BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 30, 1926.

I.—GENERAL; PLANT; MACHINERY.

Influence of exterior temperature on the temperature of standard pyrometric [electric] lamps. G. RIBAUD (Compt. rend., 1926, 182, 625—627).—The influence of the external temperature on the apparent temperature of a tungsten filament in a standard pyrometric lamp is investigated theoretically under conditions of constant current, constant resistance, and constant voltage. Short, thick filaments require appreciable corrections under the first-named condition, this being confirmed experimentally. S. K. TWEEDY.

Trichromatic colorimeter suitable for standardisation work. J. GUILD (Trans. Optical Soc., 1925-6, 27, 106-129).-A trichromatic colorimeter which may be employed to obtain the specification of any colour in a form which can be converted to any fundamental basis of specification is described. The three working primary colours are obtained by means of gelatin filters, and mixing of these colours is effected by a periscopic prism which rotates past three stationary sectors. Provision is made for adding any of the primaries to the colour under test, should this be necessary in order to obtain a match of colours, so that colours of higher saturation than it is possible to match directly may be dealt with. J. S. G. THOMAS.

Criticism of the monochromatic-plus-white method of colorimetry. J. GUILD (Trans. Optical Soc., 1925-6, 27, 130—138).—It is contended that from the practical standpoint of quantitative colorimetry, hue and saturation are not fundamental elements of colour quality, but must be regarded as derivatives of the trichromatic constitution of the colour. It is unsound to determine colour quality by methods involving a photometric measurement. Such determinations should involve only those properties of the eye which are concerned in colour-match relationships. J. S. G. THOMAS.

New method of colorimetry. J. GUILD (Trans. Optical Soc., 1925-6, 27, 139-158).—A colorimeter, employing a new method of determining the quality of a colour, as defined by its position on the trichromatic colour chart, in which only the colourmatching properties of the eye are involved, is described. The determination depends on two colour matches in which the test colour is matched by a mixture, in unknown proportions, of a standard colour and a monochromatic colour produced spectroscopically, the standard colour being different in the two matches. J. S. G. THOMAS. Laboratory mixing machine for solids.— R. M. HIXON (Ind. Eng. Chem., 1926, 18, 138). —Small quantities of powders, *e.g.*, nicotine dusts, or solids and liquids may be mixed by means of an apparatus consisting of a stirring motor to the shaft of which is attached by means of suction tubing a metal rod covered with rubber tubing and sufficiently long to take the rotating mixing bottle. Parallel with this rod is another rod which is rigidly supported from the same stand and is provided with a glass or metal sleeve to rotate with the bottle. D. G. HEWER.

Source of error in mechanical analysis of sediments by continuous weighing. J. R. H. COUTTS and E. M. CROWTHER (Trans. Faraday Soc., 1925, 21, 374-380).—See B., 1926, 31.

Silica to glass and to metal joints. BUTTOLPH. —See A., Mar., 264.

PATENTS.

Separation of the several components of a gaseous mixture. G. L. E. PATART (E.P. 232,986, 27.4.25. Conv., 26.4.24).—An elaborate succession of columns and heat interchangers for the separation of several constituents of a gaseous mixture by liquefaction and rectification is described. For example, a pure mixture of hydrogen and nitrogen is obtained from illuminating gas (subjected if desired to catalytic hydrogenation after addition of water-gas) by a reflux stream of liquid nitrogen, which is liquefied in another column the condenser of which is cooled by liquid methane. The column for methane is cooled by liquid ethylene, and the ethylene column by liquid ammonia.

B. M. VENABLES.

Low-temperature cooling, liquefaction, and separation of not easily condensable gases. A. SELIGMANN (U.S.P. 1,573,752, 16.2.26. Appl., 3.3.24).—The greater portion of the necessary decrease of pressure is obtained by the flow of the gases at a relatively high velocity through pipes adapted to absorb the pressure energy by the friction of the gases over the entire length of their walls. H. HOLMES.

Means for effecting solution of solids in liquids. WOLFF U. CO., E. CZAPEK, and R. WEINGAND (E.P. 245,683, 12.8.25).—An apparatus for effecting rapid dissolution of solids in liquids consists of two containers for the liquid, connected at top and bottom. The solid is placed in one of the containers, and on dissolving forms locally a solution of greater density which sinks through the solvent, setting up circulation between the two compartments. An adjustable valve situated in the lower connecting channel controls the rate of flow of solvent to solid, and hence the rate of solution. Modifications are described in which one container surrounds the other; by spacing the outer container but slightly from the inner the exothermic heat of solution of the substance can be utilised for preheating the solvent, thereby facilitating the process. R. B. CLARKE.

Automatic means for letting off steam from a container having in it a boiling liquid. W. CARPMAEL. From FARBENFABR. VORM. F. BAYER & Co. (E.P. 246,567-8, 28.10.24).-Steam is blown off from a vessel containing boiling liquid through an orifice within the steam-outlet pipe. In (A) the rate of flow of steam is held constant firstly because the pressure in the vessel is above the critical value, and when the pressure has dropped below that value an automatic valve is opened by the reduction of pressure in the vessel and permits additional steam to exhaust through a by-pass. In (B) the liquid in the vessel is prevented from frothing by proportioning the areas of the orifice, of the steam pipe, and of the exposed surface of the liquid in the vessel so that the rate of flow of steam is below the frothing limit. When the liquid is water the proportions may be 1:12:9000.

B. M. VENABLES.

Means for running off hot liquids from a container under pressure. W. CARPMAEL. From FARBENFABR. VORM. BAYER & Co. (E.P. 246,569, 28.10.24).—The danger of blowing down a high-pressure boiler or similar apparatus is obviated by having a pressure-tight receiver at a lower level with both steam and water connexions to the boiler. The receiver is first filled with steam up to the boiler pressure, the blow-down (water) valve is then also opened and water from the boiler discharged by gravity into the receiver. Finally, with both connexions to the boiler closed, the pressure in the receiver is reduced by blowing off steam, and the liquid then exhausted under a moderate pressure.

B. M. VENABLES.

Vacuum evaporating apparatus. E. C. R. MARKS. From SWENSON EVAPORATOR Co. (E.P. 247,246, 13.9.24).—Evaporators of the Yaryan type are described, with all parts in contact with the liquid constructed of corrosion-resisting material, *e.g.*, iron containing 11—15% Si, or glass for the tubes and earthenware for the separators. The remainder of the apparatus is made of stronger material, such as steel. B. M. VENABLES.

Evaporation or inspissation of solutions. CARRIER ENGINEERING Co., LTD., and K. J. R. ROBERTSON (E.P. 247,346, 2.1.25).—Solidified products are obtained from liquids—particularly salt from brine—by spraying the liquid saturated at ordinary temperature into a current of air at 315—370°, whereby the solution loses 5—10% of its water and is raised in temperature to 60—77°. On cooling, 2 or 3% of the liquid crystallises in the form of large crystals, and the mother liquor is returned with fresh brine to the evaporator. B. M. VENABLES.

Removing dust from gases or vapours. E. M. SALERNI (E.P. 247,274, 10.11.24).—The gases are passed in a zig-zag course through a number of hanging plates or slats which are struck by projections carried by a moving endless chain or band at the bottom of the apparatus. The collecting plates may be so arranged that, when struck, the plates of one row scrape the surfaces of the plates of the adjacent row, and the endless band may also serve as a conveyor to remove the dislodged dust.

B. M. VENABLES.

Mixing granular substances and liquids. P. LENART (E.P. 247,451, 25.6.25).—The apparatus consists of a receiving vessel, a chamber with radial blades, and a centrifugal disc arranged in order downwards. The material flung off by the disc is caught in a casing and returned through a pipe to the upper vessel. The radial blades in the intermediate chamber guide the material to the centre of the disc. B. M. VENABLES.

Grinding machine. A. E. JACOBSON (U.S.P. 1,572,722, 9.2.26. Appl., 10.9.24).-A cylindrical casing is provided with one closed side and with an axial opening in the other side, and a rotary shaft extends axially of the casing through the closed side. Spaced discs at right angles to the shaft constitute a rotary grinding head. These discs are secured to spaced arms disposed between them and carried by a member secured to the shaft. Each disc, except that adjacent to the closed side of the casing, is provided with a central opening enabling material delivered through the axial opening to pass through the casing. The disc adjacent to the closed side and the member carrying the spaced arms form a non-perforated side of the grinding H. HOLMES. head.

Drying. J. CREDO, ASST. to LOUISVILLE DRYING MACHINERY CO. (U.S.P. 1,573,144, 16.2.26. Appl., 24.5.24).—The material, during its passage through the dryer, is heated and subjected to regulated currents of heated air flowing in opposite directions. H. HOLMES.

Purification, filtration, decolorisation, and deodorisation of liquids and gases. H. W. A. BRANCO (F.P. 598,826, 27.5.25. Conv., 27.5.24).— The liquid or gas is passed through a layer of ashes from wood, coal, or peat that have been washed to remove acidic and basic substances.

A. R. POWELL.

Utilisation of residues by vacuum distillation in presence of steam. G. P. GUIGNARD (G.P. 421,787, 9.1.24. Conv., 9.11.23).—Steam or water in small quantity is fed continuously into the interior of the material being distilled *in vacuo*. The material is constantly stirred, the steam or water being introduced through the hollow vanes of the stirring apparatus. A. B. MANNING. Rotary furnace. A. H. PEHRSON (U.S.P. 1,574,932, 2.3.26. Appl., 8.8.22).—See E.P. 184,810; B., 1924, 39.

Dissolving device. E. CZAPEK and R. WEINGAND (U.S.P. 1,574,561, 23.2.26. Appl., 1.9.25).—See E.P. 245,683; preceding.

Dehydrating minerals. W. R. WADE, ASSF. to NEW YORK ZING CO. (U.S.P. 1,574,950, 2.3.26. Appl., 28.3.23).—See E.P. 213,561; B., 1925, 193.

Apparatus for drying granular material. F. H. ROSENCRANTS, ASSR. to INTERNAT. COM-BUSTION, LTD. (U.S.P. 1,576,362, 9.3.26. Appl., 15.10.24).—See E.P. 234,327; B., 1925, 577.

Disintegrating dry powders. H. PLAUSON (U.S.P. 1,575,717, 9.3.26. Appl., 3.11.22).—See E.P. 195,690; B., 1923, 534 A.

Pulverising machine. R. S. RILEY, Assr. to SANFORD RILEY STOKER CO. (U.S.P. 1,576,472, 9.3.26. Appl., 21.7.24).—See E.P. 236,769; B., 1925, 746.

Evaporator for concentrating or drying. T. RIGBY (U.S.P. 1,576,471, 9.3.26. Appl., 23.11.21).— See E.P. 181,035; B., 1922, 574 A.

Separation of larger from smaller grains of granular substances. F. H. CLAUSE (E.P. 248,082, 28.11.24).

Combined powdered-fuel and grate furnaces. C. HOLD (E.P. 248,277, 19.9.25).

Shaft furnaces for burning limestone etc. (E.P. 245,653).—See VII.

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

Bacterial decomposition of textile fibres. III. Occurrence of humus compounds in deteriorated fabrics and the bearing of their formation on the origin of peat and coal. A. C. THAYSEN, W. E. BAKES, and H. J. BUNKER (Biochem. J., 1926, 20, 210-216).-Microbiological activity is insufficient to eliminate all the cellulose present in plant tissues in the process of peat formation by decay. Humic compounds obtained from typical peats consist of two different types, one of which yields a chloro-derivative identical with or very similar to that of "natural humus" (lignin humic compound), and the other a chloro-derivative closely related to that of artificial humus compounds obtained by the action of inorganic acids on carbohydrates or from cellulose fibres decayed through ageing. It is suggested that the presence of this latter humus compound in peat may be due to the decomposition of cellulose other than by microbiological activity. A humus compound of this type was isolated from a sample of Egyptian linen dating from the 18th Dynasty. S. S. ZILVA.

Chemistry of coal. IV. Oxidation of the residue from the benzene-pressure-extraction

process. W. A. BONE and R. QUARENDON (Proc. Roy. Soc., 1926, A 110, 537-542; cf. B., 1924, 584).—When the residue obtained after extraction of a bituminous coal with benzene under pressure was treated with alkaline potassium permanganate, complete oxidation and solution resulted (cf. Francis and Wheeler, A., 1925, i., 1389). About 25-40% of the residue taken was recovered in the form of acid oxidation products from which considerable yields of mellitic and benzene-1:2:3:4tetracarboxylic acids have been obtained. Other unidentified acids are being investigated. The large yield of mellitic acid indicates that the coal substance has a six-carbon ring structure, each carbon of the ring being connected to other carbon atoms.

F. G. SOPER.

Coking of coal. H. GREGER (Braunkohlenarch., 1925, 67-101; Chem. Zentr., 1926, I., 1904-1905). -The softening of coal during coking was studied by measuring the pressure necessary to force a wire loop a fixed depth per unit of time, into compressed blocks of the powdered coal. The degree of softening first decreases and then increases with rising temperature. The softening curves and the maximum softening vary with different varieties of coal. For any particular variety the softening curve and the appearance of the coke are affected by preheating the coal, the preheating temperature and the duration of the heating both having an effect. The softening and swelling properties of the preheated coal are depreciated by storing in the absence of air after L. A. COLES. preheating.

Ash from powdered-fuel installations. J. T. DUNN (J.S.C.I., 1926, 45, 60-61 T).-Ashes escaping into the air from powdered-fuel installations have been impossible to obtain, but the author has measured the size of particles in ashes from the flues and boiler tubes in such installations, both in Britain and in the United States. The fineness of these particles ran from 1/120 in. to 1/6000 in., and over 85% of the whole were under 1/800 in. The particles escaping are presumably still finer, and the rate of settlement of these from the air must be so slow that they will be spread over an enormous area, and cause no nuisance by their settlement in the neighbourhood of the installations producing them. This accords with the fact that deposits of ash have not been obtained in the neighbourhood of powdered-fuel installations.

Resistance to the flow of gases in the fuel bed of a coke-fed furnace or water-gas generator. M. W. TRAVERS (J.S.C.I., 1926, 45, 61-63 T).--The author discusses the results of the experiments carried out by the U.S. Bureau of Mines (Tech. Paper 137), with a view of calling attention to the need for further investigation of the subject. In shallow fuel beds the fact that the pressure in the ash-pit increases as the 1.5th, and not as the 2nd. power of the rate of flow of the air into the fuel bed, is probably due to the fact that the spacing of the material of the fuel bed is dependent upon the blast velocity. The resistance to the flow of the air is the sum of two factors, the loss of head due to the increases in velocity and in mass of the gas, which attain a maximum in the hottest part of the fuel bed, and a factor which is proportional to the depth of the fuel bed, and which represents the loss of head due to viscosity.

Determination of phenols in crude ammonia liquor at coking plants and gasworks. F. ULRICH and K. KATHER (Z. angew. Chem., 1926, 39, 229-232).-Koppeschaar's method of determining phenols by the bromine they take up is applicable to mixtures of phenol and the isomeric cresols, provided the mean mol. wt. of the mixture is known. Three atoms of bromine are taken up per mol. of the phenol. Other substances reacting with bromine (e.g., hydrogen sulphide, ammonium thiosulphate) must first be eliminated, and this may be done by extracting the ammonia liquor with benzene containing 20% of quinoline. The method takes 2 hrs., and gives results in good agreement with those obtained by the gravimetric, ether extraction method. W. T. K. BRAUNHOLTZ.

[U.S.] Bureau of Mines Orsat apparatus for gas analysis. A. C. FIELDNER, G. W. JONES, and W. F. HOLBROOK (U.S. Bur. Mines Tech. Paper No. 320, 1925. Pp. 18).—Carbon dioxide is determined by absorption in caustic alkali; unsaturated hydrocarbons by absorption in fuming sulphuric acid (with subsequent removal of the sulphur trioxide fumes in the caustic alkali); oxygen by absorption in alkaline pyrogallol solution; carbon monoxide by combustion over copper oxide heated at 300° by an electric furnace (or provision may be made for the determination of carbon monoxide alone by absorption in ammoniacal cuprous chloride). By adding oxygen or air and burning in a combustion pipette, methane and ethane may be determined. The apparatus is fitted with a compensating tube for eliminating errors due to changes in pressure, temperature, and pressure of aqueous vapour. The apparatus is somewhat similar in principle and method of operation to Haldane's gas burette and some of its modifications, although it is arranged like Orsat's. The compensator, however, is a separate connexion, not being attached to the caustic alkali absorption pipette. It is essential that the copper oxide tube shall be cool when, after having been used, it is being rinsed during the methane and ethane determination. Before commencing an analysis the free spaces in the apparatus must be filled with nitrogen. Sources of error and their avoidance are indicated and a table of pertinent data relating to the commoner gases is given.

R. A. A. TAYLOR.

Karwendel oil shale. E. BERL and W. SCHMID (Brennstoff-Chem., 1926,7,49—54).—Karwendel shale yields an oil of high sulphur content which is the source of products of pharmaceutical value (ichthyol). Analysis of the shale gave : moisture 1.25%, volatile matter 37.98%, sulphur 3.98%, nitrogen 0.36%, ash 52.08%. On distillation under ordinary pressure in an iron retort holding 600 g., to a maximum

temperature of 545°, the shale yielded 13.4-14.4% of oil and 3.8-4.6% of gas, whilst at pressures of 10-50 mm. the yields were 17.9-18.8% of oil and 4.9-5.5% of gas. The average composition of the gas was: CO₂ 14.13%, H₂S 23.71%, N₂ 3.28%, H₂ 19.31%, CH₄ 31.34%, CO 2.15%, O₂ 0.86%, CnH_m 4.95%. A thick, red-brown oil with green fluorescence, unpleasant odour, and feebly alkaline reaction was obtained ; d18 0.975, S 9.98%. Fractionation under 12-20 mm. pressure gave 35-120° 26.54% (S 12.71%), $120-220^{\circ}$ 35.77% (S 11.12%), $220-280^{\circ}$ 30.07% (S 10.45%), and pitch 7.62%. The oils darkened rapidly on keeping, due to oxidation of the unsaturated constituents. The phenol content of the oil was less than 1%. Only a very small amount of the sulphur was removed by treatment with copper oxide. The crude oil was purified by treating successively with soda-lime, sodium and ammonia, dilute acid, and finally distilling under ordinary pressure from 120° to 230°. The oil thereby lost its unpleasant odour. The purified oil gave the characteristic colour reactions of dimethylthiophen. Treated with liquid sulphur dioxide at -12° , the purified oil left 8.12% undissolved. This fraction, containing the saturated hydrocarbons, was bright yellow, odourless, and contained S 1.96%. The soluble fraction, containing the unsaturated and aromatic hydrocarbons, was Bordeaux red in colour after removal of the sulphur dioxide, possessed a pleasant odour, and had a sulphur content of 8.61%. The calorific value of the dry shale was 3713 cal./g.; the calorific values of the oil fractions and gases have been estimated from their composition. A. B. MANNING.

Oil-bearing chalk at Heide (in Holstein). G. KEPPELER and J. SCHMIDT (Z. angew. Chem., 1926, 39, 220-224).-Benzene extracts from the chalk 10-20% of a heavy, black, viscous, and highly unsaturated oil, which is similar to, though not identical with, that exuded by the chalk in situ. Attempts to extract the oil by dissolving away the chalk with acid were unsuccessful. Distillation of the chalk alone yields about 75% of oil, 15% of gas, and 10% of dark, asphaltic residue (reckoned on the total oil content of the chalk), whilst distillation in a current of steam, carbon dioxide, or hydrogen yields about 89% of oil and 5-6% of gas. The oils obtained by distillation are lighter than the natural and extracted oils, and, unlike the latter, can be distilled without decomposition, giving about 2-6% of benzine, 45-50% of oil distilling at 150-350%, and 45-50%of lubricating oils. Oil obtained by distillation in a current of gas has a higher sulphur content than that obtained by direct distillation, and that obtained by distillation in hydrogen has an exceptionally low flash point and low content of unsaturated aliphatic hydrocarbons.

W. T. K. BRAUNHOLTZ.

Conversion of fatty acids into hydrocarbons. G. STADNIKOV and E. IVANOVSKI (Neftjanoe i slancevoe Chozjajstvo, 1925, 8, 470-476; Chem. Zentr., 1926, I., 1909).—On passing 940 g. of fatty

acids from linseed oil having saponif. value 188, and iodine value 179, over finely divided iron deposited upon asbestos, at 400°, a gas mixture containing mainly carbon dioxide with a little carbon monoxide and ethylene hydrocarbons, and 720 g. of liquid distillate, were obtained. The fraction of the distillate volatile in steam (b.p. 50-250°) was hydrogenated and then dehydrogenated over nickel, to convert hydroaromatic into The liquid, however, aromatic hydrocarbons. consisted almost entirely of aliphatic hydrocarbons. On treating with potassium permanganate the portion volatile in steam (iodine value 139), the greater part was oxidised to carbon dioxide, but small quantities of the lower fatty acids, up to butyric acid and valeric acid, and succinic acid, were formed. L. A. COLES.

Naphthenic acids from gas oil distillate of Californian petroleum. Y. TANAKA and S. NAGAI (J. Soc. Chem. Ind. Japan, 1926, 29, 1-7; cf. B., 1925, 196).—From the product obtained by acidifying a waste lye produced in the refining of the gas oil fraction of the mixed oil from Long Beach and Santafe Spring Fields in California, a mixture of crude naphthenic acids, d_4^{15} 0.9797, nD¹⁵ 1.4927, acid value 191.4, was separated. These were converted into the methyl esters, d_4^{15} 0.9535, $n_{\rm D}^{15}$ 1.4806, 81% of which boiled at 160–230°/ 8.9–9.0 mm. By hydrolysis of the esters pure mixed naphthenic acids were obtained, having d₄¹⁵ 0.9773, n_D¹⁵ 1.4893, neutralisation value 208.4. They distilled at 136-260°/8.9-9.0 mm., the chief portion (82%) distilling at 200-260° and having d 0.976-0.985; the fraction distilling at 210-220° had d 0.985. The specific gravity (0.976-0.985) of the main fraction is a little lower than that (0.99)of the naphthenic acids derived from Kurokawa petroleum (B., 1922, 973 A) and a little higher than that (0.97) of the acids from Nishiyama petroleum (cf. B., 1925, 870). There are two series of isomeric naphthenic acids differentiated chiefly by the specific gravity, and the acids from Californian petroleum are probably a mixture of the two series of the acids. A white crystalline mass separated in the fractions of high boiling point. K. KASHIMA.

Effects of lead tetraethyl upon the deterioration of turbine oils. S. HATTA (J. Soc. Chem. Ind. Japan, 1925, 28, 1346-1352).-Quantities of 0.05, 0.1, or 0.2% of lead tetraethyl were added to turbine oils, which were then kept at 120°, and treated with moist oxygen passed at the rate of about 150 bubbles per min. With an oil of the paraffin series, the darkening of the colour, sludge formation, and the deterioration of demulsifying power were distinctly diminished by the presence of lead tetraethyl. The density, viscosity, and acid and saponification values of the oil, however, increased gradually, both in the presence and absence of the lead compound. With an oil of the naphthenic series, the darkening of the colour was retarded, but the density, acid and saponification values, and the sludge formation were all distinctly increased by the presence of lead tetraethyl. K. KASHIMA.

Micrographic analysis of mineral oils subjected to oxidising processes. M. VANGHE-LOVITCH (Bul. Soc. Chim. Romania, 1926, 7, 93-99; cf. HOBLYN, B., 1925, 274).-The work of Hoblyn (loc. cit.) is extended. Samples of oil were heated at 250° for varying periods and examined microscopically, the time required for the first precipitation of asphalt being noted. This test gives a much more reliable indication of the value of the oil than the older tests of viscosity, inflammability, covering power, etc. Chemical analysis of the oil after heating gives results agreeing with the microscopical method, but the latter is much quicker and more delicate for detecting the first changes. By the use of various solvents on the precipitates it is shown that the oxidation proceeds in the order: oil-hard asphalt \rightarrow soft asphalt \rightarrow carbene (colloidal carbon) \rightarrow coke. The long periods of heating may be avoided by using nitric acid. A thin film of oil is placed under the microscope and a drop of a solution of nitric acid added. If the concentration of the acid exceeds a certain limit an immediate precipitation of asphalt occurs, and by noting the limiting concentration of nitric acid required by different oils, the resistance of the oils to oxidation is obtained. Results thus obtained agree with those found by prolonged heating at 250°. W. HUME-ROTHERY.

Adsorption of resins by paraffin wax and solidification of paraffin-containing products. N. TSCHERNOSHUKOV (Neftjanoe i slancevoe Chozjajstvo, 1925, 8, 640-646; Chem. Zentr., 1926, I., 1910-1911).-On removal of resin from masut of maximum and minimum solidifying pts. 25° and -2° , these values being recorded after heating to 60° and 100° respectively (cf. Tytschinin, B., 1924, 1003), the solidifying point rose to 25-27° and was independent of previous heating; on removing the greater part of the ozokerite, it fell to $2-15^\circ$, and was dependent upon previous heating; on removing both resins and high-molecular paraffins, it was 14° and was independent of previous heating. Colorimetric investigation of the adsorption of resins by kerosene saturated with paraffin wax, showed that adsorption increases with rising melting point of the wax, and the quantity of resin adsorbed at different temperatures varies according to the L. A. COLES. solidifying point of the product.

Liquid reaction products obtained by action of hydrogen on paraffin wax under high pressure at 450°. H. I. WATERMAN and A. F. H. BLAAUW (Rec. trav. chim., 1926, 45, 284—295; cf. B., 1925, 273, 746).—Careful fractionation of the gasoline boiling up to 150°, obtained by the action of hydrogen at 450° and pressures up to 280—290 atm. on Rangoon paraffin wax containing C 86·4%, H 14·8%, indicated that the gasoline contains large quantities of the successive members of the paraffin series (*n*-pentane to *n*-nonane) and probably a smaller amount of olefines (about 10%). Benzene was not detected and toluene is only present to a very small extent. R. BRIGHTMAN.

Effect of heat on mineral oils. Products of decomposition of the oil by the electric arc.

F. EVERS (Wiss. Veröff. Siemens-Konz., 1925, 4, 324—334).—When a high-tension arc is maintained under mineral oil the gaseous products are hydrogen $(50\cdot3\%)$, methane $(1\cdot0\%)$, nitrogen $(3\cdot4\%)$, oxygen $(0\cdot9\%)$, and unsaturated hydrocarbons $(44\cdot4\%)$, of which the main portion is acetylene, with a little ethylene. These unsaturated products were separated as bromides and from the less volatile fraction a *bromide*, C₆H₄Br₈, m.p. 181°, was isolated. Distillation experiments on the oil prove that some liquid products also result from the action of the arc. A dark brown solid which is produced is of variable composition but contains oxygen (4-7%), hydrogen (3%), and carbon (90-94%). The colloidal properties of this solid and the manner of its production are discussed. G. M. BENNETT.

Determination of ammonia in ammoniacal liquor. JUNGBLUT.—See VII.

Insulating oils. STÄGER.-See XI.

PATENTS.

