# BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JANUARY 4, 1929.

# I.-GENERAL ; PLANT ; MACHINERY.

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Automatic control through temperature or pressure. C. J. SWAN (Ind. Eng. Chem., 1928, 20, 1152-1155).-The electric switch used consists of a sealed glass tube containing inert gases and a small quantity of mercury. The tilting of the tube causes the mercury to make or break the circuit. The movement is effected by the pressure within a metal bellows operating against an adjustable spring. For pressure control the apparatus to be controlled is connected to the bellows. For temperature control the bellows is charged with a liquid of suitable b.p. The tension of the spring regulates the sensitiveness of the instrument. Unless the unit to be controlled is electrical a motor valve is used. The device is suitable for controlling the supply of steam or hot water to dryers, for maintaining an even gas pressure with a number of boosters, and for maintaining constant levels in tanks. C. IRWIN.

Gas flow through packed columns. S. P. BURKE and W. B. PLUMMER (Ind. Eng. Chem., 1928, 20, 1196-1200).—The pressure drop in a packed column p/l = $C(\rho V^2 S/f^3)$ , where V is the linear velocity, S the surface per unit packed volume, and f the free volume per unit packed volume. C is a function of  $\mu S/\rho V$ , where  $\mu$  is the viscosity of the gas and  $\rho$  the density. The pressure drop for air at 27° flowing with known velocity through columns packed with spheres of various sizes was determined. A further curve was then obtained by plotting C (values from above equation) against  $\mu S/\rho V$ . This curve expresses both viscous and turbulent flow. The principal difficulty in the practical application of the equations corresponding to this curve is in the accurate determination of f. They have, however, checked well with tests on scrubbers etc. Special (e.g., hollow) packing material requires empirical compensations to be applied. C. IRWIN.

Loss calculations in dissolving, leaching, and extraction. C. V. IREDELL (Chem. Met. Eng., 1928, 35, 685—686).—In extraction of one solid from a mixture of solids by means of a liquid, loss is due to incomplete removal of the desired substance, and the extent of this loss can be controlled by means of suitable analysis. Provided that the one constituent only is extracted, that any change in the insoluble residue can be easily reversed, and that the extent of chemical change in the substance removed is known both in the solution and in the residue, then the % loss (L) is given by the equation L = 100B(1-A)/A(1-B), where A is the weight of the desired substance per unit weight of dry raw material, and B is the weight of the same substance per unit weight of dry residue. When chemical change occurs modifications are introduced into this equation, and their mathematical derivation is described. R. H. GRIFFITH.

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Suspension of macroscopic particles in a turbulent gas stream. S. P. BURKE and W. B. PLUMMER (Ind. Eng. Chem., 1928, 20, 1200—1204).—The force (R) acting between a sphere and a turbulent gas stream flowing past it is :  $R = k \varphi V^2 r^2$ , where r is the radius of the sphere. For particles of other shapes  $\pi r^2$ is replaced by other expressions corresponding to the maximum cross-section. The value of k has been determined by measuring the apparent loss in weight of spheres etc. suspended from a balance arm in an upward gas current and averages 0.00084. For irregular coke particles the average value was 0.00093. This value was confirmed by data obtained by measuring the suspension velocities for coke particles in a vertical air current. These results apply only to the case where the whole body of moving gas is in turbulent flow.

C. IRWIN.

Approximate determination of the absolute magnitude of the pores of porous materials. M. RABINOVITSCH and N. FORTUNATOV (Z. angew, Chem., 1928, 41, 1222-1226).- A number of pieces of the porous material of approximately the same shape and with as smooth a surface as possible are first dried at 120°, cooled in a desiccator over phosphorus pentoxide, and weighed. They are then attached to light wires and suspended in a closed vessel over water which is heated at 60-70°, whilst the air is pumped out of the vessel; the test-pieces are then allowed to fall into the water, and after cooling are removed, the adhering water is absorbed with filter paper, and the pieces are weighed again. The volume of water absorbed gives the volume of the pores. The pieces are then placed in a desiccator containing a large volume of sulphuric acid at 0° until a constant weight is obtained; from this weight the volume of the pores still filled with water at the vapour pressure in the desiccator is obtained. The mean radius of the pores that remain filled with water can then be calculated, assuming that they are small capillaries, from Minkovski's equation (cf. Anderson, A., 1914, ii, 632). Tests on various charcoals show that the pores of active carbon consist almost entirely of these micropores having a radius smaller than 10µµ. The proportion of the volume of the micro-pores to that of the total pores for various porous materials has been determined. A. R. POWELL.

Apparatus for rapid sedimentation analysis. C. J. VAN NIEUWENBURG and W. SCHOUTENS (J. Amer. Ceram. Soc., 1928, 11, 696-705).—The apparatus for the continuous determination of the distribution curve of particle size consists of a sedimentation vessel in the form of a Schöne tube, and a long conical underpart, which, in certain experimental circumstances, prevents the formation of vortices. The powdered material (20 g.), dried at 110°, is washed through a conical sieve (30 meshes per linear cm.) into the sedimentation vessel, the desired volume being obtained by the addition of distilled In this liquid a weighted, hollow glass body water. (wt. under water 3-5 g.), which is attached to a balance of regulated movement and sensitivity, is placed at a predetermined position. After compressed air has been admitted in order to render the suspension homogeneous, readings of the movement of the balance pointer are regularly taken. The measured increase in the weight of the suspended body at any moment is related to the weight of the particles which have settled. Two further points on the curve are obtained independently, (1) by measuring the residual solid matter in the suspension after the conclusion of the experiment, (2) by conducting an elutriation in the same apparatus. The radius of the particles is calculated by the application of Stokes' formula. An allowance for a "contraction factor," due to the reduction of the effective cross-section of the tube by the suspended body, is essential. During all experiments the temperature must be maintained A. T. GREEN. constant.

Apparatus for testing filtering etc. media. LINSBAUER and VAŠÁTKO.—See II. Lovibond glasses. JUDD.—See VIII. Aeration of liquids. STICH.— See XVIII.

#### PATENTS.

Calcining furnace. J. H. KNAPP, Assr. to TATE, JONES & CO., INC. (U.S.P. 1,686,565, 9.10.28. Appl., 17.12.24).—The furnace comprises an externally-heated drum; the means for introducing and withdrawing material are connected to the drum in an air-tight manner. B. M. VENABLES.

Furnaces. C. V. A. ELEY (B.P. 299,527, 2.8.27).— In a furnace of the type having tubular or trough-like air passages below the firebars, the bottoms of the troughs are arranged as doors which can be lowered to discharge fine ash. B. M. VENABLES.

Heat interchanger. O. E. FRANK, Assr. to O. E. FRANK HEATER & ENGINEERING CO., INC. (U.S.P. 1,688,183, 16.10.28. Appl., 6.12.27).—The shell of a bundle-of-tubes-type heat exchanger is made in at least two parts so that a baffle for the outer fluid, which slides in grooves formed in the wall of the shell, can be easily removed. B. M. VENABLES.

Heat-exchanging apparatus. P. LEVEQUE (B.P. 293,759, 10.7.28. Belg., 11.7.27).—Cast metal plates fit together in an air-tight manner by means of flanges and grooves. Fins on one side of the plates receive heat from one fluid and conduct it through the plate to fins on the other side in contact with the other fluid. The fins intercalate to form sinuous passages.

#### B. M. VENABLES.

Cooling system. W. H. CARRIER, Assr. to BUFFALO FORGE Co. (U.S.P. 1,687,542, 16.10.28. Appl., 28.7.21). —The system comprises a constant body of air in circulation, with liquid sprays in one part and a liquid eliminator in another part of the circuit. B. M. VENABLES.

Rotary beater-type disintegrating and shredding mills. J. P. VAN GELDER (B.P. 284,230, 20.12.27. Austral., 25.1.27).—The flat ends of disintegrator blades are slit radially (or approximately so) and the tangs thus formed are set to either side alternately.

B. M. VENABLES.

Mixing apparatus. R. B. BROWN (U.S.P. 1,686,593, 9.10.28. Appl., 29.12.24).—A number of knives are fixed in staggered relation on a shaft and rotate within a casing. The material is admitted at the top of one end and exhausted at the bottom of the other end of the casing. B. M. VENABLES.

Apparatus for mixing materials. A. B. and C. R. SMITH (B.P. 298,929, 16.7.27).—A number of sweeps or stirrers rotate in a stationary pan, and cylindrical or conical rollers co-operate with the upper surfaces of the sweeps. B. M. VENABLES.

Drying apparatus. MASCHINENFABR. BUCKAU A.-G. ZU MAGDEBURG (B.P. 275,239,27.7.27. Ger., 27.7.26).— Hot gases are supplied through a drum which rotates in a trough containing the material, and the trough is continued upwards as a casing to collect the vapour. The upper part of the casing is divided into a number of compartments with separate dampers to regulate the withdrawal of vapour. B. M. VENABLES.

Drying machines. P. TURNER (B.P. 299,134, 20.8.27).—A number of rotating drums are arranged one above the other and are annular; the annuli are divided by radial partitions, and the inner cylindrical walls are perforated and serve for admission of drying fluid. The material itself enters the periphery at the upper part of a drum and leaves at the lower part, dropping into the next drum and so on. B. M. VENABLES.

Apparatus for extracting moisture from materials. D. M. A. G. HAWES (B.P. 298,524, 8.7.27).— The material falls on to a rapidly rotating disc and is flung on to the interior wall of a less rapidly rotating element preferably shaped as two hollow cones base to base. It is pushed down the sloping face of the cone by means of fixed scrapers, and drops on to another rapidly rotating disc below and so on downwards through many stages. The drying medium is hot air or other gas. B. M. VENABLES.

Apparatus for drying and handling grain etc. TOMLINSONS (ROCHDALE), LTD., and E. W. SMITH (B.P. 298,369, 10.10.27).—A rotary cylinder to which drying air is admitted has its peripheral wall composed of spaced slats arranged with such an overlap and angle that when the drum is rotated in one direction the material will be completely retained, but when reversed the material will be discharged through the spaces between the slats. A "feathering" device may be added to vary the openings.

B. M. VENABLES.

Separation of materials of different sp. gr. T. M. CHANCE (U.S.P. 1,685,521, 25.9.28. Appl., 28.6.27).—The materials are treated in a pulp comprising a mixture of a fluid and a comminuted solid of sp. gr. intermediate between the constituents to be separated. A number of treating vessels are arranged in staircase form with recirculation of the pulp.

B. M. VENABLES.

Treatment of foundry sand and other finelydivided materials. PNEULEC, LTD., and P. PRITCHARD (B.P. 298,384, 5.11.27).—A rotary drum screen has its screening surface constructed of a number of rods or wires loosely or tightly supported in end members, every or every alternate rod being wound with a helical wire forming a spacer. B. M. VENABLES.

Extracting substances from solid materials. N. NOBEL (B.P. 298,501, 4.7.27).—The extracting liquid flows in a closed circuit, and during part of the circuit the solid material flows with it, being conveyed solely by the flow of the liquid, and is then removed by rakes or similar means. Counter-current extraction may be effected by the use of several liquor circuits. During the extraction period the pulp is subjected to change of pressure, *e.g.*, by pumping-up siphons or by centrifugal means. B. M. VENABLES.

Filter. W. C. GRAHAM (U.S.P. 1,687,863, 16.10.28 Appl., 26.7.24).—Rocking filter trays have trunnions which are mounted on a carrier similar to two wheels abreast. The trunnions on one wheel are connected to a source of vacuum, and the other trunnions to a source of feed-pulp or wash-water according to their position. The passages through the trunnions connect below and above the filter surface in the trays respectively. B. M. VENABLES.

[Sand] filters. B. BRAMWELL (B.P. 298,835, 7.2.28). —The sand is cleaned in the filtering vessel by an upward jet of clean water in conjunction with an open-ended tube which keeps the whole sand in a state of turmoil and removes the silt with the overflow. Several cleaning jets and uptake tubes may be arranged in one vessel, and a single multiple-valve may effect the change of connexions from filtering to cleaning, and may be automatically operated by the increase of pressure due to the filter becoming choked. B. M. VENABLES.

Suction filter. FELD & VORSTMAN GES.M.B.H. (G.P. 452,514, 17.12.24).—The apparatus comprises a suction chamber, a filter surface, and a pressure chamber above it for receiving the material to be filtered, the upper part being hinged so that it can be tilted over to empty it by gravity and to facilitate cleaning and replacing the filter surface. A number of filter units may be arranged in a common pressure chamber. L. A. COLES.

Straining and filtering apparatus. J. G. McKEAN and R. F. JONES (B.P. 298,856, 23.3.28).—A number of horseshoe-shaped plates are assembled with distance pieces to form narrow spaces between for the flow of liquid. Cleaning fingers rotated by a handle are also interleaved. B. M. VENABLES.

[Dust] filters. J. J. C. BRAND and B. LAING (B.P. 299,080, 19.7.27.)—A cone of great height of filtering medium is folded back on itself many times forming a series of concentric, conical surfaces of alternate slope. The whole is hung in a cylindrical container by means of the upper circular edges or folds, and the lower circles are attached to weights or springs so that the filter fabric can be shaken. B. M. VENABLES.

Air filters for separating dust from dust-laden air. E. BUDIL (B.P. 275,257, 29.7.27. Ger., 31.7.26).— The filter is constructed of an assemblage of polygonal tubes which are perforated with many slots, the material of the slot being bent inwards. B. M. VENABLES.

Apparatus for removing dust from gases. Soc. ANON. DES ÉTABL. NEU (F.P. 628,200, 28.1.27).— Centrifugal force set up in the gas stream by direction against a central cone drives the dust particles into annular cells surrounding the cone. L. A. COLES.

Device for separating dust, water, oil, etc. from air, gases, and vapours. HUNDT & WEBER, GES.M.B.H (B.P. 298,855, 20.3.28. Ger., 10.11.27).—Within a separating vessel having a tangential supply pipe is a smaller device acting on the same principle, the supply pipes to the inner separator being downwardly inclined as well as tangential, and the final outlet for cleaned gas upwards at the axis of the inner separator. The bottom of the inner separator is held in place only by a spring, so that it can move downwards slightly to permit separated liquid and dirt to pass into the outer collector. B. M. VENABLES.

Combined filter and separator. A. C. HANDLEY (B.P. 299,648, 20.1.28).—An apparatus is described in which the bulk of the oil (from a mixture of oil and water) is separated by gravity and the remainder caught in a fibrous filter. B. M. VENABLES.

Removal in layers of material from vacuum filters, especially from drum filters. R. Wolf A.-G. (G.P. 452,392, 19.3.24).—The apparatus is provided with an outer, fixed scraper and with an inner scraper which penetrates gradually into the filtered material, and is returned automatically to its original position as soon as it reaches the innermost layer.

L. A. COLES.

Wet treatment of subdivided materials involving filtration or screening. S. C. SMITH (B.P. 298,172, 5.4. and 18.6.27).—Rubber-woven fabric or perforated sheet is used as a filtering (or percolating) medium either by itself or as a corrosion- and erosion-resisting lining or backing to other filter media.

B. M. VENABLES.

Apparatus for distillation of solid matters. H. DUPUY (B.P. 289,425, 22.3.28. Fr., 27.4.27. Addn. to B.P. 198,661; B., 1924, 497).—In the process previously described the steam is superheated by passing over incandescent coal, and an injector is used as the circulating pump. B. M. VENABLES.

Apparatus for separating liquids of different densities. F. PINK (B.P. 298,683, 29.6.27).—The apparatus is of the type through which the mixed fluid flows so slowly (less than 2 ft./min. for oil and water) that stream-line flow is obtained and the two liquids are separated by gravity. B. M. VENABLES.

Apparatus for sterilising liquids. N. V. HUYGEN & WESSEL'S INGENIEURSBUREAU (B.P. 290,555, 25.8.27. Holl., 14.5.27).—The whole steriliser is rocked, and the inner container for the goods is preferably springmounted within the outer container for the heating and cooling medium. B. M. VENABLES.

Treatment of liquids with reagents. R. JEANNIN and R. M. BERLINE (F.P. 627,822, 20.1.27).—Reagents mixed with porous or non-porous, inactive, finelydivided material are brought in intimate contact with the liquid. L. A. COLES.

Apparatus for expelling gases from liquids. A. VOGT (G.P. 452,265, 17.10.24).—A vessel attached to a source of reduced pressure is provided with up- and downflow pipes both dipping into the same container.

L. A. COLES.

Apparatus for distillation, rectification, or evaporation. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,084, 20.7.27).—The vapour outlet from a still is bent into a hook shape, the discharge being downwards into the reflux tube, so that the vapour has to double back upwards before it can finally exhaust. The reflux tube may continue downwards as a screw column, and a cooler may be provided in the upper part.

B. M. VENABLES.

Treatment of gases with liquids in scrubbing towers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299, 075, 20.6.27).—The tower has no real filling, but is provided with a spiral slide down which the liquid flows in a sheet, the centrifugal force being counteracted by sloping the surface radially as well as helically. The hand of the helix may be changed part way down, and various additional devices, *e.g.*, for temperature regulation, are claimed. B. M. VENABLES.

Gas washers. M. MALLET (B.P. 297,765, 29.3.28. Fr., 27.9.27).—In an apparatus comprising several comparatively low towers side by side, the circulating pumps draw from sumps intermediate the towers, and to ensure that all the liquid is circulated the level of the liquid in the intermediate sumps is maintained below that in the sumps under the towers.

B. M. VENABLES.

Treatment of a metallic, organic, or other compound, or a gas, by a gaseous agent serving to enter into reaction therewith. Soc. INTERNAT. DES PROC. PRUDHOMME HOUDRY (B.P. 274,846, 12.7.27. Fr., 23.7.26).—A substance is treated by a gaseous reagent in the absence of a true catalyst, but after the gas has passed through an activating (" atomic-rendering ") filter which is confined in a separate chamber. The activating filter contains substances similar to those usually used as catalysts. B. M. VENABLES,

Separation of gases and vapours by means of solid adsorbents. Soc. DE RECHERCHES ET D'EXPLOIT. PÉTROLIFÈRES (Addn. F.P. 31,976, 4.1.26, to F.P. 607,683; B., 1927, 210).—Vacuum apparatus is attached to the outlet of the second cooler in the apparatus described previously so that the water boils at a lower temperature (30—50°) in the evaporator. L. A. COLES.

Recovery of adsorbed gases from solid adsorbents. I. G. FARBENIND A.-G., Assees. of G. and R. WIETZEL (G.P. 452,456, 8.4.22).—The gases are recovered in fractions by heating the material and then treating it with steam under reduced pressure. L. A. COLES.

Effecting reactions under pressure. M. TORNOW (G.P. 452,216, 16.1.27).—The reaction vessels are constructed of natural stone. L. A. COLES.

Colour-estimating apparatus. O. ROSENHEIM, E. H. J. SCHUSTER, and TINTOMETER, LTD. (B.P. 299,194, 31.10.27).—Standard slips of transparent material are uniformly coloured individually, and a number of them are assembled in one plane to form a series, several of which may be mounted behind each other. Every series is movable in both directions in its own plane so that any part can be brought across the line of sight, which is to one side of the centre line of the instrument, while the line of sight to the object to be compared is correspondingly at the other side. B. M. VENABLES.

**Penetrometers.** E. and E. A. GRIFFITHS (B.P. 298,662, 12.7.27).—A blunt-nosed plunger surrounded by a guard ring is presented to the surface of the material to be tested. A spring is inserted between the plunger and the operating handle, and the deflexion of the spring is indicated by a pointer which is automatically locked at its reading (of the load) when the plunger has penetrated a predetermined distance.

B. M. VENABLES.

Grinding and pulverising mill. B. SCHERBAUM (U.S.P. 1,691,210, 13.11.28. Appl., 23.12.25. Ger., 31.12.24).—See B.P. 245,435; B., 1926, 695.

Valves for high-pressure and high-temperature purposes. O. D. REDDING (B.P. 286,273, 1.3.28. U.S., 2.3.27).

Apparatus for screening or washing coal, coke, ballast, ores, grain, etc. W. J. FIDDES (B.P. 299,102, 30.6.27).

[Closing device for] filter presses for extracting liquids from viscid substances. B. GowsHALL (B.P. 298,282, 12.7.27).

Centrifugal apparatus (G.P. 452,486).—See VII. Gas purification (G.P. 452,394 and 452,437).—See XI.

# II.-FUEL; GAS; TAR; MINERAL OILS.

Bristol and Somerset coalfield. Carbonisation of "Parkfield large gas " coal. Anon. (Dept. Sci. Ind. Res., Fuel Res. Survey Paper No. 12, 1928, 39 pp.). --Carbonisation tests have been carried out on a 750-ton consignment of "Parkfield large gas" coal, of which the proximate analysis gave : moisture 1.5%, volatile matter 34.3%, fixed carbon 56.6%, ash 7.6%. In the laboratory assay at 600° the coal yielded a swollen and porous coke similar to that given by typical Yorkshire and Durham gas coals. The coal worked satisfactorily in both vertical (Glover-West) and horizontal retorts; owing to its highly swelling character it presented some difficulty in first starting up in the vertical retorts, but this was overcome either by starting up with another coal or by reducing the temperature of the top combustion chambers. The yields of products per ton of coal from the vertical retorts when using 5% of steam were : coke 13.7 cwt., gas 16,030 cub. ft. (511 B.Th.U./cub. ft.), tar 11.5 gals., ammonium sulphate  $22 \cdot 8$  lb. The best results with the horizontal retorts were obtained with charges of 12<sup>1</sup>/<sub>2</sub> cwt. carbonised for a period of 12 hrs., giving exactly the rated throughput for the setting. Tests were carried out with both producer-gas and water-gas heating. The yields per ton of coal were : coke 14.0cwt. (including 3.66 cwt, used in the producer), gas 12,930 cub. ft. (568 B.Th.U./cub. ft.), tar 10.4 gals., ammonium sulphate 26.7 lb. The coal did not lend itself to easy carbonisation in low-temperature vertical retorts owing to holding up of the charge. The difficulty was largely overcome by briquetting the fines (with 7% of pitch and 24% of coke breeze) before charging to the retort. Both the high- and low-temperature cokes gave trouble when used for steam-raising purposes under a Lancashire boiler and as producer fuels in a suction-gas plant owing to the fusible nature of the ash. A. B. MANNING.

Composition of coal-its rational analysis. W. FRANCIS and R. V. WHEELER (J.C.S., 1928, 2967-2979).-A rational analysis of a coal can be given in terms of free hydrocarbons, resins, organised plant entities, and ulmin compounds ; chemical compositions of these substances, with the exception of the ulmins, do not vary very much, whether these ingredients form part of a lignite, bituminous coal, or semi-anthracite, but the composition of the ulmin compounds varies, depending on the coals from which they are obtained. The difference in character of these ulmins may be denoted by determining their reactivity with oxygen, which is obtained directly from the amount of oxygen combining with them in a given time and indirectly from the rate of production of alkali-soluble ulmins under standard treatment with an oxidising agent. Examined in this way the ulmins show a graduation in properties in the series : peat, lignite, bituminous coal, anthracite. Rational analyses are given of a series of coals from the Pittsburg seam (U.S.A.), ranging in character from semi-bituminous to semi-anthracite, as well as for a number of coals of younger formation. For the Pitts-burg seam in particular the "reactivity index," *i.e.*, degree of oxidation of the ulmins, is shown to afford a measure of its rank or "degree of coalification." Methods of estimating the proportions of hydrocarbons, resins, organised plant entities, and ulmins are given, together with the determination of the "reactivity index " of the last-named. C. B. MARSON.

