL.

Copper-magnesium alloys. W. T. COOK and W. R. D. JONES (*Inst. Metals*, Sept., 1926. Advance copy, 14 pp.).—Alloys of magnesium and copper containing from a trace to 10% Cu have been prepared by melting the metals in bottom-pouring forged-steel crucibles with a flux of magnesium chloride and fluoride (3—4% of the weight of the metal) and casting into $\frac{3}{4}$ to 1 $\frac{1}{8}$ in. (diam.) moulds, under conditions readily reproducible in workshop practice. Gas-holes of $\frac{1}{16}$ — $\frac{1}{8}$ in. in diam. which appeared on the first casting were eliminated by allowing the metal to cool slowly below the solidification temperature, then immediately re-melting and re-casting (cf. Archbutt, B., 1925, 286). The melting losses did not exceed 0.75%. Tables and graphs are given showing the tensile strengths of the chill-castings as determined on a Riehlé multiple-lever testing machine. The alloys have a comparatively low tensile strength, little ductility, and a low limit of proportionality. The addition of copper up to 2 or 3% to magnesium is beneficial. Brinell hardness tests carried out on an Amsler machine show the hardness to increase with the amount of added copper. The hardness does not appear to increase on ageing. The alloys have a low impact value. Etching with an alcoholic solution of

1% nitric and 0.5% hydrochloric acids showed the alloys to consist of grains of almost pure magnesium in a eutectic of the composition, Mg₃Cu, which had a tendency to become globular. The macrostructure developed by a 5% alcoholic solution of nitric acid shows a refinement of the grain due to copper.

A. COULTHARD.

Influence of the thermal and mechanical treatment of aluminium on its resistance to corrosion. W. WIEDERHOLT (*Korrosion u. Metallschutz*, 1926, 2, 126—133; *Chem. Zentr.*, 1926, II., 938—939).—The velocity of dissolution of aluminium sheet in acid increases with rise of annealing temperature to a maximum at 300°, at which temperature the maximum separation of impurities from solid solution in the aluminium occurs, with the consequent formation of the greatest number of local elements. Above 300° the impurities, especially silicon, diffuse into the metal to form a solid solution, and the corrosion rate decreases proportionately. Below 300° the impurities are thrown out of the solid solution at a rate proportional to the time of annealing, hence the rate of corrosion of metal annealed below 300° is also proportional to the time of annealing. The resistance to corrosion is increased by annealing in nitrogen, and to a greater extent by annealing in oxygen. The coarser the crystalline structure after annealing, the higher is the resistance to corrosion, so that the degree of deformation to which the metal has been subjected by cold-work has a considerable influence on the resistance of the subsequently annealed metal to corrosion. Reductions of 2—15% have practically the same effect in this respect, but with a greater reduction the corrodibility increases with the degree of reduction.

A. R. POWELL.

Constitution and structure of commercial aluminium-silicon alloys. A. G. C. GWYER and H. W. L. PHILLIPS. **Properties of modified aluminium-silicon alloys.** D. STOCKDALE and I. WILKINSON (*Inst. Metals*, Sept., 1926. Advance copy, 43 pp.).—The composition of the normal silicon-aluminium eutectic is confirmed to be 11.7% Si and 88.3% Al, and its m.p. is 577°. After addition of a modifying agent the all-eutectic alloy contains 12.85% Si and solidifies at a lower temperature, varying according to the modifying treatment and the rate of cooling, but it always melts at 577°. The higher the proportion of modifying agent added the lower is the f.p. of the alloy, and the more rapid the cooling the finer is the crystal structure. These facts are explained by assuming that the sodium obtained from the modifying agent acts as a colloid and hinders the separation of crystals from the melt, thereby inducing supercooling. The supercooled alloy then solidifies rapidly in the presence of a colloid, which acts as a protective colloid and retains the aluminium and silicon in the colloidal form in which they first separate from the melt. In substantiation of this theory, it is shown that addition of sodium to aluminium-copper and manganese-aluminium alloys and of aluminium to lead-antimony and antimony-copper alloys also results in a refinement of the structure. The converse of this effect is produced by adding sodium hydroxide to iron-aluminium alloys at 900°, acceleration of the separation

of FeAl_3 being induced, followed by the coagulation of this constituent into large crystals. In a similar manner, addition of sodium chloride to the remelted modified silicon-aluminium alloys causes rapid reversion to the normal form, probably owing to its effect in coagulating the colloid. Pronounced colonisation occurs along the grain boundaries of silicon-aluminium alloys modified with excess of sodium hydroxide; the same effect is produced by addition of copper or zinc to the alloys. The silicon-aluminium-iron system contains a ternary eutectic, f.p. 577° , containing 11.6% Si and 0.8% Fe, the iron occurring as the unknown X constituent. Alloys containing more than 11.6% Si also show the presence of a new iron constituent, δ , which appears to be a ternary substance and is converted into X on annealing. D. STOCKDALE and I. WILKINSON, in an appendix (12 pp.), give the results of various mechanical tests on modified silicon-aluminium alloys. Their results in general confirm those of Grogan (*infra*). The alloy containing 11% Si and the minimum amount of iron seems to have the best all-round combination of properties and the smallest tendency to reversion.

A. R. POWELL.

Mechanical properties of silicon-aluminium alloys. J. D. GROGAN (Inst. Metals, Sept., 1926. Advance copy, 13 pp.).—Silicon-aluminium alloys modified by the salts method usually have better mechanical properties than those modified by addition of metallic sodium, owing to their greater soundness. The amount of sodium retained by the modified alloys varies from 0.002 to 0.008%. The density of the alloys decreases linearly with the silicon content from 2.68 for the 8% Si alloy to 2.65 for the 14% alloy; the stress required to produce a permanent deformation of 0.5% increases regularly from 5–6 tons per sq. in. for the 8% alloy to 7.8 tons for the 14% alloy chill-cast, and from 4.8 tons to 6.5 tons respectively for the corresponding sand-cast alloys, and the ultimate stress increases from 12.1 tons per sq. in. for the 8% chill-cast alloy to 13.4 tons for the 12% alloy, a further 2% Si making little difference. The corresponding ultimate stress figures for the sand-cast alloys are 10.3 and 11.6 tons per sq. in. respectively. The elongation falls rapidly from 19% for the 8% Si alloy chill-cast and from 16.5% for the same alloy sand-cast to 11 and 9.5% respectively for the 12% alloy, further silicon up to 14% making no difference. The Charpy impact values fall with increase of silicon from 8 to 13.5% from 1.34 ft.-lb. to 0.56 ft.-lb. for chill-cast and from 0.80 ft.-lb. to 0.44 ft.-lb. for sand-cast alloys. The fatigue range of the alloy containing 8.5% Si and 0.3% Fe is ± 4.2 tons per sq. in. The hardness increases linearly with the silicon content from 8 to 14% from 50 to 65 for chill-cast and from 44 to 57 for sand-cast alloys. Addition of zinc increases the hardness and tensile strength, but reduces considerably the ductility, whereas magnesium does not affect the tensile strength but ruins the ductility.

A. R. POWELL.

Silumin and its structure. B. ÔTANI (Inst. Metals, Sept., 1926. Advance copy, 25 pp.).—The solubility of silicon in solid aluminium, as determined by chemical methods and by electrical conductivity measurements, is 1.47% at 550° and 0.43% at 360° . The most satis-

factory modified structure using metallic sodium results from the addition of 0.1% Na at $720\text{--}750^\circ$; using "salts," the best results are obtained with 1% of a mixture of 40% of sodium fluoride and 60% of potassium fluoride at 750° , and by 0.5–0.8% of sodium hydroxide at $700\text{--}800^\circ$. A structure resembling the modified structure may be obtained by quenching the alloy immediately after solidification, so that the exterior layers of chill-cast normal alloys often exhibit a fine-grained eutectiferous structure. The action of sodium in modifying the structure of silicon-aluminium alloys is discussed at some length and evidence is adduced to show that none of the existing theories fits in with all the known facts. An explanation is suggested, based on the fact that a second liquid consisting almost wholly of metallic sodium exists in the melt during its passage through the solidification range; this liquid separates out from its solution in the aluminium just as the aluminium and silicon crystals begin to form, and thereby hinders their growth and causes the formation of many more nuclei. Bismuth and cadmium bring about only a slight modifying effect, although they also form no solid solutions with aluminium; they are, however, less soluble than sodium in the molten alloy, and have relatively high densities. Hence their restraining influence on crystallisation is slight.

A. R. POWELL.

Significance of silicon in the ageing of aluminium alloys containing lithium or magnesium. P. ASSMANN (Z. Metallk., 1926, 18, 256–260).—The maximum hardness of alloys of lithium or magnesium with commercial aluminium, after quenching from above 500° and ageing at 18° , is obtained when the silicon impurity of the aluminium is just sufficient to form the silicides Li_3Si or Mg_2Si ; with more or less silicon than this the hardness of the aged alloy decreases proportionately to the excess or deficiency. With very pure aluminium neither lithium or magnesium produces ageing phenomena, thus indicating that the ageing properties of these alloys are due to the solubility of the silicide at 18° being less than that at 500° . Whilst the hardening effect of magnesium silicide is not affected by ageing at 100° , but the rate of hardening is increased, a similar treatment of alloys containing lithium silicide results in a softer alloy than is obtained by ageing at 18° . Annealing at 200° in each case destroys the effect of ageing. Copper up to 4% and zinc up to 12% increase the hardness of the lithium alloys aged at 18° , but lithium reduces the hardness of copper-aluminium or zinc-aluminium alloys aged at 100° . The maximum hardness at 18° of the lithium alloys is obtained with 1.6% Li_3Si , and of the 4% Cu or 12% Zn alloys with 0.67% Li. Thus it appears that copper and zinc depress the solubility of Li_3Si in aluminium at 500° , and that Li_3Si tends to repress the "artificial" ageing effect of copper and zinc.

A. R. POWELL.

Constitution and physical properties of alloys of cadmium and zinc. C. H. U. JENKINS (Inst. Metals, Sept., 1926. Advance copy, 35 pp.).—The physical properties of alloys of zinc and cadmium have been found to be more complex than would result from a simple eutectiferous series of these metals, and the

complexity has been traced to alterations in the alloys caused by the two polymorphic changes (especially the one about 350°) which appear to occur with zinc. The eutectic point is at 266° with Cd 82.6%. Although a considerable degree of metastability occurs in all the alloys no intermediate or solid solutions are formed, and no marked thermal arrests were observed except at the temperature of the liquidus and the eutectic point. The physical properties of the cadmium-rich, zinc-rich, and eutectic alloys have been examined, and tables of values are given for the tensile strength, Brinell hardness, and electrical conductivity of cast, annealed, and aged alloys. Somewhat in opposition to the views of Rigg and Morse (B., 1915, 1097) the properties of cast and rolled zinc are found to be improved by the addition of small amounts of cadmium, although some degree of hot-shortness occurs in alloys containing more than 3%. In the zinc-rich alloys a non-permanent hardening effect was observed which is followed by softening at room temperature over a period of months. The cadmium-rich alloys soften to minimum values much more rapidly. Cold-worked cadmium has been found to recrystallise completely at the temperature produced by cold working. The solid solubility of cadmium in zinc amounts to 1.75–2% at 250° , that of zinc in cadmium 2–2.25%, but this falls to less than 0.25% of cadmium and 0.75–1.0% of zinc at 60° . The presence of cadmium in zinc does not appear to accelerate corrosion due to rain-water, but rather the reverse. Zinc containing a small quantity of cadmium is superior for certain purposes, such as zinc-base die-casting and zinc sheeting. The eutectic alloy has a strength of about three times that of ordinary tin-lead solder, and possesses properties which should lead to its increased use as a medium hard solder. A. COULTHARD.

Bearing metals. R. T. ROLFE (J. Inst. Metals, 1926, 35, 439–440).—Addition of copper to tin-antimony alloys tends to prevent segregation; thus a 90% Sn, 10% Sb alloy segregates into two layers, whereas no segregation at all takes place in an alloy of 85% Sn, 8.5% Sb, and 6.5% Cu. The tensile strength of an Admiralty tin-base bearing metal increases with the casting temperature, and decreases with an increase in the temperature of the mould. Casting temperature, however, has no effect on the compressive load required to produce a permanent set, but a mould temperature of 200° gives better results than a higher or lower temperature; in practice it appears to be best to cast at 365° into moulds preheated to 200° . Micrographic examination of some specimens of this alloy showed a cuboid of SnSb split as though by a wedge by a needle of the hard SnCu_3 compound. A. R. POWELL.

Comparison of static and dynamic tensile and notched-bar tests. K. HONDA (Inst. Metals, Sept., 1926. Advance copy, 11 pp.).—With tension tests the energy absorbed on impact is generally greater than that absorbed in the static test. For example, in a series of steels containing 0.1–0.9% of carbon, the work of rupture was determined both dynamically and statically, and showed the following mean ratio: dyn./stat. = 1.37. In the case of bending tests it is confirmed

that the same amount of energy is required whether the test is dynamic or static. Deformation is considered the most important factor affecting the difference in the static and dynamic energies absorbed in the tension tests. A method is described by which the degree of fatigue at any given period during an impact test may be determined. A. COULTHARD.

Steel age—1876 to 1926. J. A. MATHEWS (Ind. Eng. Chem., 1926, 18, 913–914).

Fifty years' progress in aluminium. J. D. EDWARDS (Ind. Eng. Chem., 1926, 18, 922–924).

Metallurgy fifty years ago and now. W. M. CORSE (Ind. Eng. Chem., 1926, 18, 892–895).

Effect of zinc, zinc oxide, and zinc sulphide upon the health of workmen. BATCHELOR and others.—See XXIII.

PATENTS.

Refining of iron. G. S. EVANS (U.S.P. 1,590,731, 29.6.26. Appl., 9.5.25).—The addition of sodium carbonate which has been aggregated to a dense product by fusion and subsequent cooling to the charge of iron, coke, and limestone used in foundry cupolas, produces a strongly alkaline fluid slag which absorbs the greater portion of the sulphur present and forms a good coating on the molten iron. About 1–4 lb. of sodium carbonate are used per ton of metal treated. T. S. WHEELER.

Coating iron or steel articles [with tin-aluminium alloy]. I. J. KOEHLIN (U.S.P. 1,591,983, 13.7.26. Appl., 20.6.24).—Iron or steel articles are coated with an alloy of tin containing up to 20% of aluminium by dipping in a bath of the molten alloy. The alloy is harder, lighter, and cheaper than pure tin and protects the iron better against corrosion. T. S. WHEELER.

Alloy steel. R. T. WIRTH (U.S.P. 1,593,924, 27.7.26. Appl., 19.10.21).—An alloy steel contains from 2 to 30% of chromium, and not less than 10% of tungsten or molybdenum or both, the total of alloying elements exceeding 35%. M. COOK.

Treating ingot iron. W. TAFEL (U.S.P. 1,594,133, 27.7.26. Appl., 22.8.24. Conv., 16.6.23).—To produce iron or steel similar to wrought iron, a mixture or a slag containing ferrous oxide, ferric oxide, and silica is added to the molten iron in a furnace. C. A. KING.

Metallurgical furnace. F. G. BREYER, Assr. to NEW JERSEY ZINC Co. (U.S.P. 1,594,000, 27.7.26. Appl., 4.9.24).—The furnace comprises a heating chamber with a flue for conducting the metal vapour produced in the furnace to the atmosphere outside the furnace, and means for directing a blast of relatively cool gas against the stream of vapour issuing from the flue. A. R. POWELL.

Corrosion-resisting metal. C. E. JONES, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,594,061, 27.7.26. Appl., 19.6.22).—An article consisting largely of aluminium is electroplated with cadmium, to render it resistant to corrosion in a salt atmosphere. B. M. VENABLES.

Production of magnesium. H. E. BAKKEN, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,594,344, 3.8.26.

Appl., 1.11.22. Renewed 17.4.26).—Material containing metallic magnesium is heated under a pressure lower than the vapour pressure of magnesium at its m.p. to a temperature at which the metal sublimes readily.

A. R. POWELL.

Production of magnesium. H. E. BAKKEN and W. G. HARVEY, Assrs. to AMER. MAGNESIUM CORP. (U.S.P. 1,594,345, 3.8.26. Appl., 8.5.23).—Pure magnesium is obtained by subjecting material containing metallic magnesium to distillation under a pressure slightly above the vapour pressure of the metal at its m.p. and condensing the vapours evolved.

A. R. POWELL.

Magnesium product. Working magnesium. Sublimation apparatus [for magnesium]. H. E. BAKKEN, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,594,346—8. 3.8.26. Appl., [A], and [B], 1.11.22, [c], 10.2.25).—(A) A crystalline mass of metallic magnesium is obtained by sublimation. (B) This mass may be worked into a coherent product by subjecting it to pressure at a temperature just below its m.p. (C) Impure magnesium is refined by sublimation in a vertical cylindrical retort, the lower portion of which is heated in an electric resistance furnace while the upper part is cooled to allow the metal to condense on the sides. The pressure inside the retort is maintained just below the vapour pressure of the metal at its m.p.

A. R. POWELL.

Electrothermal reduction of alumina. F. C. FRARY, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,594,362, 3.8.26. Appl., 10.4.24).—In the process of producing aluminium-copper alloys by the electrothermal reduction of alumina over a bath of molten copper, the contents of the furnace are tested periodically to determine whether aluminium carbide is present. If such is the case alumina alone is added to the furnace charge so as to react with the excess of carbide.

A. R. POWELL.

Refining metals. J. L. MULLIGAN, Assr. to UNITED STATES SMELTING, REFINING AND MINING CO. (U.S.P. 1,594,424, 3.8.26. Appl., 19.6.24).—In the method of refining a molten metal by treating it with a fused alkali hydroxide, a thickening agent is added to the slag just previous to skimming.

A. R. POWELL.

Concentration of ores. J. L. STEVENS (U.S.P. 1,594,858, 3.8.26. Appl., 23.8.24; cf. U.S.P. 1,525,211, B., 1925, 300).—The reaction product of fusel oil, carbon disulphide, and alkali is added to the mineral pulp and the mixture subjected to flotation.