Drying wet fuels. E. BERL (G.P. 419,906, 6.11.23).—A liquid, insoluble or very sparingly soluble in water, is intimately mixed with the fuel, and the excess liquid then removed by centrifuging or pressing. The process is repeated several times. The oils obtained by the distillation of lignites or bituminous coals are particularly suitable for the purpose; other liquids which can be used are mineral oils, aniline, toluidine, phenols, etc.

A. B. MANNING.

Preparation of low-ash coal. ELEKTRO-OSMOSE A.-G. (GRAF SCHWERIN GES.) (Swiss P. 100,129, 18.1.22. Conv., 29.1.21).—A suitable electrolyte, *e.g.*, an alkali silicate, is added to the finely divided material, which is then subjected to a flotation process. A. B. MANNING.

Briquettes from powdered fuels. E. KOLLÁR (Swiss P. 97,593, 9.7.21. Conv., 14.7.20).—The powdered fuel is mixed with a pulp made by heating finely divided non-lignified parts of plants (leaves, stems, etc.) with water at 60—100°. To make the briquettes waterproof, plant materials containing tannin and resins may be incorporated with the pulp, or the formed briquettes may be dried in flue gases. A. B. MANNING.

Peat briquettes. J. STEINERT (Swiss P. 99,230, 9.8.21. Conv., 28.1.21).—Peat is dried in the field to a water content of 45—50%, further artificially dried to 12—15% water content by direct treatment with hot gases of low oxygen content, and then made into briquettes. The gases for drying are supplied by a producer fed with peat of about 30% water content. A. B. MANNING.

Manufacture and use of active carbons. J. N. A. SAUER (E.P. 247,241, 13.8.24).—A base of active carbon is impregnated with an organic or inorganic substance such as will by suitable subsequent treatment form an adsorbent (*e.g.*, silica gel, activated carbon) in the pores of, or on, the basis. The apparent sp. gr. of the active carbon is thus increased and also the adsorptive power per unit volume. The process may be used to effect an agglomeration of the carbon particles to give larger granules. R. A. A. TAYLOR.

Producing highly adsorbent charcoal. E. BATEMAN, ASST. to UNITED STATES OF AMERICA (U.S.P. 1,573,509, 16.2.26. Appl., 5.8.19).—Carbon in a comminuted form is treated with carbon dioxide at 600—1000°. R. A. A. TAYLOR.

Carbon from coal, lignite, peat, wood, and waste material containing these substances. J. G. AARTS (F.P. 598,023, 4.3.25. Conv., 14.1.25). —The material is distilled and the gaseous products are decomposed in the usual way to give amorphous carbon. A. R. POWELL.

Distillation of carbonaceous materials. E. M. SALERNI (E.P. 247,300. Appl., 14.11.24).-The apparatus comprises a drying chamber fitted with plates or shelves and a carbonising chamber. In the dryer the charge is scraped by means of bars on an endless chain so that it passes along and over the edge of one plate and falls on to the plate below. On the return journey the bars scrape it along and off the edge of the second plate. Finally it passes through a gas-tight valve into the carbonising chamber. This may be of the same type as the dryer, or it may consist of a series of troughs arranged side by side, the charge passing laterally through them in succession. The carbonised residue may then be passed into a cooler of similar construction to the dryer. R. A. A. TAYLOR.

Distillation of lignites. E. HENEAGE (E.P. 247,324, 28.11.24).—Several horizontal metal cylinders are arranged one above the other and the charge is passed through one by means of a screw-conveyor and then drops into the next cylinder below. The charge is dried in the topmost and carbonised in the lower cylinders, after which it passes out of the setting into a cooling cylinder. The flues are arranged for heating the setting from the bottom, but the screw-conveyors may have hollow shafts to admit of the introduction of hot air or gases into the cylinders to heat the charge by their sensible heat. The screw-conveyors are flanged in order to take up heat from the cylinder walls and pass it to R. A. A. TAYLOR. the charge.

Distillation of coal and other fuels. "ALLKOG" ALLGEM. KOHLENSVERWERTUNGS-G.M.B.H. (F.P. 598,568, 22.5.25. Conv., 16.7.24).—The coal or fuel is mixed with sand or other inert material, before distillation, in order to improve the heat transference, and to loosen the fuel. A. B. MANNING.

Production of a low-boiling tar by the carbonisation of lignite. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of P. SIEDLER and K. H. KÜSTER (G.P. 421,617, 14.2.22).—The charge of coal is heated in cylindrical, vertical or inclined retorts, and the temperature is raised so slowly and the heating carried out so regularly, that at the beginning of the CL. II .- FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

actual tar evolution the coal is sufficiently free from water. The heating is slowly continued, avoiding the formation of layers at different temperatures. The products are led off at the lower end of the retort. The tar so obtained is distinguished from the lignite tars previously described in that 95% or more distils below 200°, and it is, moreover, more or less free from phenols. A. B. MANNING.

Distillation of coal. GES. FÜR INDUSTRIE-OFENBAU M.B.H. (G.P. 423,125, 21.12.17).—Coal is so distilled that the tar is obtained from it at a lower temperature, the gas and ammonia at an intermediate temperature, and the coke at a higher temperature. The distillation is carried out by the passage through the coal of gases which are heated first by the sensible heat of the discharged coke, then by the nearly completely coked charge, which is externally heated. A. B. MANNING.

Continuous water - gas production. H. HILLEBRAND (G.P. 422,999, 28.1.22).—The necessary heat of reaction for water-gas production is supplied by a stream of gas which circulates through a heater and then through the producer. The sensible heat of the gas stream after leaving the producer is used to evaporate the water for the water-gas production. Before reheating the gas stream any excess water it contains is separated. The process permits easy regulation of the gas composition.

A. B. MANNING.

Pre-drying and pre-distilling gas-producer fuels by hot gases. F. LANDSBERG (G.P. 423,350, 30.4.19).—A small part of the gas from the drying zone is led with the fuel into the carbonising zone, while the remainder passes away above. Separation of the drying layer from the carbonising layer by a valve is not necessary. A. B. MANNING.

Continuous production of water-gas and producer-gas. A.-G. FÜR INDUSTRIEWERTE, and J. FRÜH (Swiss P. 97,229, 5.3.21).—A gas containing oxygen acts on finely divided coal in a rotary retort heated internally by electrical means.

A. B. MANNING.

Production of oil-gas. GES. FÜR CHEM. IND. IN BASEL (Swiss P. 98,743, 1.7.22).—By cracking gas-oil or gas-oil vapour in a current of oil-gas itself a considerable increase in the yield of carbon in the form of gaseous hydrocarbons is obtained. A. B. MANNING.

Production of combustible gases from finely divided or high-ash fuels. STETTINER CHAMOTTE-FABR. A.-G. VORM. DIDIER (Swiss P. 98,993, 17.12.21. Conv., 21.12.20).—Low-grade fuel is fed into a retort, ignited, and heated by passing air through the charge in such a manner that a hot porous mass of clinker is formed over the cross-section of the retort above the air port. The air, passing through this, comes into contact with the unburnt fuel in a highly heated and finely divided state.

A. B. MANNING.

Combustible gas mixture containing nitrogen. N. F. O. PALACIN (Swiss P. 112,359, 21.2.25).—A portion of a mixture of carbon monoxide and nitrogen obtained by burning coal over a mixture of calcium carbonate with coal, is caused to react with steam, yielding, after removal of carbon dioxide, a mixture of nitrogen and hydrogen, which is then mixed with the remainder of the original gas.

L. A. COLES.

Determination of the constituents of gas mixtures. ADOS G.M.B.H., and K. HENSEN (G.P. 423,142, 12.11.24).—The gas mixture is sent in succession through two capillary tubes maintained at different constant temperatures, and the pressure in a vessel lying between the two tubes is observed. The pressure varies with changing viscosity of the gas mixture. The temperatures of the capillaries are chosen to give large pressure differences, which are recorded by an attached manometer. The readings are not affected by changes in the external temperature. A. B. MANNING.

Apparatus for the examination of gas mixtures, especially flue gases. ADOS G.M.B.H., and K. HENSEN (G.P. 423,297, 30.8.24).—A shaft rotating at constant speed carries two propellers, one of which rotates in the gas mixture under examination, and the other in a comparison gas. The shaft can move axially under the resultant force of the two propellers, which act in opposition to one another. The axial motion of the shaft automatically alters the inclination of the blades of the second propeller in such a way that the force due to the propeller in the gas mixture is increasingly opposed until equilibrium is reached. A pointer attached to the shaft registers its axial displacement. A. B. MANNING.

Iron carbonyl mixtures [for use as motor fuel]. BADISCHE ANILIN- & SODA-FABE. (F.P. 597,517, 1.5.25. Conv., 5.12.24).—The mixtures contain hydrocarbons and at least 10% of iron carbonyl, a stabiliser (1:4-di-*p*-toluidinoanthraquinone) and dyes being added if necessary.

Distillation and fractional condensation of complex mixtures, such as mixed hydrocarbons. Soc. ANON. D'OUGRÉE-MARIHAYE (F.P. 598,414, 8.9.24).—The mixture is subjected to a circulatory process, in which the mixed vapours traverse the lighter condensation products in the lower, middle, and upper zones successively, of a condensing column. In the middle zone definite pure products are obtained. A. B. MANNING.

Manufacture of a highly-active decolorising charcoal. H. MÜLLER-CLEMM, Assr. to GES. FÜR CHEM. PRODUKTION M.B.H. (U.S.P. 1,575,703, 9.3.26. Appl., 2.5.24).—See E.P. 216,130; B., 1925, 490.

Apparatus for removing solid carbonaceous material from oil-cracking apparatus. L. C. HUFF (E.P. 248,232, 2.6.25).—See U.S.P. 1,528,004; B., 1925, 749. Separation of gaseous mixtures (E.P. 232,986). —See I.

Utilising energy liberated during oxidation of hydrogen compounds of sulphur, nitrogen, etc. (G.P. 421,665).—See VII.

III.—TAR AND TAR PRODUCTS.

Production of road tars without distillation. L. H. SENSICLE (Gas World, 1926, 84, Coking Sect., 31-36).—In the coke-oven plant where the method was devised, the gases passed through a cyclone tar extractor working at 95°, followed by a centrifugal extractor at 85°. Liquor was sprayed into the latter. The light oil was largely carried forward and condensed previous to benzol washing. In order to prepare road-tar, a suitable tar mixture, from which part of the light oil had been removed, was passed through a preheating boiler and into a de Laval separator at 55—60°. This reduced the water content to 1% and further dehydration was effected by air agitation in the tar receiver, a temperature of 60° being maintained. Free carbon from the tar gradually accumulated in the bowl of the separator and required removal. C. IRWIN.

Working up lignite low-temperature tar without distillation. II. R. VON WALTHER, H. STEINBRECHER, and W. BIELENBERG (Braunkohlenarch., 1925, 59-63; Chem. Zentr., 1926, I., 1908—1909).—The influence of distillation in a current of steam and *in vacuo* upon the characteristics of low-temperature tar has been studied. However carefully the process be carried out, the character of the tar is altered considerably during distillation. L. A. COLES.

Removing creosote from tars and tar-oils by means of solid calcium hydroxide. R. von WALTHER and W. BIELENBERG (Braunkohlenarch., 1925, 64—66; Chem. Zentr., 1926, I., 1906).— Considerable quantities of creosote are removed from tars and tar-oils by treatment with solid calcium hydroxide, but a small proportion always remains, the proportion varying with different tars. The use of petroleum ether or benzine as solvent for the tar during the treatment gave favourable results, but when benzol or ether was used, only moderate quantities of creosote were extracted. L. A. COLES.

Obtaining useful products by the oxidation under pressure of lignite-tar creosote. E. KÁRPÁTI (Chem. Runds. Mitteleuropa u. Balkan, 1926, 3, 3—5; Chem. Zentr., 1926, I., 1908).— Oxidation under pressure is used for purifying phenolic fractions of b.p. up to about 230°, and for converting liquid tar products containing a high proportion of creosote into solid condensation products having good electrical insulating properties.

L. A. COLES.

PATENTS.

Distillation of tar. B. Young (G.P. 408,945, 12.12.22).—Tar is distilled in a rotary retort, the

interior of which is provided with a scraping device, which removes the pitch and coke formed during the process. Tar of high water and dust content can be continuously distilled in the apparatus.

A. B. MANNING.

Working up of tar. H. SUIDA (Austr. P. 101,333, 19.10.23).—Treatment of the tar takes place in two stages; in the first the tar is distilled to liquid pitch, and in the second the pitch is distilled to coke. The coke is gasified by the introduction of air into the retort, the gas produced being used for heating the distillation plant. The pitch obtained in the first stage is led continuously into one of two generator-retorts, in which alternately the pitch is coked and the coke is gasified. A. B. MANNING.

Fractional condensation of hydrocarbons (F.P. 598,414).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Constitution of Hansa Yellow G [M.L.B.] and other yellow pigment colours. F. M. Rows, A. H. BURR, and S. G. CORBISHLEY (J. Soc. Dyers and Col., 1926, 42, 80-82).-Hansa Yellow G, NHPh·CO·CHAc·N₂·C₆H₃Me·NO₂, was prepared by coupling diazotised m-nitro-p-toluidine with acetoacetanilide. The latter could only be obtained in 30% yields by heating ethyl acetoacetate with aniline at $130{-}140^\circ$ under pressure ; at ordinary pressures, using an inert solvent, diphenylcarbamide was the chief product. The methods of E.P. 9252 of 1914 and G.P. 287,569 and 293,429 (B., 1915, 867) gave satisfactory yields. Monolite Yellow G (B.D.C.) and Pigment Fast Yellow HGL conc. new (J.W.L.) are identical with Hansa Yellow G (M.L.B.). Pigment Fast Yellow GRL Extra (J.W.L.) was made by coupling diazotised m-nitro-p-toluidine with acetoacet-toluidide. The toluidide (unlike the anilide) is obtained in satisfactory yields by heating equimolecular proportions of ethyl acetoacetate and o-toluidine at 150° under pressure. Pigment Fast Yellow RL Extra (J.W.L.) is identical with the GRL brand but is shaded with a small proportion of an insoluble azo colour. It has similar properties to, but is not identical with, Hansa Yellow R (M.L.B.). Helio Fast Yellow RL (By.) appears to be a mixture of the dibenzoyl derivatives of 1:5- and 1:8-diaminoanthraguinones. A. COULTHARD.

Composition of some products used for the production of insoluble azo colours. F. M. Rowe and E. LEVIN (J. Soc. Dyers and Col., 1924, 42, 82–93; cf. B., 1925, 797).—Naphthol AS-TR (Gr.E.), which is used in conjunction with diazotised Fast Red TR base (Gr.E.) and Fast Scarlet TR base (Gr.E.) is the 5-chloro-o-toluidide of β -hydroxy-naphthoic acid. It has been prepared by condensing equimolecular proportions of the components in boiling toluene in the presence of phosphoru trichloride. Fast Red TR base is the hydrochloride of 5-chloro-o-toluidine. The colour produced on the fibre by using it in conjunction with Naphthol

AS-TR (Gr.E., G.P. Appl. 48,892) is the compound described by Saunders (J. Soc. Dyers and Col., 1924, 48) as of outstanding fastness to kier boiling, viz.,



Fast Scarlet TR base (Gr.E.) is the hydrochloride of 6-chloro-o-toluidine. Diazotised and coupled with β -naphthol it gave red needles, m.p. 166°, and with Naphthol AS, red needles with metallic lustre, m.p. 268°. Naphthol AS-D (Gr.E.), which is also used in conjunction with the above bases, is the o-toluidide of hydroxynaphthoic acid (E.P. 23,732 of 1913, 199,771, G.P. 293,897; B., 1914, 70; 1923, 824 A). The following colouring matters have been prepared (cf. B., 1924, 704): Diazotised Fast Red TR base, coupled with Naphthol AS-TR, gives scarlet needles, m.p. 285°, with Naphthol AS-D, red needles, m.p. 261°. Diazotised Fast Scarlet TR base and Napthhol AS-TR gives scarlet needles, m.p. 268°, and with Naphthol AS-D, long scarlet needles, m.p. 272°. A. COULTHARD.

See also A., Mar., 296, Sulphonic acids of arylamine derivatives of naphthaquinones (WAHL and LANTZ). 297, Constitution of naphthazarin (DIMROTH and RUCK). 307, Transformation of 2:4-dinitro-4'-phenylazodiphenylamine into phenazine derivatives (BERETTA). 314, Potentiometric titration (diazotisation) of amines (MÜLLER and DACHSELT).

Trichromatic colorimeter. Criticism of monochromatic-plus-white method of colorimetry. New method of colorimetry. GULD.— See I.

PATENTS.

Manufacture of condensation products [dyes] of the anthraquinone series. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 246,683, 23.2.25. Add. to 205,502; B., 1924, 627).—The condensation products, suitable for use as dyes or for the production of dyes, described in the chief patent and in E.P. 220,304 and 222,125, are obtained more easily and in better yield by prolonged treatment of a 1:5-diaroylnaphthalene with aluminium chloride at a high (temperature and preferably with the simultaneous introduction of air or oxygen into the reaction mixture. According to the temperature at which the operation is conducted and to its duration dibenzopyrenequinones are obtained directly, or Bz-1-aroylbenzanthrones or their reduction products are first formed, these being capable of subsequent conversion into dibenzopyrenequinones. For example, a dye having m.p. about 385° and soluble in nitrobenzene is obtained by heating, within a closed vessel, for 2 hr. at 170-195° a mixture containing 1 pt. of 1:5-dibenzoylnaphthalene, m.p. 186.5°, and 10 pts. of freshly

sublimed aluminium chloride or alternatively by heating for 40 hr. at 120° and with the continuous introduction of oxygen, a mixture containing 29 pts. of benzanthrone, 190 pts. of aluminium chloride, and 116 pts. of benzoyl chloride, the temperature being further maintained at 180° for 24 hr.

A. J. HALL.

Manufacture of sulphide dyes. A. G. BLOXAM. From A.-G. FÜR ANILIN-FABR. (E.P. 247,378, 10.2.25).-Sulphide dyes of good fastness on cotton are obtained by melting sulphur, or a compound which yields sulphur (other than strongly alkaline sulphurising agents such as alkali sulphides or polysulphides), with leuco-indophenols in which the hydrogen of the amino-group has been substituted by the methylene or benzylidene group, the benzyl group, an acyl group of the aliphatic or aromatic series, or a substitution derivative of these groups which has a tendency to form a thiazole ring during sulphiding. For example, 15 pts. by weight of 4-amino-4'-hydroxydiphenylamine are added to 10 pts. of benzaldehyde. The solid benzylidenecompound produced is baked with 60 pts. of sulphur for 10 hr. at 200°. The crude melt is dissolved in aqueous sodium disulphide and the dye precipitated by a current of air. Cotton is dyed from an alkali sulphide bath of the dye in reddish-brown shades of very good fastness to soap, boiling, acid, and chlorine. A. COULTHARD.

Manufacture of dyes of the dibenzanthrone series. BADISCHE ANILIN- & SODA-FABR. (F.P. 598,752, 25.5.25. Conv., 11.8.24).—Aminodibenz-anthrones when treated with acylating substances under mild conditions yield derivatives which, in the presence of diluents, may be alkylated or aralkylated. For example, benzoyl chloride in nitrobenzene acts on aminodibenzanthrone to form a vat dye giving a blue shade on cotton which changes to grey in the presence of chlorine. Aminoisodibenzanthrone gives a violet dye. The dye formed by the action of o-chlorobenzoyl chloride on aminodibenzanthrone when treated with methyl toluenesulphonate gives a blue vat dye, fast to chlorine. Aminobenzanthrone and toluenesulphonyl chloride in nitrobenzene yield a dye which gives a greenish-blue shade on cotton and is converted by methyl toluenesulphonate into another dye (crystalline) A. COULTHARD. giving a similar shade.

Manufacture of trisazo dyes. FARBENFABR. VORM. F. BAYER & CO. (F.P. 598,894, 28.5.25. Conv., 13.6.24).—Diazo compounds of primary disazo dyes which have a 1-amino-2-naphthyl ether 6-sulphonic acid as end component are coupled with 2-amino-5-naphthol-7-sulphonic acid or 2-arylamino-5-naphthol-7-sulphonic acid or their derivatives, in the presence of pyridine or its derivatives. The dyes so produced, give pure greenish-blue shades on cotton, which are fast to light. For example, the diazo compound of the disazo dye from aniline-3: 6-disulphonic acid, a-naphthylamine, and 1-amino-2-naphthyl ether-6-sulphonic acid is coupled with 2-phenylamino-5-naphthol-7-sulphonic acid. A. COULTHARD.

Preparation of polyazo dyes. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of L. LASKA and F. WEBER (G.P. 423,092, 16.11.24).-One mol. of a tetraazo compound-from benzidine for exampleis combined with 1:8-aminonaphthol-3:6-disulphonic acid, in a solution of mineral acid, and the product coupled with a monodiazo-compound and 1:8- or 1:5-naphthylenediamine. The dyes are specially suited for mixed fabrics of wool and cotton. A. COULTHARD.

Production of hydroxyanthraquinones. J. THOMAS, H. W. HEREWARD, and SCOTTISH DYES, LTD. (E.P. 246,529, 22.10.24).-In the manufacture of hydroxyanthraquinones, particularly alizarin, by treating 2-chloroanthraquinone with caustic soda and an oxidising agent in an autoclave, as described in E.P. 174,101 (B., 1922, 212A), the resulting alizarin is insoluble in 20-25% caustic soda and the presence of sodium chloride in the reaction mixture has no harmful effect. A simplified and more economical method of working up the autoclave melt is thus possible, the alizarin being separated by direct filtration or filtration after formation of insoluble calcium alizarate (by addition of lime or calcium hydroxide) and the alkaline filtrate concentrated and causticised so that it may be again used although it contains sodium chloride. A. J. HALL.

Preparation of 1:2:3:4-tetrahydroxyanthraquinone. FARBENFABR. VORM. F. BAYER & Co., Assees. of R. E. SCHMIDT (G.P. 421,235, 7.2.24).-Alizarin in concentrated sulphuric acid, is treated with suitable oxidising agents, such as manganese dioxide, lead peroxide, etc., in the presence of boric acid. The process runs smoothly and no purpurin is formed. A. COULTHARD.

Preparation of a heterocyclic compound of the naphthalene series. GES. FÜR CHEM. IND. IN BASEL (Swiss P. 100,851, 2.12.22. Addn. to 92,688; cf. A., 1923, i., 938).-a-Napthathiofuran-1:2-dione, a red crystalline powder, m.p. 168°, is obtained by the action of oxalyl chloride upon a-thionaphthol, preferably in the presence of diluents, e.g., carbon disulphide or benzene, and condensing agents, e.g., aluminium chloride or sulphuric acid.

L. A. COLES.

Dyestuffs of the anthraquinone series. J. BADDILEY and W. W. TATUM, Assrs. to BRITISH DYESTUFFS CORP. (U.S.P. 1,574,748, 2.3.26. Appl., 18.4.23).—See E.P. 201,610; B., 1923, 968A.

Waste waters from dyeworks (Austr. P. 100,735).—See XXIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Influence of sunlight on wool. W. von BERGEN (Textilber., 1925, 6, 745-751; Chem. Zentr., 1926, I., 1070) .- After exposure to sunlight wool swells strongly in dilute alkali and crumples characteristically; it shows no double refraction

between crossed nicols. Acids promote the decomposition of the wool molecule, and the oxidation of the liberated sulphur to sulphuric acid. Alkalis hinder the oxidation of the sulphur, and neutralise the acids formed. Free sulphuric acid causes vellowing of the wool. After treatment with sunlight wool is dyed more deeply by basic dyestuffs, and less so by acid dyestuffs. A. GEAKE.

Behaviour of textile fibres towards colloidal solutions [of soap, Turkey-red oil, etc.]. W. HERBIG and H. SEYFERTH (Z. Deuts. Oel- u. Fett-Ind., 1926, 46, 81-84).-The wetting properties of aqueous solutions containing soap, Turkey-red oil, Turkey-red oil and Tetracarnit (Sandoz), Bäuchol (a proprietary wetting-out substance), Bäuchol and Tetracarnit, and Tetracarnit were compared by measurement of (1) the degree and rapidity with which the solutions (tinted with Benzopurpurine 4B) ascend vertically suspended cotton and woollen yarns, (2) their surface tension and viscosity by means of a stalagmometer and Ostwald's viscosimeter respectively, (3) the degree of spreading of drops of the solutions along a single horizontal cotton thread, and (4) the vertical depth of drops of the solutions suspended from a horizontal band of cotton threads. The results obtained are inconclusive and depend on factors (e.g., the tension and surface of the textile fibres) other than the wetting-out properties of the solutions. A. J. HALL.

Importance of a definite regulation of atmospheric humidity in industrial textile processes. J. OBERMILLER (Z. angew. Chem., 1926, 39, 46-51; cf. B., 1925, 94) .- For spinning and weaving processes it is important to maintain a definite and comparatively high relative humidity of the surrounding air; the strict regulation of the temperature is less vital. Curves are drawn showing the relation at 20° between the relative atmospheric humidity and the moisture content of wool, raw silk, fibroin, artificial (viscose) silk, and cotton. These are all very similar and are roughly linear between about 10% and 75% relative humidity. At a given relative atmospheric humidity the moisture content of the various fibres shows but slight variation with temperature. Cotton, a vegetable fibre which contracts when wetted, shows an increased elasticity in the wet state, whereas the reverse is true for animal fibres, such as wool and silk, and also artificial silks, which expand when wetted.

W. T. K. BRAUNHOLTZ.

Chemistry of the sulphite-cellulose digestion process. E. Hägglund (Svensk Kem. Tidskr., 1925, 37, 116–124; Chem. Zentr., 1925, 96, I., 271; cf. Hägglund and Bjorkman, B., 1925, 238, 586) 586).—In the early stages of the digestion of wood with solutions containing sulphur dioxide and sulphites, one or more ligninsulphonic acids are formed which cannot be removed from the pulp by washing with water. The lignin in wood is chemically combined with carbohydrates, and the sulphonic acids of these compounds hinder penetration of the sulphite liquor. During digestion, the sulphite liquor reacts rapidly

at first and then but slowly. Accumulation of ligninsulphonic acids within the pulp occurs if the rate of hydrolysis of the lignin-carbohydrate compounds is slower than the rate of sulphonation, but this increase is hindered by a rise in the temperature of digestion. Sufficient lime should be present so that the digestion is complete or nearly complete before combination of all the sulphite occurs. Ligninsulphonic acid is comparable in strength with mineral acids. During digestion the concentration of ligninsulphonic acids approaches the original concentration of the sulphur dioxide. At any moment during digestion, the lime present is distributed between the ligninsulphonic acids and the sulphite, and at the completion of digestion the greater part is combined with the former. The digestion liquor remains bright yellow provided that sufficient sulphite is used, and darkening is caused by the use of high temperatures. Blackening of the cellulose occurs when the acidity of the digestion liquor increases too much by formation of ligninsulphonic and sulphuric acids, insufficient lime being present. A. J. HALL.