Spontaneous heating of coal. J. D. DAVIS and D. A. REYNOLDS (U.S. Bur. Mines, Tech. Paper No. 409, 1928, 74 pp.).-The tendency of a coal to self-heat increases as the size of the particles decreases; fine coal is therefore dangerous to store. Self-heating is more pronounced with coals of low geological rank; these should be stored so that segregation of fines is prevented and foreign matter excluded as much as possible. As to what effect moisture in coal has on spontaneous heating, the authors consider that this is probably determined by the conditions of storage; if those parts of the pile liable to develop spontaneous heating could be wetted, then it might be prevented because the heat required to vaporise the water would be more than the oxidising coal could supply, but wetting down the whole surface of the pile changes the conditions of ventilation and may favour heating at points not reached by the water. Occluded gases probably have little effect. The tendency to self-heat increases with the temperature of initial exposure, hence storage near steam pipes and such like is to be avoided. Finely-divided pyrites increases the risk of spontaneous heating, and should therefore be absent from storage coals. Oxidation of the coal substance is the main cause; certain organic constituents may contribute more to the heating than others, but no single constituent exerts a predominating influence, to the exclusion of the rest. Spontaneous heating takes place in two stages; the first, which operates at room temperature, is associated with the physical absorption of oxygen and the formation of a coal-oxygen complex; as the temperature rises, this complex decomposes, yielding carbon monoxide, carbon dioxide, and water, which forms the second stage. This stage generates more heat than the first; there is no sharp transition point from one stage to another. C. B. MARSON.

Causes of oxidation of coal. D. J. W. KREULEN

(Chem. Weekblad, 1928, **25**, 642—646).—The humic acid formed by heating dry coal in air for 2 hrs. at 175° increases fairly regularly with the proportion of real volatile matter, but there are considerable deviations. The readiness with which a coal oxidises is bound up also with its friability and resistance to grinding. Methods of expressing these factors quantitatively are described. S. I. LEVY.

The sulphur problem in burning coal. J. F. BARKLEY (U.S. Bur. Mines, Tech. Paper No. 436, 1928, 7 pp.).—The tendency of a coal to clinker, often associated with its content of sulphur, is really due to the presence of pyrites which, when present, gives ferrous sulphide and ferrous silicates—the active clinkering agents. On combustion, usually not more than 10%of the sulphur originally present in a coal is retained by the ash, the remainder passing into the flue gases as sulphur dioxide and trioxide. The method of calculating the temperature of the flue gases at which condensation of (a) water, (b) sulphuric acid will occur is described, as it is considered that such condensation is necessary before appreciable corrosion of iron and steel will take place. W. H. BLACKBURN.

Laboratory apparatus for testing filtering and decolorising media. A. LINSBAUER and J. VAŠÁTKO (Z. Zuckerind. Czechoslov., 1928, 53, 25-30).—A small filter has been designed primarily for the examination of decolorising carbon according to the layer method, the active surface taking 2—10 g. of carbon per 100 cm.<sup>2</sup> Principal advantages claimed are constant working temperature and rate of flow, and an equal pressure over the whole of the filtering surface. Filtration proceeds through the layer horizontally, this being considered to be the only correct means of layer application of decolorising carbons. Liquor used for the tests is clarified, passed through a filter-press, and stored in a tank at a suitable height above the filter.

J. P. OGILVIE.

Wet gas purification. FERBERS (Gas- u. Wasserfach, 1928, 47, 1133—1137).—In dry purification of gas with iron oxide, the greatest drawback is the difficulty of employing material in a sufficiently finely-divided state to give rapid reaction; this has been overcome by suspending the oxide in water, the mixture containing 1 or 2 g. of iron hydroxide per litre in the presence of alkali. A plant capable of treating 100,000 m.<sup>3</sup> of gas per day has been built for the Mont Cenis mine and

contains three washers, 18 m. in useful height and 250 cm. in diam. A test lasting 14 days gave the following results : throughput of gas 67,975–88,585 m.<sup>3</sup> per day; hydrogen sulphide in outlet gas 6-0.6 mg./m.3, corresponding with a purification efficiency of 99.97% overall; air used 4-4.5%; daily consumption of caustic soda 250-275 kg., and of iron oxide 80 kg. The sulphur recovered contained 37.5% of water; the working temperature was 35°, and the cost of the process 0.153 pfennig/m.3 of gas treated.

# R. H. GRIFFITH.

Removal of tar fog from gases. L. Eck (Chem. Fabr., 1928, 657).-For small-scale experimental purposes tar fog is most readily separated from gas streams by washing, frictional-impact condensation, or electrical precipitation methods. The first type of separator is seldom used, but apparatus of the cyclone (Pelouze-Audouin or Lodge-Cottrell) class can readily be applied.

R. H. GRIFFITH.

Indene and styrene. Crude materials in industrial quantities. R. L. BROWN (Ind. Eng. Chem., 1928, 20, 1178-1180).-Thirteen representative samples of the oily condensate which collects in carburetted water-gas mains and attached appliances have been analysed. The fresh, dry liquid contains up to 15% of styrene and 35% of indene. Fractions containing 75% of indene are obtainable by rough fractionation, and with more efficient fractionation fractions containing up to 75% of styrene can be obtained. The remaining 25% consists almost entirely of stable, aromatic hydrocarbons which can be removed by polymerisation of the styrene or by the formation of simple derivatives. The naphthalene contents of the oils are given and the methods of analysis and identification of the components are described. Potential supplies and industrial uses of styrene are discussed (cf. B., 1924, 48). E. H. SHARPLES.

Basic nitrogen compounds from Fushun shale tar. II. T. EGUCHI (Bull. Chem. Soc. Japan, 1928, 3, 227-243; cf. B., 1927, 696).-A method for the separation of various methylpyridines based on carefully controlled fractional precipitation with picric acid and purification of the various picrate fractions has been worked out and applied to the isolation, in the pure state, of these derivatives in Fushun shale oil. The following have been isolated and their physical constants determined : 4-methylpyridine, b.p. 145.45-145.47°/ 763 · 9 mm.,  $d_4^{25}$  0 · 9502 (vacuum),  $n_p^{25}$  1 · 5029 (picrate, m.p. 167°); 3-methylpyridine, b.p. 144·17-144·18°/763·0 mm.,  $d_{\perp}^{25}$  0.9518 (vacuum),  $n_{p}^{25}$  1.5038 (picrate, m.p. 149°; chloroplatinate, m.p. 208°; chloroaurate, m.p. 187°; double mercurichlorides B,HCl,HgCl2, m.p. 145-146°, B,HCl,2HgCl<sub>2</sub>, m.p. 146°, B,HCl,3HgCl<sub>2</sub>, m.p. 170°, where B is 1 mol. of the base); 2:3-dimethylpyridine, b.p. 160·7°/760 mm.,  $d_4^{25}$  0·9419,  $n_p^{25}$  1·5057 (picrate, m.p. 188°; double mercurichloride, B,HCl,6HgCl<sub>2</sub>, m.p. 194°); 2:5-dimethylpyridine, b.p. 156.82- $156 \cdot 88^{\circ}/758 \cdot 1$  mm.,  $d_{4}^{25} = 0.9261$  (vacuum),  $n_{D}^{25} = 1.4982$ (picrate, m.p. 167°; chloroplatinate, m.p. 214°; double mercurichloride, B,HCl,6HgCl2,H2O, m.p. 203°). From the fraction of shale tar b.p. 170-172° were isolated 3:5-dimethylpyridine, b.p. 171.6°/760 mm., d<sup>25</sup>/0.9385,  $n_{\rm p}^{25}$  1.5032; 2:3:6-trimethylpyridine, b.p. 172.88- $172 \cdot 91^{\circ}/761 \cdot 2$  mm.,  $d_{4}^{25} \ 0 \cdot 9220$  (vacuum),  $n_{D}^{25} \ 1 \cdot 5018$ 

(picrate, m.p. 147-148°; chloroplatinate, decomp. 220°; chloroaurate, m.p. 139°; double mercurichlorides, B,HCl,2HgCl<sub>2</sub>,H<sub>2</sub>O, m.p. 104°, B,HCl,6HgCl<sub>2</sub>, m.p. 165°), and 2:4:6-trimethylpyridine. From the fraction b.p. 178-180° were isolated 3: 4-dimethylpyridine, b.p.  $178 \cdot 82^{\circ}/759$  mm.,  $d_{4}^{23}$  0.9537 (vacuum),  $n_{D}^{24}$  1.5099 [picrate, m.p. 163°; chloroplatinate, m.p. 276° (decomp.); double mercurichloride, B,HCl,3HgCl2,2H20, m.p.  $130 \cdot 5 - 131^{\circ}$ ; *chloroaurate*, m.p.  $187^{\circ}$ ], and 2-methyl-4-ethylpyridine, b.p.  $179 - 180^{\circ}$ ,  $d_{4}^{25}$  0.9130,  $n_{p}^{25}$  1.4953. From the fraction b.p.  $186 - 190^{\circ}$  was isolated 2:3:5-trimethylpyridine, b.p. 186.75°/761.1 mm., d<sup>25</sup>/<sub>4</sub> 0.9310 (vacuum), n<sup>25</sup>/<sub>p</sub> 1.5057 (picrate, m.p. 184°; chloroplatinate, m.p. 212°; double mercurichloride, B,HCl,3HgCl<sub>2</sub>, m.p. 110°), and 2:4:5-trimethylpyridine, b.p. 190.04-190.05°/766.1 mm., d<sup>25</sup> 0.9330 (vacuum), n<sup>25</sup> 1.5054 (picrate, m.p. 161°; chloroplatinate, m.p. 192°; double mercurichloride B,HCl,3HgCl<sub>2</sub>,2H<sub>2</sub>O, m.p. 112°). From the fraction b.p. 200-202° was isolated 2:3:4:6-tetramethylpyridine, b.p.  $203 \cdot 9^{\circ}/771 \text{ mm.}, d_4^{25}$ 0.9229, n<sup>25</sup><sub>D</sub> 1.5084 (picrate, m.p. 107°). From the fraction b.p. 187-188° was isolated 2:6-dimethyl-4ethylpyridine, b.p. 187.5-188°/758 mm., d<sup>23</sup> 0.9089, n<sup>25</sup><sub>D</sub> 1.4964 (syrupy picrate; chloroplatinate, m.p. 203°), and from the fraction b.p. 189-190° 2: 3-dimethyl-6(?)ethylpyridine, b.p. 190-190.5°/764 mm., d25 0.9107,  $n_{\rm p}^{25}$  1.4970, was obtained. From the final fraction, b.p. 200-202°, a new base pyridane (probably represented



by the annexed formula), b.p.  $199 \cdot 8^{\circ}/$ -CH<sub>2</sub> 761 mm.,  $d_4^{25}$  1.029,  $n_D^{25}$  1.541 (picrate, m.p. 181°), was isolated. From a comparison of the various accurate b.p. data it is found that the rise in b.p. produced by a methyl group in the

 $\alpha$ -position is about one half that produced by a methyl group in the  $\beta$ - or  $\gamma$ -positions in the pyridine ring, the b.p. of the various methylpyridines being expressed by the empirical formula  $T = 115 \cdot 3^\circ + 14m + 28m' + C$ , where T is the b.p. of the methylpyridine, m the number of  $\alpha$ -methyl groups, and m' the number of  $\beta$ - or  $\gamma$ -methyl groups, C is a variable correction term, being  $4^{\circ}$  for 2:3substitution, and 8° for 3:4-substitution, these values being reduced to one half if the two side-chains are situated in the 1:4-positions. J. W. BAKER.

Benzine for precipitation of asphaltenes from mineral oil. H. BURSTIN and J. WINKLER (Przemysl Chem., 1928, 12, 445-463).-Benzine used for the precipitation of asphaltenes should consist of saturated aliphatic hydrocarbons possessing the following pro-perties : b.p. 65—95° (70% boiling from 65—80°),  $d^{15}$  0.685—0.695, raffination number<20,  $n^{20} < 1.3950$ , aniline point  $> 64^\circ$ , and the fraction boiling from  $80^\circ$  to 95° should have  $d^{15} 0.700$  and  $n^{20} > 1.4000$ .

R. TRUSZKOWSKI.

Preparation of contact substances from the solar oils of Grozni. M. D. TILITSCHEEV and A. I. DUMSKI (Neft. Choz., 1926, 11, 738-743).-Acids formed by the action of fuming sulphuric acid on solar and machine oil distillates are used as catalysts in the saponification of fats, the condensation of phenols with formaldehyde, etc. After treatment with the acid, the residual oil was extracted with aqueous alcohol; the effect of repeated extraction was studied. The acid causes resinification and then sulphonation of the aromatic hydrocarbons. The residue consists chiefly of naphthene hydrocarbons. The higher is the density of the original oil, the greater is the yield (13-22%) of contact substance.

#### CHEMICAL ABSTRACTS.

Oxidation of mineral oils in presence of soluble catalysts. G. S. PETROV, A. I. DANILOVICH, and A. Y. RABINOVICH (Papers Karpov Chem. Inst., Bach Mem. Vol., 1927, 157-168).-Manganese naphthenates were most, and zinc naphthenates least, active in effecting the oxidation of mineral oils; copper and lead salts were intermediate. Manganese salts of sulpho-acids of high mol. wt. were even more active. Oils refined with sulphuric acid oxidise to about the same extent as nonrefined oils; the former yield a larger amount o' soluble acids, whilst the latter yield more oxy-acids. Distilled oils highly refined with fuming sulphuric acid do not oxidise in absence of catalysts even when tre ted with air at 150° for 40 hrs., but darken as a result of decomposition. CHEMICAL ABSIRACTS.

Determination of the composition of petroleum. B. TARASSOV (Neft. Choz., 1928, 11, 67—70).—Unsaturated hydrocarbons are determined approximately by measuring the critical solution temperature with aniline before and after treatment with 80% sulphuric acid; on removal of the sulphuric acid with aqueous alkali, naphthenic acids are removed. Aromatic hydrocarbons can be determined by nitration; suitable formulæ for converting nitro-derivatives into hydrocarbons are given for each fraction. CHEMICAL ABSTRACTS.

Mol. wt. of petroleum products. B. KAMINER (Neft. Choz., 1926, 11, 219-223).—The following mol. wts. were obtained ebullioscopically in benzene: crude petroleum 250-263; gasoline (b.p. up to 190°) 119-122; kerosene (b.p. 190-259°) 166-170; gas oil (259-264°) 227-231; solar oil (264-310°) 288-297; machine oil (310-320°) 411-420; cylinder oil (322-330°) 474-481; tar 711-732. CHEMICAL ABSTRACTS.

Fifteen years of the Burton process [for cracking heavy petroleum oiis]. R. E. WILSON (Ind. Eng. Chem., 1928, 20, 1099—1101).—The development of the Burton process is historically reviewed. G. CLAXTON.

Liquid-phase cracking processes. E. OWEN (Chem. Met. Eng., 1928, 35, 677-681).—A review of the development of oil-cracking processes and plant; the older Burton, Coast, and Fleming methods are being replaced by others such as those of Jenkins, Isom, and Dubbs. R. H. GRIFFITH.

Dossor mineral oil. B. G. TUICHININ and S. N. PAVLOVA (Papers Karpov Chem. Inst., Bach Mem. Vol., 1927, 215-29).—The characteristics of the three horizontal layers of the Dossor (Caucasus) oil fields are described. CHEMICAL ABSTRACTS.

Higher hydrocarbons from methane. H. M. STANLEY and A. W. NASH (Nature, 1928, 122, 725).— Fischer's statement (B., 1928, 844) that under suitable conditions of heating methane yields higher hydrocarbons is confirmed. -A. A. ELDRIDGE.

Higher hydrocarbons from methane. R. V. WHEELER (Nature, 1928, 122, 773).—A claim for priority (cf. Fischer, B., 1928, 844). The production of benzene during the decomposition of ethylene is believed to be by way of ethylene and butadiene.

#### A. A. ELDRIDGE.

Determination of hydrocarbon vapours in air by active carbon. E. POSNER (Z. anorg. Chem., 1928, 174, 290—294).—The determination of hydrocarbon vapours by adsorption on active carbon is unaffected by carbon monoxide and methane, but acid and alkaline gases should first be removed. The preparation of the apparatus takes about 20 hrs., and the actual determination up to about 1 hr., with a rate of flow of 60 litres per hr.

H. F. GILLBE.

Cresol-naphthene soap solutions. KOGAN.—See XII. Brewers' pitch. KUTTER.—See XVIII.

#### PATENTS.

Treatment or purification of coal or other carbonaceous material. Soc. ANON. D'OUGRÉE-MARIHAYE (B.P. 295,312, 16.9.27. Fr., 9.8.27).—Lowash coals are separated by a "float-and-sink" method in which the crushed material (0.5-10 mm.) is impregnated with liquors, e.g., solutions of calcium chloride, of successively increasing strengths up to that of the density requisite for the separation. After the separation the liquors are used in the reverse order for washing the coal, each liquor moving in a closed cycle, first wetting the coal and so becoming weaker, then washing the coal and thereby being restored to substantially the original strength. A. B. MANNING.

Apparatus for distillation of coal and other solid fuels. R. FEIGE (B.P. 279,120, 15.10.27. Ger., 15.10.26). -A retort for the carbonisation of powdered fuel is provided with a central vertical shaft which can be rotated and to which are attached a number of flat or funnel-shaped plates. Interposed between these plates are stationary funnel-shaped surfaces attached to the outer wall of the retort. The material falling on one of the moving plates is projected outwardly in a thin layer by the centrifugal action. After passing over the edge of the plate it falls by gravity down the stationary plate on to the centre of the succeeding rotating plate, and so on through the retort. The retort is double-walled and is heated by the passage of hot gases through the annular space so formed. These gases may then pass up through the retort itself counter-current to the flow of the material. Pipes may be provided for the introduction of hydrogen, hydrocarbons, etc. to different zones of the retort if desired. The apparatus may be modified to work under pressure or under a vacuum, or to permit the material to be subjected to an electrical discharge.

A. B. MANNING.

Low-temperature carbonisation of bituminous coals etc. C. H. LANDER and J. F. SHAW (B.P. 299,201, 11.11.27).—A vertical retort for the low-temperature carbonisation of coal has the horizontal cross-section in the form of an elongated figure of which the opposite sides are parallel, and is tapered vertically in such a way that the rate of increase of the cross-sectional dimensions is greater at the lower end of the retort than at the upper. The retorts are conveniently built up of channelshaped end-sections and plates which are interposed between them. Each complete section may be made to

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engage with that immediately below it by connexions of the spigot and socket type. A. B. MANNING.

Vertical retorts and the like. C. H. LANDER and J. F. SHAW (B.P. 299,202, 11.11.27; cf. B.P. 299,201, preceding).—The lower end of a retort of the type previously described (*loc. cit.*) is not rigidly secured to the outlet duct below but rests thereon in such a manner that it is free to expand laterally. The relative dimensions of these two parts are such that when the retort is fully expanded the lower end is not larger in cross-section than the adjacent part of the outlet duct. Thus the lower end of the retort may be provided with a skirt or socket which rests within a trough or spigot on the upper end of the outlet duct. To facilitate relative motion between the two parts the surfaces in contact may be coned. A. B. MANNING.

Destructive hydrogenation of carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 276,001, 11.8.27. Ger., 11.8.26).—Carbonaceous material is converted into valuable hydrocarbons by destructive hydrogenation in presence of catalysts containing up to 10% of silver (either free or combined), the remainder of the catalyst being boron, aluminium, silicon, titanium, vanadium, tantalum, or molybdenum, either free or combined, or cobalt compounds, or mixtures thereof. Hydrogen in excess or gases containing, or giving rise to, hydrogen are employed at pressures above 50 atm., and the reaction is carried out at above 400°.

#### G. CLAXTON.

Coke ovens. SIMON-CARVES, LTD., and J. H. BROWN (B.P. 298,285, 14.7.27).—In coke ovens of the crossregenerator or other type, the air for each gas burner (or group of burners) is taken from the atmosphere at a point below the structure, the inlets having valves controlled from outside the structure and preferably connected in groups for operating by a single controlling means which may be combined with the gas-reversing gear. From the valves the air passes upwards in conduits through the regenerator sole flues.

B. M. VENABLES.

Ovens of the rotary, annular sole type. TROCK-NUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES.M.B.H. (B.P. 293,449, 13.6.28. Ger., 7.7.27).—The material to be heated is spread in the thinnest possible layer on the sole, and the latter is divided into several segments each provided with charging and discharging zones. The dust-like product may be removed by a blast of gas, one for each segment, and recovered in a cyclone or other separator, one separator being common to all the segments. B. M. VENABLES.

Adsorption media. LURGI-GES. F. WÄRMETECHNIK M.B.H. (B.P. 280,505, 9.9.27. Ger., 11.11.26).—The flue dust from combustion plants is used as an adsorption medium, after being subjected to selective separation, *e.g.*, by centrifugal or electrical action, combined with known washing processes. A. B. MANNING.

Absorptive carbon. Soc. ANON. DES ENGRAIS ET NOIR ANIMAL (F.P. 624,277, 3.3.26).—Carbonaceous material is saturated with a mixture of sulphuric and phosphoric acids, heated at a temperature below the activation temperature, compressed, and then strongly heated to complete carbonisation. A. R. POWELL.

Active carbon. Soc. ANON. DES ENGRAIS ET NOIR ANIMAL (F.P. 626,983, 7.4.26).—Material rich in cellulose is impregnated with an activating compound prior to carbonisation. A. R. POWELL.

Treatment of coke. C. L. WAGGONER and F. B. THACHER, ASSTS. to BY-PRODUCTS COKE CORP. (U.S.P. 1,685,654, 25.9.28. Appl., 9.4.28).—The porous coke, produced by high-temperature distillation and free from volatile constituents, is mixed with a moistureabsorbing alkaline-earth compound. F. G. CLARKE.

Manufacture of coke and electrodes therefrom, chiefly for use in electrometallurgy. Soc. ANON. D'OUGRÉE-MARIHAVE (B.P. 295,314, 28.10.27. Fr., 9.8.27).—Coal is purified by the process of B.P. 295,312 (B., 1929, 7) and carbonised in a strongly heated retort, or briquetted and carbonised in a current of heated nitrogen or steam, producing a partly graphitised coke containing less than 1% each of ash, sulphur, and volatile matter, suitable for electrodes. A. Key.

Gas generators. E. C. R. MARKS. From BERNITZ FURNACE APPLIANCE Co. (B.P. 299,627, 2.12.27).—A gas generator is lined with hollow bricks the cavities of which communicate with each other in such a way as to form a continuous space between the inner and outer walls. The bricks corresponding with the lower half of the fuel bed are perforated to permit the circulation of gases between the wall space and the fuel bed. A few of the bricks in the upper zone may also be perforated, and vertical ducts may be provided for conducting air and steam to the upper section of the lining; or the upper zone of the wall space may be entirely separated from the lower and used as an air preheater.

A. B. MANNING.

Manufacture of water-gas. H. NIELSEN and B. LAING (B.P. 299,485, 28.5.27).—A water-gas plant for use with powdered fuel comprises a combustion chamber surmounted by a reaction chamber. The latter contains a suitable arrangement of refractory material to act as a heat accumulator, so that sufficient heat becomes stored therein during the "blow" to carry on the water-gas reaction for any desired length of time when the air supply is replaced by superheated steam.

A. B. MANNING.

Manufacture of carburetted water-gas. P. DVORKOVITZ (B.P. 299,482, 27.4.27).—Water-gas is enriched by the addition of oil-gas which is produced in a separate gasifier maintained at a constant temperature. A. B. MANNING.

Manufacture of gas from [heavy] oils. C. CHILOWSKY (B.P. 293,007, 20.10.27. Fr., 27.10.26).— Processes described in B.P. 255,423, 271,899, and 271,907 (B., 1927, 805, 867) are improved so that fouling of the apparatus and catalyst is prevented.