S. BINNING.

Production of single-crystal metal wires from liquid metal. W. FREY and E. VON GOMPERZ (G.P. 429,276, 23.11.21).—The bath of liquid metal is maintained at a temperature just above the m.p., and is cooled evenly at one spot to the m.p. A wire of the same metal is dipped into the liquid at this spot and slowly withdrawn, whereby a single-crystal wire is obtained.

A. R. POWELL.

Melting and refining of readily oxidisable metals (e.g., copper, lead, tin, zinc, or aluminium) and

their alloys. P. T. ARNEMANN (G.P. 429,666, 27.2.25).—Readily oxidisable metals are melted in a crucible by means of neutral or reducing gas flames directed on to the surface of the metal at such a pressure that they completely envelop the crucible and prevent access of air. For refining purposes slightly oxidising flames are used in the same way. High melting temperatures may be obtained by this method without any loss of valuable metal by oxidation.

A. R. POWELL.

Increasing the amount and phosphoric acid content of basic slag. H. HILBERT (G.P. 429,767, 23.9.25. Addn. to 418,102; cf. B., 1926, 162).—A mixture of calcium phosphate and calcium oxide is used in place of calcium phosphate alone to replace the limestone or dolomite added in the converter process. The formation of iron phosphate is prevented.

C. T. GIMINGHAM.

Treatment of complex ores and smelter products. F. KRUPP GRUSONWERK A.-G. (G.P. 429,938, 6.4.24).—Complex ores are subjected to a blast of air in a combustion chamber in such a way that the oxygen supplied to the zone in which decomposition of the sulphides takes place is sufficient only for the combustion of the sulphur, so that the easily volatilised metals are not oxidised, and are eliminated from the charge while it is still suspended in the blast.

A. R. POWELL.

Lead alloy, especially for cable covering. A. H. RUNDIUS (Nor. P. 40,552, 19.7.23).—A lead alloy contains tin, bismuth, and antimony or arsenic in addition to magnesium. The total amount of metal alloyed with the lead may be as high as 2%, but, preferably, should not be more than 0.5%.

C. T. GIMINGHAM.

Purification of iron. MATHIESON ALKALI WORKS, Asses. of G. S. EVANS (E.P. 250,522, 6.7.25. Conv., 10.4.25).—See U.S.P. 1,590,739; B., 1926, 753.

Complete elimination of zinc from fine zinciferous ores and the like, especially zinciferous purple ores or calcined residues. A. L. MOND. From METALLBANK & METALLURGISCHE GES. (E.P. 257,133, 17.11.25).—See G.P. 421,384; B., 1926, 412.

Coating articles particularly with metals [by the spray process]. M. U. SCHOOP (E.P. 236,175, 18.5.25. Conv., 30.6.24).

Briquette (U.S.P. 1,590,706).—See II.

Alloy for glass-melting container (U.S.P. 1,592,336).—See VIII.

Electric furnaces for bright annealing (E.P. 242,283).—See XI.

XI.—ELECTROTECHNICS.

Inside frosting of incandescence [electric] lamps. M. PIPKIN (Ind. Eng. Chem., 1926, 18, 774—776; cf. E.P. 228,907, B., 1925, 804).—Frosting of the inner surface of lamp bulbs was obtained by repeated internal spraying at 5-sec. intervals to a total of 60 secs. with a mixture of water, barium sulphate, sodium bisulphate, ammonium bifluoride, dextrin, and hydrofluoric acid

(11.9% by weight) at 50°, followed by a strengthening treatment consisting of a similar spraying with a mixture of water, barium sulphate, dextrin, and hydrofluoric acid (7.44% by weight). A suitable commercial frosting mixture was made from 48–60% hydrofluoric acid saturated with ammonium bifluoride with the addition of enough water and sodium carbonate to reduce the acidity to 25%, the bulb being covered and left for 25–50 secs. at 30–50°. Fine-grained etching was given by the mixture: ammonium bifluoride 42%, dextrin 7%, barium sulphate 20%, sodium bisulphate 3.5%, hydrofluoric acid 27.5%, water to give an acidity of 18–25% of hydrofluoric acid. Internally frosted bulbs had about the same diffusion as those externally frosted, but they absorbed less light. The absorption of light, in the case of daylight lamps, was greatly increased by internal frosting.

A. COUSEN.

Theoretical study of the yield of the high-frequency electric furnace with alternating current. G. RIBAUD (*J. Phys. Radium*, 1926, [vi], 7, 250–256).

A half-century of artificial [electric] lighting. M. LUCKIESH (*Ind. Eng. Chem.*, 1926, 18, 920–922).

Electrical resistance of refractory materials. FERGUSON.—See VIII.

Electro-chemical potentials of carbon and chromium steels. BENEDICKS and SUNDBERG.—See X.

Thermal and electrical conductivity in iron and carbon steels. BENEDICKS, BÄCKSTRÖM, and SEDERHOLM.—See X.

Determination of electrical conductivity of milk. GERBER.—See XIX.

PATENTS.

Gaseous electric conduction devices. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of E. E. CHARLTON (E.P. 237,235, 2.7.25. Conv., 17.7.24).—To improve the efficiency of electric discharge devices which operate by the ionisation of a gas, the voltage necessary to produce discharge between the electrodes may be reduced to a minimum by providing in the bulb, together with an inert gas, an oxide of an alkali metal preferably associated with a highly oxidisable metal such as an alkaline-earth metal. Methods of introducing the alkali and alkaline-earth metals are described. (Reference is directed, in pursuance of Sect. 7 (4) of the Patents and Designs Acts, 1907 and 1919, to E.P. 224,544).

M. E. NOTTAGE.

Oxide cathodes for discharge tubes. N. V. PHILIPS' GLOEILAMPENFABRIEKEN (E.P. 245,147, 23.12.25. Conv., 27.12.24).—A wire or other body of tungsten or a similar metal or alloy is at least partly oxidised at its surface and is surrounded by the vapour of an alkaline-earth metal to deposit alkaline-earth oxide on the surface. In one method of carrying out the process an easily decomposable alkaline-earth compound, such as barium azide, is introduced into the tube, and during the process of exhausting the tube is heated sufficiently to effect its decomposition and volatilise the metal. H. HOLMES.

Electric discharge devices. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of D. A. MULLANEY (E.P. 250,906, 13.1.26. Conv., 16.4.25).—In a device consisting of an evacuated envelope containing co-operating electrodes, the thoriated cathode of which is very sensitive to minute quantities of gas, a quantity of tantalum is arranged so as to be heated during operation. To this end it may constitute part of one of the electrodes, being preferably fixed to the central part of the anode or, in the case of a device containing a grid or controlling electrode, this may consist of tantalum. The tantalum exercises a cleaning-up action for gas during the entire life of the device. M. E. NOTTAGE.

Electron-emission material. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,591,717, 6.7.26. Appl., 28.9.22).—A refractory alkaline-earth oxide, such as barium oxide, is mixed with a metallic powder, e.g., tantalum or platinum, and coated on a suitable wire, e.g., platinum-iridium, by suspending the coating mixture in nitrocellulose dissolved in amyl acetate and removing the organic material by the action of heat from the coated wire. Alternatively, mixtures of tantalum and the oxide may be converted into a filament by any of the methods usually employed for the pure metal. The electron-emission material obtained retains its activity for a long period. T. S. WHEELER.

Preparation of rare metallic oxides [for electric lamps]. J. A. HEANY (U.S.P. 1,592,459, 13.7.26. Appl., 14.11.18).—Rare metal oxides, such as thoria or zirconia, when associated with the heating element of an electric-light bulb, liberate occluded oxygen which attacks the heating element. To prevent this, the purified oxide is moulded and then immersed in a bath of molten paraffin until the hydrocarbon has replaced the occluded gases in the pores of the material. It is then removed and placed in position with the heating element in the bulb, which is attached to the pump and exhausted with the electric current running. When the pump has removed all the hydrocarbon, the bulb is sealed off or filled as desired.

E. S. KREIS.

Electric incandescent device [lamp]. G. R. FONDA, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,594,057, 27.7.26. Appl., 30.8.24).—To prevent the volatilisation and crystal faulting of the tungsten filament in a gas-filled lamp, zirconium chloride is introduced, so that the filament operates in contact with zirconium chloride vapour at a pressure equal to its vapour pressure at the temperature of the lamp bulb. B. M. VENABLES.

Electric furnaces for bright annealing. SIEMENS-SCHUCKERTWERKE, Assees. of HERAEUS-VACUUM-SCHMELZE A.-G., and W. ROHN (E.P. 242,283, 28.10.25. Conv., 1.11.24).—The heating chamber of the furnace consists of a metal container enclosed in an air-tight furnace casing, the electric heater being contained in the space between them and the whole surrounded by a further heat-insulating layer. The heating current is controlled by the thermal expansion of the metal container. The protective gas passes separately or consecutively through the space between the metal container and the furnace casing and through the interior of the

metal container, and also through antechambers which are provided at one or both ends of the heating chamber.

M. E. NOTTAGE.

Electric furnace. J. J. NAUGLE (U.S.P. 1,593,879, 27.7.26. Appl., 21.4.23).—An electric furnace for treatment of carbonaceous material or the like consists of a container, along the inner periphery of which extends longitudinally a number of electrodes, means being provided for keeping the latter in motion so that they are intermittently immersed in the carbonaceous material.

M. E. NOTTAGE.

Regenerative cell of manganese dioxide, coal, and zinc [feeble current accumulator].—E. HEESE (E.P. 254,549, 21.8.25).—A regenerative cell comprises a positive element of pure zinc (as distinct from commercial zinc) and a depolarising mixture enclosed in an osmotic envelope, *e.g.*, of animal skin, fish bladder, etc., and a manganese dioxide-coal negative element. The electrolyte consists of zinc ammonium chlorate or zinc ammonium chloride, and on recharging the cell a surface of pure zinc is formed.

J. S. G. THOMAS.

Insulated electrical conductors and the like. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,599 and 1,590,608, 29.6.26. Appl., [A] 17.6.24, and [B] 18.3.26).—(A) The conductor is coated with cellulose by applying a coating of cellulose dissolved in zinc chloride or cuprammonia and removing the solvent constituents electrolytically. The apparatus consists of a tank, containing the cellulose solution, fitted with pipes for supplying more solution and maintaining a gas pressure. Through the side wall of this tank is fitted a nozzle and opposite to it, in the other wall, is a nozzle with a bore the size of the object to be coated. This projects into a second tank, containing dilute sulphuric acid, and having a tubular copper cathode. At the end of the cathode is a stuffing-box with a central perforation connecting it to a washing tank. The wire to be insulated passes through the two nozzles in between which it becomes coated with cellulose solution. In the second tank, while passing through the tubular cathode, it acts as the anode, thereby becoming coated with a film of cellulose. (B) The coating of cellulose on the conductor is transparent, homogeneous, and impermeable to oil.

R. B. CLARKE.

Electrolytic cell [for deposition of cellulose]. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,604, 29.6.26. Appl., 21.6.24).—A rotating hollow drum is mounted so that its porous periphery passes just above the copper base of a semi-cylindrical vessel. Between the copper base and the drum is a solution of cellulose in cuprammonia and inside the drum is a conducting solution. A small curved anode, of platinum if the conducting solution is dilute sulphuric acid, or of carbon if it is ammonium nitrate, is suspended from the centre of the drum; the copper base acts as the cathode of the cell. On passing a current cellulose is deposited on the outer surface of the drum, from which it is scraped and wound on to reels.

R. B. CLARKE.

Electric-battery construction. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,605, 29.6.26. Appl., 21.6.24).—The electrodes consist of

antimony-lead embedded in non-fibrous cellulose mixed with a metal oxide, *e.g.*, manganese dioxide, the whole being covered with a thin layer of permeable, colloidal cellulose. The cell is filled with dilute sulphuric acid and is closed by a cellulose lid. The electrodes are prepared as follows: a solution of cellulose in cuprammonia is evaporated to a thick paste and the manganese dioxide is stirred in. The mass is placed in an electrolytic cell, the anode of which is a grid of antimony-lead. On passing a current cellulose containing admixed manganese dioxide is deposited on the grid which then becomes the anode of a cell containing a pure solution of cellulose in cuprammonia.

R. B. CLARKE.

Electrolytic apparatus [for deposition of cellulose]. E. TAYLOR AND E. F. CHANDLER, Assrs. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,609, 29.6.26. Appl., 19.3.26; cf. U.S.P. 1,590,593, p. 818).—The electrolytic cell is closed to the atmosphere and contains four copper cathode plates. A belt carrying a solution of cellulose in cuprammonia passes over pulleys from the top to the bottom of the cell, underneath the first plate and over the top of the second, and so on.

R. B. CLARKE.

Electrolytic apparatus. W. G. ALLAN, Assr. to F. G. CLARKE (U.S.P. 1,592,512, 13.7.26. Appl., 8.4.21. Renewed 7.12.25).—A number of cells are connected to form a battery and divided into working groups. The anode and cathode liquids are removed to separating chambers, where the oxygen and hydrogen are removed. The liquids then flow to a mixing chamber before being returned to the cells.

E. S. KREIS.

Apparatus for electric formation of oxides of nitrogen. J. S. ISLAND (E.P. 257,083, 30.7.25).—See F.P. 601,740; B., 1926, 592.

Dry cell. M. MARKIEWICZ and W. RÖMER (U.S.P. 1,597,165, 24.8.26. Appl., 31.5.24. Conv., 5.7.23).—See E.P. 234,701; B., 1925, 639.

Electric welding process and apparatus. G. B. ELLIS. From T. E. MURRAY (E.P. 257,055, 30.6.25).

See also pages 818, **Conversing fibrous cellulosic material** (U.S.P. 1,590,592—3); **Treating cellulosic material** (U.S.P. 1,590,594); **Producing colloidal cellulose** (U.S.P. 1,590,596). 819, **Treating silk and cellulose material** (U.S.P. 1,590,600); **Treating cellulose** (U.S.P. 1,590,601 and 1,590,606). 833, **Electrothermal reduction of alumina** (U.S.P. 1,594,362). 840, **Production of artificial leather** (U.S.P. 1,590,602).

XII.—FATS; OILS; WAXES.

Determination of fat in oilseed [linseed] and oil-cake by the refractometer. H. ZANDER (Z. Unters. Lebensm., 1926, 51, 324—335).—25 g. of the sample are ground so that 80% will go through a No. 34 sieve, and 2 g. of the powder are weighed into a mortar, previously warmed to 70°, and triturated for 2 minutes with 4 c.c. of monochloronaphthalene. After filtration a drop is examined in a refractometer, the temperature of each reading being taken. The oil content is obtained from the readings by reference to tables given, which

have been obtained by the examination of pure linseed oil at a temperature of 25°. The refractometer readings for oilseed or oilcake must be brought to this temperature by tables or curves; every degree above 25° necessitates an addition of 0.00045 to the refractometer reading, and every degree below, a corresponding deduction. Results compared with the ether extraction method agreed closely. Methods using trichloroethylene and also glacial acetic acid in an immersion refractometer proved to be unsatisfactory, and the sources of error are discussed.

W. G. CAREY.

Determination of milk fat and coconut oil in fat mixtures and examination of milk sweets.

H. FINCKE (Z. Unters. Lebensm., 1926, 51, 357—368).—The author finds that by employing a modified method for the Kirschner value, and determining the Polenske value in the usual way, it is possible to determine approximately the coconut oil content. 5 g. of the fat, with the addition of 5 c.c. of glycerol, and 2.5 c.c. of 50% potassium hydroxide are saponified, and the Reichert—Meissl and Polenske values determined. The titrated distillate from the Reichert determination is diluted with water to 130 c.c. and 0.5 g. of finely powdered silver sulphate is added. After keeping for 1 hr. with occasional shaking the mixture is filtered and 120 c.c. are distilled after the addition of 25 c.c. of 2.5% sulphuric acid. 110 c.c. of the distillate are titrated with 0.1N-soda; the result multiplied by 1.2 gives the Kirschner value. By means of formulæ and a table the percentages of milk fat and coconut oil are obtained. Methods are also given for the examination of sweets containing milk fat. For ash, 5—10 g. of an average sample are incinerated in the usual way; if the ash is less than 0.6%, the sample is doubtful, if less than 0.45 there is an insufficiency of milk. Albumin is determined by dissolving 5 g. in hot water, precipitating with copper sulphate solution, filtering through asbestos, washing, drying with alcohol and ether, weighing, igniting, and weighing again. The normal amount of albumin in milk sweets is about 2.5%. Fat is determined on 5—10 g., after precipitating the albumin with copper sulphate, by the Röse—Gottlieb or the Kuhlmann and Grossfeld process, and for further examination the saponification value, and the Reichert, Polenske, and Kirschner values are determined.

W. G. CAREY.

Relations between the constants of fats. H. WOLFF (Z. angew. Chem., 1926, 39, 1037—1038; cf. Schay, B., 1926, 593).—The formula $n = 1 + d(0.5557 - 0.00022V + 0.000035I)$, where n is the refractive index, d the density measured at the same temperature, V the saponification value, and I the iodine value, deduced on theoretical grounds, is confirmed completely by actual determinations.

S. I. LEVY.

Fluorescence of oils in ultra-violet light. F. CRONER (Z. angew. Chem., 1926, 39, 1032).—The fluorescences observed with various fatty and mineral oils both before and after heating to specific temperatures, are recorded. A dark blue fluorescence at the surface indicates a fatty oil which has been heated above 150° or a mixture of fatty oil and mineral oil. A mixed colour indicates a mixture of different fatty oils. S. I. LEVY.

Detection of unsaponifiable oils in fats. D. HOLDE and A. GORGAS (Chem. Umschau, 1926, 33, 198).—Unsaponifiable matter frequently cannot be detected in marine animal oils by the ordinary qualitative test, although present to the extent of 10%. By the addition of cold water, drop by drop, or of a larger quantity (10 c.c.) of hot water to the saponified test mixture, the unsaponifiable constituent can be detected. Small amounts of various unsaponifiable oils when added to linseed or cottonseed oils can be detected in a similar manner. Surface tension appears to be an important factor in the detection of unsaponifiable matter in fats.