Chemical activity of cellulose and its significance in sizing paper. E. OEMAN (Papier-Fabr., 1925, 23, 725-727, 745-748, 800-802, 813-816).-Cellulose is amphoteric in character; thus, if immersed in dilute acid, hydrogen ions are taken up and hydroxyl ions are formed, and the converse takes place in an alkaline medium. The isoelectric point is somewhat on the acid side of neutrality. When sulphite-cellulose is steeped in 0.015N-sulphuric acid, washed thoroughly, and immersed in water, the amount of alkali required to bring the $p_{\rm H}$ of this water to 8 is several times as great as when the cellulose has not been previously immersed in acid. This is not due to retention of sulphuric acid because no sulphate can be found in the alkaline solution, and it is ascribed to combination of cellulose with hydrogen ions; this shifts the isoelectric point towards the acid side. With sulphatecellulose the effect is smaller, and immersion in alkali has the reverse effect. The sulphate ions, corresponding to the hydrions taken up from sulphuric acid, remain in the solution as free ions. Thus, if a trace of sulphuric acid is added to water, and this shaken with sulphate-cellulose, the $p_{\rm H}$ of the water, determined electrometrically with a hydrogen or quinhydrone electrode, is less than that determined colorimetrically. If the water is made alkaline the electrometric method gives the higher $p_{\rm H}$. Size particles are electro-negative and, by making the cellulose positive by absorption of hydrogen ions from an acid solution, combination can be made to take place without alum, but this is not a practical process. When alum is used aluminium is absorbed and the cellulose becomes electro-positive; this is enhanced by washing with acid water, and size then readily combines and is fixed in the fibre.

A. GEAKE.

Theory of resin sizing. E. OEMAN (Papier-Fabr., 1926, 24, 49—58).—Sulphite-cellulose takes up aluminium from alum solutions and calcium passes into the solution. The amount of aluminium which is absorbed depends on the calcium content of the cellulose, and becomes a minimum for ash-free filter paper. Aluminium is not absorbed as hydroxide, but probably replaces calcium in some organic combination. If cellulose containing aluminium or calcium is washed with water, some of the metal passes into solution, calcium more freely than aluminium. If more sulphite-cellulose is added to the wash water the aluminium is absorbed, and may thus be transferred from one portion of cellulose to another. Sulphite-cellulose, and even ash-free filter paper, are able to absorb and fix resin without any previous treatment with alum. The amount absorbed depends on the degree of dispersion of the resin sol. Thus from a sol prepared by pouring an alcoholic solution into water, sulphite-cellulose was able to absorb 55.5% of the resin; whereas from sols prepared by adding the calculated amounts of dilute sulphuric acid to sodium resinate solutions only 16-25% could be absorbed. After treatment with alum and washing, ash-free filter paper was able to absorb practically all the resin from the first sol, but only 15% from the second. Sulphite-cellulose after alum treatment was able to absorb as much as 20% of its weight of resin from a sol prepared from the alcoholic solution, although the excess of alum had been washed off and could not have passed into the resin sol. From sols prepared from sodium resinate only about 15% was absorbed. The amount of resin taken up by sulphite-cellulose after alum treatment may be increased by adding acids or neutral salts, which diminish the dispersion of the resin, but if too much is added poor sizing may result. Any alum passing into the resin solution assists sizing in this way. A. GEAKE.

Comparative study of paper fillers. M. B. SHAW and G. W. BICKING (U.S. Bur. Standards Tech. Papers, 1925, 19, [301], 733-748).-In continuation of previous work (cf. B., 1925, 126) experiments have been conducted on a semicommercial scale in order to compare the papermaking qualities of a number of fillers including asbestine, talc, clays, "crown filler" (artificially prepared calcium sulphate), and gypsum. All the fillers used were of relatively good colour and low grit content. Retention figures for the silicate fillers were fairly regular, talc being the best in this respect ; "crown filler" and gypsum, on the other hand, owing to their solubility in water, showed poor retentive qualities. An examination of the various samples of paper made under strictly comparable conditions showed that, in general, differences in opacity, finish, strength, and degree of sizing were due in the main to the difference in retention of the fillers. Talc, however, appeared to cause less reduction in tensile strength and bursting strain than did other fillers when present to the same extent. None of the fillers had any appreciable effect on the colour of the paper, but any outstanding differences in the colour of various fillers would be noted in the finished sheet. It is concluded that asbestine and talc are, with respect to their papermaking qualities, comparable with the better clays:

also, that relative cost is no criterion of paper-making value; the cheapest clay proved to be as good as one costing twice as much, and "crown filler" offered no advantage over the cheaper gypsum.

D. J. NORMAN.

Analysis of resin size. M. GOTTLÖBER (Papier-Fabr., 1926, 24, 125).—" Total resin" is determined by precipitating with dilute sulphuric acid, collecting the precipitate on a tared filter, drying at 105°, and weighing. Filtering the precipitate has the advantage over extracting it with ether that substances, such as casein and starch, which contribute to the sizing effect, are included. A. GEAKE.

See also A., Mar., 312, Silk fibroin (BRILL). 325, Fermentation of cellulose by thermophilic bacteria (VILJOEN, FRED, and PETERSON).

Occurrence of humus compounds in deteriorated fabrics. THAYSEN, BAKES, and BUNKER.— See II.

PATENTS.

Process for working up animal fibres, hairy skins, or hair. R. BACH (E.P. 243,301, 22.12.24. Conv., 20.11.24).-Aldehydes, ketones, or compounds capable of yielding them, more particularly bisulphite compounds, are used, either alone or in conjunction with other agents, e.g., mercury salts, as carroting agents for hair and the like. Formaldehyde is especially suitable and may be used in aqueous solution or in gaseous form. This process may be applied before or during the felting operation, or to "half-planked felt (Labraz)": alternatively, these carroting agents may be added to the planking liquor. The carroting operation is preferably fol-lowed by treatment with oxidising agents; for this purpose hydrogen peroxide and nitric acid are particularly suitable, since, apart from their function as deodorisers, they enhance the carroting effect. *Example* : Hair is exposed to the action of a 5-15%solution of formaldehyde (slightly acidified with, for instance, hydrochloric acid) at 25-80° for 1-40 hr. depending on the quality of the hair and the temperature and concentration of the solution. If desired, the operation may be conducted under pressure at temperatures above 100°. The hair is then centrifuged and treated with hydrogen peroxide. This process improves the felting properties of the fibres and enables inferior and waste hair to be successfully used in the manufacture of hat bodies. D. J. NORMAN.

Proofing cellulosic, animal, and other substances against insects and the like. S. W. KENDALL (E.P. 247,242, 14.8.24).--Cellulosic, vegetable, animal, metallic, and building materials are protected from attack by all forms of animal or vegetable organisms by impregnating or covering with an insoluble soap of a rare-earth metal, such as cerium, lanthanum, didymium, or yttrium, or of thorium, thallium, titanium, zirconium, or uranium. The soap may be applied as a true or colloidal solution in an organic solvent, or as an emulsion in water, or it may be prepared in or upon the material from an oxide, hydroxide, or salt of the metal and the

organic acid or a soluble salt of the latter. Fatty, oleic, linoleic, linolenic, clupanodonic, abietic, cocceric, ricinoleic, dihydroxystearic, japanic, chaulmoogric, resin or gum acids may be used, or crude mixtures of these, such as oils, fats, waxes, gums, or resins, or chemical derivatives, such as oxidation, reduction, hydration, or dehydration products. Textile materials may be treated with such a proportion of the preparation that they are proofed against organisms without substantially affecting their appearance or ability to absorb moisture; a larger proportion also waterproofs the material (cf. U.S.P. 1,536,254; B., 1925, 586). Timber is similarly protected from dry rot, the death watch beetle, and from marine animals or vegetation; plaster, boards and the like are protected from ants and rodents. The compositions may also be used as anti-fouling paints for timber and metal. A. GEAKE.

Material for protection [of fabrics etc.] against moths. W. SCHMITZ (G.P. 421,100, 18.7.24. Addn. to 419,463; cf. E.P. 230,203, B., 1925, 421).— The material consists of a mixture of quillaia bark and lupin or broom seeds which is extracted with a dilute inorganic acid (not sulphuric acid), and the extract neutralised with sodium or magnesium carbonate, evaporated to a thick syrup, mixed with anhydrous sodium sulphate, and reduced to a dry powder. C. T. GIMINGHAM.

Manufacture of artificial filaments, films, plates, and the like. H. HAWLIK (E.P. 242,240, 7.9.25. Conv., 29.10.24).-Filaments, films, and the like, which are impervious to water, are prepared from viscose by mixing it with a base, basic compound or salt, and adding an organic acid to the coagulating bath. The addition to the viscose may be sodium aluminate, aluminium chloride, sulphate, or acetate, or other compounds of aluminium, calcium, barium, strontium, tin, or zinc, or soap, or mixtures of salts. The organic acid may be a higher fatty acid, such as palmitic, stearic, oleic, or erucic acid, a sulphonated acid, such as sulphoricinoleic acid, a hydrogenated fatty acid, hydroxy-fatty acid, halogenated fatty acid, resin acid, wax acid, or the like, or an aromatic acid, such as salicylic or tannic acid. If soap has been added to the viscose, the bath may contain a compound, such as aluminium sulphate or calcium chloride, which forms insoluble soaps. A. GEAKE.

Manufacture of artificial silk and the like from viscose solutions. W. P. DREAPER (E.P. 245,815, 17.9.24).—The drying of freshly-precipitated viscose filaments under tension prior to further treatment may be dispensed with, and the desulphurising, bleaching, and dyeing operations conducted with the filament in the gel condition if the desulphurising solution contains an alkali or ammonium salt in quantity equivalent to at least 40% saturation, preferably about 80% saturation, at the ordinary temperature. When dyeing is carried out from sodium sulphide solution, desulphurisation and dyeing may take place simultaneously provided that the dyestuff is not salted out. It is advantageous to add an alkali salt to any other baths, e.g., dyeing or bleaching, through which the filament is passed, but in such cases about 10% of the saturation quantity is sufficient. The treated and washed filaments are subsequently dried under tension on stretching frames. Yarn produced by this process behaves like raw or "gum" natural silk in that the filaments show a certain tendency to cohere.

D. J. NORMAN.

Manufacture of artificial silk or the like. J. C. HARTOGS (E.P. 246,423, 9.11.25).—Considerably larger quantities of soaps (added either as potash soaps or suitable fatty acids) may be incorporated with viscose solution if potassium hydroxide is used instead of sodium hydroxide in the preparation and solution of the cellulose xanthate. For instance, a suitable spinning solution is obtained by adding 2% of potassium oleate (dry weight) or 1% of oleic acid to potassium-viscose. The spinning bath may optionally contain a proportion of potassium sulphate. D. J. NORMAN.

Dissolving and gelatinising cellulose esters such as nitrocellulose and like cellulose compounds. V. PLINATUS (E.P. 246,272, 4.12.24).—The gelatinisation of cellulose esters is effected by the action, in the presence of water, with simultaneous application of heat and pressure, of mono-, di-, or tri-butyrin. For example, in one method the cellulose ester, previously dispersed in and retaining more than 18% of water, is treated with undiluted mono-, di-, or tri-butyrin and the resulting mixture kneaded and rolled under pressure. The presence of water as a diluent not only ensures uniform gelatinisation but accelerates the process. D. J. NORMAN.

Production of coloured cellulose plastics and solutions. BADISCHE ANILIN- & SODA-FABR. (E.P. 247,288, 12.11.24).-Coloured cellulose plastics are produced by milling the cellulose ester and a softening or swelling agent, such as camphor, with an organic or inorganic colouring matter which is insoluble in the other constituents of the mixture. Milling is continued until, when a sample is dissolved in a liquid which is a solvent for the cellulose ester but not for the colour, no substantial separation of the colour takes place after keeping for several days or weeks. The mass then contains the dyestuff in a colloidally, or nearly colloidally, dispersed condition and, in a thin layer, it is more or less transparent. By dissolving such plastics in solvents, solutions are obtained which are suitable for producing coloured coatings on wood, glass, leather, metals, paper, fabrics, and the like. The colouring matter may be prepared in a highly dispersed condition before incorporation with the cellulose ester, and, in one example, colloidal silver is formed in the varnish itself. A. GEAKE.

Cellulose acetate film composition. S. J. CARROLL, Assr. to EASTMAN KODAK CO. (U.S.P. 1,572,232, 9.2.26. Appl., 22.4.25).—Sufficient tributyrin is added to cellulose acetate to maintain flexibility after prolonged heating at 65°.

A. GEAKE.

Reducing the viscosity of nitrocellulose. S. E. SHEPPARD and L. W. EBERLIN, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,572,248—9, 9.2.26. Appl., 15.6.25).—Nitrocellulose is treated with (A) a substantially non-nitrating gaseous mixture, containing higher oxides of nitrogen, or (B) a peroxide solution. A. GEAKE.

Reducing the viscosity of nitrocellulose. L. E. BRANCHEN, ASST. to EASTMAN KODAK CO. (U.S.P. 1,572,266, 9.2.26. Appl., 13.5.25).—Nitrocellulose is softened by treatment with a penetrant, and its viscosity reduced by separate treatment with an aqueous alkali. A. GEAKE.

Preparing and preserving fibres for pulp making. E. C. LATHROP and T. B. MUNROE (U.S.P. 1,572,539, 9.2.26. Appl., 20.6.24).—Bagasse and similar fibres are softened by suitable fermentation (cf. following abstract), and so piled that the heat generated partially dries and sterilises the material. A. GEAKE.

Preserving fibres for pulp making. E. C. LATHROP and T. B. MUNROE (U.S.P. 1,572,540, 9.2.26. Appl., 20.6.24).—By wetting green bagasse and similar fibres the growth of moulds and fungi is inhibited, and that of lactic acid-producing and similar bacteria is promoted. This fermentation is continued until the desired proportion of parenchymatous matter is destroyed. A. GEAKE.

Production of pulp. L. BRADLEY and E. P. MCKEEFE (U.S.P. 1,572,840, 9.2.26. Appl., 10.4.24). —Chemical pulp is boiled with sodium hydroxide and sodium thiosulphate, the former being considerably in excess of that required to decompose silicates and aluminates in the material treated. A. GEAKE.

Cellulose from vegetable substances. J. M. SCHMIDT (Can. P. 247,077, 29.12.23).—The fibres of peat or the like are loosened by heating or freezing, freed completely or partly from colloidal constituents, and treated many times alternately with alkali and chlorinating baths with vigorous stirring.

A. GEAKE.

Paper pulp from seaweed. A. J. and H. DE MONTBY (F.P. 595,394, 17.6.24).—The seaweed is washed with sulphuric acid, hydrochloric acid, and soft water. It is then treated in a rotating washer or autoclave with a solution of sodium hydroxide and quillaia bark and steam at 3—4 atm. pressure for 8—10 hrs. After pressing and washing with soft water, it is treated with chlorine and acidified. A. GEAKE.

Replacing a heavy or cold liquid containing cellulose, peat, or the like, in suspension, by a light or hot liquid. T. N. M. MOLIN (G.P. 416,600, 1.8.23. Conv., 9.8.22).—The mass, e.g., pulp from a cellulose boiler, is run into a diffusion vessel with a false bottom, and the light or hot liquid introduced at the top. The heavy or cold liquid runs away through the false bottom, and is replaced by the light or hot liquid. The mass may then be more readily pressed. A. GEAKE. Utilising the condensate from indirectlyheated sulphite-cellulose boilers. R. KARLBERG (G.P. 420,830, 19.11.24. Conv., 27.11.23).—The condensate is treated with manganese dioxide to oxidise and neutralise the acids; neutralisation is completed with sodium hydroxide or carbonate. Heat losses are avoided by carrying out the operations in a closed vessel under pressure. A. GEAKE.

Reclaiming used paper. G. HAMMOND (U.S.P. 1,572,478, 9.2.26. Appl., 12.3.25).—Paper, to which a pigment has been applied, is recovered by heating with caustic alkali below 43°, and then adding an alkali hydrogen sulphite at the ordinary temperature. A. GEAKE.

De-inking paper. G. HAMMOND (U.S.P. 1,572,479, 9.2.26. Appl., 24.5.22).—A composition for de-inking paper comprises a saponaceous ingredient, an ingredient of the glucose series, and water.

A. GEARE.

Recovering waste sulphuric acid from manufacture of parchment paper. H. BECHHOLD and H. KARPLUS (G.P. 418,831, 10.7.24).—The sulphuric acid is concentrated until suitable for the parchmentising process and treated with a solid adsorbent, such as silica or a silicate. Alternatively concentration is omitted, and the acid only heated before treatment with an absorbent. A. GEAKE.

Manufacturing threads of artificial silk of highest degree of fineness from viscose. L. HESSE and H. RATHERT (U.S.P. 1,575,052, 2.3.26. Appl., 13.2.22).—See E.P. 240,717; B., 1925, 986.

Pumps [for use in manufacture of viscose silk]. E. LUNGE, and COURTAULDS, LTD. (E.P. 248,038-41, 20.11.24).

Apparatus for manufacture of artificial silk. E. LUNGE, and COURTAULDS, LTD. (E.P. 248,042 and 248,046, 20.11.24).

Air-vessels for securing regular flow from reciprocating pumps [in manufacture of viscose silk]. E. LUNGE, and COURTAULDS, LTD. (E.P. 248,045, 20.11.24).

Washing textile material on bobbins (E.P. 242,612).—See VI.

Manufacture of alkali carbonates (Can.P. 248,096).—See VII.

Sugar from cellulose (F.P. 595,439).-See XVII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Theory of the dyeing process. E. ELÖD (Melliands Textilber., 1925, 6, 742—743; Chem. Zentr., 1926, I, 1039—1040).—The equilibrium hydrogen-ion concentration of solutions of acid or basic dyes in contact with wool is characteristic of the wool used and depends neither on the concentration of the dye nor on the initial $p_{\rm H}$ of the solution. The quantity of dye taken up is not directly connected with the change in $p_{\rm H}$ over the region investigated.

No marked decomposition of wool occurs with solutions having $p_{\rm H}$ equal to the so-called isoelectric point of the wool, whereas the wool proteins are broken down at hydrogen-ion concentrations removed from this value. The isoelectric point varies from one kind of wool to another and, since it is a resultant of the isoelectric points of the amphoteric substances composing the wool, may not be well defined even for an individual sample. L. M. CLARK.

Physics and chemistry of the dyeing process. K. H. MEYER (Melliands Textilber., 1925, 6, 737-739; Chem. Zentr., 1926, I, 1040).-The theory that dyeing may be due to solution of the dye in the fabric, is supported by the facts that the amount of o-nitroaniline taken up by cellulose acetate silk from aqueous solution depends on the concentration, and that the colour of a fibre so treated is uniform throughout the cross-section of the fibre. Moreover, all substances, coloured or not, which can be extracted from aqueous solution by ethyl acetate, penetrate acetate silk. With basic dyes, the term "adsorption" is justified, since here the dyes are attached only to the surface of the material; the aromatic nuclei of the dyes may be supposed to penetrate the fibre, while the ammonium radical is turned towards the aqueous the mordant, "Beize für medium. When Acetatseide," B.A.S.F., is used, both solution (as mordant-dye compound) and adsorption of the dye may take place. L. M. CLARK.

Physical changes of dyes within dyed fibres. R. HALLER and A. RUPERTI (Cellulosechem., 1925, 6, 189-192) .--- Cellulose acetate silk dyed at low temperatures with Para-Red has a yellowish shade, the dye being uniformly distributed within each fibre; after immersion in hot or boiling water, the shade becomes redder and the dye agglomerates into small particles. Similar results are obtained, although with greater difficulty (steaming under pressure is necessary), when the Para-Red is obtained from Naphthol AS (the anilide of β -hydroxynaphthoic acid) instead of β -naphthol or when aminoazobenzene is used instead of *p*-nitroaniline. Similar changes are observed in Chardonnet silk dyed with the same dyes and also with indigo, Thioindigo Red, and Indanthrene Blue. Chardonnet silk dyed cold with Naphthylamine Claret (a-naphthylamine diazotised and coupled on the fibre with β -naphthol) contains the dye evenly distributed; when heated in water under 1 atm. pressure the dye agglomerates slightly without change of shade, but when heated for a prolonged period in boiling water or subjected to a short steaming under 6 atm. pressure, agglomeration becomes complete and the dye migrates towards the surface of each fibre and is there deposited as welldefined crystals which may be removed by washing and pressing, the fibres being thereby decolorised. Thioindigo Red dyed on Chardonnet silk behaves similarly. Chrome Yellow (from lead acetate and a dichromate) dyed on Chardonnet silk is at first evenly distributed but after steaming under 4 atm. pressure it agglomerates and becomes orange (even in the absence of an alkali) although no migration of the pigment occurs. Similar changes are observed by

steaming dyed cotton, except that the agglomerated dyes migrate to the boundaries of the lumen in each fibre as well as to the cuticle, the migration, change of shade, and condensation or crystallisation of the particles of dye being favoured by prolongation of the steaming or rise of temperature. Vat dyes, Indigo and Thioindigo Red easily, and Indanthrene Red 5 GK, Indanthrene Brilliant Violet RK, and Indanthrene Blue RS with greater and increasing difficulty, crystallise and migrate within cotton fibres to the lumen and cuticle when steamed, accompanied by a change in shade. Uncertain results are obtained by steaming cotton dyed with direct dyes. Alizarin Red dyed on cotton mordanted with aluminium acetate is evenly distributed within each fibre, but when steamed for 1 hr. under 0.5 atm. pressure the pigment agglomerates and migrates to the lumen and cuticle; that deposited near the cuticle is removed by washing with water, the fibre being left colourless; under similar conditions the presence of Turkey-red oil considerably retards the agglomeration and migration, and the dye which migrates to the cuticle cannot be removed by washing. The decrease in fastness to rubbing produced by steaming cotton dyed with indigo is due to migration of the dye to the cuticle of each fibre. A. J. HALL.

PATENTS.

Weighting silk. DEUTSCHE GASGLÜHLICHT-AUER-GES.M.B.H. (G.P. 422,097, 17.10.17. Addn. to 320,783).—Zirconium salts are used instead of tin salts for weighting silk by the process described in the chief patent (cf. E.P. 116,102; B., 1920, 17 A). For example, silk is treated with zirconium nitrate solution, d 1.21, followed, after centrifuging, by treatment with ammonia, which may be diluted with inert gases. L. A. COLES.

Washing, bleaching or dyeing textile materials wound on perforated bobbins. NAAML. VENNOOTS. NEDERLANDSCHE KUNSTZIJDEFABR. (E.P. 242,612, 19.10.25. Conv., 8.11.24).—The bobbins, immersed in the dye-liquor or other liquid, are attached to a long vertical pipe, the column of liquid in the pipe supplying the suction necessary to draw the liquid through the material. The process is suitable for bleaching, dyeing, etc., and for washing out acid from freshly spun viscose silk. L. A. COLES.

Apparatus for dyeing and otherwise treating textiles, textile fabrics, and the like. P. STAPLETON and E. STROUD (E.P. 246,386, 24.7.25).—A dye vat with a curved or sloping bottom is provided with a steam injector by means of which dye liquor is withdrawn from the upper part of the vat, through a small reservoir, and is subsequently discharged through perforations in the upper side of a horizontal pipe located at the lowest part of the vat, thereby promoting uniform circulation of liquor and textile materials within the vat. The injector is provided with a by-pass which allows the dye liquor to be heated independently of its circulation.

A. J. HALL.

Processes in which cellulose acetate artificial silk and like products are treated with hot

liquors. Silver Springs Bleaching and Dyeing Co., LTD., and A. J. HALL. (E.P. 246,879, 31.7.24).-Cellulose acetate silk may be treated for prolonged periods in boiling aqueous liquors without becoming curly and wool-like and without loss of lustre and transparency if the liquor contains not less than a certain minimum amount of a protective salt, the minimum quantity being dependent on the particular salt or salts used. Suitable salts include sodium, ammonium, calcium, magnesium, barium, aluminium, strontium, and potassium chlorides; ammonium, sodium, copper, magnesium, zinc, and potassium sulphates; sodium sulphite, alum, chrome alum, sodium chlorate, potassium oxalate, and sodium nitrate. The dycing properties of the cellulose acetate silk are not altered by such treatment. In general, 10-30% solutions of the protective salts are used and they are particularly useful in the treatment of cellulose acetate materials containing other fibres in the dyeing, bleaching, scouring, finishing, or other treatment in which it is necessary to use boiling liquors or liquors at a temperature exceeding 85°. The process has particular application in the dyeing of cellulose acetate-wool union textile materials. A. J. HALL.

Dyeing acetyl cellulose [cellulose acetate] or fabrics containing it and new products [dispersing agents] for use therein. BRIT. DYESTUFFS CORP. LTD., J. BADDILEY, A. SHEP-HERDSON, H. SWANN, J. HILL, and L. G. LAWRIE (E.P. 246,984, 7.1.25. Addn. to 224,077; B., 1925, 39).-Much smaller quantities (e.g., less than 10% calculated on the weight of dry dye) of the dispersing agents consisting of condensation products of naphthalene with formaldehyde as described in the chief patent are utilised for the preparation of solubilised dyes for cellulose acetate silk. For example, a dyestuff paste consists of 10 pts. of aminoanthraquinone, ‡ pt. of the dispersing agent, and 89% pts. of water. Further, complete neutralisation of acidic dispersing agent in the solubilised dyestuff paste by addition of ammonia is preferred to the partial neutralisation with caustic soda described in the chief patent. A. J. HALL.

Dyeing cellulose esters. BADISCHE ANILIN- & SODA-FABR., Assees. of F. GÜNTHEE and F. LANGE (G.P. 420,017, 12.6.23).—Dyeing is carried out with the water-soluble sulphamic acids of the dye, and the colours obtained may be diazctised and developed with chromogens or coupled with diazocompounds. Thus, the azo dye from *p*-aminophenylsulphamic acid and β -naphthol, used in acid solution, gives brownish-red shades, while further diazotisation and treatment with β -naphthol produces a red-violet colour. L. M. CLARK.

Dyeing of textile fabrics [with basic dyes]. W. EBERLEIN, and COLLOISIL COLOUR CO., LTD. (E.P. 247,328, 29.11.24).—Textile materials are treated with colloidal suspensions of natural or artificial silicates insoluble in water, such as "green earth" or "fixing earth," before they are dyed with basic dyes or printed with pastes containing the corresponding colour bases. Alternatively, the dye and colloidal material may be contained in the same bath. The colour lakes precipitated on the fibre are subsequently fixed, *e.g.*, by heating the material or by treating it with an electrolyte. Protective colloids, such as glue, casein, or albumin, may be added to the colloidal suspensions, in which case the material is subsequently treated with dilute formaldehyde, which may be added to the dye-bath. The dyeings produced are fast to light. L. A. COLES.

Bleaching and dyeing furs and the like. H. STEIN, W. E. AUSTIN, and I. LIEBOWITZ, ASSTS. to STEIN FUR DYEING CO. (U.S.P. 1,573,200, 16.2.26. Appl., 3.5.24).—Animal fibres, particularly fur skins, are bleached with hydrogen peroxide solution in the presence of a fibre-protecting agent comprising ferrous sulphate, before they are dyed. L. A. COLES.

Azo dyes stable in steam from nitrosamine colours. CHEM. FABR. GRIESHEIM - ELEKTRON, Assees. of J. GÜRTLER (G.P. 419,431, 20.5.24).— Addition to the printing colours of aromatic nitro- or nitroso-compounds as oxidising agents, permits the development of the dye in full strength and purity of tone, even where prolonged steaming is required. Suitable compounds include nitrosulphonic acid salts of the benzene, naphthalene, and anthraquinone series as well as their halogen-substituted derivatives, and salts of nitrophenolsulphonic and nitrosonaphtholsulphonic acids. L. M. CLARK.