C. B. MARSON.

Purification of fuel and other gases. KOPPERS Co., Assees. of (A) D. L. JACOBSON and H. A. GOLLMAR, (B) H. A. GOLLMAR (B.P. 280,165 and 286,633, [A] 14.7. and [B] 16.7.27. U.S., [A] 5.11.26, [B] 7.3.27).--(A) Hydrogen sulphide and other acidic constituents are removed from fuel gases by washing with an alkaline solution of a compound of arsenic, tin, or other metal of which the sulphide is soluble in an alkali or alkali sulphide solution. The solution is reactivated by aeration, and, after being freed from the sulphur which is liberated, is recirculated. A solution of sodium arsenite containing the equivalent of about 0.5% As<sub>2</sub>O<sub>3</sub> may be used, but is preferably sulphided and aerated once before use in the plant. (B) The strength of the wash liquid is maintained by the addition, at a uniform rate, of a mixture, preferably in solution, of the alkali and arsenic (or other metal) compound, containing 1-2 atoms of alkali metal to each atom of arsenic. A. B. MANNING.

Apparatus for removing tar from gases evolved in the dry distillation of wood. J. R. RONGIER (F.P. 624,127, 5.11.26).—The apparatus comprises one or more condensers terminating in bell-shaped vessels which dip into annular troughs in which the tar is collected and which are provided internally with horizontal perforated plates containing openings for the removal of uncondensed vapours. A. R. POWELL.

Manufacture of ammonium sulphate from gasworks' liquor or the like. S. ROBSON (B.P. 299,934, 8.11.28).—Crude ammonia liquors are distilled and the resulting distillate is brought to a concentration of 7-12% NH<sub>3</sub> so that when maintained at 50° a current of air and/or inert gases passing through it volatilises somewhat more than twice as much ammonia, by vol., as water vapour. These gases are then brought into contact with sulphur gases (e.g., sulphur trioxide) as described in B.P. 289,950 (B., 1928, 523) to produce ammonium sulphate directly in one operation.

#### R. J. W. LE FÈVRE.

Recovery of calcium ferrocyanide from gaspurification residues. RÜTGERSWERKE A.-G., and L. KAHL (Dan. P. 34,820, 27.8.24. Ger., 2.10.23).—The residues are extracted with aqueous calcium hydroxide solution, and the solution, after filtration, is worked up to Prussian-blue by the usual process. L. A. COLES.

Absorbent filling material for use in acetylene storage containers. M. ROHRSCHNEIDER (B.P. 299,681, 24.4.28).—Finely-divided or shredded leather, which has been freed from acids, substances soluble in acetone, and water, is used as a filler for acetylene containers. The leather may be admixed with a powdered or granular heat-resistant material, *e.g.*, pumice stone or kieselguhr. A. B. MANNING.

Manufacture of porous masses for storing explosive gases. GAS ACCUMULATOR CO. (UNITED KINGDOM), LTD., Assees. of AUTOGEN GASACCUMULATOR A.-G. (B.P. 281,718, 5.12.27. Ger., 4.12.26).—Inorganic porous substances to be used as filling materials for containers for storing explosive gases are impregnated with a finely-divided heat-resisting material (e.g., kieselguhr), which fills up all dangerous voids therein.

#### A. B. MANNING.

Treatment of gases or vapours for the recovery or extraction of hydrocarbons therefrom. P. M. SALERNI (B.P. 299,481, 26.4.27).—A number of long, narrow, spiral passages are arranged in parallel and are attached to an axis which can be rotated. The gas and oil, or other absorbing medium, are passed together along the passages, the centrifugal force maintaining the oil in a layer against the surface of the passages, along which the gases travel in a state of turbulence. A central rotor, rotating in a direction opposite to that of the spiral device, and comprising a series of vanes on a central shaft, impels the gas and oil into the inner openings of the spiral passages. The oil is fed to distributing channels through nozzles extending radially from the shaft. The gases after emerging from the passages are whirled round by suitably placed vanes in order to free them from oil spray. A. B. MANNING.

**Treatment of shale and the like.** W. H. HAMPTON (U.S.P. 1,687,763, 16.10.28. Appl., 11.12.19).—Finelydivided bituminous solids are digested with a heavy mineral oil at 260—370°. The evolved vapours are mixed with steam, subjected to cracking at higher temperatures, and the products fractionally condensed. The residual digestion mixture is treated with a lighter oil and separated from any solids. From the residual oil a heavy fraction is obtained which, mixed with a heavy fraction from the cracking process, is used for the digestion of further material. H. S. GARLICK.

Extracting the oily bases from crude shale oil. D. T. DAY (U.S.P. 1,685,315, 25.9.28. Appl., 11.12.24). —The crude shale oil is treated with a dilute solution of an inorganic acid, and the acid layer containing the bases is separated and treated with alkali. The liberated bases are separated from the aqueous solution, redissolved in dilute acid, re-liberated by means of alkali, extracted by agitation with a hydrocarbon solvent, and finally removed from this solvent by extraction with an inorganic acid. C. O. HARVEY.

**Production of low-boiling hydrocarbons.** W. GESSMAN and E. W. SHALDERS (B.P. 299,820, 30.6.27).— A mixture of unsaturated hydrocarbons, preferably acetylene, with a gas containing carbon monoxide and hydrogen is passed at normal pressure and without heating over a metal catalyst which is irradiated at intervals with ultra-violet light. The catalyst consists of pure, powdered, electrolytic copper mixed with powdered tungsten and powdered pumice, which mixture is treated with a dilute (10%) solution of caustic alkali and then dried thoroughly. G. CLAXTON.

Cracking of [petroleum] oils. J. G. ALTHER, ASST. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,685,476, 25.9.28. Appl., 20.8.20. Renewed 9.6.26).—By using a series of heating coils of continuously decreasing diameter, the velocity of the oil under treatment is increased as the temperature of the heating chamber diminishes. The smallest coil discharges the oil into a vaporising chamber. R. BRIGHTMAN.

Distillation of petroleum oils. J. E. BELL, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,683,151, 4.9.28. Appl., 1.4.24).—The feed is introduced at or near the top of a fractionating tower having a helical passage and flows continuously down the helix, meeting the vapours from the still. The heavier reflux and the unvaporised portion of the feed enter the still from the foot of the tower, the lighter vapours being collected. Effective heat interchange and refluxing of the oil render close fractionation possible. R. BRIGHTMAN.

Decolorising, clarifying, and purifying petroleum oils. J. L. KAUFFMAN and I. A. CLARK (U.S.P. 1,684,035, 11.9.28. Appl., 28.11.24).—The oil is agitated with a finely-divided filtering material (e.g., untreated clay) by means of superheated steam, and, after heating the mixture to a temperature not exceeding the b.p. of the oil and cooling, the filtering material is separated therefrom. C. O. HARVEY.

Treating slack wax. B. L. SOUTHER and W. A. GRUSE, ASSIS. to GULF REFINING CO. (U.S.P. 1,685,008 and 1,685,058, 18.9.28. Appl., [A] 17.2.26, [B] 30.8.26). —(A) The magma of solid slack wax containing oil is finely comminuted until it is fluid and the liquid and solid ingredients are separated mechanically. (B) A liquid having preferential solvent action on the oil is added to the comminuted material to facilitate the mechanical separation. F. G. CLARKE.

Refining of [petroleum] oils. S. TIJMSTRA, ASST. to ROXANA PETROLEUM CORP. (U.S.P. 1,684,159, 11.9.28. Appl., 6.12.26).—Oil which has been treated with sodium plumbite solution is freed from lead by washing with sodium polysulphide solution. C. HOLLINS.

Recovery of vapours [waste petroleum gases]. R. T. OSBORN, ASST. to STANDARD OLL CO. OF CALIFORNIA (U.S.P. 1,685,501, 25.9.28. Appl., 11.7.22).—Gases or vapours which are themselves uncondensible at atmospheric temperature and pressure are passed into vapours from a still operating below cracking temperature and pressure, whereby a mixture is obtained which may be substantially completely condensed. C. O. HARVEY.

Atomisable mobile fuel product. A. W. BURWELL, Assr. to ALOX CHEM. CORP. (U.S.P. 1,684,125, 11.9.26. Appl., 2.3.27).—A fuel oil is oxidised with air at 120— 140° and under 250 lb./in.<sup>2</sup> in presence of 0.1% of manganese stearate; water-soluble products are removed, and the remainder, saponified with sodium hydroxide, is added to 50 pts. of fuel oil and water is driven off, leaving a colloidal solution which is capable of holding in suspension an equal weight of powdered coal (100—200mesh). C. HOLLINS.

[Stream divider for] oil [distillation] apparatus. C. F. TEARS, ASST. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,688,839, 23.10.28. Appl., 18.6.23. Renewed 28.5.28).—The apparatus comprises an enlarged container with an oil inlet and with one ordinary outlet and several relatively small manifolded outlets, all the outlets terminating as standpipes in the container with their upper ends at the same level. L. A. COLES.

Manufacture of alkyl halides, glycols, and alcohols from cracked gases of hydrocarbon oils. L. BLANCHET (F.P. 610,434, 1:5.25).—Additional to B.P. 251,652 (B., 1927, 836) is the following : A cracked gas, after washing with oils to remove pentane and amylene, b.p.  $30-37^{\circ}$ , is cooled to  $5^{\circ}$  with slight pressure, and the residual gas is freed from butane and butylenes by further cooling under higher pressure ; propane and propylene are similarly removed, or the propylene may be absorbed in sulphuric acid. Ethylene (and propylene, if desired) is converted by hypochlorous acid into chlorohydrin, which is either distilled in a vacuum or hydrolysed to glycol; the remaining gas is converted by chlorine into alkyl chlorides and hydrolysed to alcohols. The liquefied fractions, pentane

and amylene, butane and butylene, are treated with hydrogen chloride and chlorine to give amyl chlorides and butyl chlorides, from which the acetates and finally the alcohols are obtained. C. HOLLINS.

Apparatus for the distillation of coal and similar carbonaceous substances. C. H. PARKER (U.S.P. 1,689,152, 23.10.28. Appl., 27.1.28. U.K., 25.9.26).— See B.P. 287,830; B., 1928, 394.

Apparatus for distilling solid combustible carbonaceous material. O. HUBMANN (U.S.P. 1,690,933, 6.11.28. Appl., 8.10.25. Ger., 4.11.24).—See B.P. 242,618; B., 1926, 971.

Formation of [reconstructed] carbonaceous fuel. W. B. RULON (B.P. 299,611, 27.10.27).—See U.S.P. 1,656,364; B., 1928, 219.

Gas purification. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,690,437—8, 6.11.28. Appl., 6.6.24. Renewed 23.1.28).—See B.P. 235,126—7; B., 1925, 662.

Cracking of hydrocarbons. C. P. TOLMAN (B.P. 299,957, 17.9.27).—See U.S.P. 1,643,036; B., 1927, 836.

Apparatus for burning pulverised fuel. GASIFIED FUEL, LTD., H. E. HAZLEHURST, and O. MARGETSON (B.P. 299,930, 11.4.27).

Reducing excess gas pressure in the lower part of freshly charged vertical chambers or retorts. PINTSCH & DR. OTTO GES.M.B.H. (B.P. 288,337, 5.4.28. Ger., 7.4.27).

Burners for gas firing. R. EICKWORTH (B.P. 275,654, 5.8.27. Ger., 6.8.26).

Combined filter and separator (B.P. 299,648). Treatment of gases with liquids (B.P. 299,075). Gas washers (B.P. 297,765).—See I. Hydrogen from gaseous mixtures (B.P. 299,558).—See VII. Preventing explosions (U.S.P. 1,686,051).—See XI.

#### III.—ORGANIC INTERMEDIATES.

Separation of alcohols and phenols from oil mixtures. H. SCHMIDT (Chem.-Ztg., 1928, 52, 898).-The oil is heated at 80-100° under slightly reduced pressure with the theoretical quantity of boric acid to produce the borate B(OR)<sub>3</sub>. When no further water distils over the temperature is raised and the unchanged oil distilled under low pressure. The residual borate is then hydrolysed with sodium hydroxide and the liberated alcohol or phenol is recovered by distillation in steam. The method affords a rapid means of separating primary, secondary, and tertiary alcohols from admixture with one another; sufficient boric acid is first added to combine with the primary alcohol, and the unchanged mixture of secondary and tertiary alcohols is distilled off under low pressure, treated with just sufficient boric acid to combine with the secondary alcohol, and the process repeated. A. R. POWELL.

Indene and styrene. BROWN.-See II.

# PATENTS.

Production of phosphoric esters of aliphatic alcohols. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,044, 12.1.28).—Phosphoryl chloride

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vapours are passed, preferably at 25—40 mm. pressure, into an aliphatic alcohol at 50°, and the mixture is boiled under reflux for about 1 hr. so that hydrogen chloride is removed as formed. Triethyl, tri-*n*-butyl (b.p.  $135^{\circ}/5$  mm.), tri-*iso*butyl (b.p.  $112^{\circ}/4$ —5 mm.), and triamyl (b.p. 143— $144^{\circ}/2.5$  mm.) phosphates are described. C. HOLLINS.

Manufacture of concentrated acetic acid from calcium acetate. L. H. FAUQUE (F.P. 612,595, 10.3.26). —Crude calcium acetate paste (30% water) is saturated in a stoneware-packed tower with hydrogen chloride, warmed until liquid, and transferred to two containers, where dry calcium acetate equivalent to the hydrochloric acid is added, and the mixture is distilled, giving 70—80% acetic acid and a residue of tar and saturated calcium chloride solution. C. HOLLINS.

**Production of pure**  $\alpha$ -hydroxycarboxylic acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,040, 28.12.27).—Cyanohydrins are hydrolysed by a solution of hydrogen chloride in an inert solvent (dioxan, chloroform, etc.) together with sufficient water for the reaction. Preferably the cyanohydrin is prepared from hydrogen cyanide, the aldehyde or ketone, and alkali, in dioxan (or a mixture of dioxan and chloroform), and gaseous hydrogen chloride is then added to the warm solution. The preparation of lactic, mandelic, and  $\alpha$ -hydroxybutyric acids is described. C. HOLLINS.

Purification of lactic acid. J. A. VON WÜLFING (G.P. 446,865, 4.2.25).—Crude concentrated lactic acid is mixed with active carbon, silica gel, etc. to form a loose powder which on extraction with ether gives lactic acid free from coloured and odorous impurities and from higher fatty acids. C. HOLLINS.

Reduction of organic compounds. L. ZECH-MEISTER and P. ROM (G.P. 446,867, 26.11.25).—Organic compounds in methyl-alcoholic solution or suspension are reduced with magnesium and aqueous ammonium chloride without addition of an activator (cf. A., 1926, 720). C. HOLLINS.

Manufacture of primary amines. FABR. DE PROD. CHIM. ORGANIQUE DE LAIRE, Assees. of G. MIGNONAC (B.P. 282,083, 9.12.27. Fr., 9.12.26).—Oximes or nitriles are hydrogenated in presence of nickel with the addition of ammonia, which largely prevents formation of secondary and tertiary amines. Acetonitrile, ammonia, and hydrogen, passed over reduced nickel at 180°, give ethylamine in 90% yield; benzylamine is similarly obtained from benzonitrile, and *iso*propylamine from acetoxime. Phenylacetonitrile in alcoholic ammonia containing reduced nickel gives 85% of  $\beta$ -phenylethylamine when shaken with hydrogen. C. HOLLINS.

Manufacture of thiuram monosulphides. A. J. MAXIMOV, ASST. to NAUGATUCK CHEM. CO. (U.S.P. 1,682,920, 4.9.28. Appl., 18.9.26).—Thiuram monosulphides (e.g., tetramethylthiuram sulphide, m.p. 120—130°) are obtained in good yield by stirring the corresponding disulphide in aqueous suspension with sodium cyanide. R. BRIGHTMAN.

Manufacture of indoles. I. G. FARBENIND. A.-G., Assees. of O. NICODEMUS (G.P. 446,544, 26.3.25).—Suitable alkylated anylamines are passed with oxygen or a nitro-compound over silica gel or highly active carbon, especially the phosphoric acid-carbon of G.P. 407,406, to which may be added oxygen carriers such as compounds of cerium, vanadium, manganese, or copper, at 300— 400°. Diethylaniline yields 1-ethylindole, b.p. 247°, 2-methyl-1-ethylindole, b.p. 265°, and di-indyls; dimethylo-toluidine gives 15% of 1-methylindole, b.p. 240°, and traces of higher indoles. C. HOLLINS.

Preparation of derivatives of isatoic anhydride. BADISCHE ANILIN- & SODA-FABR. (F.P. 603,970, 5.6.25. Ger., 28.6.24. Cf. G.P. 433,147; B., 1927, 215).—The following is additional to the G.P.: The isatoic anhydrides from o-aminobenzoic acids or their N-monoalkyl or -monoaryl derivatives (other than anthranilic acid) yield compounds with cellulose, starch, sugar, etc. which couple with diazo compounds. Isatoic anhydrides made by the action of carbonyl chloride or chloroformic esters on 2-amino-3-naphthoic acid and on 5-nitroanthranilic acid are described. C. HOLLINS.

Preparation of naphthacridine-ms-carboxylic acids and derivatives. I. G. FARBENIND. A.-G., Assees. of K. FRIES (G.P. 446,543, 5.7.26).—A  $\beta$ -naphthacoumarandione is heated with a primary arylamine in acid solution, *e.g.*,



The products are decarboxylated by heating above their m.p. The following are described : pheno- $\beta$ naphthacridine and its 12-carboxylic acid, m.p. 284°, from aniline (cf. Stolle and others, A., 1923, i, 1125); 10-methoxypheno- $\beta$ -naphthacridine, m.p. 157°, and its 12-carboxylic acid, m.p. 286°, from p-anisidine; 10-acetamidopheno- $\beta$ -naphthacridine-12-carboxylic acid, m.p. 292°, from p-aminoacetanilide, which is converted by heat and hydrolysis into 10-aminophenonaphthacridine, m.p. 238°;  $\beta\beta$ -dinaphthacridine-14-carboxylic acid, m.p. 240°, and  $\beta\beta$ -dinaphthacridine, m.p. 216°, from  $\beta$ naphthylamine. C. HOLLINS.

Preparation of anhydrides of organic acids. F. DOERINCKEL and M. SCHLIEMANN, ASSIS. to I. G. FARBENIND. A.-G. (U.S.P. 1,691,647, 13.11.28. Appl., 21.1.27. Ger., 28.1.26).—See B.P. 289,959; B., 1928, 515.

Production of gels. Production of esters from organic halides. Hydrolysis of ester-forming compounds. E. E. AYRES, JUN., and E. H. HAABESTAD, Assrs. to B.A.S. Co. (U.S.P. 1,691,424—6, 13.11.28. Appl., [A, B, C] 21.10.24. Renewed [B] 20.4.27).—See B.P. 241,889; B., 1927, 859.

Production of anthraquinonesulphonic acids. J. THOMAS, ASST. to SCOTTISH DYES, LTD. (U.S.P. 1,691,679, 13.11.28. Appl., 10.1.23. U.K., 18.1.22).— See B.P. 200,851; B., 1923, 922 A.

Treatment of compounds by gaseous reagents (B.P. 274,846).—See I. Reduction of dyes etc. (B.P. 299,899).—See IV. Formic and acetic acids (G.P. 452,138).—See VII. Oxidation of aldoses (B.P. 293,322).—See XVII. Chloral (F.P. 612,396). Phenol ethers (G.P. 446,606).—See XX.

# IV.—DYESTUFFS.

# PATENTS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G., Assees. of A. LÜTTRINGHAUS, P. NAWIASKY, and A. EHR-HARDT (G.P. 451,122, 6.9.22).—Dihydroxydibenzanthrone is treated with the toluenesulphonic ester of glycol, glycerol, ethylene chlorohydrin, or other polyhydric alcohols in which the hydroxyl groups have been partly replaced by halogen or an arylsulphonic acid residue. Blue to green-blue vat dyes result. C. HOLLINS.

Manufacture of vat dyes of the anthracene series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,972, 30.9.27).—The glyoxylidenebisanthrones of B.P. 296,817 (B., 1928, 848) are fused with alkali to give fast bordeaux to violet vat dyes. C. HOLLINS.

Manufacture of stable diazo-salt preparations. I. G. FARBENIND. A.-G. (B.P. 275,245, 27.7.27. Ger., 2.8.26. Cf. B.P. 273,352; B., 1927, 647).—The fission of diazonium hydrogen 1: 5-naphthalenedisulphonates into normal salt and free acid in solution is prevented by admixture of 1: 5-naphthalenedisulphonic acid. Other diazonium sulphonates are similarly stabilised.

C. HOLLINS.

Manufacture of insoluble azo dyes for acetate silk. I. G. FARBENIND. A.-G., Assees. of K. HOLZACH and R. METZGER (G.P. 450,920, 24.1.26).—The dye, *m*-aminobenzaldehyde  $\rightarrow$  2:4-dihydroxyquinoline, dyes acetate silk greenish-yellow, becoming violetbrown on treatment with phenylhydrazine. *m*-Aminobenzaldehyde  $\rightarrow$  phenylmethylpyrazolone gives fast yellow shades. C. HOLLINS.

Manufacture of reduction products of vat dyes and other reducible organic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,899, 4.5.27).—Dry leuco-compounds suitable for conversion into sulphuric esters are prepared by adding the vat dye and 98% formic acid to a solution of sulphur dioxide in pyridine, dimethylaniline, or other organic base. Reduction takes place at 25—70°, according to the vat dye used; benzil, isatin, etc. may also be reduced. The soluble leuco-ester may be prepared directly from the leuco-compound without isolation.

# C. HOLLINS.

Aminodiarylsulphonepyrazolone azo dyes. H. Schweitzer and W. Neelmeier, Assrs. to Grasselli Dyestuff Corp. (U.S.P. 1,685,071, 18.9.28. Appl., 5.1.26. Ger., 8.1.25).—See B.P. 245,765; B., 1926, 734).

# **V.**—FIBRES; TEXTILES; CELLULOSE; PAPER.

Influence of acids and alkalis on the formation of mucilage from cellulose. ANON. (Papier-Fabr., 1928, 26, 722—728).—Mineral acids and alkalis assist mucilage formation in the hollander and reduce considerably the time necessary to reach a given degree of grinding, but the properties (strength etc.) of the papers obtained are so adversely affected that the use of these assistants cannot be considered. More satisfactory results are obtained by the use of organic acids (e.g., acetic acid) or of salts such as sodium nitrite, which accelerate mucilage formation during grinding without causing a diminution

in the strength of the cellulose. Their use results in a reduction of the time of grinding by 30% in the case of the knife hollander and 20% in that of the stone hollander for the same degree of grinding. When no such additions are made the time required to give a degree of grinding of  $90^{\circ}$  in the stone hollander is half that required for the knife hollander. The cost of such accelerating agents increases the cost of production of the cellulose in spite of reductions due to the decreased time necessary, and the cheapest way of obtaining rapid mucilage formation is still by use of the stone grinder. B. P. RIDGE.

Wood. IV. Chemical composition of pulp woods from North Korea. Y. UYEDA and T. MORITA (J. Cellulose Inst., Tokyo, 1928, 4, 233-234).--Analyses are given of "Ezomatsu" (*Picea jezoensis*), "Tōshirabe" (*Abies nephrolepsis*), and "Chōsenmomi" (*Abies holophylla*). These woods have a total cellulose content (by chlorination) of 50-51% on the weight of air-dry wood (10% moisture), 90% of which is  $\alpha$ cellulose. D. J. NORMAN.

Relationship between amphoteric properties and purity of cellulose and its derivatives. K. KANA-MARU (J. Cellulose Inst., Tokyo, 1928, 4, 259—284). —The amphoteric character of cellulose is not a property of the cellulose itself but is due to the presence of impurities, among the more important of which are degraded celluloses such as oxycellulose and hydrocellulose, and traces of alkali loosely combined in the double-layer state with the cellulose. D. J. NORMAN.