F. R. ENNOS.

Additive products of iodine monobromide and hypiodous acid with unsaturated compounds.

D. HOLDE and A. GORGAS (Chem. Umschau, 1926, 33, 198).—Calcium salts of the bromoiodo- and iodohydroxy-fatty acids previously described (B., 1925, 600) were prepared. Bromoiodohexane was obtained from methylpropylethylene by the action of Hanus' solution as a slightly coloured liquid, b.p. 124—126°, f.p. —18°.

F. R. ENNOS.

Deodorisation of coconut oil. W. L. BROOKE (Philippine J. Sci., 1926, 30, 201—212).—The author confirms the finding of Haller and Lassieur (B., 1910, 704) that methyl nonyl ketone is present in the sludge obtained in the deodorisation of coconut oil. Alcohols are also present. Most of the unsaponifiable constituents of the oil distil over during the first four hours of deodorisation, but in the factory process only about a tenth of the yield is recovered, the remainder being removed in the water used to maintain the vacuum. C. P. STEWART.

Distillation of fatty oils in a vacuum. D. HOLDE and A. GORGAS (Chem. Umschau, 1926, 33, 197—198).—The residue from the distillation of linseed oil *in vacuo*, when mixed with Para rubber and vulcanised, does not make so good a rubber substitute as the ordinary factis. The residue from the distillation at 20 cm. pressure is a dark, viscous liquid. The semi-solid distillate (14—19% of the linseed oil) consists chiefly of saturated acids from which stearic acid can be separated, together with 13% of unsaponifiable oils, mainly unsaturated hydrocarbons with small quantities of aldehydes, ketones, and alcohols. Distillation of cod-liver oil similarly gives 31—38% of distillate, consisting of unsaponifiable oils and saturated acids, chiefly palmitic acid, together with 54—62% of an elastic residue.

F. R. ENNOS.

Polymerisation and oxidation of unsaturated fatty acids. K. H. BAUER (Chem. Umschau, 1926, 33, 198—199).—When α - and β -elæostearic acids are heated in carbon dioxide to 200°, polymerisation products are obtained having lower acidity, higher saponification values, and lower iodine values than the parent acid. The mol. wt. of the β -polymer is 985.6 as determined by the depression of the freezing point of benzene and 490 by Rast's camphor method, the corresponding figures for the polymer of the α -acid being 4588 and 2285 respectively. Catalytic hydrogenation of the α -polymer yields chiefly stearic acid, but the β -polymer gives a thick oil of mol. wt. 1030 in benzene and 535 in camphor. The fatty acids from polymerised linseed oil give mol. wt.

values which show that the product is bimolecular in benzene solutions and unimolecular in camphor solutions, thus differing from the fatty acids from polymerised perilla oil (B., 1925, 250). On oxidation with perbenzoic acid, linolenic acid takes up 4 atoms of oxygen and its ethyl ester 3. Oleic acid similarly takes up 2 atoms of oxygen and its ethyl ester one only. The oxidation products of the acids are approximately bimolecular in solution, but those of the esters are unimolecular (cf. B., 1925, 459).

F. R. ENNOS.

Synthesis of waxes. A. GRÜN (Z. angew. Chem., 1926, 39, 1037; cf. A., 1926, 596).—Good results are obtained in the preparation of wax alcohols from higher ketones by using metals other than nickel, and mixtures of metals, as catalysts, yields of 80–90% being obtained. Combustion of the products in the ordinary way for analysis gives low results, ethylene escaping unburned, but accurate results are obtained if the material is mixed with cupric oxide in the boat.

S. I. LEVY.

Cotton seed and its products [during the past fifty years]. D. WESSON (Ind. Eng. Chem., 1926, 18, 938–940).

Progress in the soap industry during the last fifty years. M. H. ITTNER (Ind. Eng. Chem., 1926, 18, 908–910).

See also A., Sept., 938, **Isomerism of dihydroxystearic acids produced by oxidation of acids of the oleic and elaidic series (HILDITCH)**. 977, **Connexion between degree of dispersion of substrate and enzyme action. Determination of enzymic lipolysis (RONA and KLEINMANN)**. 980, **Unsaponifiable fraction of fish oil (WEIDEMANN)**. 981, **Ochna pulchra berries and oil (FACER)**; **Flower waxes: rose wax (PROPHÈTE)**.

PATENTS.

Food and other products from fish (E. P. 246,148).—See XIX.

Working up slaughterhouse offal etc. (E.P. 256,162).—See XIX.

Substances for salves (G.P. 425,331).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Examination of kauri dust. H. WOLFF (Farben-Ztg., 1926, 31, 2609–2611).—When the very divergent analytical constants determined on various samples of kauri dust are calculated to values for the portion soluble in amyl alcohol, improved concordance is obtained, and in particular the sum of the saponif. value and iodine value is nearly constant (340–370 being the suggested limits). Formation of phenolic hydroxy-groups by oxidation at the double linkings is suggested as a possible explanation for this reciprocal fluctuation in saponif. and iodine values, which had previously been observed in the case of lump kauri. Examined over a period of 20 years, a sample of kauri dust showed a gradual increase in saponif. value, while its acid value and iodine value dropped towards a minimum. The sum of saponif. value and iodine value remained approximately constant

(335–350) for 13 years, but then rose with the saponif. value, attaining a value of 385 at the end of the observations. The solubility dropped by 9% during the whole period. The determination of the amount and nature of the ash in the insoluble matter gives a possible further criterion that, taken in conjunction with the above considerations, enables adulteration (particularly with rosin) to be detected.

S. S. WOOLF.

Storch-Morawski [Liebermann] reaction and rosinate-varnishes. H. WOLFF (Farben-Ztg., 1926, 31, 2611–2612).—A critical discussion of Schulz and Krämer's work (B., 1926, 796). The limitations of the Storch-Morawski reaction for rosin are pointed out, with special reference to its application to the specifying of varnishes as rosin-free.

S. S. WOOLF.

Paint and varnish—yesterday, to-day, and to-morrow. M. TOCH (Ind. Eng. Chem., 1926, 18, 948–949).

Acid constituents of resin of *Pinus pinea*. DUPONT and DUBOURG.—See A., Sept., 954.

PATENTS.

Manufacture of lampblack. VEREIN FÜR CHEM. & METALL. PRODUKTION (G.P. 429,485, 17.10.23).—The usual starting materials (except methane, ethylene, and gases containing these) are mixed with hydrogen before combustion. Materials of higher oxygen content require more hydrogen than those of smaller oxygen content.

A. DAVIDSON.

Lining and coating pipes and other bodies. B. TALBOT (E.P. 255,546, 24.4.25).—An improved coating material for applying to the interiors of pipes etc., centrifugally or otherwise, consists of bitumen mixed with inert filling material and with 5–10% of sulphur, and heated for several hours at a temperature above the melting-point of the bitumen, until evolution of gas has ceased. The inert powdered loading material may be added before or after the treatment with sulphur. The sulphur appears to combine with gases in the bitumen, as hydrogen sulphide is largely evolved, and but little sulphur remains combined with the bitumen. A bitumen already containing about 5% of combined sulphur was greatly improved (melting-point raised and rendered less brittle when cold) by treatment with a further 5% of elemental sulphur.

B. M. VENABLES.

Manufacture of paints and the like. W. E. BILLINGHAME (E.P. 255,911, 23.1.25).—Bitumen or bituminous material is emulsified in admixture with pigments. Alternatively, an emulsion of the vehicle may be added to the pigment mixed with an alkaline solution of a soluble protein or to an emulsion of the pigment ground in oil. (Reference is directed, in pursuance of Sect. 7 (4) of the Patents and Designs Acts, 1907 and 1919, to E.P. 221,380, 154,236, 15,711 of 1910, and 11,208 of 1903.)

E. H. SHARPLES.

Purifying and stabilising hydrocarbons [turpentine oil]. F. KLEIN (U.S.P. 1,592,603, 13.7.26. Appl., 19.8.25).—Oil of turpentine is fractionally distilled and the 150–165° fraction is successively heated to

156° and cooled to 20° in one container, until no more of the liquid distils. The yield of terpene boiling at 155—156° is greater by 30—40% than that obtained by the usual method of fractional distillation. The method can be applied to the fractional distillation of a number of hydrocarbon mixtures. T. S. WHEELER.

Condensation products of phenol and the like. A. H. BROWN, and SILUMINITE INSULATOR Co., LTD. (E.P. 255,516, 17.4.25).—If to cold well-stirred phenol-formaldehyde or similar mixtures yielding resinous condensation products, with or without accelerators, there is added hot linseed, castor, or other suitable water-immiscible oil (in amount not exceeding 3.2% by volume of the remaining ingredients), a separation into two layers occurs on keeping, the whole of the water present sinking to the bottom. The upper oily layer, containing the intermediate reaction product, is run off and heated, when it will thicken to any desired extent and the condensation reaction will not proceed beyond the stage attained when the heating is stopped. The addition of commercial phenol will reduce the hardness of the mass if the reaction has been carried too far.

S. S. WOOLF.

Manufacture of artificial resins. R. SINGER, ASSR. to KUNSTHARZFABR. REGAL & Co. (U.S.P. 1,590,961, 29.6.26. Appl., 17.6.25. Conv., 19.5.25).—7 pts. of monochloro- or dichloro-aminoaldehyde are dissolved in 80 pts. of 40% formaldehyde and 100 pts. of phenol; the mixture is boiled under a reflux and then heated under reduced pressure to remove water. A clear resin is obtained.

T. S. WHEELER.

Dehydrated carbohydrate-phenolic resinous products. J. V. MEIGS (U.S.P. 1,593,342, 20.7.26. Appl., 30.11.25).—600 pts. of phenol, 5 pts. of sulphuric acid, and 500 pts. of sugar containing 84% of dextrose are heated to 180°, the water formed being removed through a fractionating column. 8 pts. of stearic acid are added as a lubricant for moulding purposes, and the excess of phenol is then removed by heating *in vacuo*. A black resin is obtained, which appears from the quantities of phenol and dextrose entering into reaction and the quantity of water produced, to be formed by interaction of 1 mol. of dextrose and 2 mols. of phenol. The resin melts to a viscous liquid and may be hardened by heating to 140° with 3—10% of hexamethylenetetramine. In place of phenol, α - and β -naphthol, aniline, and α - and β -naphthylamine may be used, whilst dextrose may be replaced by other hexoses or by pentoses.

T. S. WHEELER.

Production of oven-drying lacquers. CELLA DRAHTWERK G.M.B.H. (G.P. 428,390, 25.3.24).—Colloidal palladium accelerates the drying of lacquers. An addition of 5 g. of palladium to 50 kg. of lacquer enables drying to take place at 100° in the same time as is required at 200—230° without palladium.

A. DAVIDSON.

Refining aldehyde resins. W. O. HERRMANN and H. DEUTSCH, ASSRS. to CONSORTIUM FÜR ELEKTROCHEM. IND. G.M.B.H. (U.S.P. 1,596,971, 24.8.26. Appl., 15.6.22. Conv., 29.6.21).—See E.P. 182,459 and 184,442; B., 1923, 613 A.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Artificial ageing tests on plantation rubber. LONDON COMMITTEE, CEYLON RUBBER RESEARCH SCHEME (Bull. Imp. Inst., 1926, 24, 209—219).—When vulcanised with sulphur only, unsmoked sheet rubber is superior to crêpe rubber in tensile strength, in latitude of vulcanisation, and in ageing properties; it is, however, less plastic and more variable in rate of vulcanisation than crêpe rubber. Pressing the crêpe rubber into blocks and rolling up the sheet rubber with retention of a proportion of moisture has no effect on the ageing properties.

D. F. TWISS.

Preservatives against ageing for rubber vulcanised with sulphur chloride. W. ESCH (Gummi-Ztg., 1926, 40, 2536—2537).—A review of the precautions necessary and of the preservatives favourable to the production of stable cold-vulcanised products. The preservatives, such as aniline, naphthol, and aldol- α -naphthylamine, are applied in solution after the vulcanisation process.

D. F. TWISS.

Sols of caoutchouc nitrosite-nitrosate. K. FISCHER (Gummi-Ztg., 1926, 40, 2587—2588).—Solutions of several samples of rubber in nitric acid were examined as to their viscosity and surface tension on the addition of various proportions of water and were shown to be highly dispersed sols; the physical character of the nitrosite-nitrosate precipitable by more water was also investigated as to its physical properties and colloidal character.

D. F. TWISS.

Changes in the rubber industry during the past fifty years. G. OENSLAGER (Ind. Eng. Chem., 1926, 18, 902—905).

PATENTS.

Manufacture of coloured rubber goods. I. G. FARBENIND. A.-G., ASSCES. of W. ZIESER (G.P. [A] 427,873, 20.3.21, and [B] 428,687, 24.9.21).—(A) Mixtures of rubber with organic colours, if also containing zinc oxide, a piperidinecarbothionolate, and a reduced proportion of sulphur, can be vulcanised at low temperatures without loss of brilliancy. (B) Mixtures containing inorganic pigments sensitive to the conditions of ordinary vulcanisation can in a similar manner be vulcanised below 100°.

D. F. TWISS.

XV.—LEATHER; GLUE.

Tannins of the black cypress pine (*Callitris calcarata*) and their distribution in the bark. F. A. COOMBS, W. MCGLYNN, and M. B. WELCH (J. Proc. Roy. Soc. N.S.W., 1926, 59, 356—382).—Black cypress pine, *Callitris calcarata*, yields an important tannin-bearing bark available in large quantities. The tannin occurs principally in the phloem parenchyma, the medullary ray cells, and the epithelial cells lining the resin passages. Some specimens contain nearly 37% of tannin, and commercial samples average 20—25%. For commercial extraction the bark should be finely ground, and a moderate temperature employed, since there is a loss of tannin owing to the probable formation of starch-tannin complexes at higher temperatures.

C. J. STILL.

Use of filtered ultra-violet light for recognising and distinguishing natural and artificial tanning materials. O. GERNGROSS, N. BÁN, and G. SÁNDOR (*Z. angew. Chem.*, 1926, **39**, 1028—1032).—Nearly all the modern synthetic tanning preparations show fluorescence when examined by ultra-violet light from a quartz mercury lamp passed through a dark glass filter, which cuts off all the visible rays. Tables of colours are given for 24 fluorescent materials, most of which answer the test in dilutions of 1 : 1,000,000. A list of 14 materials which give no fluorescence at dilutions of 1 : 1000 is also given. The fluorescence exhibited by natural tannins under the same conditions is very different; with these, in many cases, there is local absorption, and they yield the fluorescent components to fibres immersed in the solutions. The violet fluorescence of unbleached sulphite-cellulose is found to be due to an original constituent of the spruce bark, which may be extracted by digestion with pure water at 120°.

S. I. LEVY.

Behaviour of neutral salt-treated hide powder towards tanning agents. K. H. GUSTAVSON (*J. Amer. Leather Chem. Assoc.*, 1926, **21**, 366—385).—Portions of hide powder were treated with molar solutions of various salts and the loss of hide substance was determined. The peptising action of cations was shown to be in the order $\text{Ca} > \text{Sr} > \text{Ba} > \text{Mg} > \text{Na}$, K, and that of anions $\text{CNS} > \text{I} > \text{Br} > \text{Cl} > \text{SO}_4, \text{S}_2\text{O}_3$. Portions of hide powder treated respectively with different neutral salts were treated with a solution of hemlock extract, and the combined tannin was determined in each case by the Wilson-Kern method. The p_{H} values of the exhausted tan liquors were the same in every case, but the combined tannin was greatest in the case of those powders which had lost most hide substance by peptisation, *i.e.*, it followed the Hofmeister series. The author considers that the combination of vegetable tannin with collagen is largely a function of the state of the hide powder in regard to its secondary valency activity and specific surface forces. Vegetable tanning cannot be explained simply as an electro-neutralisation process. Cathodic chromium in the form of chlorides and sulphates gave the same chromium fixation for all the samples of hide powder, whereas the fixation of anodic chromium depended on the previous treatment of the hide powder and followed the Hofmeister series. Extremely basic chromium sulphate liquors containing both cathodic and anodic chromium showed specific ion-effects in their reactions with hide powder treated with neutral salts. In this case it is suggested that there is primary valency reaction between cationic chromium and the carboxyl groups of the protein, formation of molecular compounds between basic protein groups and anionic chromium complexes, and true adsorption of chromium salts by the hide powder. Hide powders treated with neutral salts behaved towards basic aluminium sulphate in the same way as towards extremely basic chromium compounds.

D. WOODROFFE.

Determination of moisture in tannery materials. I. D. CLARKE (*J. Amer. Leather Chem. Assoc.*, 1926, **21**, 385—399).—The Bidwell-Sterling modification of the Dean-Stark method (*cf.* B., 1925, 268) can be applied to all the substances which offer difficulties by the

ordinary method. The sample and 80—100 c.c. of toluene are placed in an Erlenmeyer flask fitted with a delivery tube, which is attached to the upper portion of a measuring tube fitted with a reflux condenser. In this way a reasonably small volume of toluene can be used. Drops of water on the sides of the measuring tube are dislodged by a thin wire loop, whilst a brush is used for removing water from the condenser to the collecting tube. The condenser and tube should be cleaned each time with chromic acid mixture. Comparative determinations have been made using benzene, toluene, and xylene respectively, also with the direct flame method, oven drying, and evaporator and dryer method. The results with benzene, toluene, and xylene did not always agree. The simple xylene distillation method gave lower results than the Bidwell-Sterling modification or the direct flame method. The latter gave results but little higher than those with toluene. Toluene seems to be the most suitable liquid to use. Xylene is unsatisfactory in the case of substances which are liable to decompose. Benzene gives very low results. D. WOODROFFE.

Properties of shoe leather. VI. Resilience. J. A. WILSON and E. J. KERN (*J. Amer. Leather Chem. Assoc.*, 1926, **21**, 399—403).—The resilience of the 18 different leathers previously mentioned (B., 1926, 504, 600, 798) has been determined. Semichrome leather had the lowest resilience and vegetable-tanned sole leather the greatest. Increasing water content of leather lowers its resilience.