Developing salts. BADISCHE ANILIN- & SODA-FABR., Assees of H. KRZIKALLA (G.P. 421,837, 16.8.24).—Claim is made for anhydrous mixtures of salts of mineral acids, diazotisable aromatic amines (neither nitrated nor sulphonated), and nitrites, to which, if necessary, other acid or indifferent substances are added. The diazo solutions produced on dissolving the mixtures in cold water, can be used immediately for the production of dyes on the fibre. A. COULTHARD.

Production of effect threads from animal fibres. FARBENFABE. VORM. F. BAYER & Co., Assees. of G. RUDOLPH (G.P. 422,464, 26.2.24. Addn. to 407,834).—Very pure effects are obtained by using formaldehyde instead of formic acid, and reducing the proportion of tin salts, in the process described in the chief patent (cf. B., 1925, 240). L. A. COLES.

Dyeing leathers tanned differently from glacé leather. L. CASSELLA & Co., Assees. of F. KOHL (G.P. 422,465, 6.12.23).—The leather is either treated with tanning materials neutralised with alkali immediately before dyeing, or it is dyed from a bath containing both the neutralised tanning material and the dye. Under these conditions, the original tannage is very little affected and level dyeing results. A. COULTHARD.

Improving the fastness to light of insoluble dyes produced on the fibre from arylides of 2:3-hydroxynaphthoic acid which contain a hydroxyalkyl group in the molecule. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of B. SCHÄFER (G.P. 422,468, 3.7.24).—The dyeings are treated with solutions of metal salts above 100° under pressure. For example, dyed cotton is heated for $\frac{1}{2}$ hr. under about 1 atm. pressure with solutions containing 2—3 g. of sulphate of copper, zinc, or nickel, per litre. A. COULTHARD.

Process of dyeing. W. DUISBERG and W. HENTRICH, ASSIS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,575,324, 2.3.26. Appl., 26.11.24).—See E.P. 225,862; B., 1925, 583.

Dyeing apparatus. B. K. THIES (U.S.P. 1,576,378, 9.3.26. Appl., 23.10.24).—See E.P. 232,494; B., 1925, 497.

Dyeing and printing of textile goods and other materials. DURAND ET HUGUENIN S.A., Assees. of M. BADER, T. LOMBARD, C. SUNDER, and C. VAUCHER (U.S.P. 1,575,958, 9.3.26. Appl., 24.7.23).—See E.P. 203,681; B., 1923, 978 A.

Artificial silk (E.P. 245,815).-See V.

Waste waters from dyeworks (Austr. P. 100,735).—See XXIII.

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

Caro's reagent. R. H. VALLANCE (J.S.C.I., 1926, 45, 66 T).—Solutions of potassium permonosulphate, approximating to gram-molecular concentration, have been prepared by triturating potassium perdisulphate with concentrated sulphuric acid below 0°, and neutralising with potassium carbonate under specified conditions.

Catalytic oxidation of ammonia. L. ANDRUSSOV (Z. angew Chem., 1926, 39, 321-332). -The composition of the issuing gases, after passing a mixture of air and ammonia containing 8.3-9.0% NH₃ over a platinum gauze catalyst, has been determined for various temperatures and many different rates of flow. Combustion of the ammonia commences at 160°, at which temperature nitrogen is the chief product, although small quantities (3-6%) of nitric oxide are always present. At and above 400° practically the whole of the ammonia is decomposed. The amount of nitric oxide formed increases with rise of temperature until at 550° this gas is the chief product of the reaction. At 500° side-reactions involving double decomposition of ammonia with nitric oxide as well as thermal decomposition of part of the ammonia and part of the nitric oxide take place, and the proportion of the reaction products consumed in these reactions increases rapidly with rise of temperature and inversely with the velocity of the gases over the catalyst; with a sufficiently high velocity of the gas stream, however, a 90% yield of nitric oxide may be obtained, even at 1000°. These results may be satisfactorily explained on the assumption that

319

an intermediate product, NOH, is formed which may decompose into nitrogen, water, and hydrogen, into nitric oxide and water, or, thermally, into nitrogen and water. A. R. POWELL.

Determination of ammonia in ammoniacal and industrial liquors. C. JUNGBLUT (Bull. Soc. chim., 1926, 39, 336—337).—The following method for the determination of ammonia in ammoniacal liquor avoids the brown discoloration produced when carbon dioxide is expelled by prolonged boiling after acidification with sulphuric acid. The liquor containing the ammonia chiefly in the form of bicarbonate is treated with sodium hydroxide and then with barium chloride. The barium carbonate is rapidly filtered off, the filtrate made neutral to methyl-orange, and the ammonia determined in the usual way by treatment with formaldehyde to convert the ammonia into hexamethylenetetramine, followed by titration of the liberated acid, using phenolphthalein as indicator.

W. HUME-ROTHERY.

Analytical control in permanganate manufacture. H. WALDE and K. A. SCHUCH (Wiss. Veröff. Siemens-Konz., 1925, 4, 188-199).-The method of Aschoff (cf. Sackur, Ber., 1910, 43, 381, 448) is found to be the most satisfactory for determining potassium manganate volumetrically, the alkaline solution being added at 70-90° to the standard oxalic acid solution containing an excess of sulphuric acid. The endpoint is made sharper by the addition of phosphoric acid. The titration was examined electrometrically and the critical potential found to be 0.796 volt measured against a normal calomel electrode. The use of a comparison electrode with a capillary electrometer as zero instrument is also described. The potentiometric and visual methods of determining the end-point were compared in the titration of solutions at various dilutions, the results being in good agreement, but the potentiometric method is to be preferred for dilute solutions. To determine the free alkali in the manganate solutions, the manganate and permanganate are destroyed by addition of hydrogen peroxide, the solution filtered from the precipitated manganese dioxide, and titrated with standard acid. The alkali produced in the decomposition of the manganate must be subtracted from the value observed, and a further correction is necessary for the potassium hydroxide adsorbed by the hydrated manganese dioxide precipitate. The adsorption was found to be 1.039 g. of potassium hydroxide per g. of manganese in 5N-alkali, falling to 0.905 g. per g. of manganese in 0.5N-solution. For the determination of permanganate in presence of manganate a method is suggested based on the quantitative decomposition of the manganate by carbon dioxide: $3K_2MnO_4 + 2CO_2 \rightarrow 2KMnO_4 + MnO_2$ $+2K_2CO_2$. The manganese dioxide is separated, and determined by conversion into pyrophosphate, and the total permanganate is determined by titration. The original permanganate is then obtained by difference, by deducting the quantity of permanganate corresponding to the manganese G. M. BENNETT. dioxide.

Commercial calcium carbide. I. E. SCHLUM-BERGER (Z. angew. Chem., 1926, 39, 213-220).-Microscopical examination of polished surfaces of samples of commercial calcium carbide of varying composition shows that the calcium carbide-calcium oxide eutectic lies in the neighbourhood of 60% of carbide. Calcium oxide crystals cannot, however, be recognised until the carbide content is reduced to about 40%. Products containing 60-40% of carbide show small calcium carbide crystals surrounded by a eutectic mass. The relation between the density and carbide content is a linear one. Measurements of the electrical resistance of commercial calcium carbide show that this varies according to the direction of the current relatively to the arrangement of the carbide crystals as seen microscopically. With decreasing carbide content the resistance increases and reaches a maximum at 65-70% of carbide. Below this, however, it rapidly decreases again, and a 40% carbide conducts electricity 400 times better than a 94% carbide. This may be due to the presence of impurities, but not of graphite (though this is present), since the temperature coefficient of commercial carbide between 25° and 125° is greater than that of graphite. It remains to be proved, indeed, whether absolutely pure calcium carbide is really a conductor of electricity. The fact that commercial carbide of low carbide content reacts more readily with nitrogen is ascribed to the smaller size of the carbide crystals in it, as seen from photomicrographs, with which the work is illustrated. W. T. K. BRAUNHOLTZ.

Analysis of silicates. R. SCHWARZ and A. SCHINZINGER (Z. anorg. Chem., 1926, 151, 214—220). —The silicate is decomposed by fusion with lithium carbonate in a platinum crucible. The product is treated as in the sodium carbonate method except that traces of platinum dissolved from the crucible must be removed with hydrogen sulphide before precipitating aluminium, and magnesium is determined volumetrically by the method of Klingenfuss (B., 1924, 852). Small quantities of sodium and potassium are separated from the lithium salts by first precipitating the bulk of the latter as phosphate, then applying the Gooch method. R. CUTHILL.

Helium for divers. G. GAERTNER (Umschau, 1926, 30, 50—21; Chem. Zentr., 1926, I., 1871— 1872).—Nitrogen at high pressure is the cause of diving sickness, whilst pure oxygen at pressures above 2.5 atm. acts on the lungs as a rapid poison. Helium is innocuous and suitable for the dilution of the oxygen. C. IRWIN.

PATENTS.

Manufacture of sulphuric acid. METALLBANK U. METALLURGISCHE GES., Assees. of H. KLENCKE (G.P. 421,786, 3.6.24).—Deposition of sulphuric acid from mixtures of gases containing sulphur dioxide with a maximum proportion of oxides of nitrogen, is effected in such a manner that solution or precipitation of the oxides of nitrogen is prevented, *e.g.*, by the influence of an electrostatic field. L. A. COLES.

D

Manufacture of sulphuric acid. RHENANIA VEREIN CHEM. FABR. A.-G., ZWEIGNIEDERLASSUNG MANNHEIM, Assees. of W. FRANK (G.P. 422,572, 6.3.23. Addn. to 406,490).—The Gay-Lussac and Glover towers used in the process described in the chief patent (cf. E.P. 212,768; B., 1924, 419), contain no filling material. L. A. COLES.

Manufacture of sulphur dioxide and sulphuric acid from sulphur-bearing slag. GEWERKSCHAFT LUTZ III (G.P. 423,640, 23.1.24).—Enriched air or oxygen is blown through the slag. The gases, with their excess oxygen, after treatment for recovery of acid, are used in the blast furnace. C. IRWIN.

Removing arsenic from burner gases. METALLBANK U. METALLURGISCHE GES. A.-G., Assees. of O. KURZ (G.P. 423,657, 21.3.24. Addn. to 368,283; B., 1923, 400 A).—After the electrical precipitation of flue-dust the gas is cooled sufficiently to form clouds but without addition of moisture. Arsenious oxide is then deposited electrically as dust, prior to the treatment in presence of sulphuric acid described in the chief patent. C. IRWIN.

Production of phosphoric acid and hydrogen. F. G. LILJENROTH (F.P. 595,987, 31.3.25. Conv., 15.4.24).—Metal phosphides reducible by hydrogen are treated at raised temperatures with water or steam, oxygen being added if necessary. L. A. COLES.

Manufacture of phosphoric acid. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of R. SUCHY (G.P. 423,275, 18.1.25).—Phosphorus or gases containing phosphorus are burnt in a closed vessel, the walls of which are continuously moistened with phosphoric acid solution. A. R. POWELL.

Manufacture of arsenic acid and arsenates. BADISCHE ANILIN- & SODA-FABR., Assees. of J. BRODE and K. KLEIN (G.P. 423,276, 1.1.25). Arsenic trioxide or an arsenite is heated under pressure with water and gases containing oxygen to a temperature above the b.p. of the solution. A. R. POWELL.

Utilisation of the energy liberated during the oxidation of hydrogen compounds of, e.g., sulphur, nitrogen, and phosphorus, to mineral acids. E. R. BESEMFELDER (G.P. 421,665, 9.3.20).— Mixtures of oxygen with hydrogen compounds of, e.g., sulphur, nitrogen, or phosphorus, are exploded in the cylinder of a gas engine, and the exhaust gases are cooled and condensed in suitable heat-exchanging apparatus. The explosive power of the mixtures may be modified by the addition of non-reacting gases. L. A. COLES.

Manufacture of hydrochloric acid and magnesia from magnesium chloride. E. FRANK (G.P. 422,322, 24.8.24).—Magnesium chloride which has been partially dehydrated by heating to $500-600^{\circ}$ in a rotating furnace, is partly decomposed by external heating alone and decomposition is completed by external heating aided by treatment with a counter-current of steam. L. A. COLES.

Shaft furnaces [for burning limestone etc.]. E. CORNET (E.P. 245,653, 16.8.24).—The object of the invention is to ensure the combustion of carbon monoxide which is always generated in shaft furnaces for burning limestone etc. Air, preheated by waste heat in flues disposed in the furnace wall, passes thence into vertical flues in the refractory lining of the furnace. These flues are controlled by dampers and have outlets leading directly into the furnace in the zone most favourable to combustion of carbon monoxide and also in the calcining zone. Preheated air may also be introduced centrally into the furnace. R. B. CLARKE.

Lime-kiln. H. MISCAMPBELL (U.S.P. 1,572,156, 9.2.26. Appl., 16.2.25).—A lime-kiln consists of a vertical shaft with two parallel walls and two downwardly diverging walls, a port for injecting heating gas being arranged in the lower portion of the shaft. A. R. POWELL.

Treatment aluminous materials. of H. SPENCE, W. B. LLEWELLYN, and P. SPENCE AND SONS, LTD. (E.P. 247,078, 6.8 and 18.9.24).-Aluminium sulphate is prepared by circulating sulphuric acid through aluminous material, such as shale, silicious bauxite, and the like, in small lumps provided these are fairly hard and contain sufficient silica to prevent disintegration. Friable bauxite and the like cannot be used directly as the circulation would be blocked. In order to make such materials available they are agglomerated with plastic clay, sodium silicate, or other suitable binder and baked at a dull red heat. The pieces so produced may be broken so as to pass a 1-in. sieve and then extracted as usual. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1917 and 1919, to E.P. 119,924 and 112,881.) C. IRWIN.

Production of alkali silicates soluble in water. B. E. D. KILBURN. From NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB (E.P. 247,439, 16.5.25).—Hydrated silicic acid prepared by treating leucite or a similar silicate with nitric acid is heated with an alkali nitrate or nitrite. Reaction commences at 250° and is complete at 600—700° without melting. The nitrous gases evolved are recovered and the porous solid product is dissolved in water and the solution filtered. Diatomaceous earth may also be used as a source of silica. C. IRWIN.

Alkali silicate powder. F. FINZI (F.P. 597,825, 5.3.25).—Solutions of alkali silicates are atomised by the aid of compressed air and a current of hot air. A. R. POWELL.

Treating [purifying] zinc sulphate solutions. R. B. ELDRIDGE (U.S.P. 1,573,233, 16.2.26. Appl., 13.10.24).—Inorganic impurities are precipitated by the action of a soluble hyposulphite, and the solution is filtered. C. IRWIN.

Producing borax and sodium bicarbonate from lake brines. M. V. LOWRY, ASST. to WEST END CHEMICAL Co. (U.S.P. 1,573,259, 16.2.26. Appl., 10.4.23).—The brine is concentrated by evaporation, carbon dioxide passed in, and the mixture cooled. Borax and sodium bicarbonate separate as a sludge. This is mixed with colemanite and heated to boiling point. C. IRWIN.

Producing light basic magnesium carbonate. R. B. CROWELL, ASST. to WESTERN INDUSTRIES CO. (U.S.P. 1,573,632, 16.2.26. Appl., 26.6.25).— Magnesium oxide in aqueous suspension is treated with carbon dioxide until the ratio of CO_2 to MgO lies between 0.8 and 0.9. The product is then heated to a temperature not above 60°.

C. IRWIN.

Manufacture of alkali carbonates. L. BRADLEY and E. P. MCKEEFE (Can.P. 248,096, 18.6.23).— Material containing cellulose is digested with neutral alkali sulphite solution, the solution, after separation of the cellulosic material is evaporated to dryness, and the residue is calcined and fused. L. A. COLES.

Treatment of natural alkali salts of secondary and tertiary origin. A. LAMBERT (Can.P. 249,255, 12.6.24).—A solution of sodium chloride, sulphate, and carbonate obtained from native deposits is treated with carbon dioxide to precipitate sodium hydrogen carbonate, the mixture is heated to 330° under pressure, and the salt separated from the liquid without cooling. A. R. POWELL.

Decomposition of sodium ammonium sulphate. E. A. E. WACHÉ (F.P. 594,694, 31.5.24). -Sodium ammonium sulphate and sodium chloride are added in the proportions required by the equation, $Na_2(NH_4)_2(SO_4)_2, 4H_2O+2NaCl=2Na_2SO_4+2NH_4Cl+$ $4H_2O$, to a solution saturated at 30° with ammonium chloride and sodium sulphate, in such quantities that anhydrous sodium sulphate but no ammonium chloride is thrown out of solution on heating to 90-100°. After removing the sodium sulphate, the solution is cooled, ammonium chloride which crystallises out is removed, and the mother liquor is used again in the process. L. A. COLES.

Ammonium sulphate saturator. Soc. DE FOURS λ COKE ET D'ENTREPRISES IND. (F.P. 596,494, 9.4.25).—A saturator for the manufacture of ammonium sulphate is provided with a centrifugal pump in the axis of the saturator above the floor. The pump is driven by a motor on the roof of the saturator and serves to prevent settling of the ammonium sulphate on the bottom. A. R. POWELL.

Bleaching and purifying alkaline-earth sulphates, especially barium sulphate. A. A. ACKERMANN (F.P. 596,640, 15.4.25).—Alkaline-earth sulphates are purified by treatment with hydrogen fluoride or compounds derived from it. L. A. COLES.

Preparation of the gas mixture for ammonia synthesis. E. EDWIN (F.P. 596,714, 11.4.25).— A mixture of steam and the vapour of a hydrocarbon is subjected to the action of the electric arc and the resulting mixture of carbon monoxide and hydrogen is passed together with steam and air into a gasproducer. Part of the issuing gases is passed back over the arc, while the remainder is subjected to the action of a catalyst to convert the carbon monoxide and steam into carbon dioxide and hydrogen, the former being subsequently removed by known methods. A. R. POWELL.

Recovery of vanadium compounds from iron ores containing vanadium and titanium. B. P. F. KJELLBERG (F.P. 598,315, 16.5.25. Conv., 9.11.24 and 12.2.25).—The ore is roasted in such a way that the vanadium compounds are prevented from oxidising to vanadium pentoxide and the roasted material is then leached with a solvent for the vanadium. A. R. POWELL.

Preparation of pure bismuth vanadate. E. ZINTL and L. VANINO (G.P. 422,947, 9.8.24).— Bismuth vanadate for therapeutic use is precipitated by the addition of vanadates, at the ordinary or at raised temperatures, to solutions containing bismuth salts and substances which prevent the separation of basic bismuth compounds, such as acetic acid, mixtures of acetic acid and acetates, small quantities of mineral acids, polyhydric alcohols, or ketones. The product is subsequently heated in the air in case any reduction has taken place during the precipitation. L. A. COLES.

Adsorption of sulphur dioxide from gas mixtures. M. SCHROEDER (G.P. 421,725, 13.8.24). -Gas mixtures containing sulphur dioxide are treated under pressure with a counter-current of water for absorbing the sulphur dioxide, in a tower in which by regulating the escape of the unabsorbed gases, the pressure is maintained constant throughout the process. The expansibility of the residual gas, after passage through heat-exchangers in which it is heated by the heat of compression of the freshly compressed gas or by other means, is utilised, e.g., in expansion cylinders, for compressing fresh supplies of the gas before entry into the tower. The pressure prevailing in the absorption tower forces the sulphur dioxide solution continuously into apparatus at the top of the tower, where the sulphur dioxide is expelled by heating. L. A. COLES.

Production of potassium nitrate. WOLFF U. Co., Assees. of H. HAMPEL (G.P. 421,988, 24.5.21).— Potassium nitrate is produced by interaction of potassium chloride and calcium or magnesium nitrate in the cold. L. A. COLES.

Production of finely divided iron oxide. BADISCHE ANILIN- & SODA-FABR., Assees. of A. MITTASCH (G.P. 422,269, 24.5.24).—The product is obtained by burning iron carbonyl, or mixtures of it with other combustible gases, or with inert gases. L. A. COLES.

Preparation of insoluble silver alkali thiosulphates. CHEM. FABR. SCHLEICH G.M.B.H., and A. ROSENHEIM (G.P. 422,323,7.8.24).—Insoluble silver alkali thiosulphates are produced by adding approximately the calculated quantity of silver salts, particularly silver halides, to concentrated alkali thiosulphate solutions at 30°, the mixture being thoroughly stirred. The insoluble compounds are rendered water-soluble by agitation with highly concentrated alkali thiosulphate solution at 0°.

L. A. COLES.

Production of alkaline-earth chlorides. VEREIN FÜR CHEM. U. METALL. PRODUKTION (G.P. 422,470, 17.7.23).—Alkaline-earth chlorides are produced by heating alkaline-earth carbonates with solid magnesium chloride. L. A. COLES.

Production of barium, strontium, and calcium peroxides. H. SCHULZE (G.P. 422,531, 19.2.24).— Hot or cold solutions containing alkaline-earth sulphides or mixtures of these with alkaline-earth hydroxides, are electrolysed in an open or closed anode chamber, which is separated from the cathode chamber, using anodes constructed of iron or other metals, or of carbon. Air free from carbon dioxide, ozonised air, oxygen, or "active oxygen" may be passed continuously or intermittently into the solution. L. A. COLES.

Evaporation of brine. MASOHINENBAU-A.-G. BALCKE (G.P. 422,657, 25.4.22).—Brine is concentrated until gypsum settles out, in pans heated directly by furnace gases, and is subsequently concentrated to deposit salt, in evaporators heated by steam generated during the first stage of the evaporation; the steam is first superheated by waste combustion gases from the furnace. The heat of the furnace gases is also used for preheating the brine charged into the pans. L. A. COLES.

Preparation of brine free from gypsum. SALZBERGWERK NEUSTASSFURT (G.P. 423,861, 26.11.24).—To the water used for dissolving impure rock salt containing gypsum such salts are added as diminish the solubility of calcium sulphate. These salts may be added to the brine after evaporation in the pans or used in the boreholes etc. C. IRWIN.

Apparatus for the manufacture of hypochlorite solutions. BADISCHE ANILIN- & SODA-FABE. (G.P. 422,725, 3.3.25).—A vessel with a non-metallic surface is surrounded by an outer jacket containing a non-reacting liquid in which is immersed a tube supplied with liquid chlorine, and the chlorine gas generated by its evaporation is conveyed through non-metallic tubes into alkali solution contained in the inner vessel. L. A. COLES.

Production of ammonia, sulphur, and thiosulphates. FABR. CHEM. PROD. F. HEFTI, and W. SCHILT (G.P. 422,726, 23.10.23. Conv., 24.9.23).—The products are obtained by reducing nitrites, especially alkali nitrites, with two mols. of hydrogen sulphide. L. A. COLES.

Complete utilisation [of the end liquors from] crude potassium salts. Wolff & Co., and F. FROWEIN (G.P. 422,987, 24.8.24).—The liquors are subjected to two cycles of treatment in one of which the sulphate ions are converted into ammonium sulphate with the aid of calcium carbonate and in the other of which the chlorine ions are converted into ammonium chloride with the aid of lead carbonate. Sodium, potassium, and magnesium nitrates are recovered during the course of the process. The magnesium nitrate is converted into a fine-grained, stable fertiliser by mixing the concentrated mother liquors with potassium sulphate at 60—70°, and stirring, kneading, and finally grinding the mixture. A. R. POWELL.

Manufacture of alkaline-earth nitrides. F. UHDE (G.P. 423,348, 6.12.21).—The oxides, hydroxides, or carbonates of alkaline-earth metals are converted into nitrides by treating them with hydrogen or hydrocarbons in an electric furnace and subjecting the metallic regulus so obtained to the action of nitrogen. A. R. POWELL.

Working up barium sulphide. RHENANIA VEREIN CHEM. FABR. A.-G. ZWEIGNIEDERLASSUNG MANNHEIM, and F. RÜSBERG (G.P. 423,755, 8.9.22).— Crude barium sulphide is decomposed with hydrochloric acid or dilute nitric acid. The hydrogen sulphide evolved is absorbed in alkali sulphite or bisulphite in order to prepare alkali thiosulphate by the method of G.P. 370,593 (B., 1923, 499 A). C. IBWIN.

Production of nitrogen peroxide from calcium nitrate. L'Azote Français, Soc. Anon. (Swiss P. 99,037, 7.10.21).—The gas is obtained by heating calcium nitrate above 500°, but below 650°, in the presence of oxygen. L. A. Colles.

Production of material containing silicon and carbon. GEBR. SIEMENS & Co. (G.P. 421,951, 11.12.21; F.P. 554,683, 2.8.22).—The material is obtained by heating, e.g., for 5—10 hrs., mixtures of 3—5 pts. of silicon carbide, 4—8 pts. of sand, and 1—2 pts. of carbon, to $1700-2000^{\circ}$, preferably to 1900°. The properties, particularly the electrical conductivity, of the product are regulated by the duration of heating, the temperature, and the proportions of the constituents. The addition of sawdust to the constituents renders the product porous. L. A. COLES.

Production of corundum in electric furnaces. H. VIERHELLER (G.P. 422,105, 24.9.24).—In the production of corundum in the electric furnace, material evolving gases, containing, *e.g.*, peroxides, carbonates, or ammonium salts, preferably moulded into solid form together with iron filings, is immersed in the fused bath at the end of the fusion and reduction process. L. A. COLES.

Magnesium hydroxide. A./S. DE NORSKE SALTVERKER (Nor. P. 39,564, 27.5.21).—Magnesium hydroxide is produced by the electrolysis of solutions of magnesium sulphate at temperatures above 90°, preferably at the b.p. A. R. POWELL.

Recovery of neon and helium from the air. GES. FÜR LINDE'S EISMASCHINEN A.-G. (G.P. 417,572, 9.12.22).—The residual gases from the rectification of liquid air are further cooled under 10 atm. pressure to remove the bulk of the remaining nitrogen, and the uncondensed gas is passed, under pressure and at the same temperature, through a tube filled with adsorbent charcoal which removes the last of the nitrogen. The purified noble gases are then charged directly into steel cylinders.

A. R. POWELL.

Refining process for pearl essence. J. PAISSEAU (U.S.P. 1,576,454, 9.3.26. Appl., 22.12.23). —See E.P. 188,774; B., 1923, 55 A.

Manufacture of sodium formate from carbon monoxide. M. ENDERLI, Assr. to R. KOEPP & Co. (U.S.P. 1,574,875, 2.3.26. Appl., 28.2.23).— See E.P. 165,163; B., 1921, 583 A.

See also pages 303, Separation of gaseous mixtures (E.P. 232,986). 304, Evaporation of solutions (E.P. 247,346). 309, Gas mixtures containing nitrogen (Swiss P. 112,359). 331, Kilns for lime burning etc. (G.P. 416,143). 332, Production of fatty acids, glycerin, and ammonium sulphate (G.P. 421,438). 333, Blanc fixe and sodium thiosulphate (G.P. 420,251).

VIII.—GLASS; CERAMICS.