Viscose. XVIII. Spinning. VIII. G. KITA, R. TOMIHISA, and J. ONOHARA. IX. G. KITA, J. ONOHARA, and K. MASUI (J. Cellulose Inst., Tokyo, 1928, 4, 235 -237, 237-241).—VIII. Results are given showing that by maturing alkali-cellulose in hydrogen instead of air the viscose prepared therefrom shows a higher viscosity, is more easily spun, and is capable, after suitable ripening, of giving stronger threads (2 g. per denier). Attempts to produce viscose threads of equal strength by shortening the time of maturing in air to 6 hrs. at 25° were not successful.

IX. The time of maturing of the alkali-cellulose for the production of the strongest viscose threads depends on the duration of the steeping operation. Thus, with a steeping time of 3, 7, 14 days (at 7—11° in each case), the optimum maturing time of the alkalicellulose at 25° was, respectively, 3, 12, and 4 hrs. Under these conditions it is possible to prepare viscose threads of as high strength as is shown by viscose prepared from alkali-cellulose matured in an atmosphere of hydrogen. D. J. NORMAN.

Chemistry of the cellulose determination. C. E. PETERSON and M. W. BRAY (Ind. Eng. Chem., 1928, 20, 1210—1213).—A study of the changes in chemical composition of spruce wood during the Cross and Bevan chlorination process has been made, and the following constituents have been determined in the chlorinated residues of 12 samples : cellulose, total pentosans and pentosans in the cellulose, methoxyl content, and lignin. The reaction involved follows the same general course as those taken by the reactions occurring with the ordinary pulping reagents, but higher yields and greater delignification result. In spruce wood, lignin is not completely removed even after a number of protracted chlorinations, and some of the pentosans in the cellulose are removed during the first and each succeeding chlorination, whilst the pentosans not in the cellulose are destroyed very rapidly during the process of cellulose isolation. For maximum reactivity the sample of spruce to be chlorinated should contain a quantity of water at least equal to its own weight evenly distributed through the wood. Since the pentosan content of the cellulose does not increase in the various steps of the chlorination process, the results indicate that cellulose is not decomposed into substances yielding furfuraldehyde during the process of its isolation (cf. A., 1923, ii, 441). E. H. SHARPLES.

Copper and silver numbers as factors for the evaluation of cellulose products. J. RINSE (Ind. Eng. Chem., 1928, 20, 1228-1230).-The determination of copper numbers is reviewed and modifications of existing methods are suggested to improve the accuracy of, and diminish the time required for, the analysis. Braidy's method gives relative values, but it has no high degree of accuracy, and both this and the Fehling's solution procedure fail to give conclusive results. The copper numbers may differ largely from the true reducing figures, as the cellulose itself is attacked by the caustic solution and the time of boiling is too short for complete oxidation of the original impurities. Götze's method for the determination of silver numbers is modified, and the results show that reaction is nearly complete after 24 hrs., and that the values are much nearer the true value than are the copper numbers. The method is simpler and standardisation is less necessary.

E. H. SHARPLES.

Determination of the sulphur of viscose. VITALE and T. MARESCA (Annali Chim. Appl., 1928, 18, 461-465).-The determination is made in a Mahler or Parr bomb, absorbent carbon of known sulphur content being used. One fourth of the total amount of sodium peroxide is mixed with 1 g. of potassium chlorate, 0.2 g. of benzoic acid, and 0.25 g. of the animal charcoal. On to this layer the viscose (0.4 - 0.5 g.) is allowed to fall in separate drops from a Lunge-Rey oleum pipette, the next layer consisting of 0.25 g. of charcoal, and the final one of the remaining three fourths of the sodium peroxide. The subsequent operations are as usual. The barium sulphate may be weighed or determined nephelometrically by means of Parr's photometer, the error in the latter case being  $\pm$  3%. An accuracy of 99% may be attained by oxidation with alkaline hydrogen peroxide solution (cf. Petersen, A., 1903, ii, 690). T. H. POPE.

Effect on rosin sizing of heating the moist paper. E. OMAN (Papier-Fabr., 1928, 26, 705-707). —If rosin-sized paper of high moisture content (e.g., at least 50%) be heated to a certain temperature (e.g., about 75°), the size is adversely affected to a less or greater degree, and may even completely disappear. The extent of this deterioration of the size is dependent on the  $p_{\rm H}$  value of the contained water, being least for the range  $p_{\rm H}$  4-5 and increasing with both increase and decrease of the  $p_{\rm H}$  outside this range. The presence of alum in the pulp water does not prevent this deterioration since the  $p_{\rm H}$  of the latter is the deciding factor. The above facts are put forward to explain why Stöckigt and Klingner obtained unsized paper in some of their sizing experiments. B. P. RIDGE.

# Fat from silk cocoons. JELAKOV.-See XII.

# PATENTS.

Producing fibre and yarn from unretted flax plants. M. WADDELL and H. C. WATSON (B.P. 299,519, 29.7.27).—After drying the flax stalks and separating the gummy constituents, woody parts, and seeds, the fibres are tightly twisted in the form of a rove on an open reel, where they are retted by bacteria and/or chemical action, washed to remove impurities, and finally spun into yarn. F. R. ENNOS.

Extraction of cellulose or paper pulp from fibrous vegetable matter containing the same. (Sir) G. C. GODFREY, E. C. BENTHALL, E. S. TARLTON, H. F. WHEELER (BIRD & Co.), G. L. SCOTT, and E. SPENCER (B.P. 283,910, 5.7.27. India, 20.1.27).-A semi-continuous process working on the reverse-washing principle is described for the alkaline digestion of fibrous vegetable material, e.g., bamboo, grasses, etc., according to which the raw material is subjected to successive treatments at increasing temperature, pressure, and concentration of liquor until digestion is complete. The partly used liquor from each stage of the process is blown over into the next but one digester in operative sequence working at a lower pressure, the total volume of liquor and the concentration of active alkali being at the same time adjusted, if necessary, by the addition of wash liquor from a completed cook. The digesters are preferably arranged in a double-ring system. (Cf. B.P. 291,064-5; B., 1928, 925.) D. J. NORMAN.

Preparing and separating the cellulose material, suitable for paper-manufacturing, from plant raw material with pith fibre. H. KUMAGAWA and K. SHIMOMURA (B.P. 299,740, 30.6.27).—The plant raw material is beaten in a stream of water in a beater or hollander; the water-soluble materials dissolve while the short pithy fibres float on the surface of the stream and the long thin ones, suitable for paper-making, remain suspended and are removed by a suitable device. F. R. ENNOS.

Treatment of cellulose. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,512, 28.7.27).—A preliminary treatment of cellulose in the manufacture of cellulose acetate consists in passing it in sheet form through glacial acetic acid. F. R. ENNOS.

Cellulose composition. O. DROSSBACH and O. JORDAN, ASSTS. to I. G. FARBENIND. A.-G. (U.S.P. 1,677,753, 17.7.28. Appl., 2.7.26. Ger., 24.7.25).— Aliphatic esters of maleic or fumaric acid are solvents and plasticisers for cellulose nitrate, acetate, or ethers. C. HOLLINS.

Manufacture of paper and compositions therefor. H. E. POTTS. From F. W. ATACK (B.P. 299,817, 19.4.27). —Liquid emulsions which dilute immediately with water without breaking are obtained by emulsifying a grease solvent, *e.g.*, a chlorinated hydrocarbon, with water by means of Turkey-red oil, optionally with addition of a small proportion of fatty acid or of ammonia, and stabilising the emulsion with a colloidal silicate, *e.g.*, bentonite. The product is suitable, after dilution, as a detergent in laundering, carpet cleaning, etc., or to remove pitch from the wire and felts in papermaking.

D. J. NORMAN.

Manufacture of paper. G. A. MOURLAQUE (B.P. 299,965, 24.9.27).—The apparatus used consists of a single boiler arrangement having an interior hearth and lixiviator. Vegetable matter is soaked in water, subjected to the action of ozone, heated under pressure with a solution of potash obtained from the cinders of the fuel used in the hearth and of lime prepared by heating limestone therein, and finally bleached, all operations being performed in the lixiviator. After removal of the pulp, the solids in the residuary liquor are recovered by evaporation and calcination in the hearth of the apparatus. F. R. ENNOS.

Strengthening and rendering impermeable paper or similar materials. L. R. MECRE (B.P. 275,617, 29.7.27. Fr., 4.8.26).—After a preliminary immersion in alkali the paper is impregnated first with an aqueous solution containing two or more of the following: salep, gum tragacanth, alga gum, to which alum or resin may be added, and then with a solution of borax, ammonium sulphate or carbonate, or boric acid. Colouring or other substances are added during the process, if desired, and the gum-like materials are finally rendered insoluble in the usual way. F. R. ENNOS.

**Production of stencil sheets for use in duplicating.** A. DE WAELE (B.P. 298,705, 22.7.27).—In the manufacture of stencil sheets by coating Yoshino paper with gelatinising organic colloids, *e.g.*, nitrocellulose, the use of large quantities of organic solvents is avoided by applying the coating in the form of an aqueous emulsion. To prepare the emulsion, collodion cotton, in admixture with softening agents, *e.g.*, castor oil, is treated with sufficient amyl acetate to give a stiff jelly, and this is beaten in a colloid mill with water containing about 10% on the weight of the nitrocellulose of gelatin or agar. Emulsifying agents such as sulphonated sperm oil or ammonium salts of ricinoleic acid may be used, and bentonite may be added to stabilise the emulsion.

D. J. NORMAN.

Treatment of fibrous material [paper]. A. LUTZ, ASST. to R. C. LEE (U.S.P. 1,682,390, 28.8.28. Appl., 29.3.21. Renewed 26.5.26. Ger., 2.7.15).—In the sizing of paper, cardboard, etc. a size containing sodium hydroxide or lactic acid is caused to penetrate the material completely at 40°; a hardening treatment with hydroxymethylformamide follows, and the material is wound in roll form while hardening proceeds.

C. HOLLINS.

**De-inking paper.** A. R. LUKENS, ASST. to RICH-ARDSON CO. (U.S.P. 1,680,949, 14.8.28. Appl.,22.6.25).— The addition of an alkali sulphonate, after or during the treatment with alkali, shortens the time required to wash the pulp free from alkali and ink constituents.

F. G. CLARKE. [Patterned woven] fabric making. A. BODMER, Assr. to HEBERLEIN & Co. A.-G. (U.S.P. 1,688,798—9, 23.10.28. Appl., [A] 27.11.26, [B] 14.7.28. Ger., [A, B] 5.12.25).—See B.P. 262,477; B., 1927, 361. Sulphonated cellulose derivatives and their manufacture. G. KRÄNZLEIN, A. VOSS, and F. BRUNN-TRÄGER, ASSTS. to I. G. FARBENIND. A.-G. (U.S.P. 1,682,382, 28.8.28. Appl., 25.7.27. Ger., 10.9.26).— See B.P. 277,317; B., 1928, 853.

Manufacture of cellulose xanthate. P. MORO (U.S.P. 1,689,958, 30.10.28. Appl., 18.3.26. Fr., 31.3.25).—See F.P. 597,336; B., 1926, 401.

Spinning of artificial silk. H. SCHMIDT and E. HUBERT, ASSTS. to I. G. FARBENIND. A.-G. (U.S.P. 1,689,895, 30.10.28. Appl., 4.4.27. Ger., 11.6.26).—See B.P. 289,942; B., 1928, 520.

Spinning of artificial [silk] filaments. R. SAJITZ and F. POSPIECH, ASSIS. to CHEM. FABR. POTT & CO. (U.S.P. 1,689,894, 30.10.28. Appl., 14.6.26. Ger., 17.6.25).—See B.P. 280,608; B., 1928, 85.

Manufacture of artificial silk, artificial horsehair, etc. [from cellulose derivatives]. H. DREYFUS (U.S.P. 1,688,531, 23.10.28. Appl., 28.6.23. U.K., 11.8.22).—See B.P. 210,108; B., 1924, 251.

[Spinning] apparatus for manufacture of artificial threads or filaments. BRIT. CELANESE, LTD., and J. BOWER (B.P. 299,405, 26.7.27).

Manufacture of artificial textile fibres [possessing wave or curl]. W. SEVER, JUN., and J. B. SPEAKMAN (B.P. 299,291, 3.4.28).

Treatment [manufacture of ribbons or webs from pieces] of fabrics made of or containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 293,858, 15.7.27. U.S., 15.6.28).

Machine for making transparent paper or sheet cellulose from aqueous cellulose solutions. C. RUZICKA (B.P. 300,477, 15.5.28).

Treatment of gut etc. (B.P. 299,535).-See XIV.

# VI.—BLEACHING; DYEING; PRINTING; FINISHING. PATENTS.

Dyeing of fibrous materials. J. NÜSSLEIN, ASST. to I. G. FARBENIND. A.-G. (U.S.P. 1,684,881, 18.9.28. Appl., 23.11.26. Ger., 7.12.25).—Aromatic amines for the production on the fibre of oxidation dyes are applied as an aqueous dispersion, *e.g.*, with Turkey-red oil, or sodium disopropylnaphthalenesulphonate or *N*-dibenzylanilinesulphonate, or with organic solvents. R. BRIGHTMAN.

Production of fast tints on vegetable fibres [dyeing cotton with azo dyes containing chromium]. Soc. CHEM. IND. IN BASLE (B.P. 271,897, 27.5.27. Switz., 27.5.26).—Dyeings fast to washing are obtained on cotton directly by use of the chromium compounds of dyes made by diazotising an o-aminophenol (other than an aminosalicylic acid) or an anthranilic acid and coupling with the usual components. The chromium compound of the dye, 4-chloro-5-nitro-2-aminophenol  $\rightarrow$  J-acid, gives bluish-green shades; 41 other couplings are mentioned. C. HOLLINS.

**Production of effect threads.** I. G. FARBENIND. A.-G. (F.P. 623,704, 26.10.26. Ger., 13. and 25.11.25).— Cotton effect threads in woollen material are left undyed in an acid dye bath to which alkylated, arylated, aralkylated, or *cycloalkylated aromatic* (or partly hydrogenated aromatic) sulphonic acids, particularly benzylnaphthalenesulphonic acids, have been added. C. HOLLINS.

Dyeing of textile goods [with vat dyes]. I. G. FARBENIND. A.-G. (B.P. 272,941, 16.6.27. Ger., 18.6.26. Addn. to B.P. 270,307).—In the process of the prior patent a boiling hyposulphite vat liquor to which formaldehyde or acctone has been added is employed. More level dyeing results, and the spent dye liquor may be brought up to strength with fresh dye and used again. C. HOLLINS.

Dyeing by means of leuco-ester compounds of dyes of the indigo series and other vat dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,743, 26.7.27).—The streakiness resulting from drying the dyed goods on a dipping frame before development is avoided if dextrin, glue, starch, etc. is added to the dye bath. C. HOLLINS.

Dyeing of cellulose esters. I. G. FARBENIND. A.-G., Assees. of F. WEBER (G.P. 446,220, 13.8.25).—An aminonaphthoic acid is coupled on the fibre or is diazotised and developed on the fibre; e.g., 2:3-aminonaphthoic acid is diazotised and developed with  $\beta$ -naphthol (strawberry-red), H-acid (bluish-red), phenylmethylpyrazolone (yellowish-orange), or 2:3-hydroxynaphthoic acid (bluish-pink); or is coupled on the fibre with diazotised *p*-nitroaniline (red); or 2:6-aminonaphthoic acid is diazotised and developed with 2:3-hydroxynaphthoic  $\beta$ -naphthylamide (yellowreddish-brown) or 2:3-hydroxynaphthoic acid (seal-red). C. HOLLINS.

Dyeing of cellulose esters. I. G. FARBENIND. A.-G., Assees. of K. H. MEYER and H. HOPFF (G.P. 446,539, 11.6.24. Addn. to G.P. 428,176; B., 1926, 785).—The process of the prior patent is used with coloured, insoluble, weakly basic, organic compounds generally, e.g., 3-aminobenzanthrone (red), 6-anilino-N-methylpyridonanthrone (bluish-red), aminoacridone (fluorescent yellow), diaminophenanthraquinone (violet), 5:5'-diaminoindigo (blue), 1-anthraquinonylhydroxylamine (brownish-red), 1:5-dihyroxylaminoanthraquinone (redbrown), coeramidonine (yellow). C. HOLLINS.

Dyeing etc. of materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD. G. H. ELLIS, and H. C. OLPIN (B.P. 299,349, 20.7.27).— Acetate silk etc. is dyed with acetoacetyl (benzoylacetyl etc.) derivatives of colouring matters which contain amino-groups. Examples are : the acetoacetyl derivatives of aminoazobenzene (greenish-yellow), 2 : 4-dinitro-4'-aminodiphenylamine (golden-yellow), 1 : 4-diamino-2methylanthraquinone (orange). C. HOLLINS.

Dyeing of cellulose acetate. I. G. FARBENIND, A.-G., Assees. of F. WEBER (G.P. 452,213, 15.1.26).—Acetate silk is dyed with azo dyes formed by coupling a nonsulphonated diazo compound with  $\beta$ -naphthylamine- or  $\beta$ -naphthol-8-sulphonic acid; *e.g.*, 2:5-dimethoxyaniline  $\rightarrow$  crocein acid (yellow); 5-nitro-o-anisidine  $\rightarrow$  phenyl- $\beta$ -naphthylamine-8-sulphonic acid (seal-red); o-chloroaniline  $\rightarrow$  phenyl acid (reddish-yellow); 2:5dimethoxyaniline  $\rightarrow$  crocein acid (pink).

C. HOLLINS.

Dyeing, printing, or stencilling of materials made with or containing cellulose derivatives [esters and ethers]. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. W. KIRK (B.P. 299,343, 19.7.27).—Unsulphonated azo dyes having a naphthylenediamine as end-component dye acetate silk, immunised cotton, etc. Examples are: *p*-nitroaniline  $\rightarrow$  1:5naphthylenediamine (orange-red), 5-nitro-*o*-anisidine  $\rightarrow$ 5-acetamido- $\alpha$ -naphthylamine (red). Certain couplings are disclaimed. C. HOLLINS.

Printing with vat dyes. I. G. FARBENIND. A.-G. (B.P. 279,864, 26.10.27. Ger., 26.10.26).—A cellulose ether or ester, particularly methylcellulose, is added as a thickener to vat-dye printing pastes. The printing material may be stored for a considerable time before steaming. Concentrated pastes suitable for printing from a photographically engraved roller are obtainable. C. HOLLINS.

Dyeing of wool and/or silk. C. SHAW (U.S.P. 1,691,500, 13.11.28. Appl., 7.11.27. U.K., 26.11.26).— See B.P. 277,833; B., 1927, 874.

Dyeing of cellulose esters and ethers. A. GOESCHKE, Assr. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,691,517, 13.11.28. Appl., 4.11.26. Switz., 14.11.25).—See B.P. 261,423; B., 1928, 121.

Colour estimating (B.P. 299,194).—See I. Azo dyes for acetate silk (G.P. 450,920).—See IV. Colour kinematography (B.P. 283,560 and 289,827).—See XXI.

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

Determination of selenium in sulphuric acid. Z. REICHINSTEIN (Trans. Inst. Pure Chem. Reagents [Russ.], 1927, 6, 27—30; Chem. Zentr., 1928, I, 230).— Selenium is determined colorimetrically by means of 4%aqueous codeine phosphate solution. The reaction is more sensitive with acid above about 75% concentration than with dilute acid. Iron gives a similar coloration to selenium, and must therefore be adjusted to the same concentration in the acid under test and in the standard solutions. Arsenic affects the coloration only when present in high concentration. The test is sensitive with 0.0015% Fe and 0.00005% SeO<sub>2</sub>. L. A. COLES.

Leucite industry from the chemico-physical point of view. N. PARRAVANO (Giorn. Chim. Ind. Appl., 1928, 10, 501-511).-The importance of the Blanc process for the utilisation of Italian leucite (cf. B., 1925, 240) is shown by the fact that the laval masses of six extinct volcanoes of Central and Southern Italy contain 105,340 million tons of material, representing 8786 million tons of potash. The leucite, separated electromagnetically from the basaltic mass, is treated with hydrochloric or nitric acid, the products being silica and a solution containing aluminium and potassium chlorides or nitrates. Results are now given of determinations of the variation in solubility of these different salts with the temperature and acid concentration, and diagrams are shown for the systems potassium chloride (nitrate)-aluminium chloride (nitrate)-hydrochloric (nitric) acid-water at various temperatures. The heating curves of AlCl<sub>3</sub>,6H<sub>2</sub>O and Al(NO<sub>3</sub>)<sub>3</sub>,6H<sub>2</sub>O are also

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given. Investigation of the X-ray spectra shows that the alumina obtained by heating these salts differs at low temperatures from that derived from bauxite by the Bayer process, but becomes identical with the latter when heated; indications that the dehydration of hydrated alumina occurs by stages are obtained.

T. H. POPE.

Reduction of tricalcium phosphate by carbon. K. D. JACOB and D. S. REYNOLDS (Ind. Eng. Chem., 1928, 20, 1204-1210).-In laboratory tube experiments on the reduction of tricalcium phosphate free from silica by means of pure carbon, conducted in an atmosphere of nitrogen, volatilisation of phosphorus commenced at 1150° and was completed in 1 hr. at 1325°, or in 10 min. at 1500°. The rate of reaction was reduced considerably if the depth of the mixture exceeded 1 cm. Tricalcium phosphate has undoubtedly definite dissociation pressures at high temperatures, and probably the rapid reduction of liberated phosphorus pentoxide by carbon removes the vapour phase and accelerates further dissociation, which follows the course of a unimolecular reaction in the range 1250-1400°. Less than 0.2% of the total phosphorus was converted into calcium phosphide at temperatures up to 1500°. C. A. KING.

Magnesia-graphite reactions at high temperatures. F. T. CHESNUT (Chem. Met. Eng., 1928, 35, 687).—In a specially designed furnace a sintered magnesia crucible fitted internally with an Acheson graphite sleeve was subjected to a 100-kw. load by induction, observations being made by a sight hole and by means of test rods of refractory oxides. A temperature estimated at about 3000° was reached, and under these conditions much gas is evolved on account of the dissociation of the magnesia. On cooling, the magnesia had not visibly vitrified. R. H. GRIFFITH.

Reduction of thoria by hydrogen. B. SEGALL (Chem.-Ztg., 1928, 52, 897).—A review of recent researches on the behaviour of thoria in an atmosphere of hydrogen at high temperatures, both alone and in contact with tungsten. A. R. POWELL.

Thermodynamic properties of oxygen and nitrogen. R. W. MILLAR and J. D. SULLIVAN (U.S. Bur. Mines, Tech. Paper No. 424, 1928, 20 pp.).—By using available experimental data and graphical methods of integration and extrapolation, values of the volume, heat content, and entropy, per g.-mol., are calculated for oxygen and nitrogen over temperature and pressure ranges of 80—300° Abs. and 1—60 atm., respectively. The results are expressed in tables and also as Mollier charts. F. J. DENT.

Corrosive action of sulphur monochloride. E. H. HARVEY (Chem. Met. Eng., 1928, 35, 684).—Pure metals in lump, stick, or foil form were covered with excess of sulphur monochloride and stored in bottles, closed with Bunsen valves, in diffused light at room temperature for one year; the metal was then washed with carbon tetrachloride, dried, and weighed. Chromium and cobalt were entirely unaffected, whilst the change with nickel, cadmium, lead, and silver was very small, but aluminium, arsenic, antimony, copper, and manganese were badly attacked. It is suggested that the use of drums of alloy

or plated steel would eliminate existing difficulties in the transport of this substance. R. H. GRIFFITH.

# PATENTS.

Production of formic acid, acetic acid, and hydrofluoric acid. H. FISCHER (G.P. 452,138, 4.10.21). —The acids are generated in drum-shaped vessels provided with stirring apparatus which by its own rotation or by that of the drum lifts up cylindrical rollers.

L. A. COLES.

Production of hydrofluoric acid. F. SCHUCH (F.P. 628,062, 1.12.26).—Fluorides are heated with mineral acids in two or more vessels attached to one another, the second being wider than the first. L. A. COLES.