D. WOODROFFE.

Tanning, 1876—1926. J. A. WILSON (*Ind. Eng. Chem.*, 1926, **18**, 934—936).

Artificial leather [during the past fifty years]. G. C. GIVEN (*Ind. Eng. Chem.*, 1926, **18**, 957—958).

PATENTS

Treatment of hides and skins prior to tanning. J. HELL (E.P. 255,566, 1.5.25).—Raw hides and skins, with or without a preliminary soaking in water, are soaked in sulphite-cellulose waste liquor or products recovered therefrom, and the excess sulphite-cellulose material is removed by washing. The hides or skins are then limed, delimed, and tanned as usual. The deliming or pickling may be carried out in the presence of sulphite-cellulose waste liquor or products recovered therefrom and auxiliary agents for plumping the hides may be added.

D. WOODROFFE.

Manufacture of dry products [bates] from pancreas. R. LEPETIT (U.S.P. 1,590,388, 29.6.26. Appl., 22.9.22. Conv., 12.11.21).—Finely-divided pancreas gland tissue is ground with one or more anhydrous inorganic salts which form crystals containing water of crystallisation, an ammonium salt, and sodium chloride, to give a dry product which is of value as an artificial bate. Suitable anhydrous salts include calcium sulphate, sodium sulphate, and magnesium sulphate. A preferred composition comprises ground pancreas material 15—20 pts., anhydrous calcium sulphate 9 pts., dry sodium chloride 16 pts., anhydrous sodium sulphate 15 pts., and dry ammonium chloride 15 pts.

T. S. WHEELER.

Dyeing of leather. M. C. LAMB (E.P. 255,555, 27.4.25).—Vegetable-tanned, mineral-tanned, oil-dressed,

or aldehyde-tanned leathers are treated with 1—3% of a hydrogenated naphthalene, e.g., tetrahydronaphthalene, alone or in association with halogen-hydrocarbons and with or without emulsifying agents such as sodium, potassium, or ammonium ricinoleate, sulphonated or solubilised oils of mineral, vegetable, or animal origin, hydrogenated phenol soap, hydrogenated cresol soap, or salts of aromatic acids or of aromatic sulphonic acids, and afterwards dyed in the usual manner. The hydrogenated naphthalene may be mixed with halogen-hydrocarbons and a suitable oil or fatty matter for the purpose of fat-liquoring the leather subsequent to the dyeing process.

D. WOODROFFE.

Treating organic material. [Production of artificial leather.] E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,602, 29.6.26. Appl., 17.6.24).—A sheet of cellulose is passed over a felt-covered roller, partly immersed in water, so that the under surface of the sheet is moistened. The upper surface is then treated with a solution of cuprammonia which penetrates the sheet but gradually loses its solvent power as it meets the water. The sheet then passes into a closed electrolytic tank, containing dilute ammonium nitrate, between copper electrodes, where it loses the solvent constituents. The sheet is washed, dyed while still moist, and passed over hot rollers, where the treated surface is stamped to simulate leather.

R. B. CLARKE.

Preparation of plastic horny substances from albumin or albuminous materials. DEUTSCHE KUNSTHORN-GES. M.B.H., and J. SCHLINCK (G.P. 429,525, 21.10.24).—The materials are treated with a dilute solution of formamide, acetamide, or mixtures of the two either before or after the plastifying process. The hardening effect is slow and does not interfere with the plastifying process.

C. IRWIN.

Treatment of cellulose (U.S.P. 1,590,607).—See V.

Food and other products from fish (E.P. 246,148).—See XIX.

XVI.—AGRICULTURE.

Composition of the fractions separated by mechanical analysis from some Transvaal soils. B. DE C. MARCHAND and C. R. VAN DER MERWE (S. Afr. J. Sci., 1925, 22, 104—118; Chem. Abstr., 1926, 20, 1880).—In sandy soils the silica decreases, whilst alumina, ferric oxide, phosphoric oxide, lime, magnesia, and potash increase with decrease in the size of the soil aggregate. The phosphorus and potash are largely concentrated in the clay. In the red clay or heavy red loam soils the silica decreases, the alumina, magnesia, and phosphate increase, with decrease in particle size, but the other constituents exhibit irregularities. Considerable proportions of phosphorus and potash are present in the clay fractions. In the black clay soils silica and lime decrease; alumina, ferric oxide, magnesia, potash, and phosphate tend to increase from the coarser to the finer fractions, the potash and phosphate again being concentrated chiefly in the clay fraction. It is noteworthy that in the black clay soils the concentration of silica is relatively

high in comparison with that of alumina, whilst the reverse is the case for the red clays. The clay fraction of sandy soils is similar in composition to that of the heavy red soils and different from that of the black.

A. A. ELDRIDGE.

Decomposition of organic matter in soil. H. H. HILL (J. Agric. Res., 1926, 33, 77—99).—Pure cellulose, whether added to soil or to culture solutions in which plants were grown, restricted plant growth to an extent proportional to the amount of cellulose added. Treatment with 0.01% of potassium nitrate did not counteract this effect. During the cellulose decomposition hydrogen sulphide was produced by the reduction of sulphates in the culture solution. The root systems of plants grown in culture solutions containing cellulose were much discoloured and unhealthy. Maize and wheat following ploughed-in legumes showed increased yields, but wheat following ploughed-in rye-grass showed decreased yields compared with controls over a twelve-year period. The nitrogen-carbon ratio of the green crop was higher for non-legumes than for legumes, and generally the ratio was smaller in younger plants. The evolution of carbon dioxide following the incorporation of green matter with soil was greater than when hayed crops were used. In similar circumstances nitrate production was more rapid from air-dried material than from green, and was considerably greater with legumes than with non-legumes.

A. G. POLLARD.

Arsenates as soil insecticides. B. R. LEACH (J. Agric. Res., 1926, 33, 1—8).—Acid lead arsenate proved toxic to the larvæ of the Japanese beetle (*Popillia japonica*) when mixed with soil in which the larvæ were feeding. The death period varied with the amount of the arsenate added. The arsenates of lead (basic), iron, and magnesium were non-toxic under these circumstances. These arsenates were also non-toxic to plants. Acid lead arsenate was the least toxic to plants among those arsenates toxic to the larva (zinc, copper, and calcium arsenates were also examined). In the soil acid lead arsenate appears to be converted slowly into a basic form, but can be applied at the rate of 1500—2000 lb. per acre in nursery soils without affecting the growth of plants.

A. G. POLLARD.

Relative availability of phosphatic fertilisers on acid and non-acid soils. G. INGHAM (S. Afr. J. Sci., 1925, 22, 122—134; Chem. Abstr., 1926, 20, 1882).—The "availability" (subsequent solubility in 1% citric acid) of a phosphatic fertiliser depends more on the soil reaction than on the water- or citric acid-solubility of the original fertiliser, varying for superphosphate from 21% in a very acid soil to 96% in a soil free from acidity. The availability is closely related to the iron, aluminium, and organic matter content of the soil. Low availability may be due to the absorptive power of organic and inorganic colloids, especially those of iron and aluminium. Acid soils have a high absorptive capacity for phosphate as well as for lime. Removal of calcium carbonate from a soil containing much iron and aluminium doubled the absorption capacity and reduced the citric acid-solubility of the fertiliser by 50%, whereas addition of calcium carbonate to an acid soil increased the availability

of superphosphate only after 12 months. The use of carbonic instead of citric acid would probably be in closer accord with soil conditions. A. A. ELDRIDGE.

Phosphates and phosphoric acid requirements of soils. M. VON WRANGELL (Landw. Jahrb., 1926, 63, 627—668; Chem. Zentr., 1926, II., 815—816; cf. succeeding abstracts).—The concentration of phosphoric acid in the soil solution depends not only on the amount of water, but also on the presence or absence of other ions; further, it depends more upon the absorptive capacity of the soil than upon the chemical nature of the phosphate. It is markedly influenced by the presence of lime, or a change in the reaction of the soil. The amount varies from 0.02—2.0 mg. per litre in different soil types, with an average figure of 0.1—0.6 mg. The speed with which equilibrium between soil and soil solution is restored after disturbance, as by dilution or the action of plant roots, is also an important factor in connexion with the supply of phosphoric acid; it is much less in heavy, strongly absorptive soils than in light soils. The total amount of soluble P_2O_5 in the soil is probably of greater importance than the concentration of the soil solution, since plant roots and root-hairs not only draw on the soil water, but can also obtain P_2O_5 from the colloid coating of the soil particles. The determination may be made by successive extractions of a small amount of soil with distilled water, until a constant minimum concentration is reached, usually about 0.1 mg. P_2O_5 per litre. In practice, the total amount of extractable P_2O_5 may be calculated from the results of the first two or three extractions. Experiments with plants in continuously renewed solutions show that the concentration at which there is no further uptake of P_2O_5 is below 0.1 mg. per litre—usually 0.03 mg. The sap of plants may contain up to 500 mg. per litre of dissolved P_2O_5 , which is probably combined with protein. A transference of phosphate ions into colloidal solution or a greater solubility of P_2O_5 in the plasma than in water must be assumed.

C. T. GIMINGHAM.

Colorimetric method for rapid determination of phosphoric acid in very dilute solutions [soil extracts etc.]. M. VON WRANGELL (Landw. Jahrb., 1926, 63, 669—675; Chem. Zentr., 1926, II., 816).—90 c.c. of the solution, containing 0.01 to 0.04 mg. P_2O_5 , are treated with 1.5 c.c. of the molybdate reagent (equal parts of concentrated sulphuric acid and 10% ammonium molybdate solution), 0.75 c.c. of freshly-prepared 1% stannous chloride solution is added, and the whole made up to 100 c.c. and well shaken. A blue colour appears and reaches its maximum intensity in 5 min. It is compared in a colorimeter with standard solutions. Oxidising substances must be absent; the solution must not be filtered, but may be centrifuged, if necessary. The colour is increased by the presence of arsenic or silica, and reduced by fluorine compounds. With soils, the error due to these is usually negligible, though arsenic should be tested for. If the method is used for "Rhenania" phosphate, serious errors are introduced unless the silica is first removed.

C. T. GIMINGHAM.

Solubility relationships in their application to tertiary phosphates. M. VON WRANGELL and E. KOCH (Landw. Jahrb., 1926, 63, 677—706; Chem. Zentr., 1926, II., 817).—The action of water on tertiary phosphates is a process of decomposition rather than of solution. In solubility experiments with tertiary phosphates of calcium, aluminium, and iron, equilibrium between the solid and liquid phases could hardly be reached, whereas with crystalline magnesium phosphate, $Mg_3(PO_4)_2 \cdot 22H_2O$, it was obtained in a short time. By the action of water on calcium phosphate, ions of three kinds are formed— PO_4''' , HPO_4'' , H_2PO_4' —and a hydrolytic equilibrium is set up; the process of solution alters the acidity of the soil mass, or, conversely, the soil is altered by increase of acid. The more acid the solution, the higher is the content of calcium phosphate, whereas the content of aluminium and iron phosphates increases with increasing concentration of hydroxyl ions; magnesium phosphate is intermediate in this respect. The final result of the hydrolytic decomposition of difficultly soluble phosphates depends considerably on the nature of the base of the phosphate, on the influence of acid on the specific effect of the acid anions, and on the influence of base on the cations. The different effects of sodium and calcium hydroxides are specially striking. Calcium carbonate has a greater inhibiting effect on the solution of tricalcium phosphate than an equivalent amount of calcium chloride. The action is less marked with magnesium, iron, and aluminium phosphates; but soluble calcium salts reduce the PO_4 ions by formation of calcium phosphate. Solutions of ammonium salts exert a much greater solvent action on tricalcium phosphate than solutions of sodium or potassium salts; the effect of the former increases with increasing concentration, whereas that of the latter changes only slightly. Study of the solubility relationships of the phosphates suggests the possibility of controlling the supply of soluble phosphate in the soil by varying the conditions.

C. T. GIMINGHAM.

Phosphoric acid content of natural soil solutions. M. VON WRANGELL and W. HAASE (Landw. Jahrb., 1926, 63, 707—738; Chem. Zentr., 1926, II., 817—818).—Methods for obtaining the natural soil solution are discussed. Direct displacement of the solution by pressure is the most satisfactory, and details are given of a suitable type of hydraulic press. It is important that the pressure should be great in relation to the height of the column of soil. The slightly turbid liquid obtained is purified by flocculating the colloidal matter present and centrifuging. Phosphoric acid can then be determined by the colorimetric method (cf. preceding abstract). The concentration of P_2O_5 in the soil solution of different soils varies widely, but is relatively constant in any one soil at different times of the year. There appears to be no relation between the amount of P_2O_5 in the soil solution and the total present in the soil. The effect of the treatment of the soil with very dilute acid or alkali on the concentration of P_2O_5 varies greatly with the nature of the soil. With some soils, a second displacement of the solution, after remoistening of the soil, shows about the same concentration of P_2O_5

as the first; with other soils there is a marked decrease.

C. T. GIMMINGHAM.

"Available" part of the phosphoric acid in soil. M. VON WRANGELL and L. MEYER (Landw. Jahrb., 1926, 63, 739—775; Chem. Zentr., 1926, II., 818—819; cf. preceding abstracts).—Neubauer's seedling method for determining the P_2O_5 requirement of soils is critically compared with von Wrangell's method of determining P_2O_5 colorimetrically in the natural soil solution displaced by pressure. The value obtained by the former method is liable to vary from time to time under the influence of changing conditions, whereas the latter determines a relatively fixed quantity. Careful control of the quality of seed, the temperature, the water content of the soil and other factors is most important in carrying out the Neubauer method. Comparative experiments with both methods indicate a relationship between the availability of the soil P_2O_5 and the concentration in the soil solution. With increasing water content, the P_2O_5 concentration of the soil solution increases; on drying, there is a rapid decrease of concentration; and on remoistening, the original figure is reached again in a short time. These relationships are less evident with heavy soils on account of the slow rate at which equilibrium between soil and soil solution is restored after disturbance. The seedling method also shows an increasing uptake of P_2O_5 by the plants, with increased water content of the soil. Addition of sand to the soil decreases the P_2O_5 content of the soil solution, unless the water content is made up to the original figure. It is suggested that the increase of concentration of P_2O_5 in the soil solution when the amount of water in the soil is increased is due to the solution of certain forms of phosphate, probably held absorbed, owing to a change in the mechanical structure of the finest soil particles.

C. T. GIMMINGHAM.

Nitrogen availability studies on crops harvested at different stages of growth. C. H. SINGLETON (New Jersey Agric. Exp. Sta. Bull., 1925, [421], 1—28; Chem. Abstr., 1926, 20, 2040).—For oats, millet, buckwheat, and rape, the yields of dry matter after fertilisation were in the decreasing order: sodium nitrate, ammonium sulphate, tankage, calcium cyanamide. The recovery of nitrogen was respectively 62%, 54.24%, 46.62%, 25.84%. The percentage of nitrogen is highest in plants during the first 3 weeks of growth.

A. A. ELDRIDGE.

Sulphur as a plant food. J. R. NELLER (Washington Agric. Exp. Sta. Bull., 1924, [187], 37—38; Chem. Abstr., 1926, 20, 1881).—Comparatively large quantities of sulphur may be oxidised in E. Washington soils without marked retardation of organic oxidation; the addition of large amounts of sulphur to fine sandy loam caused an increase in the water-soluble potassium and calcium. Legumes, in increased yield, frequently showed an increase in total protein, phosphorus, and potassium, and a decrease in calcium. Straw humification proceeds more rapidly on the addition of sodium nitrate or ammonium sulphate and calcium carbonate. The initial stages of decay appear to be associated with the formation of a toxic substance which is subsequently oxidised to a harmless or beneficial compound.

A. A. ELDRIDGE.

Effect of nutrient conditions on the colloidal properties of certain vegetable crops. J. W. CRIST (Michigan Agric. Exp. Sta. Tech. Bull., 1926, [74], 1—27).—Lettuce, spinach, radish, beet, and tomatoes, when grown on suitably fertilised soil, had a higher ratio of green to dry weight than unfertilised plants. The treated plants had higher percentages of bound water and the hydratable components of their juices were of greater hydration capacity, nitrates being most effective. The efficiency of nitrate is reduced by potassium or phosphorus, but not by sulphur. The relation of the results to the structure and behaviour of the plants is discussed.

A. A. ELDRIDGE.

Relationship between concentration of soil solution and physico-chemical properties of leaf-tissue fluids of Egyptian and Upland cotton. J. A. HARRIS (J. Agric. Res., 1926, 32, 605—647; cf. Harris and others, B., 1924, 607; 1925, 35).—One variety of Egyptian and two varieties of Upland cotton were grown under irrigation conditions in Arizona, the planting being so arranged that sub-plots of the three varieties were replicated many times in the experimental area. Statistical methods of treatment are applied to the data obtained by determinations of (a) the concentration of the soil solution (as measured in terms of the electrical resistance of the saturated soil mass) of a large number of samples of soil, taken at depths of 1—4 ft. throughout the field, and (b) of the osmotic concentration (freezing point depression), specific electrical conductivity, and chloride content of the leaf-tissue fluids of the plants on each sub-plot. The results show the existence of marked heterogeneity of the soil in a small experimental field and demonstrate the possibility of determining, with considerable precision, the correlation coefficient of the relationship between the properties of the soil of different portions of the field and the characteristics of the plants grown thereon. Differences in the plant tissue fluids are associated with the soil differences, though there is not an exact parallelism. In general, higher concentrations of all solutes, and of dissociated electrolytes in the plant fluids, are associated with higher concentrations of the soil solution. There is also a substantial correlation (especially marked for Egyptian cotton) between resistance of the soil mass and the chloride content of the leaf fluids. The method of treatment could be extended to other properties of soils and crops, and the results are considered to indicate the possibility of carrying out physiological investigations in the field.

C. T. GIMMINGHAM.

Fertilising in relation to the disease resistance of crops. B. THOMAS (Fert. Feeding-stuffs J., 1925, 10, 478—488).—Potassium salts, particularly the chloride or sulphate (*e.g.*, the latter at 200 lb. per acre), considerably increase the percentage of healthy potato plants; superphosphate (600 lb. per acre) applied to a soil already containing an unusually large quantity of available phosphate considerably decreased the percentage. Ammonium sulphate supplied up to 800 lb. per acre as a balanced fertiliser with potash and superphosphate afforded a progressive increase in the percentage. Potassium is the determining factor in promoting the disease resistance of plants; nitrogen and phosphate in excess

sive amounts have an adverse effect. There is an optimum ratio for each variety of plant.