Physical properties of porcelain. K. WETZEL (Ber. Deuts. Keram. Ges., 1925, 6, 23-40; Chem. Zentr., 1926, I., 762-763).-Addition to the normal porcelain mixture (50%) of clay, 25% of quartz, 25% of felspar) of 3% of various ingredients gave the following results: Lime lowered the m.p. by 6 Seger cones, appreciable softening occurring at cone 10. This oxide produced a fairly low modulus of elasticity (6800 kg./sq. mm.), a resistance to bending shock of 2.06, a high value of coefficient of expansion, and a low resistance to temperature change. Much undissolved quartz could be detected microscopically, and the transparency at cone 10 was not very good. Magnesia gave the greatest depression of m.p. (to cone 17), the interval between softening and melting being approx. 280°. The modulus of elasticity was little changed (7590 kg./sq. mm.), whilst other properties were similar to those of the lime mixture. Addition of talc produced an interval between softening and melting of 420°, a modulus of elasticity of 8166 kg./sq. mm., and the highest resistance to bending shock (2.12). The coefficient of expansion and thermal endurance were about the same as with lime and magnesia. Dolomite gave similar results to magnesia but the transparency slowly increased with rise of temperature. Softening and melting results for fluorspar were similar to those for tale, but the modulus of elasticity was lower (6840 kg./ sq. mm.). Calcium fluoride produced a resistance to bending shock of 2.07, a fairly high expansion coefficient, and a very small thermal endurance. Ferric oxide gave a low sintering temperature (1150°), but a large interval of approx. 470° between softening and melting. The modulus of elasticity was 7650 kg./ sq. mm., the resistance to bending shock 1.87; the thermal expansion coefficient was high, the thermal endurance low, and transparency was poor. Zinc oxide raised the m.p. to cone 31, and gave a large interval between softening and melting (approx. 470°). The resistance to bending shock was 1.74, and the porcelain had the lowest thermal expansion coefficient (38×10^{-7}) , this coinciding with high thermal endurance. Microscopical examination

revealed no undissolved quartz. Mixtures containing alumina, titania, or zirconia sintered above 1200°. With titania and zirconia the m.p. was cone 27–28, with alumina cone 32. All three oxides, but particularly alumina increased the modulus of elasticity and the resistance to bending shock ($TiO_2 1.75$, $AI_2O_3 1.86$). The transparency with zirconia was very low, with titania nil. In all cases there was a large formation of mullite. A. COUSEN.

Pottery from the ancient "oppidum" of Vindalium (Vaucluse). L. DESVERGNES (Ann. Chim. Analyt., 1926, 8, 40–41).—Two types of pottery found on the Sève mountain on the site of the ancient town of Vindalium, classed respectively as (1) soft and fine and (2) hard and coarse, had the following characteristics :—fracture (1) ash coloured and homogeneous, (2) yellow periphery and dark brown centre; d (1) 1.718, (2) 1.885; loss at red heat (1) 21.26, (2) 8.31; SiO₂ (1) 50.07, (2) 70.65; Al₂O₃ (2) 16.92, (2) 8.55; Fe₂O₃ (1) 6.46, (2) 9.49; CaO (1) 5.13, (2) 2.79; MgO (1) 0.13, (2) 0.21; Mn, traces in both samples. D. G. HEWER.

PATENTS.

Lead - free enamels. C. TOTOT-GIBARU (F.P. 597,146, 6.8.24).—An enamel flux free from lead consists of 2—20 pts. of silica, 5—35 pts. of zinc oxide, 30—70 pts. of crystallised borax, 2—20 pts. of alkali nitrate, 0—8 pts. of alkali fluoride, and 0—30 pts. of calcium carbonate. Tin oxide or antimony trioxide is used as opacifier.

A. R. POWELL.

Manufacture of opacifiers for enamels. I. KREIDL (G.P. 422,600, 29.11.24. Conv., 24.11.24).— Zircon is decomposed by fusion with alkaline reagents and the product so obtained is treated with hydrofluoric acid to convert the combined and adsorbed silica into silicon fluoride. The amount of silica retained by the zirconia is a function of the temperature of fusion and of the quantity of alkali used in the fusion. A. R. POWELL.

Sifting of granular or pulverulent materials [potters' slip]. A. C. HARRISON (E.P. 248,121, 13.12.24).

Material containing silicon and carbon (G.P. 421,951).—See VII.

IX.—BUILDING MATERIALS.

Three-component system lime-silica-alumina and hydraulic binding materials. R. GRÜN (Zentr. Bauverwalt., 1926, 46, [1], Reprint. 3 pp.).— Ceramic bodies and cementitious materials consist essentially of lime silica, and alumina; the cementitious or hydraulic properties of the binding materials depend on their lime content, as represented by the calcium aluminates and calcium silicates present. The calcium aluminates, which harden very quickly, are the most important constituents of aluminous cements and high alumina blast-furnace slags, while the calcium silicates present determine the slower setting of Portland cement and blast-furnace cements. No hydraulic binding materials are known which do not contain lime. B. W. CLARKE.

Thermal investigations on blast-furnace slags. R. GRÜN (Mitt. Forschungsinst. Hüttenzement-Ind., 1925. Reprint. I. Latent energy of blast-furnace slags, 5-19. II. Latent energy of blast-furnace slags and the single components of the threecomponent system silica-lime-alumina, 20-39). -The hydraulic properties of blast-furnace slag depend on the development of a vitreous product, which passes into the crystalline form when heated to about 800°, with the evolution of heat. The heat evolved during this transition is not a direct measure of the hydraulic properties of the slag cement, except with slags of the same composition, but depends on the extent of vitrification, and may therefore be used as an exact method for comparing the efficiency of various granulation processes from the point of view of the hydraulic properties of the slags. Thus, preliminary cooling of the molten slag before granulation, and overheating of the slag gives a low vitreous content and poor hydraulic properties; air granulation, although giving less vitrification than the wet process, produces slag more active chemically and having the better hydraulic properties. Calorimetric determination of the heat of solution of slags of similar composition gives a more exact comparison of the energy content, i.e., extent of vitrification, than a microscopical examination can give. The heat of hydration of a slag may be calculated from the heat of solution of the slag after deducting the heats of solution of the hydroxides produced by complete hydrolysis; the relative proportions of these can be determined from the chemical composition of the slag. The heat of hydration, however, bears no direct relationship to the hydraulic properties of the These depend chiefly on the chemical composlag. sition of the material, e.g., in Portland cement the heats of solution and hydration depend chiefly on the proportion of tricalcium silicate present, while the dicalcium silicate and fluxes affect the setting or hydraulic properties of the cement. The energy contents of the single components of three- or fourcomponent systems, such as the various types of blast-furnace slag, are most conveniently studied by the determination of the heat of solution, which gives a more accurate knowledge of the physical nature of the slag, e.g., extent of devitrification, than a microscopical examination. B. W. CLARKE.

Utilisation of freshly made, hot cement. R. GRÜN and W. MUTH (Baumarkt, Leipzig, 1925, [5]. Reprint, 1 p.).—Blast-furnace cement, still hot from the grinding mills, shows no reduction in strength compared with cement which has been allowed to mature for some time before use. Similar results are to be expected with Portland cement. This disproves the view generally held that the use of freshly made, hot cement is dangerous from the strength standpoint. B. W. CLARKE.

PATENTS.

Proofing substances against insects (E.P. 247,242).—See V.

Lime-kiln (U.S.P. 1,572,156).—See VII.

Kilns for lime burning etc. (G.P. 416,143).— See XI.

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

Melting grey cast-iron in the electric furnace. E. RICHARDS (Stahl u. Eisen, 1926, 46, 249-254). -Electric furnace cast-iron has better mechanical properties, is denser, and has a finer grained fracture than that melted in the cupola. In the basic-lined furnace the sulphur may be reduced from 0.2% to 0.05% in about 30 min., the iron is deoxidised, and the phosphorus content also lowered. The foundry is made independent of the coke supply, and the use of expensive pig iron is unnecessary. Fine scrap can be melted without loss, and large castings can be made without trouble. With the acid-lined electric furnace the advantages of purification of the metal are lost, but, on the other hand, a basic lining has a tendency to flake when subjected to temperature changes, and with intermittent operation its working life may be no greater than 8 days. Neutral lining materials were investigated. A carbon hearth, although preventing the formation of slag inclusions resulting from dissolved oxides, is strongly attacked along the slag-metal line. As the electric melting process is expensive, the "Duplex" process in which the cupola is used in combination with a basic electric furnace is recommended. The coke used in the cupola should be of low sulphur content. The chemistry of grey iron melting in a basic electric furnace is discussed with special reference to the behaviour of the manganese in the bath. Oxygen is removed by both the manganese and the carbon; calcium carbide is formed in the neighbourhood of the electrodes, and sulphur is removed by the action of the lime in combination with the carbon and calcium carbide. The production of synthetic cast iron from small steel scrap in the basic electric furnace is described. Silicon is taken up from the basic slag or may be added more quickly as ferrosilicon. Ferromanganese is added in the ladle. Synthetic grey iron containing 2.65% of total carbon had a tensile strength of 32-35 kg./mm.² and d 7.5. The iron was dense and free from inclusions, the graphite being finely devided in a ground-mass of pearlite and silicon-ferrite. T. H. BURNHAM.

Influence of the grain size on the magnetic properties of sheet silicon-iron. O. VON AUWERS (Wiss. Veröff. Siemens-Konz., 1925, 4, 266—273).— A more detailed account of work the results of which have been published previously (cf. B., 1926, 15). G. M. BENNETT.

Dilatometric investigation of the A3 and A4 transformations in pure iron. S. SATÔ (Sci. Rep. Tôhoku, 1925, 14, 513—527).—In the case of pure iron, during heating, the A4 transformation is accompanied by an expansion and the A3 transformation by a contraction, these changes being reversed on cooling and the ratio of the magnitudes of the change in length being about A4:A3::10:3. As the temperature reaches 1400° an abrupt increase in length is observed due to the A4 transformation and then the rate of expansion becomes less than in the γ region and the deflection-temperature curve lies in the prolongation of that of *a*-iron, indicating that δ iron is the same phase as *a*-iron. C. A. KING.

Solidus line in the iron-carbon system. S. KAYA (Sci. Rep. Tôhoku, 1925, 14, 529-536).— The solidus line in the iron-carbon system was determined by the electrical resistance method using Swedish steels ranging from 1.60% C (solidus point 1140°) to 0.23% C (solidus point 1444°). The solidus line as determined lies between the curve as obtained by Carpenter and Keeling (B., 1904, 608) and that of Gutowsky (B., 1910, 91). The solubility limit of cementite determined from the point of intersection of the solubility and the eutectic lines was 1.7% of carbon, as at present accepted.

C. A. KING.

Determination of the heat of precipitation of cementite from a- and β -martensites. M. KAWA-KAMI (Sci. Rep. Tôhoku, 1925, 14, 559—568).— The heat of precipitation of cementite from a-martensite was measured for eight kinds of carbon steel $(0^{3}-1^{3}2\%)$ C), by tempering a specimen in a calorimeter containing a eutectic mixture of potassium, sodium, and lithium nitrates, and maintained in a thermostat. Differentiation between a- and β martensite was effected by tempering a-martensite below 200° and both the forms at above 300°. The heat of precipitation increases proportionally to the content of carbon and for eutectoid steel is 3.7 cal. per gram (a-martensite). The heat from a-martensite is about half as large as that from β -martensite.

C. A. KING.

Influence of thermal treatment on silver alloys. L. GUILLET and J. COURNOT (Compt. rend., 1926, 182, 606—609).—The results of the thermal treatment of two silver-zinc and two silvercadmium alloys containing 69.77, 62.28, 59.96, and 49.47% Ag, respectively, are recorded. On annealing the first alloy two constituents appear, one of which increases on quenching at 400°. On tempering a finely-divided precipitate appears in this constituent, accompanied by an increase in hardness and brittleness. Exactly analogous changes are shown by duralumin. The second alloy behaves in the same way. The results for the silver-cadmium alloys are less definite, though for both types of alloy the accepted phaserule diagrams are apparently not quite correct.

S. K. TWEEDY.

Hardness of cold-rolled copper. S. L. HOYT and T. R. SCHERMERHORN (Inst. Metals, Mar., 1926. Advance copy, 24 pp.).—Hardness tests were carried out by the scleroscope, the Rockwell hardness tester, and by ball indentation tests on two series of cold-rolled electrolytic copper bars, originally 0.75 in. square in section, one series receiving a 2% and the other a 10% reduction in thickness per pass. The results confirm the accepted form of the relationship between the degree of working

and the hardness of copper. The reversal in hardness with excessive cold working, noted by Rawdon and Mutchler (B., 1924, 260), was not found. The 10% reduction series gave a slightly greater increase in hardness than the 2% series. The hardness over the cross-section was found to be uniform for reductions of above 20%, but with small reductions in thickness the top surface was harder than the side of the bar. No definite relationship was found to exist between the hardness numbers of annealed and cold-worked copper as obtained by the three methods used. The time of loading in the ball indentation test was from one to several hours, since true equilibrium between the ball and the sample was not obtained in the standard time of 30 sec. Meyer's constant n (in the equation $P=ad^n$) decreases from about 2.4 for annealed copper to an approximately constant value of 2.0 at about 20% reduction in thickness, but as the hardening is by no means completed at this point, it follows that the constant n does not apply accurately to deformations due to cold-rolling. The increase noted in a after n has attained a constant figure is discussed, a possible explanation being that while further cold-rolling hardens the metal, the indentations made by the ball have no such effect.

A. W. HOTHERSALL.

Soft soldering of copper. T. B. CROW (Inst. Metals, Mar., 1926. Advance copy, 14 pp.).-The interfacial effects in the soft soldering of copper were studied by the micro-examination of sections of joints prepared by soaking the ends of electrolytic copper rods in a bath of solder of eutectic composition for 10-20 min. at temperatures ranging from 237° to 497°. The solder was allowed to solidify in situ after adjusting the gap between the rods to the desired dimension. In the case of joints made at temperatures below 300°, a chemical reaction takes place at the copper surface which involves the production in the solid state of a structureless greyish-white band of the constituent "H" (CuSn). The extent of this reaction depends upon time and temperature and may be so slight as to leave traces of original surface markings upon the copper. The constituent "H" is soluble in excess of tin, so that increased periods of soaking do not produce thicker bands. The adhesion between the "H" constituent and the copper is relatively high, fracture in the tensile test never occurring so as to expose raw copper. At temperatures higher than 300° there is formed between the "H" and the copper a blue-mauve material which has been identified with the η phase of the bronzes (Cu₂Sn). It is weaker and more brittle than the "H" constituent. The increase of tensile strength of joints with diminished thickness of solder film is progressive until the film becomes discontinuous as a result of union of the two bands of "H" constituent across A. W. HOTHERSALL. the gap.

Loss of zinc by brass in corrosion. G. MASING and L. KOCH (Wiss. Veröff. Siemens-Konz., 1925, 4, 257-265).—Systematic experiments on the selective corrosion of brass show that the process involves the complete solution of the brass and subsequent reprecipitation of the copper. It is due to the potential difference between brass and copper, and may therefore be regarded as an electrolytic phenomenon. G. M. BENNETT.

Brittle ranges in bronze. W. L. KENT (Inst. Metals, March, 1926. Advance copy, 8 pp).— The brittle ranges have been investigated by the Izod impact test at temperatures up to 700° on alloys, in both the cast and annealed condition, containing up to 25% Sn. The brittle copper-tin alloys containing δ in the form of $a + \delta$ eutectoid lose their brittleness at 520° due to the replacement of δ by β . The β constituent so formed is soft and thus these alloys are workable above this temperature. Owing to the softness of the metal the upper limit of the range of malleability in the eutectoid alloys has not been determined, but the range extends over at least 150°. M. Cook.

Die-casting of aluminium alloys. G. MORTIMER (Inst. Metals, Mar., 1926. Advance copy, 27 pp.).— The slush, centrifugal, Cothias, gravity, and pressure methods of die-casting are reviewed. Of these the last two are most generally applicable to industry. Economic considerations are an important factor in the choice between gravity and pressure methods and the former seems more suitable for industrial requirements in Great Britain. The equipment and operation of a gravity casting installation are described. M. COOK.

Effect of artificial ageing on aged aluminium alloys. K. L. MEISSNER (Metal Ind., 1925, 26, 623-626; Chem. Zentr., 1926, I., 1029; cf. B., 1925, 321).—Two aluminium alloys, one containing copper and both of which aged at ordinary temperatures, were subjected to artificial ageing. The treatment consisted of annealing at various temperatures followed by air cooling. The most favourable annealing temperature was 125°. The physical properties of the alloy containing no copper were thereby improved in contrast to those of the copper alloy. The difference is ascribed to the inertness of the compound CuAl₂. L. M. CLARK.

Influence of ageing on the corrodibility of aluminium alloys. K. L. MEISSNER (Korrosion u. Metallschutz, 1925, 1, 206—208; Chem. Zentr., 1926, I., 1704).—Artificial ageing of aluminium alloys containing copper but no magnesium, e.g., lautal, reduces the resistance to corrosion as measured by the oxidising salt test of Mylius (B., 1925, 552). This is probably due to the separation, during ageing, of the excess copper in a highly dispersed ultramicroscopic form. The best tensile and mechanical properties of these alloys are obtained after an ageing treatment that yields an alloy which loses 70 g. of metal per sq. m. per day in the salt test.

A. R. POWELL.

Specifications for aero-alloys. S. DANIELS (Foundry, 1925, 53, 1003—1006; Chem. Zentr., 1926, I., 1702).—Details are given of the metals and alloys used in the manufacture of light alloys

for aeronautical work. The six principal alloys used are as follows: (1) 8% Cualuminium alloy, d 2.85, with a minimum tensile strength of 21,000 lb. per sq. in., an elongation of 1%, a Brinell hardness of 60, and a limit of proportionality of 7000 lb. per sq. in. (2) 88.5% Al, 10% Cu, 1.25% Fe, and 0.25 M in. (2) at 100 m it a limit of proportionality of 7000 lb. 0.25 Mg; this alloy retains its hardness and tensile strength at high temperatures. Its ultimate strength is 25,000 lb. per sq. in., elongation 1%, and hardness 80; after ageing at 20° for 6 months these values become 27,000 lb., 0%, and 95 respectively and remain unchanged up to 260°. With suitable heat treatment the hardness of the sand-cast alloy may be increased to 120 and that of the chill-cast to 150°. The microstructure shows a network of CuAl, crossed by long needles of the iron constituent with traces of Mg2Si. (3) 95.75% Al, 2.5% Cu, 1.25% Fe, and 0.5% Mg. This alloy is suitable for the manufacture of small parts required to withstand shock and compression. After annealing at 525° for 2 hrs., quenching in boiling water, and ageing at 150° for 2 hrs. it has a tensile strength of 31,000 lb. per sq. in., an elongation of 3%, and a hardness of 68. (4) 94% Al, 5% Cu, 1% Si, is one of the most useful alloys on account of its high tensile strength and resistance to compression; the necessary heat treatment consists of an anneal at 510° for 24-96 hrs., quenching in cold water, and ageing for 2 hrs. at 150°. (5) 93% Al, 4% Cu, 3% Si. This alloy is suitable for large castings; its microstructure consists of thin feathery crystals of CuAl₂, dispersed masses of the aluminium-silicon eutectic, a fair amount of needles of unknown composition, and cubes of a blue grey constituent in a ground mass of aluminium-rich solid solution. (6) 92.5% Al, 4% Cu, 2% Ni, and 1.5% Mg. The cast alloy has a tensile strength of 26,000 lb. per sq. in., an elongation of 0.5%, and a hardness of 80; after ageing for 6 months at 15-20° the corresponding values are 28,000 lb., 0.5%, and 95. The tensile strength at 260° is 23,000 lb. per sq. in. and after suitable heattreatment may be increased to 36,000 lb. The microstructure shows CuAl₂, NiAl, Mg₂Si, the ternary copper-nickel-aluminium compound, and a ferruginous constituent derived from impurities in the metals used. A. R. POWELL.

Analysis of commercial magnesium. R. GUÉRIN (Ann. Chim. analyt., 1926, 8, 34-40).-Chlorine is determined colorimetrically by means of a 0.6% solution of silver nitrate if 0.01% or less is present, or gravimetrically if more than 0.01% is present. Silica is determined as in aluminium by dissolving in a mixture of equal volumes of water and a mixed acid (100 c.c. HNO₃, d 1.38, 300 c.c. HCl, d 1.18, 100 c.c. H₂SO₄, d 1.84, 500 c.c. of water), evaporating to dryness, treating with dilute sulphuric acid until clear, boiling, filtering, calcining the precipitate, weighing, and finally treating with hydrofluoric acid. Any lead or barium will be partially retained by the silica as sulphate. Iron is determined by titration with potassium permanganate. Manganese is determined colorimetrically as in steels, by means of silver nitrate and ammonium persulphate. For copper, if 0.2% or less is present, the blue colour developed with ammonia is compared with those from type samples; if more than 0.2%, after separation of silica an electrometric method is used in the cold with Hollard's electrodes. Lead is determined as sulphate. For aluminium the hydrochloric acid solution is treated with bromine water, and after boiling, with saturated ammonium chloride, then with ammonia until alkaline, whereby aluminium, iron, and manganese are precipitated. The precipitate must be freed from any magnesium oxide; the oxides are weighed and the aluminium determined by difference. Zinc is determined by electrolysis of a solution containing the zinc and magnesium after separation of copper. Calcium is separated from magnesium by a method depending on the different solubilities of the oxalates.

D. G. HEWER.

Interpretation of the macrostructure of cast metals. R. GENDERS (Inst. Metals, Mar., 1926. Advance copy, 21 pp.).-In steel ingots the commonly known regular distribution of the different types of macrostructure appears to be due largely to the low rate of solidification resulting from the relatively low conductivity of the metal. Ferrous alloys will permit of slow casting with a superheat of less than 50°, and under such conditions a maximum of equiaxed crystals with a minimum of columnar crystals is produced. Where the thermal conductivity of a cast alloy is high, however, as in non-ferrous alloys, solidification is rapid, occurring concurrently with pouring, which does not allow the turbulence of the metal during pouring and the path of the stream to be smoothed out. These facts are directly reflected in the macrostructure Observations on the distribution of metal when cast in a mould were made by pouring a molten "red" brass (90% Cu) followed immediately by a white nickel-silver of the same order of conductivity. The character of the macrostructure of an alloy is influenced by the range between liquidus and solidus. Ingots of 70:30 brass, which has a relatively long freezing range, may show a proportion of equiaxed crystals, whilst a similarly cast ingot of 60:40 brass, which has a very short range, consists entirely of columnar crystals. C. A. KING.

Relation between the equilibrium diagram and the magnetic susceptibility in binary alloys. H. ENDO (Sci. Rep. Tôhoku, 1925, 14, 479-512).-The magnetic susceptibility-concentration curve becomes a straight line in the case of alloys consisting of a mechanical mixture of two constituents, but when alloys form a solid solution the same curve takes the form of a curved line. The magnetic susceptibility was determined in relation to the equilibrium diagram in the systems, Cu-Sn, Cu-Zn, Zn-Sn, Pb-Te, Bi-Pb, Sb-Sn, Sb-Cu, Bi-Zn, Cu-Mn, Bi-Sn, Sb-Pb, Bi-Te, the equilibrium diagrams of the last three systems being revised on the basis of the magnetic analysis. C. A. KING.

Corrosion of an ancient tin specimen. C. O. BANNISTER (Inst. Metals, Mar., 1926. Advance copy,

2 pp.).-Examination of the corroded layer of a tin scabbard end of mediæval or possibly Roman period showed that the mechanism of corrosion of tin comprises the formation of hydrated stannous oxide and the subsequent oxidation and dehydration of this compound to stannic oxide. C. A. KING.

Separation of tin and antimony, especially from tin-antimony-lead alloys, by the dry method. W. LIDLE (Metall u. Erz, 1926, 23, 5-10; Chem. Zentr., 1926, I., 1862) .- Tin and antimony cannot be separated from lead by addition of alkali or alkaline-earth metals to the molten alloy, followed by liquation; e.g., if magnesium is added, mixed crystals of SnMg₂ and Sb₂Mg₃ are produced which form a eutectic with the other constituents and this liquates with the lead. Small quantities of antimony may be separated from tin-lead alloys by addition of a tin-iron alloy which will remove up to 10% of its weight of antimony. Addition of sulphur to the molten alloy removes first tin, then lead, but much of the latter is converted into sulphide before all the tin is removed as sulphide. Thus, an alloy containing 40% Sn, 50% Pb, and 10% Sb, after treatment with sulphur, gave a metallic regulus containing 98% of the antimony, 62.6% of the lead, and 23.7% of the tin in the original alloy.

A. R. POWELL.

Striation due to working or to corrosion in microscopical metallography. Mode of action of etching reagents. A. M. PORTEVIN (Inst. Metals, Mar., 1926. Advance copy 6 pp.).-The latent scratches produced during polishing a metal and developed by etching are often visible only on certain grains, at the boundaries of which they stop, to reappear again on other crystals having a similar orientation. This is particularly marked in the case of the aluminium-copper solid solution with 2.2% Al after etching with ammonium persulphate; the surface becomes covered with big lines in the same general direction caused by fine markings or striations. The intensity and direction of striation changes on crossing a twin and the striæ bear a close relationship to etching figures, being merely etching figures on a crystalline surface the lattice of which has been disturbed by a permanent deformation set up by the scratch. In the case of a-iron, regions deformed by cold work such as scratching with a pin or squeezing in a vice show characteristic markings in the form of a silhouette after etching with copper ammonium chloride and nitric acid. After removal of a pinscratch on the metal by polishing, deep etching for etching figures reveals the path of the scratch by the development of striations which deform the etching figures. After annealing metal which has been scratched, new grains develop from the regions where the lattice has been deformed and twins are formed in the parts that have suffered proportional translation. A. R. POWELL. translation.

Behaviour of some metals and alloys with plastilin and free sulphur. O. BAUER and H. ARNDT (Z. Metallk., 1926, 18, 85-88).-Copper becomes slowly converted into sulphide in contact 328

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

with plastilin (containing 40% of zinc oleate, 18% of wax and oil, 15% of kaolin, and 23% of sulphur), the depth of penetration increasing with the time to a maximum of about 0.5 mm.; further action is then prevented by the copper sulphide layer formed. The action is more rapid the higher the temperature, but the maximum depth of penetration is the same. Brass and bronze are hardly affected even by prolonged contact with plastilin, and aluminium, zinc, and tin are only slightly discoloured. Aluminium, zinc, tin, and especially manganese prevent the action of plastilin on copper and appreciably reduce the severity of the action of molten sulphur on copper. An alloy of 33.3% Mn and 66.7% Cu is not attacked by sulphur at 400° even after 5 hrs., whereas pure copper loses 2 g. per sq. dm. in the same time. Lead is almost as severely attacked by plastilin as is copper. A. R. POWELL.

Cold-deformation or corrosion fringes. A. PORTEVIN (Compt. rend., 1926, 182, 523-525).--A study has been made of the effects of chemical reagents on the polished surfaces of metals subjected to cold deformation, and in particular of the alternate bright and dark striations or "fringes" produced. These were of two distinct types, namely, those which showed distinct alternating fringes and channels, and those in which the corrosion patterns were modified. These were studied for copper and a-iron respectively. It is concluded that the patterns are characteristic of a permanent crystalline state, but that where the deformations follow directions not strictly parallel, the consequent fineness of structure and distortion of the lattice give the appearance of an amorphous layer.