Storage and transportation of acid mixtures in steel drums. J. H. GRAVELL (U.S.P. 1,678,775, 31.7.28. Appl., 11.12.25).—An element between mercury and iron in the electrochemical series, *e.g.*, tin or arsenic, is added to the acid to prevent corrosion. Compounds of the elements, *e.g.*, arsenic trioxide or sodium arsenite, may be used. Known substances having an inhibiting action such as organic bases may also be added. F. G. CLARKE.

Treatment of caustic solutions for the production of solutions and of solid caustic soda of a high degree of purity. D. A. PRITCHARD, and UNITED ALKALI CO., LTD. (B.P. 299,995, 21.10.27).—Evaporated caustic soda liquors or solutions are treated with a salt or salts of sulphuric acid or with sulphuric acid itself (these reagents are preferably added in the anhydrous form), whereby the major portion of the sodium chloride originally present is precipitated. Liquors containing as little as 0.2 pt. of sodium chloride per 100 pts. of Na<sub>2</sub>O in solution may be obtained, and are capable of producing solid caustic soda of a high degree of purity on evaporation. R. J. W. LE Fèvre.

Production of mixed crystals containing potassium and ammonium. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,984, 13.10.27).—The solid deposit is separated from an aqueous solution saturated with the chlorides and/or the nitrates of potassium and ammonium (and sodium salts, if desired) which is in equilibrium with at least three solid phases of these salts. To the separated solution solid potassium and/or ammonium nitrate or the corresponding chlorides or both kinds of salts and sodium salts, or other salts, if desired, are added, and the resulting deposit of mixed crystals is removed. Soluble phosphates may also be added to the saturated solution. R. J. W. LE FÈVRE.

Production of ammonium phosphates from mono- or di-calcium phosphate. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,796, 29.6.27).— Mono- or di-calcium phosphate or mixtures of the two may be converted into ammonium phosphate by treatment, while stirring, with ammonia in the presence of water. A mixture of calcium phosphate and ammonium monohydrogen phosphate is thereby formed. The reaction is facilitated by raising the temperature and/or pressure. The resulting tricalcium phosphate is recovered, converted by treatment with acids into monoCL. VII.-ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

and/or di-calcium phosphate, and the product returned to the first stage of the process. R. J. W. LE FÈVRE.

Making oxy-compounds of phosphorus [and fertiliser]. C. G. MINER, Assr. to PHOSPHORUS HYDRO-GEN Co. (U.S.P. 1,686,873, 9.10.28. Appl., 27.10.23).— A mixture of phosphatic and potash minerals is reduced, and the liberated potassium and phosphorus are condensed as potassium phosphide. The latter is treated with water or steam, preferably in the presence of nitrogen, whereby a mixture of nitrogen and hydrogen and a solution of potassium hydrogen phosphate are obtained. A complete fertiliser is produced by treating the phosphate solution with ammonia, obtained from the nitrogen-hydrogen mixture by the Haber or Claude process. F. G. CLARKE.

Production of commercial borax from rasorite by a wet method. T. M. CRAMER, Assr. to PACIFIC COAST BORAX CO. (U.S.P. 1,685,214, 25.9.28. Appl., 22.1.27).—Borax is produced from a natural borate, which contains 2—5 mols. of water and is difficultly soluble, by heating it under pressure with water, preferably by means of live steam in a closed vessel.

F. G. CLARKE.

Preparation of silica from silicate solutions. P. SPENCE & SONS, LTD., T. J. I. CRAIG, and A. KIRKHAM (B.P. 299,483, 28.4.27).-Aqueous solutions of alkali silicates, containing about 10% SiO<sub>2</sub> and, if necessary, sufficient alkali carbonate to ensure a molecular ratio after treatment of not less than 2 pts. of alkali carbonate to 1 pt. of silica originally present, are treated gradually (about  $\frac{1}{2}$  hr.) at 95-70° or lower with a warm (40-50°) solution of alkali hydrogen carbonate and/or carbon dioxide sufficient to convert all the alkali present into the normal carbonate. Precipitation of the silica in a flocculent, easily filterable, opaque condition is completed by cooling to about 25°. To remove the alkali still remaining after filtering and washing, the product is given further washings with dilute acid and water. The silica, after being dried and sifted, has a bulk density of 0.1. Alkali carbonate is recovered from the motherliquor and re-used as desired. P. E. L. FARINA.

Production of pure, finely-divided kieselguhr. K. PERL & DR. F. STEINITZER CHEM.-TECH. INST. (G.P. 446,411, 19.9.26).—Kieselguhr is triturated with strong acids before or after treatment with material for destroying organic substances, after which it is calcined, treated with oxidising agents, and washed.

L. A. COLES. Production of lead chloride and nitrates from material containing lead and small quantities of gypsum. SILESIA VER. CHEM. FABR. (G.P. 450,737, 25.11.25. Addn. to G.P. 449,739; B., 1928, 815).—The gypsum is decomposed by treatment with hot lead chloride solution and the lead sulphate formed is washed free from calcium chloride. L. A. COLES.

Utilisation of waste iron pickle. A. J. EVANS (B.P. 299,919, 8.8.27).—The precipitation of hydrated peroxide of iron from iron liquors is facilitated by addition of small quantities of protective colloids (liquid glue, size, etc.) to the mixtures of these liquors with alkaline-earth carbonates. Agitation and aeration then cause a

permanent froth to be formed whereby the mixture is brought into intimate contact with atmospheric oxygen as rapidly as possible. R. J. W. LE FÈVRE.

Production of aluminium sulphate from crystalline aluminium chloride. I. G. FARBENIND. A.-G., Assees. of E. C. MARBURG and F. ROSSTEUTSCHER (G.P. 452,457, 13.1.24).—Crystalline aluminium chloride is heated at not above 200° to expel the greater part of the hydrogen chloride, steam being applied if necessary, and residual acid is removed by treating the residue with sodium carbonate or bicarbonate, or with ammonia or ammonium carbonate. The residual alumina is dissolved in sulphuric acid of suitable concentration.

L. A. Coles.

Manufacture of aluminium sulphate. L. G. PATROUILLEAU, and ALUMINE & DÉRIVÉS (F.P. 627,334, 10.1.27).—Bauxite is treated with sulphuric acid, and the resulting solution is electrolysed hot between iron electrodes and subsequently diluted to precipitate basic aluminium sulphate. A. R. POWELL.

[Colloida1] chromic hydroxide. J. HÄUSLER and B. KOHNSTEIN (Austr.P. 106,829, 28.1.21).—An acid chromic acid solution and an alkaline solution of sucrose are separately atomised into a mixing chamber whereby a fog is obtained from which colloidal chromic hydroxide may be recovered. A. R. POWELL.

Anhydrous magnesium chloride. I. G. FARBENIND. A.-G. (F.P. 624,736, 17.11.26. Ger., 7.12.25).—Crystallised magnesium chloride is dehydrated in a current of a dry gas containing hydrogen chloride, the temperature being regulated according to the hydrogen chloride content of the gas mixture. A. R. POWELL.

Preparation of tungsten oxide gel. W. A. PATRICK and E. H. BARCLAY, ASSTS. to SILICA GEL CORP. (U.S.P. 1,683,695, 11.9.28. Appl., 29.3.27).—Nitric acid ( $6\cdot38N$ ) is added to an 8% solution of sodium tungstate, Na<sub>2</sub>WO<sub>4</sub>,2H<sub>2</sub>O, the addition being stopped at each appearance of a precipitate until the latter redissolves on stirring, the final acidity being  $0\cdot1-0\cdot5N$ . The sol sets on keeping to a hydrogel, or may be mixed with a silicie acid sol to produce a mixed hydrogel.

C. HOLLINS.

Centrifugal apparatus for the simultaneous neutralisation and drying of salts containing acids. W. VOGEL (G.P. 452,486, 1.7.26).—Gases containing ammonia are blown through the material by a propeller or fan attached to the shaft driving the drum. L. A. COLES.

Production of hydrogen, or hydrogen-nitrogen mixtures, or hydrogen-carbon monoxide mixtures. M. CASALE-SACCHI (B.P. 299,492, 25.7.27).-Carbon monoxide as such or in gases containing it has the decomposed completely into carbon dioxide and hydrogen by bubbling through water containing catalysts such as reduced copper, nickel, iron, thallium, platinum, palladium, or metallic oxides or salts of weak inorganic acids, or mixtures thereof, at such a temperature (180-360°) and pressure (20-150 atm.) that the water is kept liquid ; thence the gases pass through a heat exchanger to the apparatus for removal of carbon dioxide. Hydrogen or a hydrogen-nitrogen mixture is thus obtained suitable for hydrogenation, the synthesis of ammonia, etc. By modifying the process incomplete catalysis is possible with the production of hydrogen-carbon monoxide mixtures suitable for organic syntheses (methyl alcohol etc.). The compressed state of the gases is an advantage in the process, and also favours the carbon dioxide elimination. P. E. L. FARINA.

Recovery of hydrogen from gaseous mixtures rich in same. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,558, 29.9.27).-The impure hydrogen, containing about 8% of methane (and homologues) and small quantities of potential catalyst poisons, is washed at -150° and at a pressure up to 400 atm. with a suitable organic solvent, of which liquid methane, obtained in the process, is preferred. Final traces of the impurities are removed by adsorption in active charcoal, silica, alumina gel, natural substances like chabasite, brown coal, etc., either separately or together. The hydrogen remaining is exceptionally pure and particularly suitable for the synthesis of ammonia. The liquid methane and cold hydrogen may be used to precool the impure gas and the adsorbents. When methane has been used in the preliminary washing the adsorbent is sufficiently regenerated by raising the temperature to -80°.

P. E. L. FARINA.

Production of phosphorus. I. G. FARBENIND, A.-G. (F.P. 628,179, 28.1.27. Ger., 2.2.26).—Phosphates are heated with reducing agents and alumina or material containing it. L. A. Coles.

Manufacture of phosphorus and aluminium. E. COLLET (N.P. 41,453, 17.9.24).—A mixture of 10 pts. of bauxite, 15 pts. of phosphate rock, 3 pts. of coke, and 1 pt. of sodium carbonate is heated in the electric furnace whereby phosphorus distils and aluminium is obtained. A. R. POWELL.

[Mould for] production of roll sulphur. RHEN-ANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assees. of W. VON DIETERICH, F. ADEN, and J. LOOSER (G.P. 451,796, 4.2.26).—The moulds are constructed of metal, preferably of aluminium or its alloys. L. A. COLES.

Control of rate of oxidation [manufacture of sodium monoxide]. H. R. CARVETH, ASST. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,685,520, 25.9.28. Appl., 29.1.26. Ger., 6.6.25).—See B.P. 264,724; B., 1927, 218.

Treatment of gases bearing gaseous synthetic ammonia. W. H. KNISKERN, ASST. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,690,585, 6.11.28. Appl., 16.8.23).—See B.P. 220,655; B., 1925, 283.

Manufacture of alkali hypochlorites. A. Oppé (U.S.P. 1,689,748, 30.10.28. Appl., 26.6.26. Ger., 17.4.26).—See B.P. 258,821; B., 1926, 1013.

Treating earthy minerals. R. W. Hyde, Assr. to DWIGHT & LLOYD METALLURGICAL CO. (U.S.P. 1,688,422, 23.10.28. Appl., 4.4.25).—See B.P. 275,463; B., 1927, 777.

Preparation of sulphur of a high degree of dispersion. H. SCHRADER and H. SCHOELLER (U.S.P. 1,688,357, 23.10.28. Appl., 14.8.24. Ger., 16.8.23).— See B.P. 220,597; B., 1925, 99.

Treatment of gases with liquids (B.P. 299,075). Treatment of compounds or gases by a gaseous reagent (B.P. 274,846).—See I. Ammonium sulphate from gas-works' liquor (B.P. 299,934). Calcium ferrocyanide from gas residues (Dan.P. 34,820).—See II. Titanium oxide (B.P. 299,835).— See XIII.

# VIII.—GLASS; CERAMICS.

Effect of temperature change on the colour of red and yellow Lovibond [tintometer] glasses. B. D. JUDD (U.S. Bur. Stand. Res. Paper No. 31, 1928, 8 pp.).—From determinations of the spectral transmission at 15° and 40° of the Lovibond glasses 35Y and  $7\cdot 2R$ , separately and in combination, the effect of an increase of temperature of 25° on glasses within the range 35Y to  $35Y + 7\cdot 2R$  was found to be practically equivalent to the addition of only  $0\cdot 2$ Lovibond red unit. Two samples of cottonseed oil gave even smaller colour changes over the same temperature range, and, if these were typical, temperature variations of less than 15° need not be considered in colour grading. A. COUSEN.

Viscosity of glass. S. ENGLISH (J. Soc. Glass Tech., 1928, 12, 106—113 r).—A critical examination of the available methods of determining viscosity. The viscosity range of glass for working was taken as  $10^{2\cdot5}$ — $10^{3\cdot5}$  e.g.s. units, and for the annealing point at  $10^{13}$ .

A. COUSEN.

Solidification of amorphous matter [glass]. S. ENGLISH (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 3—6).—A brief synopsis of earlier work of the author on the viscosity of soda and soda-lime glasses. The experimental methods adopted are described and some of the results obtained are shown in a series of graphs. A. R. POWELL.

Annealing of glass from the point of view of factory operation. E. A. COAD-PRYOR (J. Soc. Glass Tech., 1928, 12, 204—213).—An examination of the principles underlying efficient lehr design.

A. COUSEN.

Relationship between physical properties of glasses and their suitability for manipulation by machine. F. W. ADAMS (J. Soc. Glass Tech., 1928, 12, 114—119 P).—Limiting compositions of colourless and green glass for working on flow and suction-feed machines are given, and conditions conducive to the production of homogeneous glass. The physical properties which the glass should possess to work successfully in the machine are also briefly treated.

A. COUSEN.

Effect of oxidising and reducing agents on selenium glass colours. A. SILVERMAN (J. Amer. Ceram. Soc., 1928, 11, 81).—Selenium produces a ruby colour in zinc-potash glasses in the presence of cadmium sulphide and a reducing agent; an amber shade in lead glasses with an oxidising agent; a pink colour in lime glasses with an oxidising agent without cadmium sulphide; and an orange shade when cadmium sulphide and a reducing agent are present. F. SALT.

Loss of silica during glass melting. S. R. SCHOLES (J. Amer. Ceram. Soc., 1928, 11, 79).—Silica is the component which suffers greatest loss during the melting of glass; the finer are the particles of silica introduced, the greater is the loss. When round-grained and dustless sand is used, the silica in the glass is lower by 0.64%(average) than the value calculated from the batch; when sharp-grained, dusty sand is used the loss is greater, viz., 1.13%. This difference is suggested as the reason why sharp-grained sand is the easier to melt. F. SALT.

Acid-resistant enamels. A. MALINOVSKY (J. Amer. Ceram. Soc., 1928, 11, 110-113).—Three enamel batches formulated by Kerstan (Ceram. Ind., 1927) were made up and tested for resistance to hydrochloric acid in various strengths. All three were badly attacked. F. SALT.

Vacuum slip process of removing air from clay bodies. G. W. LAPP (J. Amer. Ceram. Soc., 1928, 11, 61-67).—The body slip is passed to the top of a vacuum treating tank before going to the filter press. The slip flows in a thin layer down the sides of the vacuum tank, and is thus exposed to the full action of the vacuum. The benefits of the vacuum treatment to the manufacturing process and to the finished ware are pointed out. F. SALT.

Determination of workability of plastic clays. S. J. McDowell (J. Amer. Ceram. Soc., 1928, 11, 99— 102).—Apparatus is described which measures the amount of deformation of plastic clay specimens under a given load (8000 g.) applied vertically. It is simple and robust in construction, and has given good results in practice. F. SALT.

Warpage study of terra-cotta clays. R. M. MURPHY (J. Amer. Ceram. Soc., 1928, 11, 103—104).— Several types of terra-cotta clays, in bars measuring 9 in. by 1 in. by 1 in., were tested for warpage during drying and firing. Clays of low plasticity reduce the drying shrinkage and warpage; moderate amounts of a sandy clay reduce the firing warpage and shrinkage. Plastic clays have high dry shrinkage and warpage, but they add strength to the body. F. SALT.

Equations and tables for shrinkage, expansion, and design calculations [in ceramics]. A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1928, 11, 715—723).— Formulæ and data eliminating the use of certain incorrect equations are given. To facilitate calculations fourfigure tables have been computed. A. T. GREEN.

Use of fused silica as raw material in the manufacture of porcelain. A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1928, 11, 82-89).—The effect of substituting fused silica for ground quartz sand on the firing behaviour and thermal expansion of a porcelain body was studied. Two bodies were examined, one containing 20% of flint sand, the other 20% of fused quartz. In a reducing atmosphere the fused silica body matured 3-4 cones lower than the other, forming a very dense product. The thermal expansion of the fused silica body when fired under reducing conditions to cone 8 was much lower and more regular than that of the normal body fired to cone 11 under oxidising conditions. At cone 11 small crystals resembling mullite appeared in the fused silica body. The name "fusilain" is suggested for this body. F. SALT.

Measurement of thermal expansion of pottery bodies. T. YOSHIOKA (J. Japan. Ceram. Assoc., 1927, 35, 568—572).—A new dilatometer is described comprising a silica tube, a mechanism to rotate a mirror, a lamp, and a scale. S. KONDO.

Analysis of bauxite and of refractories of high alumina content. G. E. F. LUNDELL and J. I. HOFFMAN (U.S. Bur. Stand. Res. Paper No. 5, 1928, 13 pp., and Bur. Stand. J. Res., 1928, 1, 91-104) .-Although the method follows that usually adopted in the analysis of silicates, precautions are necessary when the material has a high content of alumina. Raw bauxite should be dried for 2 hrs. at 140°, and is then hygroscopic. It may be decomposed almost entirely by a mixture of hydrofluoric, sulphuric, and nitric acids, though fusion is necessary with calcined samples. Silica is determined after two evaporations with either hydrochloric or sulphuric acid followed by heating the precipitate at not below 1200°, the usual correction for silica in the ammonia precipitate being applied. Alumina is determined by difference by subsequent treatment of the mixed oxides of aluminium, iron, titanium, zirconium, phosphorus, vanadium, and chromium, precipitated by means of ammonia. The solution of these oxides is treated with 10% caustic soda solution containing a little sodium peroxide, precipitating the oxides of iron, titanium, and zirconium, and chromium is determined colorimetrically in the filtrate, which is then treated with ammonia. After filtering, the ammonia precipitate is dissolved in 10% nitric acid together with a few drops of hydrogen peroxide, and the tint of the solution compared with that of a standard vanadium solution under similar conditions. After oxidation with potassium permanganate phosphorus is precipitated with ammonium molybdate and titrated or weighed. Separation of iron is effected by treating the alkaline solution of iron, titanium, and zirconium oxides with hydrogen sulphide, platinum having been removed first in faintly acid solution. The filtrate containing titanium and zirconium, acidified to give a 10% sulphuric acid solution and cooled in ice water, is precipitated with a cold 6% aqueous solution of cupferron. The curdy precipitate, collected on a paperpulp filter, is heated at 1200°, weighed as zirconia and titania, and fused with potassium pyrosulphate, zirconium then being precipitated as the phosphate. A new and larger sample (1-5 g.) is generally required for the determination of lime and magnesia. The silicafree solution is nearly neutralised with caustic soda and poured into a 10% solution of caustic soda containing carbonate, the presence of iron, either natural or introduced, aiding agglomeration of calcium and magnesium. The impure precipitate is dissolved, precipitated with ammonia, and lime and magnesia are separated from the filtrate by ordinary methods. Alkalis can be determined satisfactorily by Smith's method. A more rapid method suitable for routine work and depending on the precipitation of aliquot portions of the solution by different reagents is also C. A. KING. given.

#### PATENTS.

Strengthened glass. P. H. HEAD (B.P. 299,900, 30.7.27 and 19.4.28).—Diacetone alcohol, alone or mixed with industrial alcohol, tetrachloroethane, *cyclohexanone*, etc., is used for effecting adhesion between celluloid and glass sheets. L. A. COLES.

Shaping of silica. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of H. P. HOLLNAGEL (B.P. 292,624, 23.6.28. U.S., 23.6.27).—Hollow articles are formed by causing silica rendered plastic by heating at about 1400° to flow over the surface of a rotating convex mould by the application of local pressure. L. A. COLES.

# IX.—BUILDING MATERIALS.

Cause and prevention of kiln and dry-house scum and of efflorescence on face-brick walls. L. A. PALMER (U.S. Bur. Stand. Tech. Paper No. 370, 1928, 22, 51 pp.).-A distinction is made between efflorescence, kiln scumming, and dry-house or "fixed" scumming. Two sets of tests on various makes of facebricks have been made, and several types of lime and cement mortars are described. In the first series the bricks alone are stood for 6 months in trays containing about 1 in. of water; in the second, three bricks are built up into small panels with the various mortars and subjected to the same treatment. The soluble salts which cause the efflorescence are for the most part in the form of sulphates, and the results of the tests indicate that both the brick and mortar materials contribute to the efflorescence on a face-brick wall. In an attempt to reduce the efflorescent effect by incorporating a water-repellent substance with the mortar materials, it is shown that the addition of 2% of ammonium or calcium stearate is very effective. The formation of sulphates during drying and burning is next considered. If the waste heat from the kilns is utilised in the dryer, small amounts of sulphur dioxide which are present may be oxidised and adsorbed on the surface of the bricks. Quantitative tests made in a small laboratory oven show that the formation of soluble sulphates in the dryer is minimised by completing the drying process as rapidly as possible. Conditions which make this possible are high temperature, low humidity (and consequently good draft), and a minimum amount of mixing water in the clay. The formation of sulphates during burning was found to be rapid during the water-smoking period and also from 370-540°. This range should be passed through as quickly as possible. At the higher soaking temperature, however, the sulphates begin to be eliminated, and more rapidly in a reducing atmosphere. If this latter condition is impracticable, the soaking temperature should be raised and the process extended. Dry-house scum is caused by soluble salts already in the clay which accumulate on the surface during drying. They are chiefly sulphates of sodium and potassium, which melt before their decomposition and elimination is effected. This "fixed" scum is minimised by rapid drying, a strongly reducing atmosphere at 870-930° (below their m.p.), and by barium treatment of the clay. It is recommended that the soluble salts of barium rather than barium carbonate be used. A brick containing not more than 0.05% of water-soluble sulphuric anhydride in the outer  $\frac{1}{8}$  in, of its exposed surface is not likely to develop efflorescence. J. A. SUGDEN.

Properties of breeze and clinker aggregates [for concrete], and methods of testing their soundness. F. M. LEA (Dept. Sci. Ind. Res., Building Res. Bull. No. 5, 1928, 17 pp.).—Whether a particular breeze or clinker is suitable for use as an aggregate depends not so much on the amount of combustible matter present, which is not harmful if well carbonised, but on whether it contains uncarbonised coal, particularly of high oxygen content. Such coals undergo a progressive expansion when in contact with the setting cement, and this expansion may be checked by a rapid drying of the mixture, only to assert itself again should it become wetted later. Failures of clinker concretes are rarely due to the presence of sulphur compounds, though limits of not more than 0.45% S (as sulphide) and 0.4% S (as sulphate), or 0.75% of total sulphur excluding sulphates, are indicated. The corrosion of reinforcement in concretes is due to permeability, and the accelerating effect of sulphur compounds is secondary. A test for the resistance to cracking consists in forming a pat of a mixture of 3 pts. of breeze (through 76-mesh) and 1 pt. of a mixture (1:1) of Portland cement and plaster of Paris. The upper surface of the pat formed on a glass plate 4 in. square should be of convex shape and about 1/2 in. deep at the centre. After setting for 3-4 hrs. in a moist atmosphere the pat is immersed in water and examined for visible cracks during 1-4 days. The unsuitability of breeze is indicated also by its excessive absorption of methylene-blue. C. A. KING.