A. A. ELDRIDGE.

Chemistry's contributions to the fertiliser industry [in the past fifty years]. J. E. BRECKENRIDGE (Ind. Eng. Chem., 1926, 18, 941—943).

Insecticides and fungicides [during the past fifty years]. E. C. HOLTON (Ind. Eng. Chem., 1926, 18, 931—933).

Losses of nitrogen caused by soil bacteria in pure cultures. LEMOIGNE and DOPTER.—See A., Sept., 979.

Variation in moisture and nitrogen content of potato during growth. FAGAN.—See XIX.

Effect of hydrogen-ion concentration of soil on formation and composition of essential oil of wood sage. DEEL.—See XX.

PATENTS.

Manufacture of mixed fertilisers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 255,726, 7.1.26).—As an improvement on E.P. 236,494 according to which a mixed fertiliser is prepared by mixing together urea, ammonium phosphate, and potassium nitrate and/or sulphate, it is now proposed to replace the ammonium phosphate wholly or partially by dicalcium phosphate. It is claimed that the latter compound is of value on account of its buffer action. Potassium may be used in the form of any fertilising salt but the sulphate and/or nitrate are preferable. (Cf. B, 1925, 732.)

G. W. ROBINSON.

Decomposition of raw phosphates. LANDWIRTSCHAFTLICHE WARENZENTRALE A.-G. (G.P. 430,095, 4.7.25).—The decomposition of crude phosphates by means of sulphur dioxide is facilitated by the addition of an equivalent, or smaller amount, of chloride, nitrate, oxalate, silicate, permanganate, or persulphate, or mixture of these salts, with or without a sulphate. The introduction of a small quantity of free acid (which must not exceed one-tenth of the phosphoric acid present) is also advantageous.

R. B. CLARKE.

Storage of mixed fertilisers containing ammonium nitrate. J. HEPPES (G.P. 430,096, 24.12.24).—As usually stored, mixtures of ammonium sulphate and ammonium nitrate, or similar mixtures, set to an extremely hard mass after a short time. This is avoided if the storage heaps are maintained at a temperature above 32°.

R. B. CLARKE.

Insecticidal or other toxic substances from plants of the *Derris* species. NETTAI SANGYO KABUSHIKI KAISHA (E.P. 247,140, 25.6.25. Conv., 4.2.25).—Fresh roots of *Tuba* or other of the *Derris* species are crushed, mixed with a small quantity of water, and the mixture is filtered in a centrifugal filter. The liquor is allowed to settle in a tank for about 3 hrs. and the supernatant liquid transferred to a vacuum evaporator. The residues in the tank consist mainly of resinous matter and may be dried separately. The juice is evaporated at a temperature not exceeding 70° and should be constantly agitated to prevent the formation

of a greyish, oily skin on the surface. When reduced to a grey-brown paste the contents of the evaporator are spread on glass or iron plates and dried in a drying chamber at 40—45°. The resulting brown powder can be stored indefinitely without deterioration, and on the addition of water produces a milky-white emulsion.

A. G. POLLARD.

Method of combating plant diseases. J. D. RIEDEL A.-G. (G.P. 425,496, 8.5.21).—The plants are sprayed with a solution developing sulphur and sulphur dioxide. A dilute, aqueous solution of sodium thiosulphate acidified, just before use, with 5% hydrochloric acid is suitable. A coating of colloidal sulphur becomes deposited on the plants.

R. B. CLARKE.

Material for combating plant diseases and treating seeds. R. FALCK (G.P. 425,707, 8.10.22).—A solution especially suitable for disinfecting seeds consists of an alkaline solution of a formaldehyde-phenolic resin incorporated with a copper salt. For instance, such a resin is mixed with a solution of a cupric salt. In the complex formed copper is probably present in the phenolic nucleus as it can be detected only on breaking down the molecule. The product is soluble in ammonia solution and also in fixed alkalis, in contrast to copper hydroxide. Carbon dioxide re-precipitates the compound from such solutions. When a solution in caustic soda is sprayed on to young plants a resinous film, impermeable to rain, is formed. The copper applied in this form exerts a considerably greater destructive action on fungi than a corresponding amount applied as an acid solution of copper sulphate. After destruction of fungi the copper compound is subsequently decomposed by the carbon dioxide of the air. It does not affect injuriously the germinating power of seeds.

R. B. CLARKE.

Materials for spray fluids and seed pickling. A. W. BEER (F.P. 580,438, 5.1.23).—Tanning extracts or protein precipitants with tanning properties are claimed as fungicides; e.g., 0.25—0.5% aqueous solutions of protocatechuic acid, gallic acid, or pyrogallol will kill fungus spores in 10—20 min. Tannins insoluble in water may be dissolved in appropriate organic solvents or made soluble in water by the addition of a little sulphuric acid. The fungicidal action of tannins may be increased by addition of substances which increase their diffusibility, such as mineral or organic acids, ethers, ketones, or salts. Such additions also increase the efficiency of known fungicides; e.g., the time required for a 0.5% solution of copper sulphate to kill "smut" spores is reduced from 15—16 hrs. to 8—10 min. when the solution contains 0.1% of monochloroacetic acid. The wetting power of tannin solutions is improved by addition of phenol, acetin, sulphite-cellulose waste lye, or pentosans. The above-mentioned materials can be used for the protection of seeds against animal pests, such as rodents and birds, if a small amount of naphthenic acid or its salts is added.

C. T. GIMMINGHAM.

Increasing the amount and phosphoric acid content of basic slag (G.P. 429,767).—See X.

Food and other products from fish (E.P. 246,148).—See XIX.

XVII.—SUGARS; STARCHES; GUMS.

Increase in alkalinity of sugar solutions on evaporation. B. MINAEV (Zapiski [Russia], 1925, 2, 234—239; Chem. Abstr., 1926, 20, 2085).—Small amounts of amide-nitrogen present in the beets are decomposed during evaporation; thus conditions favourable for a decrease of alkalinity could not exist. Sulphitation is preferred to carbonation at the third saturation.

A. A. ELDRIDGE.

Increase in alkalinity on evaporation of sugar juices. L. SHAPIRO (Zapiski [Russia], 1925, 3, 50—54; Chem. Abstr., 1926, 20, 2085).—Normally the [beet] syrup is unlikely to have the proper reaction after saturation with carbon dioxide at the third saturation; addition of sulphur dioxide is necessary.

A. A. ELDRIDGE.

Determination of ash in sugar manufacture by measuring the electrical conductivity of the aqueous solutions. A. G. ARKHIPOVICH (Zapiski [Russia], 1926, 3, 95—101; Chem. Abstr., 1926, 20, 2086).—The conductivity method for the determination of ash, using nickel or platinum electrodes and 2–4 c.c. of a solution of 40° Brix, diluted to 100 c.c., gives satisfactory results.

A. A. ELDRIDGE.

Practical methods of p_H measurement and the scheme of automatic electrometric control of cane juice defecation. R. T. BALCH and H. S. PAINE (Planter and Sugar Mfr., 1925, 75, 347—350; Chem. Abstr., 1926, 20, 1915).—Quinhydrone electrodes are unsuitable; for continuous recording a tungsten-manganese sesquioxide electrode is preferred. Charts are given showing changes in p_H for continuously limed defecated juices. The liquid must be cooled and sterile conditions maintained.

A. A. ELDRIDGE.

Honey [analysis]. F. LUCIUS (Z. Unters. Lebensm., 1926, 51, 351—357).—Reduction or polarisation methods of sugar determination are not satisfactory owing to the presence of other reducing substances, but a separation of sugars from dextrans can be effected by precipitation with ether in alcoholic solution. A mixture of 10 g. of honey and 10 g. of water is treated with 200 c.c. of 96% alcohol, and 100 c.c. of ether are added gradually with continuous agitation. The mixture is left for 24 hrs. and is filtered from the dextrans, which are dried and weighed. The alcohol and ether are evaporated, the residue is made up to 100 c.c., and the sugars are determined by the polarimeter after inversion. In a separate portion the levulose is decomposed by boiling for 3 hrs. with 20 c.c. of 5*N*-hydrochloric acid and then, after adding alkali till the solution is only slightly acid, the solution is filtered, decolorised with animal charcoal, and examined in the polarimeter.

W. G. CAREY.

See also A., Sept., 941, **Structure of fructose, γ -fructose, and sucrose** (HAWORTH and HIRST); **Constitution of sucrose** (McOWAN).

PATENTS.

Purifying carbohydrate solutions etc. C. B. DAVIS (U.S.P. 1,592,598, 13.7.26. Appl., 26.2.23).—Raw sugar is made into a magma with 10% of water and

centrifuged. On diluting the brown syrup thus obtained to d 1.075, some of the impurities are precipitated, and after addition of a small quantity of diatomaceous earth and heating to 70°, the solution is filtered through bone char. The resulting colourless liquid is used to dissolve the washed sugar, or other unrefined sugar, until a syrup of d 1.263 is obtained, which is purified by treatment with diatomaceous earth, heating to 70°, and filtration through bone char as before. The purified syrup is grained *in vacuo* to obtain a pure sugar.

T. S. WHEELER.

Crystallisation of sugar. C. GRAHAM (Austral. P. 19,812, 29.9.24).—A quantity of sugar juice is introduced into a vacuum pan and boiled until minute crystals of sugar are perceived in a sample of the boiling massecuite when held up before a strong light. The crystals are then caused to grow by running in a syrup which gradually increases in sugar content as the pan fills up. The crystals thus obtained are of uniform size and purity. The essential feature of the process is the blending of the syrups etc. in such a way that the crystals may be caused to grow uniformly.

R. B. CLARKE.

Synthetic bone char (U.S.P. 1,592,599).—See II.

XVIII.—FERMENTATION INDUSTRIES.

Technical control of purchase of barley, malt, and yeast for distilling. S. H. HASTIE (J. Inst. Brew., 1926, 32, 343—351).—Methods for the evaluation of barley, malt, and yeast for pot-still distillery purposes are indicated, and their value is discussed.

C. RANKEN.

Calculation of percentage of alcohol and other factors in fermented worts. A. SLATOR (J. Inst. Brew., 1926, 32, 359—360).—A table is given by means of which it is possible to calculate the percentage of alcohol present in beer, the number of degrees of gravity lost, the percentage fermented and other factors, when only the original gravity and attenuation are known. The figures in the table were calculated from the "Mean Brewery Table" of Thorpe and Brown, and are arranged in six columns, recording respectively the degrees of attenuation lost, the degrees of gravity lost per degree of attenuation lost, of alcohol per 100 c.c. per degree of attenuation lost, increase of carbon dioxide concentration per degree lost in attenuation, gravity lost, and increase of alcohol per degree of attenuation lost.

C. RANKEN.

Pectins of grapes and the mellowness of wines. L. SEMICHON and FLANZY (Compt. rend., 1926, 183, 394—396).—When a must or wine is acidified to the extent of 1% with hydrochloric acid, the addition of alcohol precipitates a mixture of pectins and gums. When the precipitate is dissolved in water the pectic acid separates as calcium pectate and the gums are separated from the filtrate by the addition of alcohol. An examination of a large number of musts and wines has shown that musts contain only pectins, whilst the wines have only gums or mixtures of gums and pectins. It is concluded that grape pectins arise as in other fruit from the decomposition of cellulose tissue, and that the formation of gums during the fermentation of a must is not due to

transformation of the pectins, but that the gums are the products of bacterial action. The bouquet of a matured wine is due largely to the dissociation of the methyl pectic esters or pectins.

A. E. MITCHELL.

Rapid determination of alcohol in distilled spirits and of colour in whisky. J. F. WILLIAMS (Ind. Eng. Chem., 1926, 18, 841—843).—The alcoholic distillate from wines and cordials and the concentrated distillates from vinegar, ale, cider etc. are extracted with a special reagent consisting of 70 c.c. of amyl alcohol, 28 c.c. of toluene, and 2 c.c. of a 50% aqueous solution of tartaric acid. The extraction is carried out at 20° in a graduated 20-c.c. glass-stoppered cylinder. The height of the lower layer, which consists of the water contained in the distillate, is read and the percentage of alcohol obtained from a table containing the results obtained from a prepared set of 24 standard alcoholic solutions ranging from 5 to 66%. No appreciable interference was noted by the presence of small amounts of glycerol or sucrose. For the determination of colour in the whisky, the cylinder containing the treated spirit is placed against a sheet of white paper, and the approximate percentage of colour in the upper and lower layers noted. So-called natural colour in whisky appears in the upper layer, while any caramel or other water-soluble colours dissolve in the lower or aqueous layers. The test is not a precision method, and is intended for use primarily where a pycnometer and balance are not available, or where the sample is too small for convenient use with these instruments.

C. RANKEN.

See also A., Sept., 976, Separation of enzymes of barley malt. Lichenase and cellobiase (PRINGSHEIM and BEISER); Protein nature of invertase (WILLSTÄTTER). 977, Formation of polysaccharides by yeast preparations (NAGANISHI). 978, Function of phosphates in dissimilation of hexoses (KLUYVER and STRUYK); Effect of organic substances on glycolysis of yeast (SCHOELLER and GEHRKE); Pyruvic acid in alcoholic fermentation (TRAETTA-MOSCA); Simpler nitrogenous constituents of yeast. Choline and nicotinic acid (VICKERY); Determinations of permeability with *Saccharomyces cerevisiæ* (SÖHNGEN and WIERINGA); Enzymic decomposition in lactic acid bacteria and yeast (NILSSON and SANDBERG); Acid production of *Aspergillus niger* (BERNHAEUER).

Lactic ferment preparations. PLACERES.—See XX.

PATENTS.

Manufacture of pressed yeast. J. I. A. EFFRONT (F.P. 601,484, 18.4.25).—Wort prepared from molasses is purified by treatment with alkali carbonates and nitrogen is passed in during fermentation, so that 30 to 150 mg. of nitrogen are present per litre of wort. The process of assimilation is hastened by leading in very small amounts of oxides of nitrogen during fermentation.

C. RANKEN.

Bulgarian milk (U.S.P. 1,593,899).—See XIX.

Preparation of citric and other organic acids (G.P. 426,926).—See XX.

XIX.—FOODS.

Determination of protein [in cereals etc.].

H. L. THOMPSON (Amer. Miller, 1926, 54, 396; Chem. Abstr., 1926, 20, 2029).—Wheat proteins (gliadin, glutenin, leucosin, globulin, and proteose) cannot be separated without change in structure. The most accurate factors for calculating total protein from the percentage of nitrogen are: wheat 5.7, rye 5.62, oats 6.31, maize, rice, mixed feed, bran, and "shorts," 6.25.

A. A. ELDRIDGE.

Influence of storage on the composition of flour.

J. E. GREAVES and C. T. HIRST (Utah Agric. Exp. Sta. Tech. Bull., 1925, [194], 1—21; Chem. Abstr., 1926, 20, 2031).—The milling of wheat in the production of high-grade white flours removes 70% of the ash, 63% of the phosphorus, 90% of the calcium, and 79% of the magnesium. During storage, the soluble carbohydrate and the water-soluble phosphorus increase, whilst the alcohol-soluble phosphorus decreases; the hydrogen-ion concentration first decreases and then increases. No evidence was obtained of protein cleavage during storage. From 1.39 to 3.52% of the nitrogen of these flours occurred as water-soluble basic nitrogen.

A. A. ELDRIDGE.

Changes in flour during storage.

J. E. GREAVES (Amer. Miller, 1926, 54, 376; Chem. Abstr., 1926, 20, 2031; cf. preceding abstract).—Only inferior flours greatly increase in acidity during storage for four years. It is concluded that the improvement of the bread-making properties of flours on storage is due to the action of enzymes on the carbohydrates, fats, and proteins.

A. A. ELDRIDGE.

Colloid chemistry of wheat gluten.

A. KUHN and G. RICHTER (Koll. Chem. Beih., 1926, 22, 421—448; cf. Ostwald, B., 1920, 499 A).—The baking quality of a flour is apparently intimately connected with the nature of the gluten which it contains, and viscosity measurements have therefore been made with sols of the gluten from various types of flour. In every case the viscosity decreases with rising temperature and increasing age of the sol. The gluten from an extract flour (degree of milling 30%) has a higher viscosity than that from a baker's flour (degree of milling 74%) made from the same wheat, the difference depending on the milling. Of the baker's flours examined, the best were those giving the most viscous gluten sols. As the gluten content of a flour decreases with increasing degree of milling, it is necessary, in order to arrive at a correct estimate of the value of the flour, that the sol examined should be of unit concentration. Measurements by Ostwald and Steiner's membrane method (A., 1925, ii, 771) show that the surface tension of the sols decreases with increasing degree of milling, and that the better quality flours give the more elastic sols. R. CUTHILL.

Practical method for the determination of the electrical conductivity of milk. V. GERBER (Z. Unters. Lebensm., 1926, 51, 336—351).—The electrical conductivity is a valuable criterion of the quality of milk, but owing to the cost of the apparatus, as well as the skill and time required, the process is not in general use. The author adopts the method used for determining

the electrical conductivity of other liquids, and an apparatus is described and illustrated in which the ordinary Wheatstone bridge method is used, with a telephone or a galvanometer to determine the position of zero current. Results obtained with many different samples of milk are given.

W. G. CAREY.

“Cryolac” number of milk and milk products as a means to determine quantity of added water. P. POST (Pharm. Weekblad, 1926, 63, 983—995).—The name “cryolac” is proposed for the osmotic concentration of lactose and chloride, expressed in Raoult units. The lactose content, chlorine content, and acidity are determined; from the first and last, by reference to prepared tables, the depression of the freezing point which should be caused by the lactose originally present is calculated, and by adding the depression which should be caused by the soluble chloride the cryolac number is obtained. This bears a constant ratio to the total depression, so that the latter may be calculated; figures so obtained for a great variety of samples agree well with figures obtained by actual determination from the milk originally used. The cryolac number of unadulterated milk being taken as 425, the determination enables the proportion of water added to the milk, examined as such or in the form of butter milk, ice cream etc. to be calculated.