J. GRANT.

Volumetric determination of uranium, vanadium, copper, and iron in uranium ores. A. S. RUSSELL (J.S.C.I., 1926, 45, 57-60 T).-Methods for determining volumetrically some or all of the elements named in solution in sulphuric acid have been worked out, and applied principally to the determination of these elements in uranium ores. Uranous sulphate is used as a new reducing agent, and previous work on liquid zinc amalgam as a reducing agent, and of diphenylamine as an indicator of vanadic acid, has been made use of. The results for the vanadium, uranium, and iron contents of carnotite; the uranium, copper, and iron contents of torbernite; and the uranium and iron contents of pitchblende, thorianite, autunite, and other ores agree satisfactorily with those obtained gravimetrically. A modification of the usual method for determining quadrivalent uranium in the oxide U₃O₈ is suggested.

"Atomised" coal for smelting non-ferrous metals. R. BLACK and C. L. SHAFER (Metal Ind., 1925, 23, 403—404; Chem. Zentr., 1926, I, 1030— 1031).—The use of "atomised" coal (all of which passes through a 200-mesh sieve) instead of coal "dust" (75% through the 200-mesh sieve) brings definite advantages. Starting from cold, combustion is complete after a period of 3—4 min.; the ash melts and is found as slag on the walls of the furnace, the outside of the crucible, and the surface of the charge. Weekly removal of this loose and friable slag suffices. The preparation of the carbon as well as the furnaces suitable for its use, and the cost are discussed. L. M. CLARK.

a-Phase boundary in the copper-tin system. D. STOCKDALE (J. Inst. Metals, 1925, 34, 111-124). --See B., 1925, 809.

Constitution of alloys of aluminium, copper, and zinc. D. HANSON and M. L. V. GAYLER (J. Inst. Metals, 1925, 34, 125-170).—See B., 1925, 809.

Constitution of zinc-copper alloys containing 45-65% Cu. M. L. V. GAYLER (J. Inst. Metals, 1925, 34, 235-244).—See B., 1925, 808.

 β -Transformation in copper-zinc alloys. J. L. HAUGHTON and W. T. GRIFFITHS (J. Inst. Metals, 1925, 34, 245-260).—See B., 1925, 808.

Physical properties of the copper-cadmium alloys rich in cadmium. C. H. M. JENKINS (J. Inst. Metals, 1925, 34, 85-102).—See B., 1925, 764.

Zinc-cadmium alloys. R. B. DEFLEY (J. Inst. Metals, 1925, 34, 193-206).—See B., 1925, 810.

Properties of some aluminium alloys. H. HYMAN (J. Inst. Metals, 1925, 34, 207-234).—See B., 1925, 764.

Passification and scale resistance in relation to the corrosion of aluminium alloys. L. H. CALLENDAR (J. Inst. Metals, 1925, 34, 57-84).— See B., 1925, 809.

Influence of pouring temperature and mould temperature on the properties of a lead-base anti-friction alloy. O. W. ELLIS (J. Inst. Metals, 1925, 34, 301-322).—See B., 1925, 809.

Colloidal separations in alloys. J. H. ANDREW and R. HAY (J. Inst. Metals, 1925, 34, 185-192).-See B., 1925, 809.

Thermal conductivities of industrial nonferrous alloys. J. W. DONALDSON (J. Inst. Metals, 1925, 34, 43-56).—See B., 1925, 810.

The high-temperature tensile curve. D. H. INGALL (J. Inst. Metals, 1925, 34, 171-184).---See B., 1925, 764.

Influence of time factor on tensile tests as high temperatures. J. S. BROWN (J. Inst. Metalt, 1925, 34, 21-42).—See B., 1925, 810.

Effect of temperature on the behaviour of metals and alloys in the notched-bar impact test. R. H. GREAVES and J. A. JONES (J. Inst. Metals, 1925, 34, 85-102).—See B., 1925, 765.

Electrodeposition of zinc from sulphate solutions.—A. L. MARSHALL (Trans. Faraday Soc., 1925, 21, 297—318).—See B., 1925, 636.

PATENTS.

Production of high-tensile steel. R. W. JAMES. From EISENWERK-GES. MAXIMILIANSHÜTTE (E.P. 247,276, 11.11.24).—Steel having high resistance to wear combined with great toughness, suitable for rails, tyres, and the like, contains 0.3-0.7% Si and 1.25-2.5% Mn, the combined amount of silicon and carbon being not greater than 0.7% and the proportions of these two elements being interdependent, the silicon rising from 0.30% to 0.50% as the carbon falls from 0.40% to 0.20%. By this means the formation of brittle iron carbide (Fe₃C) is retarded, Fe₃Si or Fe₃C/Si being predominantly formed, and as large a quantity of the carbon as possible is combined with manganese in the form of Mn₃C or Mn₃C/Si. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 131,980.)

T. H. BURNHAM.

Desulphurisation and concentration of sulphide iron ores. W. S. MILLAR (E.P. 247,471, 12.8.25. Addn. to 236,256; B., 1925, 725).-The ore is subjected to the action of sulphur dioxide gas of at least 50% purity in a furnace, e.g., a mechanical roasting furnace, at a gradually rising temperature, with or without the admission of steam. Part of the sulphur is first driven off leaving the ore with the approximate composition FeS, and then, without fusion, the remainder of the sulphur is driven off as free sulphur at a temperature not exceeding 950°, leaving a porous, highly magnetic product. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 236,256). T. H. BURNHAM.

Electrothermic refining of metals [steel]. J. L. DIXON, ASST. to PITTSBURGH RESEARCH CORP. (U.S.P. 1,569,464, 12.1.26. Appl., 5.9.19).—Steel is refined in an electric arc furnace having a number of electrodes, low-voltage current being supplied between one pair of electrodes and relatively high-voltage current between other electrodes. J. S. G. THOMAS.

Rust-proofing bath for iron and steel. J. H. GRAVELL (U.S.P. 1,572,354, 9.2.26. Appl., 9.5.22. Renewed 3.11.25).—A bath for treating iron in order to prevent rusting comprises a solution of phosphoric acid and sodium dihydrogen phosphate. The latter serves to neutralise the pickling acid without forming a sludge. A. R. POWELL.

Manufacture of steel. Soc. DES PROC. MÉTALLURGIQUES CONSTANT-BRUZAC (F.P. 594,547, 19.5.24).—The finely divided ore is heated to the temperature required for reduction and treated with carbon moxoxide until the iron is saturated with carbon. The product is separated from gangue either before or after melting in the absence of air, and is treated with lime, ferric oxide, or the like to remove deleterious metalloids. The carbon content is then reduced in the usual way until steel of the required composition is produced, oxygen being subsequently removed by the addition of manganese; or alternatively, the iron saturated with carbon is melted with the necessary amount of iron free from carbon. L. M. CLARK.

Steels. M. BAEKE (F.P. 595,530, 20.3.25).—Steels, which are very resistant to acids and alkalis, and are not affected by air, contain Cr 8—25%, Mn 2—25%, and C 0.4—1.5%. L. M. CLARK.

Reduction of poor colitic iron ores. J. BING (G.P. 422,030, 25.7.23).—Poor colitic iron ores are reduced at high temperatures with the hot carbon monoxide produced as a by-product in the manufacture of calcium carbide in the electric furnace. The product is smelted in the electric, cupola, or open-hearth furnace. A. R. POWELL.

Reduction of ores, especially iron ores. GEWERKSCHAFT ALFLEN VII (G.P. 423,072, 28.3.24). —Iron ores are reduced by heating with semi-coke in revolving or reverberatory furnaces. The reducing power of semi-coke is greater than that of metallurgical coke and resembles that of charcoal. A. R. POWELL.

Reduction of iron oxide ores and residues with reducing gases. R. SCHENOK and T. THORSELL (G.P. 423,501, 23.6.22).—Iron oxide ores and residues are reduced with gases at a temperature below the sintering temperature. The composition of the reducing gas mixture and the partial pressure of the reducing gases and their combustion products are so adjusted in relation to the temperature at which the reduction is carried out that the reduction is carried to the desired stage. With suitable composition of the reducing gases it is possible to produce iron free from carbon or containing more or less carbon, iron carbide, or solid solutions of iron carbide in iron. A. R. POWELL.

Spectroscopic testing of the metal bath in the refining of iron in the electric furnace. FELTEN U. GUILLEAUME CARLSWERK A.-G. (G.P. 423,436, 27.6.24).—An arc is struck between one part of the metal bath and a secondary electrode of known spectrum and the vapour so produced is observed through a spectroscope. A. R. POWELL.

Manufacture of steel or malleable iron. LINKE-HOFMANN-LAUCHHAMMER A.-G., Assees. of E. WIEGAND (G.P. 423,475, 17.12.24).—Scrap-iron is heated to a high temperature by external heat, and oxygen or oxidising substances are introduced so that partial combustion of the iron takes place while the heat thus generated fuses the remainder of the scrap ; subsequently the iron oxide formed in the first stage is reduced in the usual way. A. R. POWELL.

Case-hardening. W. J. MERTEN (U.S.P., 1,571,289, 2.2.26. Appl., 2.12.20).—Case-hardening is effected by means of cyanogen gases produced by heating a cyanide, and the molten cyanide is agitated by bubbling the unabsorbed gases through it. M. COOK.

Direct case-hardening of iron articles in molten cyanide baths. Allgem. Elektricitäts-Ges., Assees. of H. BLOMBERG (G.P. 422,305, 13.3.25). 330

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

-To increase the life of the cyanide bath small compressed pieces of charcoal are added to prevent the formation of cyanate. A. R. POWELL.

Recovery of nickel or nickel-iron alloys from nickeliferous rolling-mill scale and similar residues. M. STERN (G.P. 422,325, 26.6.18).— Rolling-mill scale and similar residues containing nickel are smelted with iron in a cupola or on the hearth of a reverberatory furnace in such a way that the nickel is precipitated from the fused oxides by the metallic iron. A. R. POWELL.

Metallurgical process [for treating copper ores]. W. E. GREENAWALT (U.S.P. 1,572,114-5, 9.2.26. Appl., [A] 2.9.24, [B] 1.12.24).--(A) Oxidised copper ore or roasted concentrate is leached with an acid solution and the resulting copper solution is subjected alternately to the action of a reducing agent (e.g., roaster gases containing sulphur dioxide) and to electrolysis to deposit the copper. The cycle is repeated until the solution becomes foul, when a quantity of the liquid is diverted to a separate circuit where it is partially neutralised and the same cycle of operations is repeated on this solution until only a small amount of copper is left. This is precipitated with hydrogen sulphide and the solution is treated for the separation of nickel, cobalt, and zinc, while the copper sulphide is returned to the circuit. (B) The copper solution obtained by leaching copper ores with acid is electrolysed in a series of units each comprising a feed tank, a reducer tank, and an electrolytic cell, and a substantially constant flow of electrolyte in a closed circuit is maintained in each unit, excess electrolyte being by-passed occasionally through the feed tanks. A. R. POWELL.

Pickling [bath for metals]. H. S. MARSH and R. S. COCHRAN, ASSTS. to AMER. COPPERAS CO. (U.S.P. 1,572,155, 9.2.26. Appl., 14.11.24).—A pickling solution for cleaning metal articles is saturated with hydrogen sulphide. A. R. POWELL.

Treating mixed lead-zinc sulphide ores. N. C. CHRISTENSEN (U.S.P. 1,572,268, 9.2.26. Appl., 31.12.23).—By treating the finely ground ore with acid, concentrated brine, the lead and part of the zinc are dissolved as chlorides. The evolved hydrogen sulphide is absorbed by lime, and the calcium hydrosulphide formed is used to precipitate first the lead, and then the zinc. A. GEAKE.

Recovery of lead and zinc from ores. H. and P. FAIVRE (F.P. 596,147, 3.7.24).—Finely divided oxidised lead ores are treated with boiling ammonium chloride solution, and the lead ammonium chloride solution so formed is separated from the gangue and cooled, whereby the bulk of the double salt crystallises out and the solution can be used again. Similarly oxidised zinc ores are leached with boiling ammonium sulphate solution which converts any lead into sulphate and dissolves the zinc as zinc ammonium sulphate, which is recovered by cooling the filtered solution. A. R. POWELL. Recovery of lead and silver from sulphide ores and metallurgical products. H. HEY (U.S.P. 1,572,388, 9.2.26. Appl., 20.1.21).— Sulphide ores are heated in the presence of gaseous hydrogen chloride under non-oxidising conditions to convert the lead and silver present into chlorides while inhibiting the conversion of the other sulphides in the ore. The lead and silver chlorides are then leached out with suitable solvents.

T. H. BURNHAM.

[Aluminium] alloy. R. L. JOHNSTON, ASST. to ALUMINUM DIE-CASTING CORP. (U.S.P. 1,572,357, 9.2.26. Appl., 26.12.22).—An aluminium alloy containing 5—10% Sn and a substantial amount of silicon is claimed. A. R. POWELL.

Refractory lining for crucibles [for aluminium alloys]. F. C. FRARY, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,572,475, 9.2.26. Appl., 15.1.24). —A crucible or ladle for handling aluminium alloy at high temperatures consists of a steel shell lined with finely ground alumina bonded with sodium silicate. A. R. POWELL.

Aluminium alloy for motor pistons. ALUMINIUM-INDUSTRIE A.-G. (Swiss P. 112,246, 25.9.24).—An aluminium alloy for the manufacture of pistons for motor-cars, contains 8-17% Cu, 1-3% Fe, and up to 2%Mg, Mn, or similar hardening element. The castings are either cooled very slowly or annealed below 350° so as to prevent subsequent changes in the hardness and shape. A. R. POWELL.

Apparatus for refining aluminium. F. WINZENRIED and R. .TIMENS (Swiss P. 98,838, 3.6.21).—An apparatus for refining aluminium consists of a tilting furnace with a charging opening in the roof. The opening is constricted towards the lower end and bent upwards. The whole is covered with a hood carrying a tubular flue which is built into the furnace lining and ends in a retort also built into the lining. The retort serves for the vaporisation of reagents which are passed through the tube into the aluminium during the refining process. A. R. POWELL.

Alloy. E. R. CROSBY. Assr. to ALUMINUM MANUFACTURERS, INC. (U.S.P. 1,572,382, 9.2.26. Appl., 7.7.21).—An aluminium alloy contains 3—8% Si and up to 5% Sn. T. H. BURNHAM.

Production of [refractory] metals. J. W. MARDEN and C. C. VAN VOORHIS, ASSTS. to WESTINGHOUSE LAMP CO. (U.S.P. 1,573,083, 16.2.26. Appl., 13.6.21).—Refractory metals are produced by heating their oxides with an alkali metal and a halide of another alkali metal or of an alkaline-earth metal. A. R. POWELL.

Concentration of oxidised ores. S. CROASDALE (U.S.P. 1,573,226, 16.2.26. Appl., 8.1.23).—Ores containing oxidised minerals may be concentrated by oil-flotation by treating the ore pulp with calcium pentasulphide, a frothing agent, and a relatively large amount of a hydrocarbon mixture containing a large proportion of olefines. No visible film of metal sulphide is formed on the surface of the mineral particles during treatment.

A. R. POWELL.

Recovering metals from slags. H. ZANICOLI (F.P. 594,928, 23.9.25).—The molten slag is heated to a high temperature and treated with reducing vapours, such as those of heavy petroleum hydrocarbons, in one chamber of a container divided by a bridge. The greater part of the heavy metal silicates present is reduced to the metal, which flows over the bridge and is mechanically separated. L. M. CLARK.

Bearing metal alloys with a bronze foundation. T. GOLDSCHMIDT A.-G. (G.P. 420,068, 6.7.21; F.P. 594,914, 10.3.25).—A bearing metal has the composition Pb12—25%, Ni $2 \cdot 5 - 5\%$, Sn 2—10%, and the remainder copper. Part of the tin may be replaced by antimony and zinc. The addition of a small quantity of phosphor-copper as a deoxidising agent often improves the casting. L. M. CLARK.

Lead – zinc – antimony bearing metal. F. DOEBLIN (G.P. 423,450, 15.5.23. Addn. to 367,978 and 382,545; B., 1923, 408; 1924, 388).—Part of the lead, specified in the previous patents, is replaced by an equal weight of copper, up to 10% of the alloy. A. R. POWELL.

Alloy for soldering lead alloys to iron, bronze, and the like. METALL-VERARBEITUNGSGES. M.B.H. (G.P. 423,293, 28.8.24).—A solder for joining lead bearing metal alloy containing alkali or alkaline-earth metals to iron or copper alloy bases consists of lead with the addition of other metals which lower its m.p. and render it plastic between 160° and 220°. A suitable alloy comprises 52.5% Pb, 42.5% Sn, and 5% Zn. A. R. POWELL.

Coatings of fusible substances [metals]. M. U. SCHOOP (Swiss P. 108,499, 10.3.24).—The coating substance is melted and sprayed from a jet in such a way that the stream is broken up into fine droplets. This may be effected by means of shaking devices inside the orifice of the jet, by spraying the substance from two jets inclined at an angle towards each other, or by using pulsating pressures to force the substance through the jet. The coatings so obtained are less brittle and softer than those produced by the ordinary metal spraying process. A. R. POWELL.

[Ore] separating process. R. ELLIS, Assr. to ELLIS FLOTATION CO. (Reissue 16,279, 2.3.26, of U.S.P. 1,425,186, 8.8.22. Appl., 1.8.24).—See B., 1922, 765 A.

Osmium alloy. E. HAAGN, Assr. to W. C. HERAEUS G.M.B.H. (U.S.P. 1,574,966, 2.3.26. Appl., 29.3.21).—See G.P. 350,703; B., 1922, 505 A.

Recovering light metals from scrap and the like. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of A. BECK (U.S.P. 1,576,080, 9.3.26. Appl., 11.4.24). —See E.P. 219,287; B., 1925, 75. Vanadium compounds (F.P. 598,315).-See VII.

Sulphur dioxide and sulphuric acid from sulphur-bearing slag (G.P. 423,640).—See VII.

Preparing calcium amalgams (G.P. 420,721).— See XI.

XI.—ELECTROTECHNICS.

Insulating oils. H. STÄGER (Z. angew. Chem., 1926, 39, 308—316).—The dielectric constant of insulating oils increases rapidly with rise of temperature so that breakdown of an insulating layer of oil may be due to local overheating. A very small proportion of moisture rapidly decreases the resistance of oil but further addition of moisture has little effect. The most serious cause of breakdown is the presence of acidic constituents produced by atmospheric oxidation, and the various methods proposed for determining the rate of oxidation are discussed and criticised at length. A. R. POWELL.

Influence of exterior temperature on temperature of standard pyrometric electric lamps. RIBAUD.—See I.

Products of decomposition of mineral oil by electric arc. EVERS.—See II.

PATENTS.

Electric resistance furnaces. G. E. TAYLOB, and ELECTRIC FURNACE CO., LTD. (E.P. 247,254, 20.7.25).—The cross sections and lengths of the resistors and the thickness of the refractory walls and roof of a furnace supplied with two-phase or threephase current are proportioned so that the full load taken by the furnace on heating up is as nearly as possible respectively twice or three times the power required to balance the loss by radiation and to maintain the furnace at the desired temperature.

J. S. G. THOMAS.

Electric-arc furnace. J. L. DIXON, Assr. to PITTSBURGH RESEARCH CORP. (U.S.P. 1,569,463, 12.1.26. Appl., 5.9.19).—An electric furnace is provided with three upper electrodes and a transformer of three-phase alternating currents having unequal transformer ratios in the several phases and supplying the electrodes with currents at different voltages. J. S. G. THOMAS.

Kiln [with electric heating for lime burning etc.]. SIEMENS U. HALSKE A.-G., Assees. of R. GROSS and M. STADLHUBER (G.P. 416,143, 22.2.22).— A kiln for burning lime, magnesite, cement, or the like consists of a circular rotating horizontal hearth covered with a dome and sealed by a sand or liquid seal. The material to be treated is introduced on to the periphery of the hearth, and is heated by several electric arcs arranged around the hearth or by radial resistances. The heat-radiating surface may be increased by providing partitions between the arcs or resistances. At intervals the heated material is impelled to the centre of the hearth, where it is withdrawn. The inlet and outlet valves and the rakes for impelling the material towards the centre of the hearth may be coupled mechanically, so that they work in unison. T. S. WHEELER.

[Coating electrodes of] discharge tubes, especially low-voltage glow lamps. F. SKAUPY (G.P. 414,517, 3.5.22).-Electrodes for use in discharge lamps are formed of a cheap metal, such as iron, and, before or during the manufacture of the lamp, are coated with a solution or a suspension of the azide of a metal of good electron-emitting properties. An alkaline-earth azide such as barium azide is suitable, or a mixture of azides may be used. The lamp is heated during evacuation either externally or by passing a current through it, and the azide decomposes forming the corresponding metal, and nitrogen, which is pumped off. Alternatively the decomposition may be accomplished while the lamp is filled with a suitable inert gas. The alloy or coating formed on the surface of the electrodes increases the life of the lamp and enables a good light to be obtained from it even on a low voltage.

T. S. WHEELER.

Preparation of high-percentage calcium amalgams by electrolysis. A. EILERT (G.P. 420,721, 21.9.24).—In the preparation of highpercentage calcium amalgams by electrolysis the mercury used as cathode and the vessel in which the electrolysis is performed are heated before introducing the electrolyte; the electrolyte is freed and kept free from suspended particles, such as undissolved calcium hydroxide, and the amalgam is compressed from time to time so that proper contact may be kept between the mercury and the lead bringing in the current. L. M. CLARK.

Protecting gas-absorbing substances [in electric cells] from penetration by liquid. R. OPPENHEIM, Assr. to Soc. ANON. LE CARBONE (U.S.P. 1,574,844, 2.3.26. Appl., 26.4.23).—See E.P. 206,471; B., 1924, 986.

Electric battery. R. OPPENHEIM, Assr. to Soc. ANON. LE CARBONE (U.S.P. 1,574,845, 2.3.26. Appl., 6.9.24).—See E.P. 236,884; B., 1925, 813.

Process for manufacturing a lead accumulator. K. KAWAKAMI (U.S.P. 1,575,167, 2.3.26. Appl., 3.10.21).—See E.P. 193,087; B., 1923, 363 A.

Cooling the electrodes in ozonisers. R. GRAEF, Assr. to A.-G. FÜR OZON IND. (U.S.P. 1,575,049, 2.3.26. Appl., 10.2.25).—See E.P. 241,125; B., 1925, 963.

See also pages 319, Sulphuric acid (G.P. 421,786). 320, Removing arsenic from burner gases (G.P. 423,657). 321, Gas mixtures for ammonia synthesis (F.P. 596,714). 322, Barium, strontium, and calcium peroxides (G.P. 422,531); Alkalineearth nitrides (G.P. 423,348); Corundum (G.P. 422,105); Magnesium hydroxide (Nor. P. 39,564). 339, Flour-bleaching gas (E.P. 246,979).

XII.—FATS; OILS; WAXES.

Composition of olive oil. K. TAUFEL and J. G. SARRIA (Anal. Fís. Quím., 1926, 24, 25—40).— The following data were obtained for a Spanish olive oil: $d^{18\cdot5}$ 0.914, oleorefractometer reading at 25° 61.9, acid value, 0.62, saponif. value 192.6, iodine value (Winkler's method) 82.7, Reichert-Meissl value 0.13, Hehner value 95, m.p. of fatty acids 28.2°. It contained stearic acid 2.27%, palmitic acid 7.55%, oleic acid 83.94, linoleic acid 0.51%, glycerol (calc.) 4.44%, and unsaponifiable matter 0.79%. Triolein and *a*-palmitodiolein were isolated. Arachidic acid was absent.

G. W. ROBINSON.

Surface properties of soap solutions. Structure of the fluid surface. J. F. CARRIÈRE (Chem. Weekblad, 1926, 23, 118-126; cf. B., 1923, 563).-The theory is put forward that the surfaces of soap solutions are composed of molecules of the fatty acids, oriented according to the theories of Langmuir and Harkins; experiments in support of this view are described. Thus the motion of camphor particles on the surface of water, inhibited by the presence of small quantities of sodium oleate, is momentarily re-induced by passing ammonia over the surface of the liquid ; sulphur dioxide has no such effect. So also extraction of a soap solution with neutral oil increases the alkalinity of the solution. A previous experiment has been repeated with air free from carbon dioxide, and it is found that the froth carried over from the soap solution contains free fatty acid, whilst the residue is more strongly alkaline than before. The surface tension-concentration curves of Lascaray (B., 1924, 432), Walker, (B., 1921, 856 A), and others are discussed, and the theories previously put forward shown to be untenable. S. I. LEVY.

See also A., Mar., 268, Saturated acids of highest m.p. from arachis oil (Holde and Godbole). 269, Additive products of iodine bromide and hypoiodous acid with unsaturated compounds (Holde and Gorgas); Fractional dissolution in alcohol of zinc salts of liquid acids from linseed oil (Agde); Octadecenoic acids (Grün and CZERNY). 286, Naphthenic acids (NAPHTALI).

Converting fatty acids into hydrocarbons. STADNIKOV and IVANOVSKI.—See II.

Ajowan and mohua cakes as fertilisers. REGE.—See XVI.

PATENTS.

Separating glycerides from fats and oils. ALLGEM. GES. F. CHEM. IND. M.B.H. (F.P. 595,250, 17.3.25).—Saturated or nearly saturated glycerides, *e.g.*, those containing oleic acid, are separated from less saturated glycerides by suitable solvents, *e.g.*, liquid sulphur dioxide. A. GEAKE.

Continuous production of fatty acids, glycerin, and ammonium sulphate. V. GRAUBNER (G.P. 421,438, 8.6.24).—An emulsion of neutral fat and ammonia, or a mixture of the fat with gaseous, liquid, or dissolved ammonia, is run continuously into a large quantity of hot ready-formed ammonium soap. A corresponding amount overflows from the vessel and is decomposed with sulphuric acid or sulphur dioxide. The ammonium soap is preferably under a slight pressure. A. GEAKE.

Obtaining fat from fat-bearing vegetable rinds. CHEM. FABR. GRIESHEIM-ELEKTRON (Swiss P. 111,359, 5.5.24).—The rinds are mechanically disintegrated and extracted with a fat solvent, *e.g.*, trichloroethylene. A. GEAKE.

Hydrogenation of oils, fats, and fatty acids with carbon as carrier for the metallic catalyst. N. V. ALGEM. NORIT MAATSCHAPPIJ (Dutch P. 12,851, 14.11.21).—Active or decolorising vegetable charcoal, freed from mineral constituents, and retaining its structure, is used as a carrier for the catalyst; it takes up more of this than kieselguhr or the like. A. GEAKE.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Determination of the covering power, opacity, and grain size of paints. W. REGLIN (Z. angew. Chem., 1926, 39, 334-335).-A simple apparatus for use in determining the covering power, opacity, and grain size of paints and the evenness of paint films comprises a rectangular box containing a 32-c.p. electric lamp and fitted with a glass lid on which transparent films of gelatin-coated paper may be fixed. The paint may be spread on the film by means of a brush or by spraying, and the irregularities of distribution are readily apparent when the apparatus is transferred to a dark room and the lamp in the box switched on. The size of the individual particles of pigment may then be measured by means of a microscope. The films produced on gelatin-coated paper are in every way similar to corresponding films on metallic surfaces, so that this apparatus permits deductions to be drawn as to the suitability of a paint for rust-protecting coatings. A. R. POWELL.