Fuel economy in burning Portland cement clinker. R. D. PIKE (Ind. Eng. Chem., 1928, 20, 1155— 1163).—Whilst progress has been made in fuel economy by the use of waste-heat boilers, there has been little advance in reducing the consumption of fuel in the cement kiln itself. It is shown by consideration of theoretical heat balances that in a properly designed kiln using the dry process a saving of 40—50% over the wet process should be achieved. Partial corroboration of this view is obtained from the working results of dry shaft kilns. C. A. KING.

Studies on Portland cement compounds by the X-ray diffraction method. W. C. HANSEN (J. Amer. Ceram. Soc., 1928, 11, 68-78; cf. Harrington, A., 1927, 715).-Methods of preparing compounds of lime with alumina and silica which may occur in Portland cement are examined, and the preparation of 5CaO,3AI2O3, 3CaO, Al<sub>2</sub>O<sub>3</sub>, 3CaO, SiO<sub>2</sub>, Y-2CaO, SiO<sub>2</sub>, and β-2CaO, SiO<sub>2</sub> is described. In an attempt to produce &-2CaO,SiO2, a product was obtained which consisted mainly of twinned crystals, but the X-ray diffraction pattern of which was identical with the β-form. Two mixtures of lime, alumina, and silica were heated at four different temperatures, and the products were studied by the X-ray method. The compounds identified in these products were 3CaO,SiO<sub>2</sub>,  $\gamma$ - and  $\beta$ -2CaO,SiO<sub>2</sub>, 3CaO,Al<sub>2</sub>O<sub>3</sub>, and CaO. F. SALT.

# PATENTS.

[Portland] cement manufacture [by the wet process]. W. A. SCHMIDT, ASST. to INTERNAT. PRECIPI-TATION CO. (U.S.P. 1,688,882, 23.10.28. Appl., 21.9.27). -The flue dust is recovered, allowed to react with water, and then introduced into the raw-mix slurry.

#### F. G. CLARKE.

**Production of dolomite cement.** F. W. BROWN, Assr. to G. R. AUSTIN (U.S.P. 1,682,479, 28.8.28. Appl., 10.11.24).—Dolomite free from iron and silica is rapidly heated to 760°, and maintained at 870° until carbon dioxide is removed from the magnesia but not from the lime; the cooled product is then dyed. F. G. CLARKE.

Manufacture of porous masses from mineral substances capable of setting. J. MEYER and E. ASMUS (B.P. 292,468, 7.6.28. Ger., 18.6.27).—Finelypowdered alloys of calcium with zinc and/or magnesium, preferably containing 70—80% Ca, are added to cement etc. L. A. Colles.

Manufacture of wall board. A. E. WHITE. From UPSON Co. (B.P. 300,041, 30.12.27).

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

Utilisation of burnt pyrites as an iron ore. C. FICAI (Giorn. Chim. Ind. Appl., 1928, 10, 450-459, 511-521).-A description is given of the plant and method used by the Montecatini Co. at Porto Maghera, where burnt pyrites is used for the preparation of about 400 tons per day of agglomerated ore, mostly utilised by the iron industry. The agglomeration of finely-divided iron ore, in particular of burnt pyrites, by the briquetting process consists in heating the material, previously compressed into blocks, to about 1350°. The various hypotheses advanced in explanation of the changes taking place, viz., (1) formation of silicates in the fused and solid states, (2) semi-fusion (sintering) of the iron oxides, and (3) recrystallisation of these oxides, are examined. Hypothesis (1) is excluded as a result of microscopical examination and in virtue of the possibility of agglomerating pure ferric oxide, and (2) by reason of the difference between the m.p. of the oxides (about 1550°) and the actual temperature of agglomeration, which in certain circumstances can be effected even at about 1200°. The experimental results favour hypothesis (3), and it appears that the red, earthy hæmatite contained in the burnt pyrites is transformed by the heating into a black, crystalline ferric oxide, magnetite being formed as an intermediate product possessing catalytic properties.

### T. H. POPE.

Some constituents of basic open-hearth slag. J. M. FERGUSON (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 9—18).—Three well-crystallised constituents have been isolated from basic slags of low phosphorus content to which fluorspar has been added to decrease the viscosity, viz., tricalcium silicate in white, orthorhombic plates, tricalcium ferrite in lustrous, black, monoclinic needles (d 4), and a blood-red magnetic constituent crystallising in the cubic system. The lastnamed contains 30—32% CaO, 13.5—14.5% MnO, 5—6% FeO, 5% MgO, 5% Al<sub>2</sub>O<sub>3</sub>, 32—34% Fe<sub>2</sub>O<sub>3</sub>, and 8—5% SiO<sub>2</sub>. The mechanism of the formation of these compounds in the slag and their function in the refining of iron are briefly discussed. A. R. POWELL.

Production of high-alumina slags in the blast furnace. T. L. JOSEPH, S. P. KINNEY, and C. E. WOOD (U.S. Bur. Mines Tech. Paper No. 425, 1928, 32 pp.) .-Prior to furnace experiment m.p. determinations showed that slags containing 47-48% Al<sub>2</sub>O<sub>3</sub>, 41-42% CaO, 4-5% SiO<sub>2</sub>, together with the usual traces of impurities, were within operating possibilities, though the ternary eutectic (49.5% CaO, 43.7% Al<sub>2</sub>O<sub>3</sub>, 6.8% SiO<sub>2</sub>) has a relatively low viscosity in a considerable range of temperature above the m.p. In a two-weeks' test slags were produced covering a range of composition 6-30% SiO<sub>2</sub>, 35-52% CaO, 18-53% Al<sub>2</sub>O<sub>3</sub>, about 2% TiO<sub>2</sub>, about 1% Fe<sub>2</sub>O<sub>3</sub>, and 0.5% S. No operating difficulties were encountered. Calcium aluminates desulphurise metal rapidly at 1500-1525°, but high-alumina slags lose the avidity for sulphur at lower temperatures. During the experimental run silicon was oxidised from the scrap charge, though this would be less pronounced in full-sized furnaces. The making up of the charge is largely a matter of local costing, but the silica in the coke ash places a limit on the permissible C. A. KING. silica in other materials.

Microscopical studies of a passive layer in carbon steel and the resulting etch structure. C. BENEDICKS and P. SEDERHOLM (Z. physikal. Chem., 1928, 138, 123—134).—In studying the etch figures produced on a carbon tool-steel by alcoholic nitric acid solutions, an unusual fissured effect was noticed which is attributed to the existence of an extremely thin passive layer; this is produced even with etch fluids containing only 0.1% of nitric acid. The probable origin of this film is discussed. F. G. TRYHORN.

Secondary hardening of alloy steel. J. H. ANDREW (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 7—8).—The secondary hardening of hypereutectoidal nickel-chromium steels on tempering above 400° is ascribed to the removal of iron carbide from solid solution, leaving a solid solution which undergoes a critical transformation ( $\gamma$  to  $\alpha$ ) at 150° with the formation of the hard martensite phase. No secondary hardening takes place when the composition of the steel is such that the  $\gamma$ — $\alpha$  transformation takes place above the decomposition temperature of martensite. A. R. POWELL.

High-speed steel. J. S. G. PRIMROSE (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 29—34).— An account of the microstructure, heat-treatment, and working properties of high-speed tungsten steels.

A. R. POWELL.

Manufacture of ferromanganese at the Makievka Works in Central Russia. V. G. KOTELNIKOV (Rev. Mét., 1928, 25, 541—556).—An account is given of the operation during a period of 18 months of a blast furnace producing high-grade ferromanganese from Russian ores. The best results were obtained by the use of a charge giving an acid slag containing 30% SiO<sub>2</sub>, 32— 34% CaO, 18—20\% MnO, 2% MgO, and 9—10% Al<sub>2</sub>O<sub>3</sub>. The manganese ore should contain at least 45% Mn and not more than 10—14% SiO<sub>2</sub>, and the temperature of the blast should be between 750° and 830°. A rapid descent of the charge in the furnace gives a better yield than the usual slow working; it is recommended that the time the ore is in the furnace should not exceed 14 hrs. With an ore containing 46-48% Mn, ferromanganese containing 76-77% Mn can be obtained under the above conditions with a coke consumption of  $2 \cdot 4$  tons/ton of metal. The extraction of manganese as metal exceeds 70%; 18% remains in the slag and 12% passes into the flue dust. The latter contains about 30% Mn and 28% SiO<sub>2</sub> and is utilised, together with the slag produced, in the smelting of iron ores in the blast furnace for the production of pig iron. Operating with a basic slag results in higher losses of manganese in the slag and a lower content of manganese in the metal. A. R. POWELL.

Accelerated laboratory corrosion test methods for zinc-coated steel. E. C. GROESBECK and W. A. TUCKER (U.S. Bur. Stand. Res. Paper No. 10, 1928, 41 pp.).—In duration tests consisting of spraying galvanised steel sheets with a normal solution of sodium chloride or ammonium chloride, the results of the former showed consistent relationship between the "life" of the coating and the thickness of zinc, though the spray method caused local corrosion depending on the distribution of the condensed spray over the surface. A simulated atmospheric attack consisting of a cycle of 5 hrs.' exposure to a warm, moist, gaseous mixture of, by vol., 5 pts. of carbon dioxide, 1 pt. of sulphur dioxide, and 94 pts. of air, followed by 1 hr. of water spraying and 18 hrs. of air drying, resulted in a progressive removal of the coating in the reasonable period of 3-4 weeks. "Copper-steel" (0.2% Cu) produced no observable effect on the life of the zinc coating, though known imperfect coatings, e.g., grey-galvanised, stringy, and blistered, were consistent in showing a lower life than regular sheets of good quality. No attempt was made to correlate results with the life in actual working practice. C. A. KING.

Recovery of copper and zinc from brass scrap. I. A. KAZARNOVSKI and V. M. ZVENIGORODSKAYA (Papers Karpov Chem. Inst., Bach Mem. Vol., 1927, 101—109).— The oxides formed on calcination are dissolved in sulphuric acid and the solution is boiled with brass turnings ; precipitation of copper is practically complete.

#### CHEMICAL ABSTRACTS.

Determination of sulphur in copper alloys containing tin. H. LEYSAHT (Z. anal. Chem., 1928, 75, 169 -182).-Methods for determining small quantities of sulphur are examined and details are given. Sulphur may either be oxidised and ultimately precipitated as barium sulphate, or, assuming that sulphur is present entirely as sulphides (as is apparently the case here), the alloy may be treated with a suitable acid and the evolved hydrogen sulphide absorbed in a suitable medium. The whole of the sulphur is completely oxidised by fuming nitric acid, but much of the sulphur is contained in the insoluble stannic oxide. This precipitate must therefore be separately examined, or the whole taken to dryness several times with hydrochloric acid. It is unnecessary to remove copper before precipitation of barium sulphate. The resulting barium sulphate is usually contaminated with tin etc., and requires treatment with fusion mixture and reprecipitation of sulphate. Removal of tin etc. by treatment with ammonia or hydrogen sulphide prior to precipitation of sulphate

leads to untrustworthy results. Accurate results are obtained when tin is removed as bromide by repeated evaporation with hydrobromic acid, but care must be taken to avoid loss by spurting during the evaporations. Oxidation by bromine is dependent on the actual experimental conditions, but satisfactory results are obtained when a saturated solution of potassium chlorate is poured over the material and a considerable excess of bromine is added. When reaction has ceased, hydrochloric acid is added and sulphate precipitated from the boiled, diluted solution. Oxidation methods are, however, tedious, and the use of large quantities of reagents necessitates the carrying out of a large number of blank determinations. The whole of the sulphidesulphur is evolved when the alloy is treated with a solution of hydrogen bromide. Suitable absorbing media are brominated hydrochloric acid, in which case sulphur is precipitated as barium sulphate, and a solution of cadmium acetate. In the latter case sulphide is determined iodometrically, or converted into cupric sulphide by treatment with a solution of copper sulphate and weighed as oxide. The whole of the sulphur is not evolved when hydrobromic acid is replaced by hydrochloric acid. This is especially true when the alloy is in the form of coarse turnings etc. J. S. CARTER.

Mechanism of crystal growth by annealing. T. SUTOKI (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 857-876) .- The crystal growth of aluminium containing 0.4% Fe and 0.2% Si has been followed after straining to 1-2% elongation and annealing at 570-630°. Crystals of macroscopic size in contact with one another do not grow, however prolonged the annealing, but a single crystal in contact with strained microcrystals grows steadily at their expense. Repeated heating and cooling produces many macrocrystals if the rate of cooling is rapid, but intermediate sudden coolings by quenching cause the formation of very large crystals surrounded by minute grains. From annealing experiments on tin containing 1.5% Sb the crystal growth of unstrained metals has been shown to proceed along concave grain-boundaries in such a manner that the radius of curvature decreases with time of annealing. In this case large crystals may grow at the expense of smaller ones, and small crystals at the expense of larger ones, and a crystal may grow on one side and be encroached upon on another side. Thus it appears that to cause crystal growth on annealing the presence of some minute gaps in the grain-boundary or of nuclei along slip-planes caused by straining is necessary.

A. R. POWELL.

Production of tungsten in rod or powder form from a single crystal. I. TARJÁN and P. TÚRY (Chem. Runds. Mitteleuropa Balkan, 1927, 4, 165—168; Chem. Zentr., 1928, I, 237—238).—The tenacity of tungsten wire used in the electric incandescence lamp is largely dependent on its production from a single crystal. Reduction of tungsten trioxide is best effected by heating at 700—950° in hydrogen; the size of the particles, which should average 0·1 $\mu$ , should not be greater than 1·0 $\mu$ . The tungsten, containing some thorium, is cast into rods weighing 85 g. in an atmosphere of hydrogen and heated for a short time only in the neighbourhood of its m.p. Crystal formation takes place in cylindrical surfaces starting at the interior and working outwards. L. A. COLES.

Corrosive action of sulphur chloride. HARVEY.— See VII. Effect of lead and zinc fumes on soil. LUNDEGÅRDH.—See XVI.

#### PATENTS.

Blast furnaces. J. P. DOVEL (B.P. 299,941, 25.8.27). —Hollow bronze cooling blocks are inserted into the lining of a blast furnace and rigidly bolted to the shell, so that as the refractory lining erodes a new lining formed from the furnace charge constituents collects and bonds about the cooling elements. In the upper part of the furnace, as far as the temperature permits, detachable metallic wear plates are fitted and spaced to allow of free expansion. For the proper formation of the stock column the charging bell should have an area, at its base, equal to about one third that of the shaft at the stock line. C. A. KING.

[Open-hearth] furnace. R. H. STEVENS, Assr. to BETHLEHEM STEEL Co. (U.S.P. 1,689,563, 30.10.28. Appl., 2.9.21).—The forward end of a tuyère port provided with a flue connexion to a regenerator projects within the rear end of a tuyère entrance port likewise provided with a flue connexion to another regenerator. Additional openings controlled by valves are arranged near the furnace end of the regenerators, and means are provided for controlling the flow of gases to the ports.

J. S. G. THOMAS.

[Mill] furnaces [for steel bars]. D. WILLIAMS (B.P. 298,507, 9.7.27).—The furnace comprises a combustion chamber, heating chamber, and flues under the heating chamber through which the products of combustion pass to and fro before reaching the stack. Over the firebridge between the fire and goods chamber is a perforated wall. B. M. VENABLES.

Welding high-carbon steel. J. H. DEPPELER, Assr. to METAL & THERMIT CORP. (U.S.P. 1,686,603, 9.10.28. Appl., 29.8.25).—A thin sheet of iron with a low carbon content is placed between the two parts to be joined, and the edges are then butt-welded by aluminothermic methods. A. R. POWELL.

Increasing the yield point in hollow bodies from steel alloys, particularly austenitic steel alloys. F. KRUPP A.-G. (B.P. 292,937, 5.6.28. Ger., 23.6.27).— Austenitic steels in the form of hollow rotary bodies are worked in a hot state and turned or expanded, *e.g.*, on a mandril, so that the stress induced by this means exceeds the original yield point. C. A. KING.

[Nickel-chromium] alloy steel and articles made therefrom. F. B. DEHN. From THOMPSON PRODUCTS INC. (B.P. 298,250, 8.4.27).—The steel contains  $11 \cdot 5$ — 13% Cr, 7—8% Ni, 2—3% Si, and  $0 \cdot 25$ — $0 \cdot 35\%$  C. The structure is rendered non-austenitic by annealing at 870—900° for 6 hrs. and cooling at the rate of 30°/hr. for 5 hrs. After working into the desired shape the alloy is further annealed at 620—675° and air-cooled or quenched in oil. A. R. POWELL.

Stable-surface iron-chromium-silicon alloy. P. A. E. ARMSTRONG, ASST. to LUDLUM STEEL CO. (U.S.P.

1,686,223, 2.10.28. Appl., 19.2.24).—An iron alloy containing more than 13% Cr, 0.3—5.0% Si, and less than 0.05% C is prepared by adding a preheated ferrochromium alloy to degasified molten iron. The ingots are forged hot and rolled first at 1040—1260°, then heavily rolled at about 600°. A. R. POWELL.

Iron alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 299,502, 27.7.27).—An iron alloy, suitable for the manufacture of armoured wire, having a tenacity of the order of 100 tons/in.<sup>2</sup>, developed by cold work, contains up to 15% of copper and silicon in about equal proportions; a suitable composition is 3-6% Cu, 3-6% Si, and remainder iron, with or without the addition of a small quantity of a deoxidiser such as manganese. M. E. NOTTAGE.

Method of making alloy steels. C. MCKNIGHT, JUN., ASST. to INTERNAT. NICKEL CO. (U.S.P. 1,689,043, 23.10.28. Appl., 8.6.25. Renewed 13.3.28).—Highsilicon scrap metal is melted under oxidising conditions to reduce the silicon content below a critical point not exceeding 0.1%, then subjected to reducing conditions with the addition of alloying materials. C. A. KING.

Metallurgical furnace. H. BUTT, Assr. to RILEY STOKER CORP. (U.S.P. 1,687,470, 9.10.28. Appl., 25.3.25).—The combustion chamber is provided with a retort to which the fuel is supplied and with a "fuel support" (fire-grate) to the side thereof (away from the firebridge) on to which the fuel overflows from the retort. Air passes through the fire-grate and a smaller amount is forced into the retort, producing a large quantity of combustible gas. The roof of the furnace is close to the bed of fuel to restrict combustion, and sloping to reflect heat into the charge in the retort; it is low over the fire-bridge, and immediately after dips down over the charge in the heating chamber so as to cause the gases to mix thoroughly and burn as a long flame.

B. M. VENABLES.

Smelting apparatus. C. E. GLESSNER (U.S.P. 1,686,912, 9.10.28. Appl., 9.6.27).—Crushed ore is fed uniformly into the top of a "centrifugal extractor" having a vertical axis and annular grooves. The bottom of the extractor is sealed by a bath of the metal produced. Air is drawn downwards through the apparatus.

B. M. VENABLES.

Centrifugal concentrating and amalgamating apparatus of the vertical type. F. D. LEWIS (U.S.P. 1,684,870, 18.9.28. Appl., 17.10.27).—The material is fed through an axial pipe to the bottom of a rotating tapering "cylinder" on the interior of which are formed annular grooves, and passes upwards into a bowl also provided with grooves and rotating. A disc sliding on the feed pipe controls the opening between the cylinder and bowl. B. M. VENABLES.

Concentration of ores and minerals by flotation. W. A. DOUGLASS, ASST. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,674,166, 19.6.28. Appl., 25.6.26).— Thionurethanes, NHR·CS·OR', are used with frothing agents for ore flotation; examples in which R = H, Me, or Et, and R' = Me, Et,  $Pr^{a}$ , or Bu<sup>a</sup> are mentioned. C. HOLLINS.

Apparatus for treating [metallic] fumes. M. I. DORFAN, ASS. to ALLIS-CHALMERS MANUF. CO. (U.S.P. 1,685,229,25.9.28. Appl., 21.11.23).—Combustible gases, escaping from the mouths of a bank of retorts for reducing zinc or other ores, enter combustion zones formed by hoods into which air is introduced. The products of combustion are passed from the hoods to a separator to deposit solids. The forward and rear walls of each hood are movable in order to control the air supply and to give access to the retorts. F. G. CLARKE.

Treatment of complex minerals containing tin, and/or copper etc. and associated metals. F. L. WILDER, E. MORRIS, E. SCHIFF, and E. S. KING (B.P. 299,750, 30.7.27).—Complex tin ores containing sulphides of iron, copper, etc. are treated with a solution containing persulphates and chlorides of the alkalis together with ferric sulphate or chloride and a small proportion of free chlorine. From the leach liquor so obtained copper or tin, or both, may be recovered by electrolysis or by precipitation by hydrogen sulphide and the waste liquor regenerated by electrolysis. A. R. POWELL,

Aluminium alloy. H. C. HALL and T. F. BRADBURY (B.P. 300,078, 3.4.28).—An alloy of aluminium contains 0·2—1·5% Ni, 0·2—5% Mg, 0·5—2% Cu, 0·2—0·5% Si, 0·7—1·5% Fe, and not more than 0·25% Mn (Ti). C. A. KING.

Manufacture of articles of refractory metal or metallic mixtures. J. W. MARDEN, Assr. to WESTING-HOUSE LAMP CO. (U.S.P. 1,675,119, 26.6.28. Appl., 10.4.22).—To avoid fashioning the articles, e.g., X-ray targets, crucibles, at high temperatures, a cream is formed of a refractory metal powder, cryolite, phosphoric acid, and water, and a plaster of Paris mould is filled therewith. After settling, the liquid is poured off and the deposit dried. The article is removed and fired in hydrogen. F. G. CLARKE.

Metal pickling. J. H. GRAVELL and A. DOUTY (U.S.P. 1,678,776, 31.7.28. Appl., 12.11.26).—Stannous chloride is added to prevent pitting, but in insufficient quantity to dissolve the scale removed from the metal. Organic substances, such as glue, formaldehyde, or preferably those which produce a foam (e.g., flour and sulphite-cellulose waste), may be added to trap noxious gases and prevent oxidation of the tin. F. G. CLARKE.

Coating non-conducting substances with metals. A. I. and E. L. GATES-WARREN, and PRECIOUS METAL INDUSTRIES, LTD. (B.P. 299,903, 2.8.27 and 2.4.28).— A solution of a metallic salt, e.g., silver nitrate, and a finely-divided metallic precipitate dispersed in a suitable carrier, e.g., silver, are applied successively or simultaneously to the abraded surface of the substance, e.g., a manufactured protein product, and the metal salt is reduced by formaldehyde or other reducing agent.

J. S. G. THOMAS.

Using direct metal from blast furnaces. R. Mol-DENKE, ASST. to NEW PROCESS MULTI-CASTINGS CO. (U.S.P. 1,691,401, 13.11.28. Appl., 19.12.24).—See B.P. 267,590; B., 1927, 369.

Solder for aluminium. O. SPENGLER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,691,532, 13.11.28. Appl., 13.7.25. Ger., 24.9.24).—See B.P. 240,411; B., 1925, 996.

Production of magnesium. W. KOEHLER (B.P. 300,149, 29.7.27).—See U.S.P. 1,650,893—4; B., 1928, 95, 96.

Apparatus for charging tilting smelting furnaces. DEMAG A.-G. (B.P. 286,224, 25.2.28. Ger., 28.2.27).

Calcining furnace (U.S.P. 1,686,565). Treatment of foundry sand (B.P. 298,384).—See I. Waste iron pickle (B.P. 299,919).—See VII. Electric furnace for metal bands (U.S.P. 1,689,369). Electrodeposition of metals (U.S.P. 1,689,597). Electrodeposition of chromium (B.P. 300,043).—See XI.