S. I. LEVY.

Destruction of vitamin-A in milk by ultra-violet light. R. W. TITUS, J. S. HUGHES, W. R. HINSHAW, and J. B. FIRCH (Ind. Eng. Chem., 1926, 18, 843).—Irradiation of milk, whilst it increases the antirachitic properties (cf. Steenbock, Hart, Hoppert, and Black, A., 1926, 437), simultaneously causes a large decrease in the vitamin-A content.

J. W. BAKER.

Chemical study of the flesh of emaciated cattle. R. HOAGLAND and W. C. POWICK (J. Agric. Res., 1925, 31, 1001—1013).—Analysis of the flesh of very emaciated cattle indicated a higher moisture content and a lower content of fat, protein, and probably sugar than normal. The average ratio of moisture:protein was greater than 4:1, whereas for the flesh from fat cattle the average value was 3.5:1. This ratio is suggested as valuable for the purpose of classifying flesh for food purposes.

A. G. POLLARD.

Nutritive value of protein in veal and calf sweetbreads; in beef cheek meat, lips, tongues, brains, spleen, and tripe; and in hog brains and tongues. R. HOAGLAND and G. G. SNIDER (J. Agric. Res., 1926, 32, 679—688).—When fed to young rats, in rations containing 10% of protein, for periods of 30 or 60 days, veal, ox brain, tongue, and spleen, hog brain and tongue had approximately the same value as milk as sources of protein for growth and maintenance; ox lips, caseinogen, and beef cheek meat had a somewhat lower value; and sweetbreads and tripe were distinctly inferior. The protein in tripe was insufficient for optimum growth even when fed as 20% of the ration.

C. T. GIMMINGHAM.

Nutritive value of protein in voluntary muscle, heart, liver, and kidney, from cattle, sheep, and hogs. R. HOAGLAND and G. G. SNIDER (J. Agric. Res., 1926, 32, 1025—1040).—Feeding experiments with young albino rats are reported. The rations contained 10, 12.5, and

15% of protein from the various tissues tested and were prepared so as to have practically the same energy value. The hog tissues (at 10 and 12.5% of protein) had, on the whole, somewhat higher values for maintenance and growth than the others. The differences when 15% of protein was fed were hardly significant.



C. T. GIMMINGHAM.

Determination of moisture in meat extract. C. DEDLOW and D. T. SMITH (Ind. Eng. Chem., 1926, 18, 858—860).—The usual methods of determining moisture in meat extracts (drying *in vacuo* or in the air oven) yield results which are from 2 to 6% too high, owing to decomposition of the extract at the temperatures used (70° *in vacuo*, 100—105° in the air oven). This degradation continues over a long period, the extract apparently never reaching a constant weight. Improved results are obtained by distillation with toluene under atmospheric pressure and measuring the water carried over, but the most accurate results are obtained by distillation for 2 hrs. at 50° under a pressure of 30—50 mm. with xylene. A detailed description is given of the apparatus used and the procedure followed.

B. W. CLARKE.

Vitamin-A in poultry flesh and fat. R. HOAGLAND and A. R. LEE (J. Agric. Res., 1926, 33, 29—40).—Nutrition experiments with rats indicated that poultry fat was considerably richer in vitamin-A than the flesh. The flesh of chicken, ducks, geese, turkeys, and guinea-fowl was examined, but no definite conclusions as to vitamin content were obtainable.

A. G. POLLARD.

Variation in the moisture and nitrogen content of the potato during growth and storage. T. W. FAGAN (Welsh J. Agric., 1925, 1, 110—115).—The moisture content of potatoes gradually decreases during growth, the total nitrogen and protein nitrogen steadily increasing. The ratio of protein nitrogen to total nitrogen also increased to 55—58% at harvest, when the composition was: water, 77.33—78.12; total nitrogen, 0.316—0.297; protein nitrogen, 0.181—0.164%. The leafy portions of the plants exhibited similar variations, the composition at harvest being: water, 22.19—30.32; total nitrogen, 1.149—1.014; protein nitrogen, 0.970—0.812%. During storage the percentages of total nitrogen and dry matter slightly increased, but the ratio of protein nitrogen to total nitrogen remained fairly constant.

A. A. ELDRIDGE.

Physical and chemical changes in celery during storage. L. W. CORBETT and H. C. THOMPSON (Proc. Soc. Amer. Hort. Sci., 1925, 346—353).—Calcium pectate and pectose are converted into pectic acid and pectin more rapidly in celery treated with nitrogenous fertilisers only than after treatment with phosphorus and potash. Changes in the soluble and insoluble nitrogen, and in reducing and total sugars, are recorded.

A. A. ELDRIDGE.

Composition of loganberry juice. J. A. DAWSON (Canad. Chem. Met., 1926, 10, 81—84).—The relatively high acid and pectin contents of the loganberry make it a useful ingredient for jams and jellies, and it is probable that the fresh berry has a positive anti-scorbutic value. The juice amounts to about 70% by weight of

the berry, and the results of analyses of juice from various sources are given, approximate figures being as follows:— $d^{15.6}$ 1.038—1.048; total solids, 9.9—12.6%; reducing sugars, 5.6—7.9%; sucrose, 0.01—0.08%; alcohol precipitate, 0.36—0.56%; citric acid, 2.09—2.30%; ash, 0.312—0.514%; phosphate as P_2O_5 , 20.0—33.4 mg. per 100 c.c. C. O. HARVEY.

Detection of saccharin in foods. L. SOEP (Chem. Weekblad, 1926, 23, 361—362).—Saccharin may be extracted from cakes, pastries etc., by crushing with slaked lime and extracting with alcohol; the extract is subjected to a lengthy process of purification, and saccharin, if present, is eventually obtained by ether extraction from an aqueous solution acidified with phosphoric acid. This final extract contains also any aromatic acids present or formed during the treatment, and is tested for saccharin by taste, the resorcinol test, hydrolysis, etc. S. I. LEVY.

Detection of preservatives containing fluorine in foods. H. LÜHRIG (Pharm. Zentr., 1926, 67, 513—518, 531—535).—The methods of detecting the use of fluorine derivatives as preservatives in foodstuffs etc. are reviewed. The author advises the general use of tests depending on the method of Meyer and Schulz (Z. angew. Chem., 1925, 38, 203—206), in which the presence of fluorides is shown, in the absence of silicates and phosphates, by the precipitate given by lanthanum acetate in the presence of acetic acid and ammonium acetate, the test being confirmed by the glass-etching method. Tests for fluorides in fats, butter, margarine, milk, beer etc., are worked out on this basis.

B. FULLMAN.

Detection of colouring matters in foods. L. SOEP (Chem. Weekblad, 1926, 23, 362—365).—Metanil Yellow and Orange IV, the latter not being regarded in Holland as poisonous, are distinguished by means of cobalt acetate, manganous chloride, and silver nitrate. The material is shaken with cold 70% alcohol, the residue after filtration and evaporation dissolved in water, and the test solutions added to separate quantities. The colorations and precipitates are described. The results of numerous experiments to differentiate New Blue R and New Blue B from Water-soluble Induline are tabulated; the most suitable reagents are potassium iodide, potassium thiocyanate, and zinc iodide; the colour reactions with the common acids and alkalis on wool have been repeated, and modified results are given. S. I. LEVY.

Loss of chlorine in the incineration of foodstuffs. J. D. FILIPPO and W. ADRIANI (Z. Unters. Lebensm., 1926, 51, 374—375). J. DROST (*Ibid.*, 375—376).—Filippo and Adriani do not agree with a previous statement of Drost that loss of chlorine during incineration is due to mineral matter, especially acid phosphates, and state that there is considerable loss in phosphate-free material; also that chlorides when heated with ash-free carbon compounds, or with pure carbon itself, react and give an alkaline ash, due to sodium carbonate. Drost agrees with these statements and states that on heating a mixture of sodium chloride and potassium chloride with pure lactose there was a loss of chlorine of 22—33%.

For the determination of chlorine in foodstuffs he recommends methods which do not entail incineration.

W. G. CAREY.

Effect of milk salts on the whipping ability of ice-cream mixes. H. H. SOMMER and D. M. YOUNG (Ind. Eng. Chem., 1926, 18, 865—866).—Sodium citrate added in an amount greater than 0.4% to the ice cream before the ageing of the mix causes a marked increase in the whipping ability of the mix. Similar results are obtained by the addition of 0.7% of disodium phosphate; 0.5% of calcium lactate shows a marked lowering effect. These results indicate the possibility of effecting economies in ice-cream manufacture. B. W. CLARKE.

"Finger and toe" disease of swedes. [Composition of swedes.] T. WHITEHEAD (Welsh J. Agric., 1925, 1, 176—184).—The roots of Magnum Bonum, Danish 25, Danish 4, and Yellow Turnip varieties of swedes yielded results within the following limits:—dry matter, 8.5—11.1; total sugar, 3.32—6.25; reducing sugar in juice, 3.30—6.20; sucrose in juice, 0.15—0.31%. A. A. ELDRIDGE.

Acid formation in silage. K. SCHMIDT (Landw. Jahrb., 1926, 63, 776—779; Chem. Zentr., 1926, II., 837; cf. following abstract).—The conclusion of Mangold is confirmed that the production of acids in silage reaches a maximum after two days. Lucerne silage made in evacuated silos or in an atmosphere of an inert gas contains considerably less acetic and butyric acids than under normal conditions, and the breaking down of nitrogen compounds to ammonia is diminished, *i.e.*, the silage is of better feeding value. Addition of very dry material, *e.g.*, maize straw, to the green fodder similarly reduces the formation of butyric acid.

C. T. GIMINGHAM.

Preservation of young green fodder. II. K. SCHMIDT (Leopoldina, 1926, 2, 107—116; cf. preceding abstract).—Laboratory and large-scale experiments were made on the effect on the composition of lucerne silage of evacuating the silos after filling and then introducing carbon dioxide or carbon disulphide vapour. Feeding experiments with sheep and cows showed that the silage made in this way gave satisfactory results. In the laboratory experiments, the formation of butyric acid was entirely suppressed. Treatment with carbon disulphide also reduced the amounts of acetic acid and ammonia produced. Samples of the gases in the silos were analysed at intervals. There was a markedly greater production of hydrogen in the silage treated with carbon dioxide than in that treated with carbon disulphide; and, in the large silo, an increased amount of hydrogen in the lowest layer. Temperature changes and the rate of formation of acids in the silage are also discussed. C. T. GIMINGHAM.

See also A., Sept., 959, Tryptophan-aldehyde reaction. Determination of tryptophan in proteins (KOMM). 970, Liberation of phosphorus from caseinogen by enzymes and other agents (RIMINGTON and KAY); Effect of heat on milk (MAGEE and HARVEY). 980, Fearon's "pyrogallol" test as possible basis for determination of vitamin-A (WILLIMOT and MOORE); Solubility of vitamin-B in benzene

(WILLIAMS and WATERMAN). 981, **Antirachitic substances**. Polymerisation of cholesterol (BILLS and McDONALD); Dietary requirements for reproduction (SURE). 982, **Lecithin, kephalin, and so-called cuorin of soya bean** (LEVENE and ROLF).

Determination of fat in oil-cake. ZANDER.—See XII.

Examination of milk sweets. FINCKE.—See XII.

PATENTS.

Manufacture of food [and other] products from fish. SOC. FRANC. DES PROD. ALIMENTAIRES AZOTÉS (E.P. 246,148, 13.1.26. Conv., 14.1.25).—The fish is autolysed at 37–55° in presence of 5–10% of sodium chloride, the temperature being lower the greater the proportion of salt used. A portion of the oil collects at the surface and is recovered, e.g., by decantation. The residue is sieved and from the skeleton débris are obtained more oil and a powder constituting a manure rich in nitrogen and phosphorus. The liquid portion is filtered, and the filter residue is used for tanning or tawing or as a further source of oil and manure. The filtrate may constitute the final food product or may be concentrated to a paste. H. HOLMES.

Working up slaughterhouse offal, animal carcasses, and fish. W. STEINMANN (E.P. 256,162, 20.4.26).—The material is sterilised and disintegrated by means of steam, and the resulting pulp is dried, and then immediately extracted with a fat solvent, such as carbon tetrachloride or benzene. A better separation of the fat is obtained than with other steam processes, and there is no loss of protein. C. O. HARVEY.

Bulgarian milk. H. BUEL (U.S.P. 1,593,899, 27.7.26. Appl., 30.4.20).—Milk is freed from cream and inoculated with *Bac. lacticus Bulgaricus*, together with a type of *paralacticus*, *Streptococci lacticus*, or *Diplococci lacticus* and kept at 30–36°. The cream after inoculation as above, but without the *Bacillus lacticus Bulgaricus*, is mixed with the cultured milk, cooled to 13–18°, and, finally, to 2–8°, at which temperature the product is held for a time. C. RANKEN.

Manufacture of leavened bread. H. A. KOHMAN and R. IRVIN, Assrs. to FLEISCHMANN Co. (U.S.P. 1,593,977, 27.7.26. Appl., 13.12.19).—A substance used in the manufacture of leavened bread is prepared by cooking hominy in an aqueous solution of ammonium sulphate until the solution is absorbed by the hominy, adding calcium carbonate, and inoculating with an *Aspergillus* fungus, which is grown until the hominy is bound together. The product, after being dried at a temperature which does not injure the enzymes, is ground. C. RANKEN.

Manufacture of soluble cocoa. C. and G. BREHIER and F. P. JEAN (F.P. 601,199, 24.10.24).—Cocoa powder is treated with proteolytic enzymes to render soluble the contained proteins. C. RANKEN.

Pectin preparation. A. E. A. P. LIOT and L. A. MACÉ (F.P. 604,529, 8.1.25).—A liquid obtained by

extraction of apple and pear pulp in presence of an alkaline-earth carbonate is caused to coagulate by treatment with an extract containing pectase obtained from plants (lucerne etc.).

Purification of flour. L. G. PATROUILLEAU (F.P. 604,950, 22.10.25).—The flour is purified by an oxidising agent (NO₂) which converts the allyl sulphide in the husks into allyl sulphoxide, an odourless liquid product, soluble in water. C. RANKEN.

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

Suitability of chloroform for alkaloidal assay. H. R. WATKINS and S. PALKIN (Ind. Eng. Chem., 1926, 18, 867–869).—Purity tests commonly used to determine the suitability of chloroform for alkaloidal assays are shown to be inadequate, the specimens giving the best results by these tests often giving the largest errors. The suitability of a given sample is best determined on actual test on the alkaloid by the automatic, continuous extraction method. Unsuitable samples may be improved by prolonged refluxing with brucine, followed by recovery of the chloroform from the extract by distillation. In all cases extraction by the continuous process using benzene as a solvent gave excellent results, and this solvent is recommended as a substitute for chloroform in alkaloidal assays. J. W. BAKER.

Detection and approximate evaluation of ergot. A. TSCHIRCH (Pharm. Acta Helv., 1926, 1, 89–90; Chem. Zentr., 1926, II., 927).—If a solution of ferric chloride in concentrated sulphuric acid be introduced under a glacial acetic acid solution of histamine or tyramine (both commercially known as tenosin) an orange-yellow ring is formed; a glacial acetic solution of a trace of ergosterol shows a green fluorescence if a layer of strong sulphuric acid be introduced under it. For the identification and approximate evaluation of ergot, it is treated with ether and ammonia. A red solution is formed (sclererythrin). After 2 hrs. the ether is separated and distilled, the solution of the residue in glacial acetic acid being treated as above with a solution of ferric chloride in strong sulphuric acid. A bluish-violet coloration (ergotamine) should appear at the zone of contact, but hardly any or no yellow colour (amines derived from proteins), and the acetic acid layer should show a green fluorescence (ergosterol). For this test the ergot should contain at least 0.2% of ergotamine. B. FULLMAN.

Suitability of monel metal for vanilla flavouring containers. T. E. HOLLINGSHEAD and T. J. OTTERBACHER (Ind. Eng. Chem., 1926, 18, 871–872).—When various types of vanilla flavours, prepared from Bourbon beans and, in some cases, fortified with vanillin and coumarin, were placed in contact with monel metal, the extracts showed a slightly weaker flavour at the end of 6 weeks, although the odour seemed the same. At the end of 10 weeks both odour and flavour were weaker. Throughout 12 weeks no precipitate was formed, and the colour remained unchanged. The action in the light was the same as in the dark. The polished surface

of the metal was dulled at the end of the eighth week. The standard extract produced a greater effect than did the fortified flavours, while the corrosion was practically twice as great in the light as in the dark. The normal lead number of the true vanilla bean extracts was higher after treatment than before. This is probably due to the resins forming compounds with nickel and copper, which precipitate more lead. In the fortified extracts the normal lead number fell considerably.

C. RANKEN.

Pharmaceutical lactic ferment preparations.

J. PLACERES (Rev. Fac. Cien. Quím., 1926, 4, 73—93).—Certain commercial lactic ferment preparations are either inactive, or else harmful on account of their proteolytic properties. The method proposed for determining the activity of lactic ferment preparations consists in incubating 100 c.c. of skim milk containing 3% of lactose with 0.5 g. of the preparation at 37° for 48 hrs., and determining the acidity by titration with standard sodium hydroxide, using phenolphthalein as indicator. A control determination is also made without the preparation. Butyric acid is produced by the decomposition of fats present in the medium, and is not a secondary product of lactic fermentation. Commercial lactic acid preparations do not yield harmful volatile products.

G. W. ROBINSON.

Aliphatic tertiary alcohols and their industrial possibilities. H. S. DAVIS and W. J. MURRAY (Ind. Eng. Chem., 1926, 18, 844—846).—The physical and chemical properties of the tertiary aliphatic alcohols are compared with those of the primary and secondary, and their possible industrial uses outlined. As a general rule the b.p., density, and solubility in water of the tertiary alcohol are similar to those of the homologous secondary alcohol with one carbon atom less, or the primary alcohol with two carbon atoms less. Chemically, the tertiary alcohols are characterised by their ease of dehydration (*e.g.*, *tert.*-butyl alcohol yields *isobutylene* on treatment with oxalic acid), lability of the hydroxyl group, resistance to oxidation, stability towards halogens, and very slow reaction with sodium. With acetyl chloride *tert.*-butyl alcohol yields *tert.*-butyl chloride and acetic acid.