Use of tung oil. H. KÖLLN (Farben-Ztg., 1926, 31, 920—921).—An account of the difficulties encountered in the use of tung oil in varnishes, the value of various incorporated resins—synthetic and natural—and a general discussion of the attempts made to solve the two main problems arising in practice, *i.e.*, avoidance of the irregularity in the drying of tung oil of normal viscosity and complete control of the consistency of the oil in the process of thickening. S. S. WOOLF.

Softening point of resins. W. NAGEL (Wiss. Veröff. Siemens-Konz., 1925, 4, 321-323).—A modification of the Krämer-Sarnow method for determining the softening point of pitches is described which makes it suitable for work with resins. The finely powdered material is pressed down in a small tube with a somewhat constricted but open lower end, and 5 g. of mercury are placed on it. The

tube is then heated in an air-jacket in a glycerin bath and the temperature observed at which the mercury falls through the resin. The results are consistent to about 5° or 10°. Values for a number of resins are recorded. G. M. BENNETT.

Trichromatic colorimeter. Criticism of monochromatic-plus-white method of colorimetry. New method of colorimetry. GUILD.— See I.

PATENTS.

Preparation of pure blanc fixe and sodium thiosulphate. CHEM. FABR. GRÜNAU, LANDSHOFF & MEYER (G.P. 420,251, 10.2.24).—A solution of sodium sulphate is added to barium sulphide and air is led through until the product is colourless. Sulphur dioxide is then passed into the heated mixture until the solution has a faintly acid reaction. By this method, blanc fixe free from sulphur is obtained. L. M. CLARK

Preparing a painting material of powder form for use with either water or oil. M. TSUBATA (U.S.P. 1,574,854, 2.3.26. Appl., 24.7.23).—See E.P. 224,273; B., 1925, 45.

Manufacture of printing colours or ink. H. CAJAR (U.S.P. 1,576,408, 9.3.26. Appl., 5.12.23).— See E.P. 219,562; B., 1924, 797.

Manufacturing linoleum. G. DUBSKY (U.S.P. 1,576,181, 9.3.26. Appl., 14.7.25).—See E.P. 235,590 ; B., 1926, 202.

Proofing substances against insects (E.P. 247,242).—See V.

Coloured cellulose solutions (E.P. 247,288).— See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Crêpe rubber prepared with different reagents. H. P. STEVENS (Bull. Rubber Growers' Assoc., 1926, 8, 77—79; cf. B., 1926, 68, 288).—Crêpe rubber prepared with the use of *p*-nitrophenol is not yellower in colour than much commercial "pale crêpe" rubber. As with smoked sheet rubber (*loc. cit.*) the joint use of *p*-nitrophenol and sodium silicofluoride tends to eliminate, by compensation, the effect of each of these on the rate of vulcanisation.

D. F. Twiss.

Mastication [of rubber]. E. A. HAUSER and H. DANNENBERG (Kautschuk, 1925, Nov., 6—8).— The "two-phase" theory of the structure of rubber suffices to explain the phenomena of the mastication process; e.g., the "recovery" of rubber on storage after mastication is attributed to globules which during the milling operation have not lost the whole of their less viscous interior, once more becoming closed, thereby restoring part of the elastic character to the material. The aggregation theory of Le Blane and Kröger (B., 1925, 932), which is particularly applicable to the vulcanisation process, is not incompatible with the two-phase theory (cf. Hauser, B., 1925, 514). D. F. TWISS. CL. XV.-LEATHER : GLUE.

Structure of stretched rubber. E. A. HAUSER and H. MARK (Kautschuk, 1925, Dec., 10-11). In the X-ray investigation of rubber the position of the interference spots, within an accuracy of 1%, is independent of the stretch whilst their intensity is approximately proportional to it; increase in the degree of extension also does not affect the position of the amorphous ring but decreases its intensity. When rubber is stretched a proportion of a crystalline phase results, the axes of the crystals lying parallel with the chief line of extension. If a sheet is stretched uniformly in every direction a ring "fibre diagram" is obtained, whilst a twisted rubber rod gives a spiral "fibre diagram." If a piece of stretched rubber is gradually heated the interference spots decrease in intensity and disappear just when the tension disappears and the rubber becomes plastic; cooling to -4° , however, does not affect the intensity of the interference although the tension again disappears. Mechanical working, *i.e.*, mastication, of rubber causes a gradual decrease in the intensity of the interference of the stretched material until finally it is no longer observable.

D. F. Twiss.

XV.—LEATHER; GLUE.

Fluorescence test for natural vegetable tanning extracts in ultra-violet light. O. GERNGROSS and G. SÁNDOR (Collegium, 1926, 1-10; cf. Gerngross, Bán, and Sándor, B., 1926, 23).-A large number of natural tanning extracts show fluorescence. Not only quebracho and tizerah but also pine bark, malet bark, and donga extract in neutral solution show slight but distinct fluorescence in ultra-violet light. In slightly alkaline solution, the bluish fluorescence of pine bark and malet bark is increased appreciably and is changed to a bright green, whilst the fluorescence of donga extract changes to a beautiful orange-red. The yellow fluorescence of fibrous tissues treated with quebracho or tizerah solutions is not confined to these two materials, but is also exhibited by fibres treated with mimosa, chestnut, and hemlock extracts but not in so marked a degree. Cotton wool dipped in pine bark and malet bark extracts shows a beautiful violet fluorescence. This is also given to a smaller extent by oakwood extract. On mixing yellow and violet fluorescing extracts (e.g., equal volumes of pine bark and quebracho extracts) and treating wadding with the mixture, a snow-white fluorescence is produced. Mangrove bark extract gives a slight whitish fluorescence. The violet fluorescing substance will combine only loosely with hide powder so that it can be separated from tannin by detannisation. The fluorescent material is fixed irreversibly by cotton wool. Sulphite-cellulose extracts in aqueous solution exhibit a change in colour from blue to green on adding alkali in the same way as pine bark and malet bark, but the latter can be differentiated from sulphite-cellulose because cotton wool treated with sulphite-cellulose extract gives scarcely any fluorescence and the slight fluorescence produced can be removed by washing. D. WOODROFFE.

Comparative durability of chrome- and vegetable-tanned sole leathers. R. C. BOWKER and M. N. V. GEIB (U.S. Bur. Standards Tech. Papers, 1925, 19, [286], 267-286).-Hides were prepared for tanning in the usual way, cut down the back, one side of each was tanned with a blend of hemlock, chestnut, wattle bark, and quebracho; the other side was chrome-tanned. Some of the chrome-tanned sides were filled with greases and mineral fillers. Under the test conditions, chrometanned leather, regardless of filling, wore approximately twice as long as vegetable-tanned leather per unit of thickness. Natural chrome-tanned leather gave the longest wear ratio, which decreased according to the types of filling materials used. Chrome-tanned leather filled with 25% of paraffin wax wore 115% longer than vegetable-tanned sole leather, whereas chrome leather filled with grease, glucose, and barium sulphate only showed 23% longer wear. The chrome leather had a content of hide substance double that of the vegetable-tanned leather. The chrome leather was more hygroscopic. Fillers prevented the rapid absorption of water. Natural chrome leathers swelled in water 25%, whilst filled leathers showed only 11% swelling. Natural chrome leathers in wear became very soft and pliable, had a tendency to spread, fray, and curl up at the edges, allowed water to penetrate rapidly, and were slippery on wet surfaces. Paraffin wax-filled chrome leather was similar but waterproof. It lost this property during wear. Leather filled with paraffin wax and pyroxylin retained its water-resisting property longer. The average thickness of vegetable-tanned bends was 25% greater than that of the chrome-tanned. Topographical charts have been made to represent the variation of the thickness of the leather in sides from the same hide but of different tannages. The filling materials increase the thickness of the leather. Very heavy hides are necessary to produce chrome leather $\frac{9}{48}$ in. to $\frac{11}{48}$ in. thick. A hide tanned by the chrome process will yield from 10-12% less area in leather than if tanned by the vegetable process. D. WOODROFFE.

Analytical standards for leather manufacture. A. M. GOLDENBERG (Collegium, 1926, 10-21).— The following standards for sole leather and Russia leather have been adopted for introduction into the factories controlled by the Ukraine Leather Trust. Soaks: g. per litre, proteins 0.5, ammonia 0.03, sulphides (as Na₂S) 0.3. Lime liquors (first lime): g. per litre, proteins 5 (sole leather), 7 (Russia leather), ammonia 0.1, lime 4, total alkalinity 3.5. Sole leather pelts: hide substance 28%, lime and ash <1%, total alkalinity (as NaOH) 0.8%. Russia leather after colouring liquors: degree of tannage 15%, hide substance (for 18% water) 70%, lime <0.3%, water-soluble matter 2-2.5%. Sole leather after colouring liquors: degree of tannage 25-30%, hide substance 45%, water-soluble matter 4%. Hide after layers: degree of tannage 55-60%, hide substance 45%, water-soluble matter 6%. Colouring liquors (sole leather): d 1.007-1.023, tannin 0.15-2.5%, acid (as acetic) 0.5%. Colouring liquors (Russia leather): d 1.004-1.011, tannin 0.10-0.15%, acid (as acetic) 0.0-0.05%. Pit liquors, first layer: d 1.021-1.028, tannin 2.5-3.0%, acid 0.5%; second layer: d 1.028-1.035, tannin 3.5-4.0%, acid 0.3%. Drum liquors for sole leather: d 1.084-1.098, tannin 13%. Sole leather: water 18%, ash 1.5%, fat 1.2%, water-soluble matter 14-17%, leather substance 65.3%, degree of tannage 85-90%, "rendement" figure 290\%, hide substance 35.5-34.4%, combined tannin 29.8-30.9%, water resistance 30%. Russia leather: water 18%, ash 0.8%, fat 14%, water-soluble matter 28%, leather substance 55.2%, degree of tannage 60-66%, "rendement" figure 270-278%, hide substance 37.1-35.9%, combined tannin 22.2-23.3%. D. WOODROFFE.

Fumaric acid as a hydrolytic product of gelatin. AbderHalden and Haas.—See A., Mar., 312.

PATENTS.

Treatment of chrome leather waste for use in the manufacture of glue. E. MEIER (Swiss P. 112,400, 8.8.24).—Waste from chrome leather which has been treated with formaldehyde is prepared for glue manufacture by treatment successively or simultaneously with alkali solution and lime.

L. A. COLES.

Process for treating blood. A. STERNBERG (E.P. 224,227, 27.10.24. Conv., 29.10.23).— Coagulated blood, preferably defibrinised, is brought in the wet condition (50% of water being the most favourable proportion) to the highest degree of fineness by triturating it with wire brushes, and is then dried. A suitable device consists of two intermeshing brushes rotating in opposite directions, and preferably at different speeds. The product is specially suitable for making artificial horn etc.

D. G. HEWER.

Utilising casein- and other proteinformaldehyde compounds. INTERNAT. GALALITH-GES. HOFF & Co., Assees. of A. BARTELS and G. EBERHARDT (G.P. 419,536, 23.10.23).—Proteinformaldehyde compounds, *e.g.*, artificial horn scrap, are treated with substances, such as bisulphites, which have a greater affinity than proteins for formaldehyde, and of which the formaldehyde compounds do not harden proteins. The proteinformaldehyde compound may first be treated with a protein solvent, *e.g.*, an acid or alkali. The recovered protein may be used again for preparing protein masses to be hardened with formaldehyde.

A. GEAKE.

Dyeing leather (G.P. 422,465).-See VI.

XVI.—AGRICULTURE.

Plate counts of soil micro-organisms. N. R. SMITH and S. WORDEN (J. Agric. Res., 1925, 31, 501-517).—The use of an electric vibrator for prepreparing suspensions of soil for making counts of micro-organisms (cf. Whittles, B., 1923, 466 A) is unsatisfactory on account of the difficulty of ensuring complete sterilisation of the apparatus. When this is got over, counts from suspensions prepared in this way agree quite closely with those obtained by the usual method of hand shaking. Soil extract-agar gives higher and more uniform results than other media. Field soils of fair fertility, irrespective of their general character, are suitable for the preparation of the soil extract-agar medium. Soil samples taken horizontally from a level about 4 in. below the surface give slightly higher and more uniform counts than those taken vertically with an auger.

C. T. GIMINGHAM.

Ammonia content of soil, and its relation to total nitrogen, nitrates, and soil reaction. H. J. HARPER (J. Agric. Res., 1925, 31, 549—553).— No correlation was observed between the ammonia content and the content of total nitrogen or nitrates or the $p_{\rm H}$ value of some 40 soils investigated. The ammonia content is evidently in equilibrium with the products of protein decomposition and nitrification. The majority of soils contain less than 10 pts. per million of ammonia and the amount very rarely exceeds 20 pts. per million. C. T. GIMINGHAM.

Formation of humus in dead surface layers of forest soils. A. NEMEC (Compt. rend., 1926, 182, 590-592).-Surface layers of the soil in forests have been investigated by treatment with hydrogen peroxide, which renders humic substances soluble in The proportion of humus in the organic water. matter of the soil increases as the acidity of the soil decreases. The rate of nitrification follows the same general rule. The $p_{\rm H}$ of the soils investigated ranged from 3.8 to 6.5, the humus content from 20.7 to 82.2% of the organic matter in the soil, and rate of nitrification from -10.3 to +194 mg. of nitrogen gained in 30 days in 1 kg. of soil (calculated on the dry weight). L. F. HEWITT.

Decomposing action of peat on phosphorite. D. PRIANISCHNIKOV (Fortschr. Landw., 1926, 1, 1-4; Chem. Zentr., 1926, I., 1612).—When calcium sulphate is added to peat, the hydrogen ions of the humic acid present are replaced by calcium and a more acid reaction is developed; in the presence of phosphorite, water-soluble phosphorus is formed. If the amount of calcium sulphate is adjusted so as not to exceed the capacity of the peat to remove calcium ions from solution, the phosphorite can be completely decomposed. Peat in presence of phosphorite and calcium sulphate becomes more acid and yields 30% more water-soluble phosphorus than with phosphorite and potassium chloride. These results were confirmed by vegetation experiments.

C. T. GIMINGHAM.

Industrial wastes as manures. I. Ajowan and mohua cakes as fertilisers. II. Utilisation of refuse. R. D. REGE (J. Ind. Inst. Sci., 1925, 8A, 245-265).-Ajowan cake, from the seeds of *Carum copticum*, contains about 0.9% of nitrogen, which is in a readily available condition. Traces of thymol are present, but the amount is insufficient to check bacterial activity in the soil. Mohua cake,

from the seeds of Bassia longifolia, contains 2.7% of nitrogen and about 8.0% of oil. When mixed with activated sewage sludge and aerated for 6 days, no decomposition of the protein of the cake occurs. The same result is obtained if the oil is first extracted. The non-availability of the nitrogen was traced to the presence of a saponin which inhibits bacterial action. Experiments with town refuse (in India), containing from 0.8 to 1.7% of nitrogen and small quantities of phosphoric acid and potassium, indicate that about 75% of the nitrogen is in a comparatively unavailable condition. In mixtures of powdered refuse and activated sewage sludge to which ammonium sulphate was added, nitrification took place rapidly and the soluble nitrogen was removed from solution. There was no loss of nitrogen if the proportion of carbon was not too high. It is suggested that raw sewage might be treated with sludge and refuse material with the object of conserving the soluble nitrogen. C. T. GIMINGHAM.

Method of obtaining size distribution of particles in soils and precipitates. D. WERNER (Trans. Faraday Soc., 1925, 21, 381-394).—See B., 1926, 31.

PATENTS.

Manufacture of phosphatic fertilisers. CHEM. FABR. HEPPES U. Co., and J. B. CARPZOW (G.P. 421,271, 7.7.23).—Fertilisers are produced by drying and grinding mixtures of phosphates with fresh sludge containing a high percentage of colloidal silica, such as sapropel or saprocol. L. A. COLES.

Manufacture of a stable mixed fertiliser containing calcium nitrate. BADISCHE ANILIN-& SODA-FABR., Assees. of H. WEITZEL (G.P. 421,331, 31.7.24).—Calcium nitrate is mixed with about two equivalents of ammonium phosphate or ammonium sulphate, or mixtures of these, to form nondeliquescent fertilisers. L. A. COLES.

Manufacture of fertiliser containing urea. A.-G. FÜR STICKSTOFFDÜNGER (G.P. 421,852, 27.1.22).—The fertiliser is produced by treating calcium cyanamide with acids and water or steam, at a temperature and under conditions such that water not combined, or liberated during the process, is removed by evaporation. L. A. COLES.

Attractant for insects. L. B. SMITH, E. A. RICHMOND, and P. A. VAN DER MEULEN, Assrs. to the People of the United States (U.S.P. 1,572,568, 9.2.26. Appl., 12.8.25).—A preparation containing geraniol as the active ingredient is claimed as an attractant for insects, particularly for the Japanese beetle. C. T. GIMINGHAM.

Utilisation of end liquors from crude potassium salts (G.P. 422,987).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Influence of salts on the crystallisation of sugar. W. J. KUSNETZOV (Z. Ver. Deuts. Zucker-Ind., 1926, 19-24).—Alkali carbonates inhibit the crystallisation of sucrose from its aqueous solution, sodium carbonate to a greater degree than the potassium salt, this effect diminishing with falling temperature. Calcium nitrate and aspartate behave similarly. On the other hand, the presence of calcium chloride promotes crystallisation, though to a comparatively small extent. These effects are influenced by the viscosity of the solution.

J. P. OGILVIE.

Treatment of molasses with zeolites in applying the Steffens process. F. R. BACHLER (Ind. Eng. Chem., 1926, 18, 180—182).—Treatment of beet molasses with zeolites, so as to remove the whole or greater part of the alkali salts, previous to submitting it to the Steffens desaccharification process, is advocated. J. P. OGILVIE.

Commercial caramels and molasses colouring substances. H. SIMMICH (Z. Ver. Deuts. Zucker-Ind., 1926, 1-18).-German commercial caramels were found to contain caramelan, caramelen, and caramelin (cf. Cunningham and Dorée, B., 1917, 973); some nitrogenous substances were also present, probably originating from the ammonia used in the manufacture of the caramel. Absorption curves determined with the König-Martens spectrophotometer did not show resemblance to those of "Kassler Braun," a humic acid prepared from lignite. Beet molasses colouring matters were precipitated with lead acetate, and three colouring substances containing nitrogen were isolated; these gave the characteristic absorption curves of the colouring matters found in commercial caramels. A fourth substance separated showed absorption phenomena similar to those observed with humic substances. J. P. OGILVIE.

Studies on starch. C. L. ALSBERG (Ind. Eng. Chem., 1926, 18, 190-193).-Most varieties of starch when heated in water suspension do not disintegrate to form a colloidal solution, the granules merely swelling gradually (cf. Harrison, B., 1911, 534). If natural starch be ground in a pebble mill until most of its granules are injured, though still recognisable under the microscope as starch, it becomes incapable of yielding paste in ordinary concentration. Most of the starch granule substance has become colloidally soluble in cold water. From such ground starch without heat or the use of any reagent clear solutions can be obtained in cold water. these containing material which gives the characteristic iodine reaction, does not reduce Fehling's solution, and is capable of dialysing through thin colloidal membrane. J. P. OGILVIE.

See also A., Mar., 273, Revision of structural formula of dextrose (CHARLTON, HAWORTH, and PEAT). 275, Relationship between optical rotatory power and structure in chemistry of polysaccharides (PRINGSHEIM and LEIBOWITZ). 276, Amylose octadextrin (KLASON and SJÖBERG).

PATENTS.

Refining of sugar. A. W. MATHYS. From F. KESSLER (E.P. 246,976, 23.12.24).—Raw sugar is subjected to a sudden change in temperature, for example, by treatment with a large volume of superheated steam, whereby innumerable fine fissures are produced in the crystals, or they are broken into fragments. The crystals are then readily purified by treatment with a pure sugar solution or one containing refining agents. If the refining agents be added to the raw sugar the pure sugar solution may be produced *in situ* by the action of the steam. D. G. HEWER.

Manufacture of cane-sugar [from molasses]. H. S. PAINE, C. F. WALTON, JUN., and V. BIRCKNER; dedicated to Citizens of U.S.A. (U.S.P. 1,572,359, 9.2.26. Appl., 8.2.24).—The invert sugar present in molasses is destroyed by selective fermentation and the sucrose is then precipitated as saccharate, which is worked in the usual way. A. R. POWELL.

Producing sugar from cellulose. N. KRANTZ and L. DE MOLTKE-HUITFELDT (F.P. 595,439, 10.3.25).—Cellulose-containing substances, e.g., sawdust, are treated with dry hydrogen chloride in the presence of water or the like. A. GEAKE.

Treating sugar juices or the like with hyposulphite. R. DUTILLOY (F.P. 595,714, 30.6.24). —Crude diffusion or press juices, or syrup or mother liquors from crystallisation, are treated while neutral or feebly acid, with a determined amount of hyposulphite at 50—60°. A. GEAKE.

Treating sugar-containing juices or solutions to decolorise or purify them, or render them suitable for filtration. C. W. SCHONEBAUM (Dutch P. 12,990, 9.11.21).—The juice or solution is treated with ozone at a raised temperature in the presence of a substance which will combine with acids and keep the solution neutral. Decomposition of sugar is thereby avoided. A. GEAKE.

XVIII.—FERMENTATION INDUSTRIES.

A., 324, See Mar., Relation between alcoholic fermentation and hydrogen-ion concentration (HÄGGLUND and AUGUSTSON); Formation of acetylmethylcarbinol and $\beta\gamma$ butylene glycol (LEBEDEV); Synthesis of coproporphyrin by yeast (FISCHER and FINK); Synthesis of Biogens, inactive mother-substances of the two "bios" (EASTCOTT); Reproduction of yeast in solutions to which no bios had been added (WHITEMAN); Influence of electrolytes on electrophoretic migration of bacteria and of yeast cells (WINSLOW and FLEESON); Acetic fermentation (NEUBERG and WINDISCH). 325, Mechanism of lactic acid formation by bacteria (NEUBERG and GORR); Fermentation of cellulose by thermophilic bacteria (VILJOEN, FRED, and PETERSON). 328, Viscosimetric determination of amylase (DAVISON).

PATENTS.

Preparing yeast nutrient solutions and manufacturing yeast therefrom. R. L. CORBY and R. GLASGOW, Assrs. to FLEISCHMANN CO. (U.S.P. 1,571,932, 9.2.26. Appl., 13.3.19).—To a nutrient solution consisting of molasses, cereal material, and nutrient salts, and having an acidity higher than the optimum for yeast propagation, a compound containing inorganic yeast-assimilable nitrogen and having an alkaline reaction is added, in amount requisite to produce the most satisfactory conditions for yeast propagation in respect to acidity and content of assimilable nitrogen.

D. G. HEWER.

Denatured alcohol. H. C. FULLER (U.S.P. 1,573,697, 16.2.26. Appl., 6.6.21).—The denaturant is a halogen-containing glyceryl ester. H. HOLMES.

Denatured alcohol. H. C. FULLER, ASST. to UNION CARBIDE AND CARBON RESEARCH LABORATORIES (U.S.P. 1,573,698, 16.2.26. Appl., 21.6.22).—The denaturant is a halogen fatty acid ester of a polyhydric alcohol. H. HOLMES.

Manufacture of yeast. C. HOFFMAN, N. M. CREGOR, and H. D. GRIGSBY, Assrs. to FLEISCHMANN Co. (U.S.P. 1,575,761, 9.3.26. Appl., 22.3.22).— See E.P. 195,347; B., 1923, 1039 A.

XIX.-FOODS.

Nature of the agglutination of fat globules. IV. Relation of serum globulins to the "creaming" of milk. E. BROUWER. V. Influence of natural and separated skim milk on the creaming of washed milk-fat globules. E. HEKMA (Jaarversl. 1924, Vereen. tot. exploit. proef. te Hoorn, 1925, 18—35; 36—43; Chem. Zentr., 1926, I., 524).—The two proteins, euglobulin and pseudoglobulin (prepared from the blood serum of cattle), when added to milk, differ markedly in their effect on the rising of the cream. The former greatly assists the process, whereas the latter has little action. The blood serum of new-born calves, which contains scarcely any euglobulin, has little or no effect. There is not, however, a direct correlation between the amount of euglobulin in blood serum and the power of promoting "creaming." The possibility that euglobulin plays a part in the natural "creaming" of milk is discussed.

Skim milk from a separator has a greater influence on the agglutination of washed milk-fat globules than natural skim milk and this difference is still noticed if the fat globules are heated to $80-90^{\circ}$ for 5 min., thus causing a considerable reduction in the capacity for "creaming." It appears that some substance derived from the leucocytes in the milk is concerned with "creaming."

C. T. GIMINGHAM.

Leucocyte content and catalase number of natural and separated cream. E. HEKMA (Jaarversl. 1924, Vereen. tot. exploit. proef. te Hoorn, 1925, 4—10; Chem. Zentr., 1926, I., 524).—The number of leucocytes in natural cream is very much greater than in separated cream—approximately 4,000,000 per c.c. in the former and 35,000 in the latter. The catalase number is also much higher in natural cream. The conditions are reversed in the corresponding skim milks.

C. T. GIMINGHAM.

Chloropicrin as a fumigant for cereal products. R. N. CHAPMAN and A. H. JOHNSON (J. Agric. Res., 1925, 31, 745—760).—The high toxicity of chloropicrin to the granary weevil is demonstrated. A lethal concentration can be rapidly obtained, even in large bulks of cereal products, if the chloropicrin is atomised. The presence of very small amounts in flour affects the quality of the bread; not only is the physical condition of the gluten changed but yeast activity in the dough is retarded. Disappearance of chloropicrin from fumigated flour and wheat is slow but eventually complete, given proper exposure to the air. C. T. GIMINGHAM.

Apparatus for the determination of water [in foodstuffs, fat, etc.]. H. KREIS (Ann. Chim. Analyt., 1926, 8, 33—34).—An apparatus for the determination of water by distillation with toluene or similar immiscible liquid, consists of a flask carrying an upright tube of large diameter with a side tube extending from a point just above the cork of the flask to a point above the open upper end of a smaller graduated tube within the wide tube. The upper end of the wide tube carries a condenser. The arrangement is such that the vapours pass only up the side tube, are condensed, and the condensed liquid collects in the graduated inner tube. D. G. HEWER.

Rapid determination of water content [in cereals etc.]. J. TAUSZ and H. RUMM (Z. angew. Chem., 1926, 39, 155—156).—The substance is mixed with dry tetrachloroethane in a flask carrying a 3-bulb fractionating column, the upper end of which is bent over and elongated to form the inner tube of a Liebig condenser. 'The distillate is collected in a tube with levelling arm, and the supernatant water finally forced up into a narrow graduated tube and measured. The method is particularly useful in the case of cereals and other substances which give up their moisture with difficulty in an oven at 105°. W. T. K. BRAUNHOLTZ.