# XI.—ELECTROTECHNICS.

Effect of hydrogen-ion concentration on the voltage of the Leclanché dry cell. B. M. THOMPSON (Ind. Eng. Chem., 1928, 20, 1176-1178).-Improved technique in which the hydrogen-ion concentration of a solution is obtained from a Lechanché cell under equilibrium conditions has shown that mixtures of manganese dioxide and graphite give straight lines when the voltage, referred to the normal calomel electrode, is plotted against the  $p_{\rm H}$  value of the solution. Natural manganese ores gave a line with a slope of -0.07, as compared with the theoretical value -0.059, the difference being probably attributable to the presence of oxidising ions other than quadri- or ter-valent manganese ions. Graphite acts as an inert electrode without influence on the reaction. Artificial oxides vary greatly in properties, depending on the method of their preparation. C. A. KING.

Wood separators for lead storage batteries. C. A. ROBAK (Ind. Eng. Chem., 1928, 20, 1151—1152).— As a result of long-period trials of accumulators under working traffic conditions the destructive effect of the positive plate on the wood substance cannot be considered an objection to placing a flat side of a wood separator next to the positive plates. After several years' use separators next to the negative plates showed a mechanical strength about 30—45% higher than those in contact with the positive plates, though the values were only about 60% that of the original wood.

C. A. KING.

Automatic control. SWAN.—See I. Reduction of thoria by hydrogen. SEGALL.—See VII. Control in beet-sugar manufacture. BALCH and KEANE.— See XVII. Purification of water. ATEN.—See XXIII.

#### PATENTS.

Electric furnace. F. T. COPE, Assr. to ELECTRIC FURNACE CO. (U.S.P. 1,690,273, 6.11.28. Appl., 12.1.27). —Independently detachable spaced blocks having ledges on the under side of a refractory furnace roof are supported independently of the roof, and the end parts of rigid resistor grids are supported only on the ledges. J. S. G. THOMAS.

Electric furnace. G. SAGRAMOSO (U.S.P. 1,690,795, 6.11.28. Appl., 19.5.24. Italy, 28.5.23).—A doublewalled jacket, secured to a cross-beam adjustably mounted on standards and surrounding an electrode secured to the beam and movable therewith, is adapted to slide within a double-walled bell supported by the furnace. Means are provided for connecting the bell with the beam in such manner that the electrode, jacket, and beam can be elevated with respect to the furnace.

J. S. G. THOMAS.

Electric furnace for glowing metal bands by induction currents. M. TAMA (U.S.P. 1,689,369, 30.10.28. Appl., 29.11.27. Ger., 17.9.27).—Metal strip to be annealed is coiled round a drum and forms one of the coils of a transformer designed to generate the induction current. J. S. G. THOMAS.

Electrical gas-purifying apparatus. METALLBANK & METALLURGISCHE GES. A.-G., and R. LADENBURG (G.P. 452,394, 17.11.22).—Particles in the gases under treatment having an opposite charge to that of the emission electrode, in apparatus comprising an emission electrode with a precipitation electrode around it having a considerably lower current density, have their charge neutralised by adding oppositely-charged ions before the gases enter the electric field. L. A. COLES.

Cleaning gas-purifying apparatus by heating the electrodes. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 452,437, 3.8.24).—A precipitation electrode in the form of an endless band is heated, e.g., by hot rollers as soon as it leaves the electric field, passed through liquid to clean and cool it, and brought in contact with scrapers to remove adhering liquid before it is returned to the field. L. A. COLES.

Gas-filled incandescence cathode discharge tube. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPEN-FABR. (B.P. 299,931, 12.8.27).—A gas-filled discharge tube adapted for the emission of ultra-violet radiation comprises an oxide cathode or an incandescence cathode containing oxide, *e.g.*, a tungsten wire wound with nickel wire coated with barium oxide, one or more solid anodes, *e.g.*, of carbon, a gaseous filling of mercury vapour and a rare gas, *e.g.*, argon, and a wall composed of quartz or partly of quartz and partly of glass, the respective coefficients of expansion of the glasses increasing from the quartz to the glass. An auxiliary anode is arranged near the incandescence cathode to start the discharge. J. S. G. THOMAS.

Electron-discharge device. J. E. HARRIS, ASST. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,689,338, 30.10.28. Appl., 19.11.21).—A cathode composed of mischmetal is used. J. S. G. THOMAS.

Electron-emitting cathodes. RADIOTECHNIQUE Soc. ANON. (B.P. 281,262, 10,11.27. Fr., 26.11.26).—A wire core, e.g., of tungsten, of fairly large diameter, is covered with an adherent coating of refractory and insulating material, e.g., magnesia, by being heated to about 3200° Abs. in a bed of the powdered refractory, and is then covered with a sheath of electron-emitting material, e.g., thoriated tungsten, wound helically, the respective m.p. of the core and coatings decreasing from the inside outwards. J. S. G. THOMAS.

Producing rapidly-moving electrons and subjecting matter thereto. H. E. POTTS. From H. PLAUSON (B.P. 299,735, 30.5.27).—Cathode rays are subjected to the action of intense electron fields and/or rotating electromagnetic fields in a reaction chamber. J. S. G. THOMAS.

Method of exhausting vacuum devices. H. C. RENTSCHLER, ASST. to WESTINGHOUSE LAMP Co. (U.S.P. 1,689,297, 30.10.28. Appl., 16.8.22).—A plate to which a piece of aluminium is attached is sealed into a bulb, which is then exhausted to a moderate vacuum. The plate is heated to drive off occluded gases and the heating continued; the aluminium is vaporised and residual gases are eliminated. J. S. G. THOMAS.

Accumulator with negative zinc electrodes. A. BRANDES and P. O. H. RICHTER (G.P. 452,573, 12.5.26).—The positive electrode is constructed of strips of lead covered with a mixture of lampblack etc. with lead monoxide wrapped around with suitable material and connected with a supporting plate, and the electrolyte consists of zinc or an alkali sulphate solution. L. A. COLES.

Production of [accumulator] plates of spongy lead stable in the air. ACCUMULATOREN-FABR. A.-G. (G.P. 451,492, 18.8.26).—A small portion of the charge is withdrawn from the fully-charged plates by short exposure to a strong current before they are washed and dried in the absence of oxygen. L. A. COLES.

[Gas] accumulator. Soc. ANON. LE CARBONE (B.P. 291,420, 15.11.27. Fr., 4.6.27. Addn. to B.P. 230,307; B., 1925, 364).—A metal, e.g., lead, forming with chlorine a chloride which is, preferably, insoluble is incorporated with the active mass of porous impermeable carbon used in the accumulator. J. S. G. THOMAS.

[Porous electrodes for] galvanic cells. P. PÖRSCKE (B.P. 299,591, 8.7.27).—The porous paste produced by adding an acid or base to a mixture of metals and/or oxides is moulded or spread on a carrier.

J. S. G. THOMAS.

Means for preventing explosions. H. C. P. WEBER, ASST. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,686,051, 2.10.28. Appl., 23.3.22).— Natural, sewer, or other explosive gas is burned in a Davy safety lamp provided with an electrically-heated filament comprising a catalyst such as nickel. A thermocouple may be incorporated to indicate when explosive gas is present. F. G. CLARKE.

Preparation of organic articles for electrolytically covering them with a metallic layer. I. AINSTEIN (W. I. EINSTEIN) (B.P. 300,060, 22.2.28).—The organic article is dipped in soluble glass, dried, and its surface sanded. It is then given a thin preparatory metallisation with an alloy of easily fusible metal, *e.g.*, lead, antimony, bismuth, tin, or zinc, by pulverisation with a gun; if desired, this is followed by a similar second metallisation with copper or its alloys. The article is then lightly plated in an electroplating bath at below 10° before being subjected to the main plating process in a hot bath. J. S. G. THOMAS.

Cell for electrodeposition of metals. R. D. PIKE, G. H. WEST, and B. P. LITTLE, ASSTS. to R. D. PIKE (U.S.P. 1,689,597, 30.10.28. Appl., 23.9.25).—A cell comprises cathode and anode compartments, separated by porous diaphragms, means for maintaining a constant level of catholyte in the cathode compartment and for removing anolyte from anode compartments, common channels connected with the anode compartments on each side, and means for automatically admitting catholyte in order to maintain the level of the anolyte about constant. J. S. G. THOMAS.

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Electrodeposition of chromium. E. V. HAYES-GRATZE (B.P. 300,043, 5.1.28).—When electroplating with chromium the anode is made of pure electrodeposited chromium of the reverse shape of the surface of the article to be plated; *e.g.*, a cast or mould of the article may be plated with chromium and used as an anode, or the article itself may be first used as a mould and the deposit of chromium stripped off for use as the anode. C. A. KING.

Insulating material. BRIT. THOMSON-HOUSTON Co., LTD., H. W. H. WARREN, R. I. MARTIN, and A. E. SMITH (B.P. 299,906, 3.8.27).—Layers of fabric, paper, or asbestos are held together by a synthetic resin of the phenolic condensation type; the whole is wrapped with a thin sheet of paper or fabric impregnated with shellac and bonded by subjection to heat and pressure.

J. S. G. THOMAS.

Construction of electroplating vats or troughs. K. BARANOVSKI (B.P. 300,038, 21.12.27).

Coke electrodes (B.P. 295,314).—See II. Electrodeposition of rubber (B.P. 299,738).—See XIV.

# XII.—FATS; OILS; WAXES.

Fat in the residual material remaining after the unwinding of the silk cocoon. J. JELAKOV (Oil Fat Ind. Russia, 1926, No. 10–11, 35).—The unwound cocoon contains a fat (25%) having d 0.919, saponif. value 191.0, iodine value 105.2, Reichert-Meissl value 3.24, mol. wt. 283, m.p. 30°. The residue contains protein (60%) and phosphorus. CHEMICAL ABSTRACTS.

Fluorescence of oils and fats. J. F. CARRIÈRE (Chem. Weekblad, 1928, 25, 632-634).—A criticism of the conclusions of van Raalte (B., 1928, 792). The statement that crude materials do not fluoresce, but refined do, is incorrect. The material under examination should be placed below the lamp and viewed from above, since the surface generally shows the strongest fluorescence. The fluorescence of many vegetable oils is actually diminished by refining, but no definite rule can be laid down. The phenomenon is very complicated, but the possibility that vitamins play any part is extremely remote. S. I. LEVY.

Determination of fats. A. TICHONOV (Oil Fat Ind. Russia, 1926, No. 6, 38-40).—Soxhlet's method of fat extraction, using ether or light petroleum, extracts 97.6% in 1 hr., or all in 5-6 hrs. Cold extraction for 2-3 days extracts nearly all the fat. In Soxhlet's apparatus a separating funnel and a tube are placed between the flask and the extractor so that the solvent can be collected after the final extraction. Fat can be determined by measuring the density of the extracted material, and by weighing the residue after extraction.

CHEMICAL ABSTRACTS.

Hydrogenation of fatty oils. VII. Formation of isooleic acid by the hydrogenation of soya-bean oil. VIII. Hydrogenation of the methyl esters of oleic and linoleic acids. T. MAZUME (J. Soc. Chem. Ind., Japan, 1928, 31, 467-469, 470-472).-VII. Formation of isooleic acid was less when the temperature of hydrogenation was low, and increased with the amount of catalyst used. When the nickel catalyst contained a

small amount of copper (0.3%) the formation of the acid was very great; large amounts of copper (4%) considerably reduced the amount of acid formed. Nickel catalysts that had, after reduction, been exposed to air at a low temperature produced more *iso*oleic acid than those exposed at high temperatures.

VIII. During the hydrogenation of methyl oleate a large amount of *iso*oleic acid was formed at the beginning of the process, and the acid so formed was gradually transformed into oleic acid by further hydrogenation. In the case of the methyl ester of linoleic acid the transformation was linoleic acid  $\rightarrow$  oleic acid  $\rightarrow$ *iso*oleic acid  $\rightarrow$  stearic acid. Y. TOMODA.

Polymerisation of tung oil and the ethyl ester of elæostearic acid by heat. T. MAZUME and S. NAGAO (J. Soc. Chem. Ind., Japan, 1928, **31**, 473—476).—When tung oil was heated at 200—300° its mol. wt. was doubled, but its iodine value did not much decrease; it is considered, therefore, that the change was one of extramolecular polymerisation. In case of the ethyl ester of elæostearic acid the change was similar, but unlike the oil the ester did not solidify. Y. TOMODA.

Nutritive value of hardened oils. II. S. UENO, M. YAMASHITA, and Y. OTA (J. Soc. Chem. Ind., Japan, 1928, 31, 393—399).—Tests on animals showed the nutritive value of the low-temperature hardened oils (sardine oil, herring oil, finback whale oil, chrysalis oil) to be superior to that of the original oils.

Y. TOMODA.

Examination of cresol-naphthene soap solutions. G. KOGAN (Pharm. Zentr., 1928, 69, 681-684). —The material is shaken with ether and dilute sulphuric acid saturated with sodium sulphate; the aqueous layer contains the bases. The naphthenic acids are removed from the ether solution by shaking with sodium carbonate solution, and after separation the cresols are removed by shaking the ethereal layer with sodium hydroxide solution, leaving only hydrocarbons in the ether solution. S. I. LEVY.

Vulcanisation of oils. II. P. STAMBERGER (Rec. trav. chim., 1928, 47, 973-976; cf. B., 1928, 23).-When triolein (5 pts.) is heated with sulphur (1 pt.) at 160° a viscous fluid is obtained which is soluble in benzene, but only partly soluble in acetone. The mol. wt. of the acetone-soluble and insoluble products are 1204 and 2040, respectively. The fluid products are converted into elastic solids (cf. loc. cit.) by (a) keeping at room temperature for several months; (b) heating to 180°; (c) irradiation with ultra-violet light; and (d) treatment with some chemically indifferent solvents (acetone or water). Whilst the solids produced are only slightly soluble in benzene, they swell considerably in this liquid, and the amount of benzene absorbed varies with the hardness of the solid. The benzene-soluble product previously described (loc. cit.) changes to an elastic solid on keeping for 3-4 months at room temperature. This solid swells in cold benzene, is soluble in hot benzene, and the product from this solution redissolves in the cold solvent. The solvent thus appears to have some action on the gel, probably causing an aggregation or polymerisation of the molecules (cf. Stevens, B., 1928, 203). H. BURTON.

Prevention of autoxidation of vegetable oils and their fatty acids. V. NOVIKOV (Oil Fat Ind. Russia, 1927, No. 2, 17—20).—The rate of absorption of oxygen by linoleic acid, and the action of various organic substances in retarding the oxidation, were studied.  $\beta$ -Naphthol (1%) prevents the oxidation of linoleic acid in oxygen at 60°. Linseed oil containing 1% of  $\beta$ naphthol did not change in iodine value or viscosity during 2 years. CHEMICAL ABSTRACTS.

Differences in crude soya-bean oils from various sources. J. F. CARRIÈRE (Chem. Weekblad, 1928, 25, 630-632).—The crude expressed oil from Manchuria has an abnormally low sp. gr. ( $d^{20} 0.9219$ ), an abnormally high content of unsaponifiable matter (0.80-1.07%), and a characteristic bluish fluorescence in ultra-violet light. Extracted Manchurian oil on the other hand is normal in all three properties. The expressed oil appears to be contaminated with about 0.4% of mineral or resin oil. S. I. LEVY.

Oil from apricot stones. V. TEMNOV (Oil Fat Ind. Russia, 1926, No. 10—11, 67—68).—The oil (yield 37%out of 47%) had d 0.915, iodine value 101.4, saponif. value 196, acid value 3.11. CHEMICAL ABSTRACTS.

Obtaining average samples of oil cake. F. Go-GOLEV (Oil Fat Ind. Russia, 1926, No. 4–5, 46–49).— Variations of the oil content in different parts of the cake are recorded, e.g., 7.03% in the middle, 8.03% at the edges; uppermost layer in the press 0.5% more than others. CHEMICAL ABSTRACTS.

Volumetric analysis of oil cakes for their oil content and moisture. A. LAPTEV (Oil Fat Ind. Russia, 1926, No. 4—5, 49—52).—The finely-divided substance is kept overnight in ether, and then extracted for 3—3.5 hrs.; the bottom of the apparatus contains a filter plug of defatted wadding. The last traces of ether are expelled in an oven in a current of carbon dioxide. CHEMICAL ABSTRACTS.

Determination of the oil content of linseed by the results of mechanical analysis of the purity. J. ORLOV (Oil Fat Ind. Russia, 1927, No. 1, 1–16).— The oil content is 0.355a + 0.05b, where *a* is the content (%) of pure linseed and *b* the content of other oil-bearing seeds. CHEMICAL ABSTRACTS.

Saponification with the Twitchell and Pfeilring reagents. G. PETROV (Oil Fat Ind. Russia, 1926, No. 6, 25–28; No. 7–8, 21–27).—The aromatic thio-fatty acids of anthracene, phenanthrene, benzene, and xylene are less active than those of naphthalene. The Pfeilring cleavage agent is examined.

CHEMICAL ABSTRACTS.

Seed oil of Citrus limomum. OccHIPINTI.— See XX.

#### PATENTS.

Generation of oxygen from washing and bleaching agents containing persalts. G. LESCHIK (B.P. 273,711, 27.6.27. Ger., 5.7.26).—A catalyst, e.g., a manganous compound, or tyrosinase expressed from fresh potato skins, is added to such washing and bleaching powders, during use, to obtain quantitative development of oxygen. W. G. CAREY. Production of material of the consistency of ointment from colophony. A. RIEBECK'SCHE MONTAN-WERKE A.-G. (G.P. 451,180, 23.6.22).—Material suitable for use in the manufacture of soap, textile preparations, etc. is obtained by the catalytic hydrogenation of a mixture of colophony and castor oil at raised temperature and pressure. L.A. COLES.

Soap product and its manufacture [by spraying and drying]. INDUSTRIAL SPRAY-DRYING CORP., Assees. of D. R. LAMONT (B.P. 291,041, 8.8.27. U.S., 25.5.27).

#### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Microscopical examination of lithopones and zinc whites. V. Allard (Ann. Falsif., 1928, 21, 445-449). -"" Molecular " lithopone, which is obtained by double precipitation, may readily be distinguished by means of the microscope from "mixed " lithopones, obtained from a mixture of zinc sulphate and chloride with barium sulphide such that 60-70% of zinc sulphide is produced, and to which is added white baryta, to reduce the proportion of zinc sulphide to about 30%. The "molecular" product presents under the microscope a homogeneous, amorphous structure, whilst such products as baryta and chalk have a translucent crystalline appearance. The effect is more marked if polarised light is used with interposition of a layer of gypsum to give a red background, when the dark, amorphous, "molecular" lithopone is in marked contrast to the brilliant colours exhibited by the salts of the mixture.

# D. G. HEWER.

#### Brewers' pitch. KUTTER.—See XVIII.

#### PATENTS.

Preparation of pigments. L. A. LEVY and D. W. WEST (B.P. 299,797, 1.7.27).—A very finely divided condition of the zinc sulphide or zinc cadmium sulphide used for phosphorescent and fluorescent paint is ensured by the addition, before firing, of up to 20% of white rare-earth or alkaline-earth oxides, and discoloration in sunlight etc. is prevented by the addition, after firing, of a solution of trisodium phosphate, sodium carbonate, or borax, the solvent being then evaporated. W. G. CAREY.

Manufacture of titanium oxide [pigments]. F. G. C. STEPHENS, L. J. ANDERSON, and W. A. CASH (B.P. 299,835, 2.8.27).—Titanium sulphate solution containing about 16% TiO<sub>2</sub>, preferably prepared as described in B.P. 256,734 (B., 1926, 876), is added slowly with agitation to a solution containing, *e.g.*, 100 pts. of barium chloride per 750 pts. of water at  $25-40^{\circ}$ (preferably at  $30-35^{\circ}$ ), and the mixture is heated at 100° by steam until all the titanium is thrown out of solution. The precipitate is removed, washed, and suspended in borax or boric acid solution before calcination. L. A. COLES.

Improvement of recent natural acid resins. CHEM. FABR. DR. K. ALBERT G.M.B.H. (G.P. 440,003, 9.8.17).—Natural acid resins are combined with phenolaldehyde resins, the remaining acidity being reduced by esterification or addition of lime. In this way the less valuable natural resins, such as pine rosin, acquire properties resembling those of the copals. Colophony, crude cresol, and formaldehyde are heated to give a clear resin, volatile products and water are removed, and warm glycerin is gradually added, reaction being completed at 250—260°; or the preformed cresolformaldehyde resin may be dissolved in molten colophony. Glycol, arabitol, or other multivalent alcohols may replace glycerin, or lime or calcium carbonate may be used. C. HOLLINS.

Synthetic resin and its manufacture. L. T. RICHARDSON, ASST. to CUTLER-HAMMER MANUF. CO. (U.S.P. 1,682,934, 4.9.28. Appl., 15.5.22).—Furfuraldehyde is treated with an inorganic acid (e.g., 0.2 pt. of concentrated sulphuric acid) to form a dense, hard product which is characterised by brilliant natural superficial finish and lustrous fracture.

R. BRIGHTMAN.

Manufacture of an artificial resin. I. G. FARB-ENIND A.-G., Assees. of W. PUNGS (G.P. 451,116, 30.8.22). —Oxidised paraffin, freed from hydrocarbons, is chlorinated in carbon tetrachloride at 70°. On removal of solvent, a hard, flexible, yellow resin (55% Cl) is obtained, which may be further hardened by heating at 110°. C. HOLLINS.

Manufacture of benzene- and spirit-soluble polymerisation products of vinyl acetate, and of lacquers therefrom. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H., Assees. of W. HAEHNEL and W. O. HERRMANN (G.P. 446,562, 16.5.26).—The polymerisation of vinyl acetate by means of ultra-violet light is stopped as soon as the product begins to be insoluble in alcohol. The polymerisation may be performed with the aid of benzoyl peroxide in hot alcohol, and yields thus a viscous 50% alcoholic lacquer directly; a benzene lacquer is similarly prepared. C. HOLLINS.

Manufacture of condensation products from urea, thiourea, or their derivatives, and an alcohol or a ketone. I. G. FARBENIND. A.-G. (B.P. 280,238, 7.11.27. Ger., 6.11.26. Addn. to B.P. 278,390; B., 1928, 648).—The resinous products of the prior patent are also obtained at 15—60° in presence of strong acid (concentrated sulphuric acid), an excess of alcohol or ketone being used. C. HOLLINS.

**Production of plastic masses from casein.** G. JAKOVA-MERTURI (F.P. 623,650, 25.10.26).—Alkalineearth oxides or carbonates are worked up to a pasty mixture with a solution containing casein, borax, and ammonia previously prepared at 100°, dyes being added if desired. The product is moulded into shape, dipped into magnesium chloride solution, dried, dipped into formaldehyde solution, and again dried, or, when required for the manufacture of gramophone records, sheets of the product are dipped into a solution containing borax and acetic acid. L. A. COLES.

Insulating material (B.P. 299,906).—See XI. Material from colophony (G.P. 451,180).—See XII.

# XIV.-INDIA-RUBBER; GUTTA-PERCHA.

Effect of repeated reclaiming of rubber. G. W. MILLER (Ind. Eng. Chem., 1928, 20, 1165-1169).-A sample of reclaimed rubber from whole motor covers was vulcanised with sulphur (1.9%) and a small

proportion of organic accelerator and then " reclaimed " by heating with 10% sodium hydroxide solution for 9 hrs. at  $184 \cdot 4^{\circ}$ ; these operations were then repeated thrice more. With repeated reclaiming the tensile strength after re-vulcanisation was little affected, but the material each time became less extensible and drier; the chloroform extract decreased from 22% to 5%, while the combined sulphur increased from 2.75% to 8.05%. When the successive grades of reclaimed material were incorporated in a vulcanisable mixture containing new rubber and carbon black as the other chief ingredients, the tensile strength decreased each time. The repeated reclaiming appeared to have little influence on the behaviour of this mixture towards accelerated ageing. D. F. Twiss.