J. W. BAKER.

Comparative tests on the sensitiveness of some usual reagents for methyl alcohol. B. OLSZEWSKI (Rocz. Farm., 1925, 3, 77—88; Chem. Zentr., 1926, II., 801—802).—The author has investigated dilute solutions of methyl alcohol in water and ethyl alcohol (the latter sometimes containing fusel oil) by oxidising with potassium permanganate and testing for formaldehyde. The most characteristic test is that with apomorphine hydrochloride in sulphuric acid, which will indicate 1% of methyl alcohol in water and ethyl alcohol mixtures after direct oxidation, 0.2% on oxidation after one distillation, and 0.1% on oxidation after two distillations. In the absence of other aldehydes, 0.001% of formaldehyde (or 0.02% of methyl alcohol on oxidation) gives a positive reaction on pouring the liquid on to the apomorphine solution without mixing, a coloured ring being formed.

B. FULLMAN.

Acidimetric and thiocyanometric determination of mercuric chloride [in sublimate pastilles].

E. RUPP with K. MÜLLER and P. MAISZ (Pharm. Zentr., 1926, 67, 529—531).—Mercuric chloride in pastilles containing it may be thus determined: the solution of the substance is added to alkaline (sodium hydroxide) hydrogen peroxide, whereby the mercuric chloride is reduced to mercury thus: $\text{HgCl}_2 + 2\text{NaOH} = \text{HgO} + 2\text{NaCl} + \text{H}_2\text{O}$, followed by $\text{HgO} + \text{H}_2\text{O}_2 = \text{Hg} + \text{H}_2\text{O} + \text{O}_2$. The excess of sodium hydroxide is titrated with hydrochloric acid. Alternatively, the mercury obtained as above is coagulated by addition of hydrochloric acid, the supernatant liquid decanted, and the mercury dissolved in concentrated nitric acid, excess of the latter being removed by addition of 1% permanganate solution to permanent coloration. After decolorising with a crystal of ferrous sulphate, iron alum solution and dilute nitric acid are added, and the solution is titrated with standard ammonium thiocyanate. Organic substances etc. retard the coagulation of the mercury, and in the case of pastilles the acidimetric method is therefore preferable.

B. FULLMAN.

Loss of free iodine from alcoholic solutions of varying purity and the effect of potassium iodide.

R. M. CARTER (Ind. Eng. Chem., 1926, 18, 827—828).—The loss of iodine from ethyl alcoholic solutions on keeping is greatly influenced by the purity of the solvent, being much greater when low-grade alcohol is used. Except with such alcohol, addition of 75% of the theoretical amount of potassium iodide required to form KI_3 largely inhibits the loss, which is then less than 2% after 1 year. The loss of iodine from solutions in commercial methyl alcohol is due mainly to the impurities present in the solvent.

J. W. BAKER.

Geraniol and its determination; notes on citronellol.

L. G. RADCLIFFE and E. CHADDERTON (Perf. Ess. Oil Rec., 1926, 17, 254—264, 350—355).—Methods for the determination and purification of geraniol and citronellol have been examined and are critically reviewed. For the determination of geraniol in commercial samples, the usual acetylation method is the best. The method of Verley and Bölsing (A., 1902, ii., 54), *i.e.*, acetylation in presence of pyridine, is shorter, but gives results about 2% low. A new method is described in which phthalic anhydride in presence of pyridine is used for acylation. This gives results about 2% low, but is more convenient than the pyridine-acetic anhydride method. For the determination of citronellol in commercial samples the acetylation method is again superior, but the new method gives results almost as good. In presence of pyridine, phthalic anhydride is selective in its action on alcohols, linalool, for example, not being attacked, and this method is the best for the determination of free geraniol in citronella oil, but for the total alcohols the figure given by Verley and Bölsing's method is the most accurate, Schimmel's method often giving variable results. For the determination of citronellol in presence of geraniol, the formylation process is unsatisfactory, and it is suggested that the process of Tiemann and Schmidt (A., 1896, i, 382), *i.e.*, isolation of the citronellol with phosphorus trichloride, would give a more correct figure. A rapid test for determining the

purity of geraniol samples has been devised, based on the speed of the reaction of geraniol with finely ground calcium chloride and the hardness of the product; the presence of quite small amounts of impurities has a very pronounced effect on the rate of formation and consistency of the additive product, which has been shown to have the constitution, $\text{CaCl}_2 \cdot 2\text{C}_{10}\text{H}_{17} \cdot \text{OH}$. E. H. SHARPLES.

Effect of the hydrogen-ion concentration of the soil on the formation and composition of essential oil of wood sage. H. and H. DEEL (Bull. Soc. chim., 1926, [iv], 39, 946—948).—Under comparable conditions, the largest crop of wood sage, *Salvia sclarea*, is obtained from a soil of p_{H} approx. 4.5, and the resulting essential oil has also a maximum ester content. The content of essential oil in the plants is, however, almost independent of the p_{H} variation. Plants from a soil of p_{H} 4.65 yielded 0.157% of essential oil, d^{15} 0.897, $[\alpha]$ —18.4°, n 1.4757, acid value 0.28, containing 46.55% of esters. H. E. F. NOTTON.

Determination of essential oils in spices. C. GRIEBEL (Z. Unters. Lebensm., 1926, 51, 321—324).—A mixture of 10 g. of ground spice with 300 c.c. of distilled water is distilled, and 200 c.c. of the condensed distillate are received in a separating funnel. This distillate is then shaken with 60 g. of common salt and 20 c.c. of pentane, and after complete settling, the pentane layer is transferred to a weighed flask, and the pentane is evaporated on a water bath, the last traces being removed by a dry air blast. The residue of essential oil is dried in a desiccator and weighed. The pentane used must not leave a residue on evaporation, and must be free from non-volatile or high-boiling impurities. W. G. CAREY.

German essential flower-extract oils. W. TREFF, F. RITTER, and H. WITTRISCH (J. pr. Chem., 1926, [ii], 113, 355—360).—Essential oils from a number of German-grown plants, obtained by extraction with light petroleum, solution in alcohol, and triple steam distillation (cf. Soden, A., 1904, i, 515), have been examined. Leaves of *Viola rossica*, "Queen Charlotte," yield 0.0166% of yellowish oil with a stupefying odour, optically inactive, d^{15} 0.912, acid value 52, ester value 76.1, acetyl value 172. Flowers, with calyx-leaves, of the carnation, *Dianthus caryophyllus*, L., yield 0.0498% of a pale yellow oil with a pleasant carnation odour, d^{15} 1.010, α —0° 36', acid value 28, ester value 132, acetyl value 249. Flowers of the German jasmin, chiefly *Coronarius*, yield 0.06% of a yellowish oil, with a powerful, characteristic odour, differing from that of true jasmin oil, optically inactive, d^{15} 0.947, acid value 28, ester value 73, acetyl value 224, probably containing methyl anthranilate (cf. Verschaffelt, Chem. Weekblad, 1908, 25). Yellow lupin flowers, *Lupinus luteus*, L., yield 0.0195% of a yellowish oil with a penetrating, sweetish odour, d^{15} 0.900, α_{D} +7° 30', acid value 38, ester value 31, acetyl value 143 (cf. Schimmel's Ber., 1926, 195). Flowers of broom, *Genista tinctoria*, L., yield 0.0364% of a yellowish oil, of oppressive odour, d^{15} 0.9335, α_{D} —9° 10', acid value 18, ester value 35, acetyl value 156.

F. G. WILLSON.

Evolution of synthetic medicinal chemicals. H. V. ARNY (Ind. Eng. Chem., 1926, 18, 949—952).

Fifty years' progress in standardisation and evaluation of medicinals. P. N. LEECH (Ind. Eng. Chem., 1926, 18, 953—954).

Development of the aromatic chemical industry from 1876 to 1926. M. SZAMATOLSKI (Ind. Eng. Chem., 1926, 18, 933).

See also A., Sept., 954, Sesquiterpene family in camphor blue oil (KOIKE). 959, Synthesis of 5- β -aminocethylacridine (JENSEN and HOWLAND). 963, Alipine hydrochloride (SEVILLA); Alkaloids of *Calumba* root (SPÄTH and BURGER). 964, Synthesis of cryptopine and protopine (HAWORTH and PERKIN). 965, Alkaloids of *Corydalis cava* (SPÄTH and MOSETTIG); Colour reactions of morphine (EKKERT); Reactions of certain nitro-compounds with alkaloids (NAVARRO); Synthesis of iodine compounds of the salvarsan group (MACALLUM). 967, Determination of small quantities of water in methyl alcohol (RISING and HICKS); Thalleioquinine reaction (HARGREAVES); Electrometric titration of alkaloids. Application of the quinhydrone electrode (WAGENER and MCGILL). 974, Chemical constitution and physiological action. Position isomerism in relation to miotic activity of some synthetic urethanes (STEDMAN). 979, Preparation and properties of purified diphtheria toxoid (WATSON and LANGSTAFF). 980, Preparation, properties, and source of parathyroid hormone (DAVIES, DICKENS, and DODDS). 981, New Zealand pepper-plant (FINLAY). 982, Kombe-strophanthin- β and other Kombe-strophanthins (JACOBS and HOFFMANN); Composition of Scilla and its tonicardiac principle (HENRIJEAN and KOPACZEWSKI).

Additive products of iodine monobromide and hypiodous acid with unsaturated compounds. HOLDE and GORGAS.—See XII.

PATENTS.

Manufacture of benzimidazoles. FARBENFABR. VORM. F. BAYER & Co. (E.P. 243,766, 1.12.25. Conv., 1.12.24).—*o*-Phenylenediamine or its homologues and derivatives react with alkoxy-acids or their derivatives, yielding, directly or via an intermediate compound, 2-alkoxymethylbenzimidazoles, the latter being anti-pyretics. For example, 1 pt. of *o*-phenylenediamine and 3 pts. of ethoxyacetic anhydride are heated together for 5 hrs. at 120°. Addition of water precipitates the diacyl-*o*-phenylenediamine, which on heating for 1 hr. at 180° yields 2-ethoxymethylbenzimidazole, m.p. 153°. The following are also described:—2-ethoxymethyl-5-ethoxybenzimidazole hydrochloride, m.p. 162°; 2-phenoxy-methylbenzimidazole, m.p. 190°, and 2-phenoxy-methyl-5-ethoxybenzimidazole, m.p. 129°. B. FULLMAN.

Manufacture of alkylated aromatic sulphonic acids. I. G. FARBENIND. A.-G. (E.P. 246,817, 9.1.26. Conv., 30.1.25).—One or more *n*-butyl groups may be introduced into the nuclei of aromatic sulphonic acids by condensing the latter with *n*-butyl alcohol, with or without condensing agents, such as sulphuric acid, or

by using the salts of the sulphonic acids, in presence of a condensing agent, or by condensing aromatic hydrocarbons with *n*-butyl alcohol in the presence of a condensing agent, subsequently (or when sulphuric acid is used, simultaneously) sulphonating. For example, naphthalene, *n*-butyl alcohol, and sulphuric acid of *d* 1.84 are heated together. From the reaction product butylated naphthalenesulphonic acid may be isolated.

B. FULLMAN.

Manufacture of the pure glucoside of *Bulbus scillæ*. A. HOME-MORTON. From F. HOFFMANN-LA ROCHE & Co. (E.P. 255,689, 14.10.25).—The pure glucoside of *Bulbus scillæ* (sea-onions) is obtained by removing the glucoside from the aqueous extract of the fresh or dried drug by means of an adsorbing agent such as charcoal or fuller's earth, and extracting the latter with an anhydrous organic solvent. For example, the charcoal is extracted with hot chloroform, the latter distilled off, the residue dissolved in methyl alcohol, and the inactive substances present removed by extraction with light petroleum. The methyl alcohol is distilled off and the residue dissolved in absolute alcohol, from which the pure glucoside is precipitated by pouring into ether.

B. FULLMAN.

Process for making arsonophenylaminoalcohols. E. C. R. MARKS. From ABBOTT LABORATORIES (E.P. 255,971, 6.5.25).— ω -Chloroalkyl chloroformates condense with equimolar proportions of arsanilic acids or their substituted derivatives to give ω -chloroalkyl arsinophenylcarbamates, which, on refluxing with excess of alkali and acidification yield arsinophenylaminoalcohols of the type $H_2O_3As \cdot R \cdot NH \cdot [CH_2]_n \cdot OH$, and where R is a benzene (or substituted benzene) nucleus. These substances are used in the treatment of syphilis, especially in the tertiary stage. The ω -chloroalkyl chloroformates are prepared from alkylene chlorohydrins of the type $Cl \cdot [CR_2]_n \cdot OH$ (where R = H or alkyl) and phosgene. For example, 21.7 g. of *p*-arsanilic acid are dissolved in 200 c.c. of water containing 4 g. of sodium hydroxide, and 14.2 g. of β -chloroethyl chloroformate are added, the temperature being kept below 35°. After acidification, the precipitated β -chloroethyl *p*-arsinophenylcarbamate is filtered off. On refluxing it for 4 hrs. with 50 c.c. of 20% sodium hydroxide and 50 c.c. of water, cooling, and adding hydrochloric acid, β -[*p*-arsinophenylamino]ethyl alcohol, m.p. 171—173° (decomp., corr.), separates. The following are also described: γ -[*p*-arsinophenylamino]propyl alcohol, m.p. 167—168° (decomp., corr.), from γ -chloropropyl *o*-arsinophenylcarbamate, m.p. 239—240° (decomp., corr.); β -[*o*-arsinophenylamino]ethyl alcohol, m.p. 145—146° (decomp., corr.), from β -chloroethyl *o*-arsinophenylcarbamate, m.p. 156—157° (decomp., corr.); γ -[*o*-arsinophenylamino]propyl alcohol, m.p. 84—85° (decomp., corr.), from γ -chloropropyl *o*-arsinophenylcarbamate, m.p. 139—140° (decomp., corr.); and β -[2-methyl-5-arsinophenylamino]ethyl alcohol, m.p. 144—145° (decomp., corr.), from β -chloroethyl 2-methyl-5-arsinophenylcarbamate, m.p. 194—195° (decomp., corr.).

B. FULLMAN.

Sulphonated thianthrens useful as pharmaceutical compounds. H. HERMINGHAUS, Assr. to

WINTHROP CHEMICAL Co. (U.S.P. 1,589,390, 22.6.26. Appl., 24.5.24. Conv., 4.8.23).—Thianthren, or its derivatives, e.g. dimethylthianthren or the compounds obtained by the methods described in U.S.P. 1,426,430 and 1,427,182 (B., 1922, 786, 837), are treated with sulphuric acid or chlorosulphonic acid at 100—160° until the product is completely soluble in water. The sodium salt of the monosulphonated thianthren derivative thus obtained is readily soluble in water, and has a strong "antiphlogistical" action. T. S. WHEELER.

Catalytic oxidation of organic compounds. C. R. DOWNS (U.S.P. 1,589,632, 22.6.26. Appl., 21.1.24).—In the oxidation of toluene to benzaldehyde by passing it in the vapour phase with air over an oxide of molybdenum at 525°, this optimum temperature is readily maintained and excess heat of reaction removed by surrounding the catalyst chamber with sulphur boiling under reflux at the pressure corresponding to that temperature. The method is of wide application in the temperature control of catalytic reactions. T. S. WHEELER.

Anæsthetic compound. R. ADAMS, E. E. DREGER, and E. H. VOLWILER, Assrs. to ABBOTT LABORATORIES (U.S.P. 1,590,792, 29.6.26. Appl., 22.8.24).—The *p*-aminobenzoates of amino-alcohols having the general formula $CHMe(NR_2) \cdot CH_2 \cdot OH$ (R = alkyl) have anæsthetic properties both in the form of the free base and as the mineral acid salt. They are prepared by treating the amino-alcohol in benzene solution with *p*-nitrobenzoyl chloride and reducing the nitro-group in the product. β -Diethylamino-*n*-propyl alcohol, b.p. 166—169°, $d^{27} 0.8665$, $n_D^{24} 1.4305$, β -diethylamino-*n*-propyl *p*-nitrobenzoate hydrochloride, m.p. 155—156°, β -diethylamino-*n*-propyl *p*-aminobenzoate hydrochloride, m.p. 159—160°, β -di-*n*-butylamino-*n*-propyl alcohol, b.p. 112—114/10 mm., $d^{20} 0.8533$, $n_D^{18} 1.4426$, β -di-*n*-butylamino-*n*-propyl *p*-aminobenzoate monohydrochloride, m.p. 200—201°, β -diallylamino-*n*-propyl alcohol, b.p. 145—147/123 mm., $d^{20} 0.9103$, $n_D^{20} 1.4466$, β -diallylamino-*n*-propyl *p*-aminobenzoate monohydrochloride, m.p. 182—183°, β -*n*-butylallylamino-*n*-propyl alcohol, b.p. 123—125/130 mm., and β -*n*-butylallylamino-*n*-propyl *p*-aminobenzoate monohydrochloride are described.

T. S. WHEELER.

Manufacturing benzoic acid from benzotrichloride. F. E. STOCKELBACH, Assr. to MATHIESON ALKALI WORKS (U.S.P. 1,591,245, 6.7.26. Appl., 26.5.21. Renewed 20.2.25).—700 pts. of calcium carbonate or other alkaline-earth carbonate are suspended in 3000 pts. of water at 80—90°, and 760 pts. of crude benzotrichloride are added with agitation during 3—4 hrs. Heating is continued for 1 hr. and the calcium benzoate formed is filtered off.