Testing and estimation of decay in preserved eggs. A. SCHMID (Mitt. Lebensm. Hyg., 1925, 16, 137—143; Chem. Zentr., 1926, I., 1320).— Satisfactory results are obtained by the following method, due to Sudenorf (private communication): 1-2 g. of the dry substance are warmed with 30—60 c.c. of neutral ether-alcohol, and titrated quickly with 0·1N-sodium hydroxide solution in the presence of phenolphthalein, till a strong red colour persisting for 30 sec. results. The acidity thus determined is a measure of the deterioration. J. GRANT.

Determination of unsaponifiable matter in flour, alimentary pastes, and eggs. R. HERTWIG and L. H. BALLEY (J. Assoc. Off. Agric. Chem., 1926, 9, 122—124).—The direct ether extract of wheat flour and eggs does not contain all the unsaponifiable matter of these materials, and the acid hydrolysis method for extraction of fat yields low results for flour and eggs. The method of Rask and Phelps (B., 1925, 224) and Hertwig's "neutral" method (B., 1923, 1040 A) yield quite similar results. The A.O.A.C. neutral method for the lipoid extraction of flour, alimentary paste, and eggs is shown to be the most satisfactory. The lipoids from 5 g. of wheat flour or alimentary paste, or from 10 g. of liquid eggs or 2 g. of powdered dried egg, are extracted according to the A.O.A.C. methods (1925, 40, 58), and the unsaponifiable matter is determined either on the dried crude extract or on the purified and weighed lipoids (cf. B., 1925, 600). C. O. HARVEY.

Effect of chemical preservation of eggs upon the stability of their vitamin contents. E. Tso (Biochem. J., 1926, 20, 17–22).—In the Chinese preserved egg "pidan." (ducks' eggs preserved in a mixture of slaked lime, straw ash, salt, and water), vitamin-B is practically completely destroyed whilst vitamin-A and vitamin-D are hardly affected.

S. S. ZILVA.

Variations in individual sugars in the Jerusalem artichoke during growth. S. H. COLLINSAN d R. GILL (J.S.C.I., 1926, 45, 63—65 T).— Following investigations on oat straw (J.S.C.I., 1922, 56 T) artichokes have been grown and analysed. The tubers contain much inulin, and the stalks much lævulose at active stages of growth, but mere traces when growth is over and the tubers are formed. The relative proportions of dextrose and lævulose in artichokes are reversed to those in oats. Detailed figures are given, showing the composition of both stalk and tubers at all stages of growth. A striking difference from cereal grains, or potatoes, is the continued increase in percentage of proteins in the tubers at late stages of development.

Occurrence of arsenic in apples. H. E. Cox (Analyst, 1926, 51, 131—137).—Out of 39 samples of apples (American) only 5 were found free from arsenic, which was present in proportions ranging from 0.5 pt. to 15 pts. As_2O_3 per million. The proportions of lead found in the samples examined indicated that the fruit had been sprayed with lead arsenate. The distribution of arsenic on apples which had been heavily sprayed with lead arsenate in the laboratory, and then suspended for 48 hours, was in two cases : whole apple, 24 and 7 pts. per million ; on the skin 100 and 33, and in the "flesh" 3.3 and 1.3. In spite of washing sprayed apples in running water a small but definite retention of arsenic was found, but if 2% caustic soda solution is used for washing, the removal of the arsenic is almost complete.

D. G. HEWER.

Examination of cacao. H. LÜHRIG (Pharm. Zentr., 1926, 67, 129—135).—Many samples of cacao beans and cocoa from industrial and other sources have been examined chemically and microscopically. Samples of material taken at different stages during treatment in a Bauermeister refining machine showed that the husk-content of the broken and purified material increased with increased fineness of grinding. Tables are given of the analyses and microscopical examination of various samples, including a very extensive table of the iron (ferric oxide), manganese (manganous oxide), and sand content of many samples (nibs, husks, and cocoa). B. FULLMAN.

Vitamin content of plant juices. R. SUCHARIPA (Konserven-Ind., 1925, 12, 623-624; Chem. Zentr., 1926, I., 1594).—Vitamins are not more stable in fruit juices prepared without heating than in cooked juices, on account of the action of oxydases in the former. For preservation of the vitamin content, the fruit should be rapidly pressed and the juice collected in small closed containers. Sterilisation should be carried out immediately. The concentration of sugar in the final product should not exceed 35% and storage should be in a cool, dark place.

C. T. GIMINGHAM.

Formaldehyde in marine products. D. B. DILL and P. B. CLARK (J. Assoc. Off. Agric. Chem., 1926, 9, 117-122).-The natural development of formaldehyde in canned Crustacea, discovered by Ishida (J. Pharm. Soc. Japan, 1917, [422]. 300), has been confirmed by the authors, who show that this development is not related to the growth of organisms, and that it is not affected by the nature of the container. It takes place in the absence of free oxygen. Concentrations of formaldehyde in canned Crustacea as high as 1 pt. in 30,000 have been found, and, as acidification and steam distillation failed to recover more than one-third of the formaldehyde added to formaldehyde-free salmon, it is concluded that the concentration of formaldehyde actually present may be as high as 1 pt. in 10,000. Formaldehyde formation has also been found to take place in canned red rock cod (Sebastodes sp.) and herring, but not in sardine, salmon, and other canned fish meat. C. O. HARVEY.

See also A., Mar., 313, Van Slyke's method of determination of nitrogen distribution in proteins; Distribution of nitrogen in proteins of eggs; Direct determination of arginine (PLIMMER and ROSEDALE). 321, Milk-diastase (CHRZASZCZ and GORALOWNA). 326, Accessory growth factors (VON EULER and STEFFENBURG); Vitamin-B and metabolism (PLIMMER and ROSEDALE).

PATENTS.

Flour bleaching gas. F. STACEY (E.P. 246,979, 5.1.25).—Nitrogen peroxide is generated electrically in a tube with a contracted passage. The electrodes are placed within the tube in front of the contraction and a nozzle fed from an air compressor is placed between them. The electrodes are supplied with electrical energy by a magneto. D. G. HEWER.

Preservation of fresh fruit and vegetable. H. R. FULTON and J. J. BOWMAN (U.S.P. 1,571,938, 9.2.26. Appl., 3.10.25). — An aqueous solution containing the sodium oxide and boric acid radicals in equal proportions and of such strength as to retard the development of organisms causing stem and blue mould rots and other forms of decay (e.g., 5 pts. by weight of borax and 1.39 of anhydrous sodium carbonate in 100 of water) is applied to the surface of fresh fruits and vegetables.

D. G. HEWER.

Pectin. DISTILLERIE DES DEUX-SÈVRES (F.P. 595,349, 11.6.24).—Potatoes or carrots are boiled with acidified water, and the mass is filtered and pressed. The extract is evaporated first to $d \ 1.050$ —1.060, and then to $d \ 1.300$. A. GEAKE.

Recovering oil or fat from milk and cream. A. E. WHITE. From MILK OIL CORP. (E.P. 247,617 and 247,660, 30.11.23).—See U.S.P. 1,485,701 and 1,494,698; B., 1924, 397, 690.

Treating blood (E.P. 224,227).-See XV.

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

Valuation of digitalis. R. WASICKY, F. LASCH, and K. SCHONOVSKI (Arch. Pharm., 1926, 264, 92-98).-Chiefly a review in which chemical and biological methods are compared. The colorimetric method of Knudson and Dresbach (cf. A., 1922, ii., 882), which depends on a colour reaction with picric acid in alkaline solution, is shown to be valueless for testing the crude drug, though it is of some value when pure glucosides are under investigation. For commercial digitalis preparations only biological methods give satisfactory results, and the method of Pick and Wasicky (Wiener Med. Woch., 1917, 6) is recommended because of its comparative simplicity and the rapidity with which it is carried out. There is a definite connexion between the bitterness of a digitalis extract and its potency as a medicament. The value of this as the basis for a simple test is being investigated.

W. A. SILVESTER.

Distillation of aqueous formaldehyde solutions. E. W. BLAIR and R. TAYLOR (J.S.C.I., 1926, 45, 65—66 T).—Aqueous formaldehyde solutions below 30% strength (by vol.) yield distillates of higher concentration than the residual liquors, whilst the reverse holds for solutions above 30%strength. The 30% formaldehyde solution approximates to a mixture of constant minimum boiling point (about 98.7°).

Essential oil of Daucus carota, L. I. Y. ASAHINA and T. TSUKAMOTO (J. Pharm. Soc. Japan, 1925, [525], 1—4; Chem. Zentr., 1926, I., 1820).— Oils from the fruit, fruit and stalks, and stalks alone have d^{22} , 0.9088, 0.9270, 0.9584; $[a]_{\rm D}^{22}$ —5.98°,+ 1.04° , $+5.06^{\circ}$; acid values nil, 2.86, 24.91, and saponif. values 74.08, 69.69, 65.97, respectively. An oil from the fruit and stalks (d^{22} 0.9220, acid value 1.17, saponif. value 66.0, acetyl value 156.8) on fractionation under 7 mm. pressure gave six fractions between 110° and 148°. Each fraction on oxidation with potassium permanganate gave a trihydric sesquiterpene alcohol, $C_{15}H_{28}O_8$. The corresponding monohydric alcohol was isolated from the oil as its mercuric acetate compound, from which the

hydroxide, R·Hg·OH, was obtained by treatment with alkali. The hydroxide, after purification through the chloride, R·HgCl (m.p. 194°), was a starch-like powder m.p. 203° (sulphate, m.p. 135-140°; nitrate, m.p. 153-155°), which on treatment with alcoholic hydrogen sulphide yielded the sesquiterpene alcohol carotol. C15H26O, b.p. 109°/1.5 mm., d415 0.9646, $n_{\rm D}^{15}$ 1.4912, [a]_D+27.9°, +28.03° (1.491 g. in 10 c.c. of alcohol, l = 0.5 dm.). Judging from the molecular refractivity and non-absorption of hydrogen (H + Pt)it appeared to be saturated, but potassium permanganate oxidised it to the above trihydric alcohol dihydroxycarotol, $C_{15}H_{28}O_3$, m.p. 142°, $[a]_D$ —2·41° (monobenzoate, plates, m.p. 83—83·5°, $[a]_D$ +27·36°). Carotol and bromine in glacial acetic acid yielded a dibromide (prisms, m.p. 58.5°), which on boiling with two mols. of silver benzoate and hydrolysis of the product gave daucol, C15H26O2, m.p. 118.5°, [a]p-15.10° (acetyl derivative, m.p. 81.3°). Richter (B., 1909, 1222) gives 116° and 79° respectively. As daucol is very stable and carotol easily loses water the following explanation of the above reactions is suggested :

> $R \cdot OH \rightarrow R(OH)_3 \rightarrow R(OH) : O$ Carotol. Dihydroxycarotol. Daucol. E. H. SHARPLES.

Oil from leaves and twigs of the lemon-tree (petitgrain). P. LIOTTA (Riv. Ital. essenze e profumi, 1925, 7, 107—108; Chem. Zentr., 1926, I, 1309).—The oil prepared by the author had d^{15} 0.8668, and a^{20} 24°15′, acid value 0.97, as acetic acid 0.11%, ester value 51.7, as linally acetate 18.10%, citral (Romeo method) 13.27%, and was soluble in 19 vols. of 80% alcohol. E. H. SHARPLES.

Essential oils of the Crimea. B. RUTOVSKI, I. WINOGRADOVA, and A. KONDRATSKI (Arb. Chem.-Pharm. Inst. Moskaus, 1925, 11, 59—86; Chem. Zentr., 1926, I, 1304—1306).—A survey of the Crimean essential oils and observations on the influence of previous comminution of the plants on the yield of oil by distillation. E. H. SHARPLES.

Essential oils from Sukhum district [Caucasia]. B. RUTOVSKI, I. WINOGRADOVA, and W. KOSLOV (Arb. Chem.-Pharm. Inst. Moskaus, 1925, 11, 93—117; Chem. Zentr., 1926, I, 1306—1307).— Physical and chemical characteristics of 23 essential oils are given. E. H. SHARPLES.

Essential oils of Sochi district [Caucasia]. B. RUTOVSKI, I. WINOGRADOVA, and G. KOLOTOV (Arb. Chem.-Pharm. Inst. Moskaus, 1925, 11, 118— 126; Chem. Zentr., 1926, I, 1307—1308).—Physical and chemical characteristics of 17 essential oils are given. E. H. SHARPLES.

Essential oil of Andropogon citratus D.C. from Sukhum [Caucasia]. W. KRASTELEVSKI (Arb. Chem.-Pharm. Inst. Moskaus, 1925, 11, 159—161; Chem. Zentr., 1926, I, 1308).—A yield of 0·19— 0·35% of lemon-grass oil was obtained from Andropogon citratus, D.C. E. H. SHARPLES.

Yield of some essential oils from Sukhum [Caucasia]. W. KRASTELEVSKI (Arb. Chem.- Pharm. Inst. Moskaus, 1925, 11, 163—166; Chem. Zentr., 1926, I, 1308).—The following yields of oil are recorded: Mentha pulegium, L., 0.63% $(d^{15} 0.930, a_D+12\cdot3^\circ)$ from the fresh plant, Mentha crispa, L., 0.84% $(d^{15} 0.8843)$ from the fresh plant, Mentha piperita, L., 2.49% from the dried plant, Liquidambar orientalis, 0.26% from the green fruits, Satureja hortensis, 0.37% from the dried plants, Thymus vulgaris, L., 0.28—0.36% from the dried plants, Valeriana officinalis, 0.36% from the roots. E. H. SHARPLES.

See also A., Mar., 305, Action of mercuric salts (FLEURY). 311, on dialkylbarbituric acids Organic bismuth compounds (MASCHMANN). 320, Narcotic action of propylene (HALSEY, REYNOLDS, and PROUT); Bacterial chemotherapy, with reference to mercury dyes (RAIZISS, SEVERAC, and MOETSCH); Chemotherapy of bacterial infections (WALKER and SWEENEY). 322, Preparation of standards for colorimetric determination of pepsin (SMORODINCEV and ADOVA). 325, Tuberculin (LONG and SEIBERT). 328, Detection of bismuth in organic material (DANCKWORTT and PFAU); Toxicological investigation of mercury (KOHN-ABREST); Pharmacological determination of ergotamine content of commercial ergot preparations (MAHN and REINERT); Pharmacological determination of ergotamine-ergotoxin titer of ergot (MASUDA).

PATENTS.

Manufacture of organic mercury compounds. FARBENFABR. VORM. F. BAYER & Co. (E.P. 242,669, 9 11.25. Conv., 10.11.24; cf. G.P. 234,851).-Halogen-, nitro-, or halogen-nitro-substituted phenols may be converted almost quantitatively into the corresponding nuclear-substituted mercury derivatives by running a mercury salt solution into a heated solution of the phenol in so much sodium carbonate that the reaction-mixture becomes acid only when decomposition is complete. For example, 50 parts of mercuric sulphate in about 27 pts. of sulphuric acid of d 1.84 and 140 pts. of water are allowed to flow with stirring into a solution of 24.8 pts. of o-nitrophenol in 47 pts. of sodium carbonate and 170 pts. of water at 75°, during $2\frac{1}{2}$ hrs., maintaining the temperature at 72-75°. After stirring for 1 hr. at 70° a 96.5% yield of mercury o-nitrophenol (containing 55-55.5% Hg) is obtained. B. FULLMAN.

Manufacture of tetraglucosan. J. KERB (E.P. 243,348, 14.11.25. Conv., 20.11.24).—Tetraglucosan is obtained by heating dextrose in the presence of a small quantity of metal or metals or their salts other than platinum or zinc and their salts, under diminished pressure or in an inert atmosphere, preferably with an inert diluent. If iron salts be used the iron is converted into a partially insoluble compound. For example, 1 kg. of anhydrous dextrose is treated with 10 drops of 10% ferric sulphate solution and the mass mixed after keeping for a short time. 1 kg. of a mineral jelly oil is added, and the mixture heated under 15 mm. pressure, with stirring, for 1 hr. at

135°. The tetraglucosan (equivalent to 90% of the dextrose) is separated and powdered. B. FULLMAN.

Manufacture of a remedy for foot-and-mouth disease. A. VAN DER WAL (E.P. 247,018, 28.2.25). -An aqueous solution of lead monoxide and lead acetate is treated with potash alum and filtered; an excess of lead must be used, which will remain as a soluble compound. For example, a filtered solution containing 10 g. of lead monoxide and 26 g. of lead acetate per 100 g., is treated with a solution of 15 g. of potash alum, filtered, and the filtrate diluted to 1000 g. To this may be added boric acid as disinfectant; colouring matter free from tannin (such as solution of caramel or extract of chicory); substances to act as accessories and to repel insects, e.g., a mixture of tinctures of eucalyptus, myrrh, and aloe, alcohol, citronella and eucalyptus oils. The above mixtures are suitable for application to the mouth. For application to the hoofs, they are thickened by, e.g., the addition of a mass containing wood-tar, resin, oil (rape, linseed, etc.), beeswax, yolk (grease), zinc oxide, boric (or benzoic or salicylic) acid, eucalyptus and/or citronella oil. B. FULLMAN.

Production of acridine derivatives and their solutions. C. SCHNORF and F. HEFTI (E.P. 247,250, 14.10.24).-Derivatives of 9-aminoacridine react with carbohydrates or mixtures of the latter, with elimination of water, forming compounds soluble in water, and stable to light, air, and in solution, and possessing other therapeutic advantages over such substances as 6:9-diamino-2-ethoxy-acridine. For example, 289 pts. of 6:9-diamino-2-ethoxy-acridine hydrochloride and 180 pts. of galactose in 5000 pts. of 90% alcohol are heated on the water-bath till precipitation of the product ends. After crystallisation from methyl alcohol, and removal of the solvent by heating in air and in vacuo, a bright yellow substance, $C_{21}H_{26}O_6N_3Cl$, m.p. 208–209 is obtained. The compounds may be prepared in solution ready for use by, e.g., adding 100 g. of 6: 9-diamino-2-ethoxy-acridine hydrochloride to 400 g. of lactose dissolved in 600 g. of boiling water, boiling till solution is almost complete, filtering, and treating the cold filtrate with 75 g. of glycerin, 25 g. of N-hydrochloric acid, and 100 g. of water, when the liquid is ready for use.

B. FULLMAN.

Separation of lipoids from ovaries and similar human or animal organs. O. FELLNER (G.P. 420,438, 13.1.21).—The crude product obtained from the organ by extraction with alcohol, ether, and acetone is extracted with 60—75% alcohol, and the extract evaporated. For example, the product obtained by extracting dried and powdered animal placenta successively with alcohol, acetone, alcohol, and ether in the warm, and evaporation, is dissolved in 75% alcohol. Residues of cholesterols and their esters and lipoids of unknown constitution remain undissolved, and on concentration there is obtained a physiologically active mixture of lipoids as a brown, viscous, fatty mass, soluble in alcohol, ether, acetone, etc. (cf. A., 1924, i., 1017).

B. FULLMAN.

Preparation of solutions of medicaments insoluble or difficultly soluble in water. GES. FÜR CHEM. IND. IN BASEL, and W. MINNICH (G.P. 420,649, 13.9.24; Swiss P. 111,560, 21.8.24).— Using phenylacetylene as solvent, solutions may be prepared of camphor, cholesterol and other lipoids, quinine, pyrazolone derivatives, such as phenyldimethylpyrazolone, halogenated alcohols, such as trichlorobutyl alcohol, and oils such as sandalwood oil. Solutions of high concentration can be obtained, which are non-irritant, stable, and can be applied as injections or percutaneously. B. FULLMAN.

Rapid fixing of ethylene by means of sulphuric acid for the purpose of obtaining ethylsulphuric esters. A. A. L. J. DAMIENS, M. C. J. E. DE LOISY, and O. J. G. PIETTE (U.S.P. 1,574,796, 2.3.26. Appl., 13.4.22).—See E.P. 180,988; B., 1923, 440 A.

Bismuth vanadate (G.P. 422,947).-See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Non-static [photographic] film. H. J. HOFF-MAN, Assr. to E. I. DU PONT DE NEMOURS AND CO. (U.S.P. 1,570,062, 19.1.26. Appl., 12.10.21. Renewed 22.6.25).—The film is coated with a composition consisting of a conductivity-increasing substance in a solvent for the film, and then dried. For example, an electrolyte dissolved in a cellulose ester solvent may be applied to a cellulose ester film. A suitable electrolyte is ammonium nitrate applied in the form of a 2% solution in a cellulose ester solvent, sufficient in amount to fix 0.25% of the nitrate on the weight of the film. W. CLARK.

Non-static photographic film. E. C. PITMAN, Assr. to E. I. DU PONT DE NEMOURS AND CO. (U.S.P. 1,570,076-9, 19.1.26. Appl., 16.9.20. 1,570,077 renewed 22.6.25).—(A) The film has a backing containing sodium acetate. (B) A transparent, flexible, thin, substantially neutral film has a backing of a carbohydrate ester, *e.g.*, cellulose acetate, containing glycerin. (c) The film backing contains a substance, such as saponin, capable of reducing surface tension. (D) The film backing contains starch acetate. W. CLARK.

Light-sensitive photographic materials. S. E. SHEPPARD, Assr. to EASTMAN KODAK CO. (U.S.P. 1,574,943-4, 2.3.26. Appl., 6.6.24).—See E.P. 235,210-1; B., 1925, 694.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Drying finely-divided materials [nitrostarch]. J. B. BRONSTEIN, ASST. to TROJAN POWDER CO. (U.S.P. 1,573,673, 16 2.26. Appl., 23.10.19).—Nitrostarch is dried in long narrow tubular fabric bags by exposure to a drying atmosphere. Moisture evaporates from the surface of the bags, and the moisture content of the nitrostarch is reduced by capillarity. C. O. HARVEY,

341

XXIII.—SANITATION; WATER PURIFICATION.

Water purification at East Liverpool, Ohio. D. H. RUPP (Fourth Annual Rep. Ohio Conference on Water Purif., Nov., 1924, 47-59).—The plant in-cludes a grit chamber, 2 coagulation basins, 6 1-million gall. gravity rapid sand filters, and a pedestal type chlorinator. Coagulation removes 81-87% of the bacteria present in the raw (Ohio River) water; filtration 77-84% of the bacteria in the water supplied to the filters; and chlorination 94-97% of the bacteria surviving filtration. The river water contains an average of 4 p.p.m. of soluble iron, and there is probably several times that amount of soluble alumina. This explains the comparatively small amount of coagulant required, 0.7 to 1.5 grain per gal. Considerable difficulty has been experienced with regard to tastes due to pollution of supply by wastes containing phenol. Study of the conditions indicates that tastes complained of are phenol tastes and not chlorophenol tastes as a result of chlorination When the content of phenols, as determined with the Folin-Denis reagent after distillation, was greater than 0.1 p.p.m., there were invariably complaints from consumers. Taste appears to be independent of residual chlorine content, which varies inversely with the organic content, being less when the efficiency of coagulation decreases. Distinct removal of phenols and substances reacting with the Folin-Denis reagent is effected by the purification process as a whole. The phenolic substances removed are not in suspension, and it is believed that they are removed in the coagulation process. When the filter sand was cleaned to remove incrustation, the material removed yielded, per 100 g., 15 mg. of phenol on acidification with sulphuric acid and distillation. Chlorine treatment does not effect any removal of phenols, the phenol content of the filtered and the disinfected water being practically identical. In laboratory experiments the phenol content of distilled water was reduced from 1 to 0.5 p.p.m. by treatment with mangan-permutit. Phenol appears to undergo oxidation on long contact with river water; hence proximity to source of pollution is a factor in taste production. R. E. THOMPSON.

Experiences in the operation of the new municipal water softening plant at Newark, Ohio. C. T. KAISER (Fourth Annual Rep. Ohio Conference on Water Purif., Nov., 1924, 60-64).-The plant (capacity 61 million gals.) consists of mixing chambers, Dorr clarifier, settling basins, rapid sand filters, and chlorinator. Split chemical treatment is employed, an average of 9 grains of lime, 1.5 of soda ash, and 1 of alum per gal. being applied to onethird of the raw water flow; after 20 mins. the treated portion is mixed with the remaining twothirds of the raw water. Hardness is reduced from average of 275 to 85 p.p.m. When incrustant hardness is less than 40 p.p.m. the use of soda ash is dispensed with. An average removal of 98.5% of the suspended solids is effected by the Dorr clarifier. Carbonation is effected by means of carbon dioxide applied through diffusers in the outlet of the settling

basins. The use of scrubbed flue-gas from hand-fired boilers for this purpose was found to give rise to chlorophenol tastes after chlorination, and a coke furnace has been installed for generation of carbon dioxide, 100 lb. of coke being required per million gals. The gas applied contains an average of 5% CO₂, while that emerging as bubbles from the carbonation chamber contains 0.5%. The process is controlled by hourly tests of the phenolphthalein alkalinity, a concentration of approx. 1 p.p.m. of carbon dioxide being maintained in the water applied to the filters. If free carbon dioxide exceeds 2 p.p.m., an increase in the total alkalinity of the filtered water occurs, undoubtedly due to re-solution of the mat " on the filters. R. E. THOMPSON.

New water purification plant at Toronto, Ohio. D. H. RUPP (Fourth Annual Rep. Ohio Conference on Water Purif., Nov., 1924, 65-67).-The plant consists of mixing chamber, coagulation basins, gravity rapid sand filters, and chlorinator. Double coagulation is used at all times, this having been found to give the best results with least application of chemicals. The amount of alum used has averaged 3 grains per gal. in the approximate ratio of 2:1 to first and second basin, respectively; the lime used has averaged 2 grains per gal. With the exception of the first month of operation the filtered water before chlorination has met the requirement of the original U.S. Publ. Health Service standard (2 B. coli per 100 c.c.), and after disinfection with approximately 0.2 p.p.m. of chlorine, the final effluent has easily conformed to the revised standard (1 B. coli per 100 c.c.). R. E. THOMPSON.

Use of sodium aluminate as a coagulant. (1) J. P. BROWNSTEAD. (2) E. T. EDWARDS (Fourth Annual Rep. Ohio Conference on Water Purif., Nov., 1924, 31-36).-(1) During a 4-month period in 1924 when aluminate and alum were used, the cost of coagulants averaged \$9 per million gals., and the reduction in bacteria was more than 90%, compared with a cost of \$12 and bacterial reduction of 70% with alum and lime over a period in 1922 when somewhat similar river conditions prevailed. (2) The results of a 2-month experimental period during which aluminate was substituted for the lime ordinarily used with alum are summarised. When the coagulant was applied in the primary and secondary basins in ratio of 2 to 1 respectively, a slightly higher efficiency was obtained in the primary basin, the floc settling more rapidly and producing a clearer effluent. This resulted in poorer filter efficiency and more algal trouble in the basins, but altering the ratio of coagulant added in the primary and the secondary basins to 1:1 improved the filter influent and efficiency. When sodium aluminate below the normal lime application was used the final effluent contained too great a concentration of free carbon dioxide and had a tendency to be corrosive. R. E. THOMPSON.

Industrial wastes as manures. Rege.—See XVI.