T. S. WHEELER.

Preparation of micro-organisms free from nucleic acids, containing only lipoids and lipoproteins. BEHRINGSWERKE A.-G. (G. P. 424,657, 2.11.24).—The cells of micro-organisms are treated with mineral acids, to hydrolyse the nucleoproteins and all the acid proteins, the resulting acid-soluble products being washed out of the cells. The micro-organisms thus treated serve as intermediate products for therapeutic preparations. For example, pressed yeast is shaken for several hours in 6.5% nitric acid, and the simpler

nucleic acids are subsequently washed out with water. The cells then contain only lipoids and lipoproteins, and the yeast is free from purine bases. If pressed yeast be treated with 5% sulphuric acid or dilute hydrochloric acid the nuclear proteins remain in the cells.

B. FULLMAN.

Preparation of metal-containing yeasts and other micro-organisms. BEHRINGSWERKE A.-G. (G.P. 424,658, 4.11.24).—Micro-organisms are treated with solutions of metal salts, and the cells washed with distilled water till the wash-water is free from metal. The products are free from nucleic acids and are better tolerated therapeutically than the known metal nucleates or yeast-metal compounds. For example, a uniform suspension of yeast is poured into 2% silver nitrate, and after 24 hrs. the supernatant liquid decanted off and the silver-yeast washed with distilled water. It has a high silver content. If it be washed with physiological sodium chloride solution or tap water, almost all the silver is washed out from the cells, and simultaneously proteins pass out. Mercury-containing yeast cells are obtained if 2% mercuric chloride be used.

B. FULLMAN.

Preparation of aqueous emulsions and sols. E. MERCK, Assees. of W. EICHHOLZ (G.P. 424,748, 31.3.22. Addn. to 330,673, B., 1924, 35).—In place of the camphor of the original patent, substances insoluble in water but soluble in lipoids, fats, or lipid solvents or in lipid solutions (except cholesterol) are used to emulsify fatty acid salts of bismuth, copper, silver, mercury, quinine and its derivatives. For example, bismuth linoleate and lecithin are dissolved in ether, the ether is evaporated off, and the residue emulsified with distilled water.

A. DAVIDSON.

Preparation of aqueous emulsions and sols. E. MERCK, Assees. of W. EICHHOLZ (G.P. 426,743, 31.3.22. Addn. to 424,748; cf. preceding abstract).—The lipoids, or the lipoids and fatty acid salts together, specified in the previous patent may be emulsified by dissolving them in easily volatile solvents which are miscible with water, adding water to the solution, and driving off the solvent. For example, lecithin is dissolved in warm alcohol, the solution filtered, poured into distilled water, and evaporated in a vacuum. There is thus obtained a 10% lecithin emulsion completely free from alcohol, of high dispersion and good stability.

A. DAVIDSON.

Preparation of 5:7-di-iodoindole-3-acetic acid and its homologues. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of W. SCHOELLER and K. SCHMIDT (G.P. 425,041, 28.5.24).—2:4-Di-iodophenylhydrazine is condensed with aldehyde-acids or their esters and the condensation products are heated with alcoholic acids. For example, γ -aldehydopropionic acid is refluxed at 100° with an acetic acid solution of 2:4-di-iodophenylhydrazine for 2 hrs. The resulting 2:4-di-iodophenylhydrazide of the γ -aldehydopropionic-2:4-di-iodophenylhydrazene, m.p. 185°, is boiled with alcoholic sulphuric acid for 6 hrs. Di-iodophenylhydrazine splits off and 5:7-di-iodoindole-3-acetic acid, m.p. 106°, is formed. Similarly, from δ -aldehydopropionic acid is obtained 5:7-di-iodoindole-3-propionic acid, m.p. 111°. The products find therapeutic application. A. DAVIDSON.

Preparation of substances which alone or with other greasy substances form salves. A. ENGEL (G.P. 425,331, 20.6.23).—The fatty acids from marine animal fats are treated with halogens in the presence of red phosphorus or the halides or oxyhalides of phosphorus or sulphur, the halogenated derivatives obtained being heated with water or ammonia. The products swell up in water, in which they are insoluble. Vaseline or animal fat mixed with 5% of these products takes up large quantities of water, forming a stable grease. For example, the dry fatty acids from fish oil are ground with red phosphorus. Chloroform, benzene, or light petroleum is added, and chlorine passed in. On pouring into water, a mass is obtained which melts to a grease on heating to 100°; or the chlorinated liquid may be poured into aqueous ammonia and boiled; or gaseous ammonia may be passed into the chlorinated liquid. B. FULLMAN.

Manufacture of technical triacetin. M. SONN (G.P. 425,611, 30.4.24).—A mixture of glycerol, fused sodium acetate, and glacial acetic acid is treated at 100–110° with a slow current of anhydrous hydrogen chloride; the liquid is separated from the solid sodium chloride by suction, and the crystals are washed with glacial acetic acid. The triacetin is recovered from the filtrate by distillation without steam at atmospheric pressure. By redistillation a yield of 85–95% of the theoretical of colourless and odourless triacetin is obtained free from chlorine and acrolein, whilst the excess of acetic acid is recovered in the glacial form for use again in the process.

A. R. POWELL.

Preparation of 6-nitro-3:4-dimethoxytoluene (6-nitrohomoveratrole). E. MERCK, Assees. of M. OBERLIN (G.P. 425,739, 7.8.24).—6-Nitro-3:4-dimethoxybenzaldehyde is condensed with hippuric acid and the oxazolone derivative so formed is hydrolysed with alkali. For example, a mixture of 6-nitro-3:4-dimethoxybenzaldehyde, hippuric acid, anhydrous sodium acetate, and acetic anhydride is gently boiled under reflux for 1 hr. The cooled mixture is extracted with boiling water or acetone and crystallised from ethyl acetate and then from alcohol, when 2-phenyl-4-[3':4'-dimethoxy-6'-nitrobenzylidene]oxazolone, m.p. 220.5–221°, is obtained. The crude oxazolone derivative is boiled under reflux with 10% sodium hydroxide for 4 hrs. to obtain 6-nitro-3:4-dimethoxytoluene, m.p. 117.5–118°; yield 85%. It is used as intermediate in the synthesis of therapeutic compounds.

A. DAVIDSON.

Preparation of carbamide from cyanamide. H. GOLDSCHMIDT & VON VIETINGHOFF CHEM.-TECHN. GES.M.B.H. (G.P. 426,671, 9.3.20).—Adsorption compounds of the heavy metals with the hydrogels of silicic acid, zirconium dioxide, titanate, thorium dioxide, and the like are used as catalysts. They are prepared by filtering an alkaline solution of the heavy metal concerned through the gel. For example, a 25% cyanamide solution is mixed with copper-silicic acid, obtained by filtering an ammoniacal cupric oxide solution through silicic acid gel, and kept for some time with frequent agitation. After 3 hrs., 39%, after 24 hrs. 54%, and after 72 hrs. over 70% of the cyanamide is converted into carbamide.

A. DAVIDSON.

Preparation of tartaric acid. DIAMALT-A.-G. (G.P. 426,864, 6.10.20, and 427,415, 26.11.21).—Dextrose in aqueous solution or suspension is treated with hydrogen peroxide or other peroxides, *e.g.*, those of potassium, sodium, calcium, or barium, in presence of organised catalysts, especially peroxydases and oxydases. As peroxydase solution, an aqueous extract of dormant corn embryos, especially rye embryos, is used. The process is carried out in presence of carbonates of the alkaline-earths, and pure carbon dioxide is passed through the reaction mixture during oxidation. For example, to a 5–10% dextrose solution at 25–30° a concentrated fresh peroxydase solution is added and, slowly and regularly, an aqueous solution of pure hydrogen peroxide run in, with constant stirring. Calcium carbonate is added to neutralise the resulting acid which, at a certain p_H , would stop the action of the catalyst. Finally, the liquid is heated to boiling point, when calcium tartrate separates. The yield of calcium tartrate is 40% of the dextrose used, but this is raised to 45% if pure carbon dioxide is passed in during oxidation. A. DAVIDSON.

Preparation of citric and other organic acids [by fermentation]. R. FALCK (G.P. 426,926, 31.5.21).—*Hyphomycetes* (thread fungi) are allowed to grow on solid substrates containing starch, and the mixture is worked up in the usual way for the resulting organic acids. Since *hyphomycetes* of the species *Aspergillus*, *Citromyces*, and *Penicillium* are not sensitive to tannins and poisons, starch-containing materials unsuitable for food or fodder purposes, such as horse-chestnuts etc. may be used. Examples are given of the preparation of a mixture containing citric, succinic, malic, and tartaric acids from wheat flour, which is rubbed up with ammonium nitrate, calcium carbonate, and water, spread out hot on glass plates, and, after cooling, inoculated with *Citromyces D.* After keeping for 11 days at 13°, the mass is dried and powdered. The yield of acid is 25%. (Cf. Falck and van Beyma thoe Kingma, B., 1924, 649.)

A. DAVIDSON.

Preparation of quinine solutions suitable for subcutaneous injection. CHEM.-PHARM. A.-G. BAD HOMBURG (G.P. 427,668, 15.7.24).—Stable quinine solutions of high concentration in olive oil and similar oils may be obtained by the addition of terpenes or of essential oils containing them. For example, a 10% solution of quinine may be obtained by dissolving quinine in terpineol and adding olive oil. B. FULLMAN.

Method for working up animal organs. G. EICHELBAUM (G.P. 428,531, 1.12.22).—Animal organs are mixed with efflorescent indifferent salts, and the plasmolysing mixtures are dried by warming them in a stream of dry, warm air, with or without a vacuum. Stable preparations are obtained, the active components being unchanged. Extraction with solvents yields the lipoids, *e.g.*, the phosphatides dissolved in fat may be thus obtained, and on addition of indifferent fat and removal of the solvent may be used for injection. The hormones are extracted from the dry, fat-free residue with water, albumin being removed by concentration and excess salts by intense cooling. During the working up small quantities of phenol may be added. For example, finely ground testicle is mixed with anhydrous

sodium sulphate, and dried at 40–50° as above. After a few hours the material is dry, friable, and almost indefinitely stable. B. FULLMAN.

Antiseptic anæsthetic medicament. F. K. THAYER, ASSR. to ABBOTT LABORATORIES (U.S.P. 1,596,259, 17.8.26. Appl., 27.2.24).—See E.P. 230,089; B., 1925, 900.

Manufacture of methyl alcohol (F.P. 605,530).—See II.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic sensitivity; a colloid-chemical problem. S. E. SHEPPARD (Colloid Symposium Monograph, 1925, 3, 76–98; Chem. Abstr., 1926, 20, 1959).—The accessory sensitivity of high-speed silver bromide in the blue and violet is not due to an optical sensitiser but follows the same absorption as that of the silver halide; hence the sensitivity-promoting substance does not affect the photochemical reaction. Evidence is adduced that this substance consists of silver sulphide nuclei acting by earlier formation of development nuclei in the production of the latent image.

A. A. ELDRIDGE.

Effect of desensitisers in development. J. I. CRABTREE and M. L. DUNDON (Brit. J. Phot., 1926, 73, 404–406, 418–421, 434–437).—The efficiency of certain Wratten safelights for use with different types of emulsion is discussed. The essential requirements of practical desensitisers are considered, and the properties of Phenosafranine, Pinakryptol Green, Pinakryptol Yellow, Basic Scarlet N, and Aurantia are studied in the light of these requirements. The limits of safety in the use of Pinakryptol Green with motion-picture negative and panchromatic emulsions are determined. Desensitising by Pinakryptol Green is directly proportional to the concentration of dye over the ranges studied. Desensitising with Pinakryptol Green reduces the colour sensitivity of a sensitised emulsion much more than it does the blue sensitivity. Pinakryptol Yellow is more effective and Phenosafranine less effective in lowering colour sensitivity. With increased time of bathing in desensitiser, sensitivity falls off very rapidly at first, but after about 5 min. the rate of decrease is very low. The comparative safety to Wratten safelights of untreated film and film desensitised for varying times with varying concentrations of Pinakryptol Green was studied. By bathing panchromatic film in 1 : 10,000 Pinakryptol Green, or after it has been in a developer containing 1 : 25,000 of the dye for 2 or 3 minutes, the film may be safely inspected with a Series 4 Wratten safelight containing a 25-watt lamp at a distance of 1 ft. Under similar conditions motion-picture negative film may be safely examined with a Series 0 safelight. A latent image on a desensitised emulsion tends to bleach when exposed to red light. The bleaching is most marked with non-colour-sensitive emulsions; with panchromatic emulsions it is not serious, and after development has commenced no appreciable bleaching occurs. With desensitised non-colour-sensitive emulsions the safe time of exposure to a red safelight is determined by the time required to destroy the latent image, and not the time

required to produce fog. Data are given for the fogging action of desensitisers with a number of developers, and an exhaustive study is recorded of the effect of Pina-kryptol Green used in the developer instead of as a preliminary bath. No desensitiser is known which is stable in a developer rich in quinol. Desensitisers affect the rate of development to an extent which varies with the developer and with the dye used. W. CLARK.

Production of photographic images with diazo-compounds. M. BATTEGAY and P. BRAUN (Bull. Soc. Ind. Mulhouse, 1926, 92, 274—276).—Paper is bathed in a solution of Diazo Yellow Light G or 2 G (Bayer), washed, diazotised in the dark in a solution of sodium nitrite acidified with hydrochloric acid, and dried at a low temperature. Printing is carried out under a negative in the shade, and the back of the paper is given a uniform exposure to prevent its coloration in the subsequent processes. After exposure the print is treated in a bath containing a coupling agent, the most satisfactory of which are resorcinol (for yellow), phenylmethylpyrazolone (red-purple), α -naphthylamine (deep violet), chromotropic acid (garnet), and H-acid (violet). The stability to light of the images is further increased by bathing in copper sulphate solution, or basic dyes such as Methylene Blue or Malachite Green. W. CLARK.

Fifty years of photography. C. E. K. MEES (Ind. Eng. Chem., 1926, 18, 915—916).

PATENTS.

Photographic film and method of marking it. E. TAYLOR and E. F. CHANDLER, Asses. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,597, 29.6.26. Appl., 17.6.24).—A cellulose film prepared as described in U.S.P. 1,590,593 (p. 818) is impregnated with a sensitive silver salt for photographic purposes. For instance, to sensitise the film it is impregnated on one side with silver nitrate solution and then exposed to the action of a halogen. The silver halide is formed inside the film so that scratching of the latter is not injurious to the photograph. R. B. CLARKE.

Production of photographic gelatin reliefs. S. DE PROCODINE-GORSKY and N. POZNIAKOW (E.P. 243,338, 10.11.25. Conv., 19.11.24).—See F.P. 589,422; B., 1926, 220.

XXII.—EXPLOSIVES; MATCHES.

Laws of combustion of colloidal powders. II. H. MURAOUR (Bull. Soc. chim., 1926, [iv], 39, 1115—1119).—Detailed results are given in support of the generalisations previously stated (B., 1926, 722), and showing that nitroglycerin powders having very different explosion temperatures obey the same laws of combustion. R. CUTHILL.

Explosives, 1876—1926. H. SCHLATTER (Ind. Eng. Chem., 1926, 18, 905—907).

PATENTS.

Blasting-powder composition. C. D. PRATT, Assr. to ATLAS POWDER Co. (U.S.P. 1,590,393, 29.6.26. Appl., 4.6.25).—The addition of 5—35% of a carbohydrate, such as starch or cellulose, and of 1—15% of an inorganic chloride, such as ammonium chloride, sodium chloride, or barium chloride, to black blasting powder yields a

cool explosive which may be safely used in gassy or dusty mines. T. S. WHEELER.

Making smokeless powder and the like. E. TAYLOR, Assr. to TAYLOR LABORATORIES, INC. (U.S.P. 1,590,598, 29.6.26. Appl., 17.6.24).—Cellulose obtained as described in U.S.P. 1,590,594 (p. 818) is partially dried after leaving the washing tank and granulated in the manner usually adopted for smokeless powders. If a dense powder is desired, the grains are thrown, in a continuous stream, into a mixture of nitric and sulphuric acids. After nitration they are placed in a vessel, perforated at the bottom, and the nitrating mixture is removed by displacement with weak acid. The product is then centrifuged, boiled with water, and dried. The smokeless powder thus obtained consists of tough, dense grains of glass-like appearance. R. B. CLARKE.

Dynamite composition. W. R. SWINT, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,594,861, 3.8.26. Appl., 13.4.22).—An explosive of low density contains about 7—15% of a normally liquid high explosive, about 5—15% of finely divided balsa wood, and ammonium nitrate. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Chlorine treatment of water and sewage. G. ORNSTEIN (Z. angew. Chem., 1926, 39, 1035—1037).—A résumé of the work carried out in Germany since 1920, fully confirming the advantages of the treatment. S. I. LEVY.

Clinical and laboratory investigation of the effect of metallic zinc, of zinc oxide, and of zinc sulphide upon the health of workmen. R. P. BATCHELOR, J. W. FEHNEL, R. M. THOMSON, and K. R. DRINKER (J. Ind. Hyg., 1926, 8, 322—363).—Detailed clinical and laboratory studies on 24 workmen of the New Jersey Zinc Co. over periods varying from 2 to 35 years, during which they were exposed to the inhalation of fumes of metallic zinc, zinc oxide, and zinc sulphide produced from ores containing only very small proportions of lead, cadmium, and arsenic, have shown that no chronic or acute illnesses follow prolonged exposure to zinc fumes, and that all diseases previously ascribed to zinc poisoning must be due to the presence of toxic impurities, such as lead, cadmium, arsenic, and antimony, which are usually associated with zinc ores. Zinc workers absorb considerable amounts of zinc, but practically all of it is excreted, although the blood contains a slightly higher content than the normal. This absorption and excretion of zinc can go on for many years without causing any affection of the kidneys, stomach, or intestines, and has no effect on the metabolism of the living cells. A. R. POWELL.

Water supply and sewerage during the past fifty years. R. S. WESTON (Ind. Eng. Chem., 1926, 18, 899—901).

PATENTS.

Heating and de-aerating boiler feed and other water. COCHRANE CORP., Asses. of G. H. GIBSON (E.P. 240,417, 5.8.25. Conv., 26.9.24).

Tricalcium cyanide (U.S.P. 1,592,127).—See VII.
Insecticides from *Derris* species (E.P. 247,140).—See XVI.