Comparison of scorching qualities of accelerators [for vulcanisation of rubber]. H. R. THES (Ind. Eng. Chem., 1928, 20, 1223—1227).—The relative tendency of various accelerators to induce prevulcanisation is measured by comparing the opacity of the dispersions obtained when a sample disc, 3 mm. thick and 13 mm. diam., after storage at a definite temperature, is shaken with a circular motion with 50 c.c. of gasoline ( $d \ 0.70$ ) for 1 min., giving 105 shakes. The storage temperatures recommended for the test are 99.5° representing milling temperatures, 66.5° representing a high storage temperature for milled stock, and the ordinary room temperature.

D. F. Twiss.

Heat transfer in the vulcanisation of rubber. T. K. SHERWOOD (Ind. Eng. Chem., 1928, 20, 1181-1185). -On account of the low thermal conductivity of rubber, non-uniformity of vulcanisation arising from "temperature lag" occurs with all but the thinnest articles. Curves are given illustrating the effect of increase in the thickness of the rubber layer on the observed course of rise of temperature in a motor cover during vulcanisation. From a knowledge of the temperature at different times during the heating process it is possible to calculate the relative vulcanisation effect at different points in a massive rubber article. Results are given demonstrating the advantages, in time consumption and in uniformity, gained by vulcanising pneumatic tyres on an inflated former filled with circulating superheated water instead of with air.

D. F. Twiss.

The accelerator [of vulcanisation] "Tuads" and its substitutes. W. ESCH (Kautschuk, 1928, 4, 255—258).—A discussion of the application of tetramethylthiuram disulphide ("Tuads") as an accelerator of vulcanisation, including some reference to the relative efficacy of its ethyl and monosulphide analogues. D. F. Twiss.

Ultra-accelerators [of vulcanisation of rubber]. A. D. CUMMINGS and H. E. SIMMONS (Ind. Eng. Chem., 1928, 20, 1173—1176).—Tetramethyl-, tetraethyl-, dimethyl-, diphenyldimethyl-, and dimethyldiethylthiuram disulphides were prepared by the action of alcoholic iodine (I mol.) on an alcoholic solution of carbon disulphide (1 mol.) and the necessary amine (2 mols.); dimethyldiethylthiuram disulphide has m.p. 72°. The inactivity of dimethylthiuram disulphide

as an accelerator is attributed to its instability and rapid decomposition under the conditions of vulcanisa-The vulcanising power of tetramethylthiuram tion. disulphide is substantially identical with that of a mixture of the monosulphide and sulphur in the equivalent proportions, thus indicating that the same accelerating substance, presumably zinc diethyl-dithiocarbamate, is produced by both. Diethylamine diethylmonothiocarbamate, obtained by the interaction of carbonyl sulphide and diethylamine, reacted with iodine giving an oily disulphide, and with zinc acetate yielding zinc diethylthiocarbamate; the latter has only feeble accelerating power. Comparative vulcanisation experiments with the thiuram disulphides and dithiocarbamates derived from dimethylamine and diethylamine, respectively, indicated that the ethyl compounds had somewhat greater acceleration activity.

#### D. F. Twiss.

Determination of sulphur in rubber by the perchloric acid method. E. WOLESENSKY (Ind. Eng. Chem., 1928, 20, 1234-1238; cf. Kahane, B., 1927, 532).-Kahane's method is improved in accuracy, experimental examples being given. The finely-divided sample (1 g.) in an 800-c.c. Kjeldahl flask is heated on a water-bath with 10 c.c. of 41% nitric acid until reaction subsides; concentrated nitric acid (10 c.c.) is then added, and heating continued for 15 min. until the rubber is dissolved. After adding 5 c.c. of a 60% solution of perchloric acid the inclined flask is heated until dense white fumes appear and the liquid is colourless. If free carbon is present a further addition of 3-5 c.c. of perchloric acid may be made or the carbon may be removed by filtration. After cooling, concentrated hydrochloric acid (5 c.c.) is slowly added and heating repeated until white fumes appear. Water (200 c.c.) is then added and the sulphate is precipitated hot with barium chloride. Any insoluble mineral matter may be removed before treatment with barium chloride and examined separately. D. F. Twiss.

Determination of iodine value of raw rubber. A. GORGAS (Kautschuk, 1928, 4, 253-254).—The iodine value of rubber can be conveniently determined by using iodine bromide at the ordinary temperature; the reagent is prepared by dissolving 10 g. of iodine monobromide in 500 c.c. of carbon tetrachloride, and is stored in glass-stoppered bottles. The comminuted rubber (0.15 g.) is allowed to swell in carbon tetrachloride (50 c.c.). When sufficiently swollen (e.g., after 24 hrs.), 50 c.c. of the reagent are added with shaking. After 15 min. in the dark, 30 c.c. of 10% solution of potassium iodide are introduced, together with 100 c.c. of water, and the mixture is titrated with 0.1Nthiosulphate. Two independent tests are made concurrently, together with a blank test. D. F. Twiss.

Inaccuracies in determination of acidity of raw rubber by water extraction. A. D. CUMMINGS and H. E. SIMMONS (Ind. Eng. Chem., 1928, 20, 1213—1214). —Soxhlet extraction with water yields high results for the water-soluble acids present in rubber; the prolonged treatment probably leads to hydrolysis of esters in the rubber. It is better to treat the acetone-extracted rubber with water on a water-bath until the aqueous extract shows no further increase in acidity.

D. F. TWISS. **Physical properties of rubber.** M. RUHEMANN and F. SIMON (Z. physikal. Chem., 1928, 138, 1—20).—Determinations have been made of the sp. heats, thermal expansions, and X-ray interference figures of a series of samples of rubber at temperatures between —100° and 40°. Abrupt increases in sp. heat occur with smoked rubber sheet near — 70° and near 0°, and with synthetic rubber near 0°. Less marked anomalies were found in the thermal expansion curves of some specimens of chilled rubber and of patent gums. These anomalies are attributed in part to a hardening process in one phase of the rubber, and in part to a change of state, probably from the solid to the liquid, of another phase. F. G. TRYHORN.

Solubility of sulphur in rubber. H. LOEWEN (Kautschuk, 1928, 4, 243—249).—The phenomena of dissolution and crystallisation of sulphur in rubber are reviewed, and reproductions of microscopical enlargements are given in illustration of the appearance of the separated sulphur under different conditions.

D. F. Twiss.

Crystallised and light-sensitive gutta-percha. F. KIRCHHOF (Kautschuk, 1928, 4, 254—255).—A solution of deresinised gutta-percha in carbon tetrachloride on evaporation yields a turbid film which under the microscope reveals distinct crystalline appearance; the turbidity disappears at 43—44° and reappears on cooling, probably on account of the fusing point of crystals lying near this temperature. Crystals of gutta-percha can also be formed by slow evaporation of a solution in acetic acid. Such colourless gutta-percha on exposure to ultra-violet light becomes more opaque and brittle, although still completely soluble in benzene. As this change takes place equally well in an atmosphere of hydrogen it is presumably due to more complete transformation into the crystalline form.

D. F. Twiss.

# PATENTS.

**Preservation of india-rubber.** RUBBER GROWERS' Assoc., INC., G. MARTIN, and W. DAVEY (B.P. 299,585, 26.9.27).—A mixture of evaporated serum from rubber latex and alkali such as sodium hydroxide or carbonate (whether added before evaporation or subsequently), e.g., in the proportion of 3—5 pts. and <u>1</u>—1 pt., respectively, to 100 pts. of rubber, has an anti-oxidant effect superior to that of serum solids alone and, unlike other anti-oxidants, does not lead to subsequent reduction in extensibility of the rubber. D. F. TWISS.

Stabilising natural or artificial types of caoutchouc. SILESIA VER. CHEM. FABR. (B.P. 291,431, 30.3.28. Ger., 2.6.27).—Ageing of rubber, vulcanised or not, is retarded by incorporating reaction products of amines, *e.g.*, aniline, with polyhydroxy-aldehydes such as dextrose. D. F. Twiss

Electrodeposition of rubber or homologous substances. W. A. WILLIAMS (B.P. 299,738, 30.6.27. Cf. B.P. 289,965; B., 1928, 494).—There is added to rubber latex, compounded or otherwise, an electrolyte, *e.g.*, ammonium chloride, which will not readily cause coagulation, but will enter into chemical action with the anode of two inserted electrodes and also act as the excitant of an internal electric current. The electrophoretic action of this current is assisted by applying to the electrodes an additional external source of current of voltage insufficient to decompose the added electrolyte, and such that the total electric potential shall be insufficient to effect electrolysis of water while causing deposition of rubber on the anode. D. F. TWISS.

Manufacture of articles from rubber or similar materials. DUNLOP RUBBER Co., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 299,974-5, 5.10.27).-(A) For the production of bulbous rubber articles, a former consisting of a rod bearing a bulbous enlargement of rubber with a roughened surface is dipped as many times as necessary in concentrated and/or compounded latex; after drying and vulcanising, the rod is removed, leaving the bulbous enlargement as part of the finished article. Between individual dippings in the latex the coated former may be immersed in a dehydrating and setting medium. (B) Articles with bulbous enlargements are made by dipping as in (A), but using smooth collapsible formers which are subsequently withdrawn from the D. F. Twiss. formed articles.

Production of shaped objects from aqueous dispersions of organic substances. DUNLOF RUBBER Co., LTD. From P. KLEIN and F. GABOR (B.P. 299,737, 28.6.27).—In the production of articles from dispersions such as rubber latex by dipping, formers are used which serve to produce the agglomerated deposit as well as to shape it. The formers, for instance, may be wholly or externally of alum, citric acid, acetic acid solidified with collodion, or of swollen or pasty masses containing coagulant substances. An aluminium former also may be used with alkaline latex, or a zinc former with latex containing ammonium sulphide. Such dipping formers may be provided with an outer semipermeable coat of inactive material. D. F. TWISS.

Rubber vulcanisation accelerator. D. H. POWERS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,674,122, 19.6.28. Appl., 28.7.26).—The thioanhydrides,  $(RO \cdot CS)_2S$ , obtained by the action of cyanogen bromide or chloride on sodium or potassium alkylxanthates, are used, especially in presence of ammonia or amines (aniline), as vulcanisation accelerators. The butyl and ethyl compounds are specifically claimed.

C. HOLLINS.

Rubber and resin compound. S. P. MILLER, Assr. to BARRETT Co. (U.S.P. 1,682,397, 28.8.28. Appl., 25.11.24).—From 10% of crude unvulcanised rubber is dissolved in "cumar" resin, m.p. 125—128°, giving a waxy product, m.p. about 123°. R. BRIGHTMAN.

Treatment of gut or other strings or strands. DUNLOP RUBBER CO., LTD., H. WILLSHAW, S. N. GOOD-HALL, and C. FOLLISS (B.P. 299,535, 4.8.27).—In vulcanising strings or strands the heating fluid which is forced into the apparatus exerts a pressure which actuates devices maintaining a continual longitudinal pull on the strands.. D. F. TWISS.

Securing the adhesion of bodies of dense, hard, or soft caoutchouc to other articles. H. BECKMANN (B.P. 300,008, 8.11.27).—The rubber surface to be attached is first coated with a thin film of porous rubber which is vulcanised in position; this film is then caused to adhere to the desired articles by means of adhesives such as glue. Porous rubber of the type described in B.P. 240,430 (B., 1925, 1000) is very suitable.

D. F. Twiss.

Treatment of subdivided materials (B.P. 298,172). —See I.

# XV.-LEATHER; GLUE.

# PATENTS.

**Production of leather.** A. EHRENREICH (B.P. 299,599, 11.10.27).—The great intestine and the wall of the stomach of chondropterygii, plagiostomi, and other similar fishes are prepared, cleaned, and treated (a) for 2—4 days with a lime liquor containing 0.1-0.2% of carbolates and/or their derivatives, then with a pickle, and tanned with basic chrome liquors or with vegetable or other tanning materials to produce leather, or (b) fulled with fish oil and sea salt to produce drum skins.

D. WOODROFFE.

Treatment of leather. A. O. T. BEARDMORE (B.P. 300,077, 3.4.28).—Vegetable-tanned sole leather hides, particularly those with grain blemishes, or other tanned skins are moistened so as to contain 20-25% of water, placed in a closed room at ordinary temperature and free from air currents, left to condition, sprayed on the surface with a mixture of mineral and vegetable oils at  $60^\circ$ , rough rolled, and finally compressed while still moist at  $27-45^\circ$  and under 12,000 lb./in.<sup>2</sup>

D. WOODROFFE.

Production of solid glue and gelatin in dropor lens-shape. C. GREINER (B.P. 287,866, 17.12.27. Ger., 28.3.27).—Drops of liquid gelatin or glue are arranged to fall on to a conveyor, preferably an endless band of metal gauze, through which drying air may be passed, and conveyed through freezing and thawing zones, arranged alternately, then scraped off the band, conveyed to a drying chamber, and dried with hot air. D. WOODROFFE.

Filler for acetylene containers (B.P. 299,681).— See II.

# XVI.—AGRICULTURE.

Suggested nomenclature and classification of Dutch soils. D. J. HISSINK (Mem. Com. Internat. de Pedologie Helsingfors, 1924; Proc. Internat. Soc. Soil Sci., 1928, 3, 289—290).—The general classification of mineral soils into clays, loams, sands, and combinations of these is amplified by considerations of mechanical analysis,  $p_{\rm H}$  values, and the contents of chalk and exchangeable calcium. Peat soils are classified primarily as high-moor, low-moor, and transition soils, and are further characterised by the contents of organic matter and lime as well as of the nitrogen and phosphorus of the organic material. Mixed soils are classified according to their content of clay, sand, humus, chalk, exchangeable calcium, and  $p_{\rm H}$  value. A. G. POLLARD.

Soil acidity. C. R. RUNK (Delaware Agric. Exp. Sta. Bull., 1927, No. 152, 6—7).—Delaware soils have a wide range of acidity. A  $p_{\rm H}$  value of 5·4—6·5 is optimal for lucerne. Experiments on lime requirement and

the effect of continued fertiliser treatment on reaction are described. CHEMICAL ABSTRACTS.

Significance of the determination of soil acidity in agricultural practice. D. J. HISSINK (Rev. Internat. Rens. Agric. N.S., 1924, 2, 827—830; Proc. Internat. Soc. Soil Sci., 1928, 3, 274).—Comber's method for determining soil acidity gives results in general accord with direct measurements of hydrogen-ion concentration, and if the colour measurements are previously standardised by means of the hydrogen electrode the method is suitable for general use among farmers.

A. G. POLLARD.

Important physical quality of a heavy soil in natural deposits. KÖTTGEN and KLITSCH (Forstwiss. Cent., 1927, 705—720; Proc. Internat. Soc. Soil Sci., 1928, 3, 252).—Mechanical analysis of soils fails to give a true representation of the true state of aggregation of soils and subsoils. Measurements of water percolation are indicative of soil pore-space and afford a single-value determination of soil structure. Changes in structure resulting from alterations in soil reaction are of practical importance, and are not detectable by the customary sedimentation processes.

A. G. POLLARD.

Influence of various forest types on nitrate content and formation in forest soils. A NEMEC and K. KVAFIL (Z. f. Forst- u. Jagdwesen, 1927, 322-412; Bied. Zentr., 1928, 57, 487-489).-The nitrate content of different soil horizons under various types of forest was studied, and nitrifying ability correlated with the type of vegetation. Nitrifying ability was determined from the rate of accumulation of nitrate in samples of soil. Nitrate content varied with the depth of the horizon, surface litter and mould being generally richest. In both coniferous and deciduous forests free from undergrowth there was little or no nitrification at any horizon, and even a tendency to denitrification when the surface layer was highly acid. The existence of an undergrowth was accompanied by considerable nitrifying power in the humus layers; certain plants were suggested as indicators of nitrifying ability in forest soils. In clearings there was a striking increase in nitrifying ability, attributed to the accelerating action of direct sunlight on the decomposition of litter material.

H. L. RICHARDSON. Utilisation of phosphates by rye seedlings in different soils. S. GERICKE (Illustr. Landw. Ztg., 1927, 47, 396; Proc. Internat. Soc. Soil Sci., 1928. 3, 276—277).—In general, the smallest proportional utilisation of phosphate occurred in soils of low phosphorus content, the mean values for marsh soils being  $4 \cdot 1\%$ , sandy soils  $7 \cdot 5\%$ , and humus sands  $14 \cdot 0\%$ .

A. G. POLLARD.

Influence of various fertilisers on the manurial action of "insoluble" phosphate. A. RINDELL (Nordisk Jordbrugsforskning, 1921—2, 3—4, 1; Bied. Zentr., 1928, 57, 491—492).—In pot experiments other salts increased or decreased the manurial action of difficultly soluble phosphate in accordance with their effect on solubility, as predicted by physico-chemical considerations. In the field there was no positive or negative effect attributable to solubility, but some agreement between the manurial action of phosphates and the changes in reaction of the soil solution produced by longcontinued application of physiologically acid or basic fertilisers. H. L. RICHARDSON.

Determination of phosphoric acid requirements of field soils. G. HAHN (Botan. Arch., 1927, 20, 223; Bied. Zentr., 1928, 57, 490—491).—The phosphoric acid requirements of 30 soils were determined by Lemmermann's (relative solubility) and Neubauer's (seedling) methods and by treatment with carbonic acid. Neubauer's method gave results that did not agree with those obtained by the other two. The author concludes that no method gives trustworthy numerical values, and that, until the seedling method is further improved, Lemmermann's is a simpler and quicker way of getting an idea of the phosphoric acid requirement of a soil.

H. L. RICHARDSON.

Determination of phosphates [in soil extracts] by the phosphomolybdate method. O. ARRHENIUS (Arch. Suikerind. Ned. Indië, 1927, 2, 903; Proc. Internat. Soc. Soil Sci., 1928, 3, 247).—10 c.c. of a 2%citric acid extract of soil are diluted to about 80 c.c. To this are added 1 c.c. of concentrated sulphuric acid, 5 c.c. of ammonium molybdate solution (25 g. of ammonium molybdate dissolved in 300 c.c. of water to which are added 25 c.c. of concentrated sulphuric acid diluted with water to 200 c.c.), 1 c.c. of sodium sulphite solution (20 g. of sodium sulphite in 80 c.c. of water), and 1 c.c. of quinol solution (0.5 g. per 100 c.c., plus 1 drop of sulphuric acid). The whole is diluted to 100 c.c. and after 24 hrs. the colour is compared with a series of standards containing 0.03—0.003 mg. P<sub>2</sub>O<sub>5</sub> per 100 c.c. A. G. POLLARD.

Action of alkali chloride on plants and soils. C. DUPONT (Ann. Sci. Agron. franç. et étrang., 1924, 41, 369; Bied. Zentr., 1928, 57, 492—493).—A discussion of the action of added chlorides on alkali soil with especial reference to the use of sylvinite. The interaction of the potassium chloride in the sylvinite with soil calcium is said to lessen the harmful action of the sodium chloride it contains, but harm may result if the soil is poor in lime and is treated with an excessive amount of sylvinite. Certain precautions necessary in its application are mentioned. H. L. RICHARDSON.

Sodium salts, used in conjunction with potash, as a plant food. H. HEINRICH (Z. Pflanz. Düng., 12A, 252; cf. B., 1928, 343).—A correction.

A. G. POLLARD.

Influence on the development of plants of zinc and lead precipitated on the soil from factory fumes. H. LUNGEGÅRDH (Aveldeln. f. Landbruksbotanik, 1927, No. 42; Proc. Internat. Soc. Soil Sci., 1928, 3, 272).—No toxicity resulted from the presence of zinc in soils at lower concentrations than 0.027%. Germination tests with oats, wheat, and clover in nutrient solutions containing zinc sulphate at concentrations between N/300,000 and N/60,000 showed in some instances a weak stimulation and in others a slight checking of germination. Oats germinated normally in N/3000 zinc sulphate solutions. Growth of plants in culture solutions showed slight injury at concentrations of N/500,000 for zinc and N/5,000,000 for lead. These metals are assumed to form nearly insoluble compounds in soil.

A. G. POLLARD.

Influence of the factor "soil" in cultural and manurial experiments. R. STORP (Diss. Danzig, 1928; Proc. Internat. Soc. Soil Sci., 1928, **3**, 286).— The value of examining soil profiles in field experiments is emphasised. Conditions favourable to plant growth are : high humus content and considerable depth of the A-horizon; compact formation in the B- and C-horizons, ample humus, subsoil water, and capillary water in Band C-horizons. A. G. POLLARD.

Effect of manurial constituents on the quality of sugar-cane juice and gur. P. B. SANYAL (Agric. J. India, 1928, 23, 277–286).—Juices and gurs of the highest purity and sucrose content were obtained (in the case of cane variety Co. 213) when superphosphate and potassium sulphate had been used as manure; organic nitrogen (as mustard cake) gave intermediate results, whilst cyanamide and ammonium sulphate gave the lowest results of all. A superior quality of gur was produced from juices clarified by Horne's " superdefecation " process (B., 1924, 685). J. P. OGILVIE.

Nitrogen question in the sugar industry of Java. O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, No. 3; Proc. Internat. Soc. Soil Sci., 1928, **3**, 284—285).—From sand-culture experiments it is calculated that the nitrogen requirement of sugar cane is 620—800 lb. per acre. Nitrogen assimilation increases with the nitrogen concentration of the nutrient solution. It is probable that the cane can utilise ammoniacal nitrogen. Cane soils in Java contain little or no nitrite even when waterlogged. The  $p_{\rm H}$  value of these soils has no influence on nitrate production. A. G. POLLARD.

Influence of concentrations of chlorine on development of the sugar cane. O. ARRHENUS (Arch. Suikerind. Ned. Indië, 1928, 1, 90; Proc. Internat. Soc. Soil Sci., 1928, 3, 284).—Sugar-cane plants were grown in sand culture with nutrient solutions containing chloride. Injury was observed with concentrations of more than 0.060% of chlorine. Remedial measures consist essentially of leaching. A. G. POLLARD.

Relation between different properties of sugarcane soils. O. ARRHENIUS (Arch. Suikerind. Ned. Indië, 1927, 2, 1197; Proc. Internat. Soc. Soil Sci., 1928, 3, 285).—A relationship between soil acidity and phosphate content is attributed to the different solubilities of mono-, di-, and tri-phosphates. The phosphate content of the soil influences nitrification. A. G. POLLARD.

Soil acidity and sugar cane. O. ARRHENIUS (Arch. Suikerind. Ned. Indië, 1927, 2, 1027; Proc. Internat. Soc. Soil Sci., 1928, 3, 285).—Optimum yields of cane are produced on neutral soils. A. G. POLLARD.

Noxious effect of molasses on soil. O. AREHENIUS (Arch. Suikerind. Ned. Indië, 1927, 2, 791; Proc. Internat. Soc. Soil Sci., 1928, 3, 284).—Injury to Amarantus tricolor, L., soon after the application of molasses to the soil was observed. The period of injury was shorter in sandy soils than in clays, and is attributed to the withdrawal of nitrate from the soil by organisms fermenting the molasses. A. G. POLLARD.

Red- and sheep's fescue. K. BÄR (J. Landw., 1928, 76, 255-284).—The dependence on external conditions of the growth, form, colour, and general botanical char-

acteristics of the fescues is examined. The digestibility of the protein of the stems of the grasses is less than that of the panicles, but in all cases is independent of the total protein content. A. G. POLLARD.

Vitamin content of silo juices. F. DUSCHEK (J. Landw., 1928, 76, 197—216).—The vitamin content of the juice from Serradella silage was demonstrated by the live weight increases and improved conditions of health of rats previously fed on a diet deficient in vitamins. With the freshly-pressed juice from maize and red clover silage under similar conditions no beneficial effects were observed in growth and development, but health conditions were improved. Neither effect was apparent when juices from cold-pressed silos were used. Losses of valuable food material in electrical ensilage practice can be avoided by the incorporation of the juices with absorbent material which can subsequently be fed to cattle.

A. G. POLLARD.

Insecticides. PEET.-See XXIII.

#### PATENTS.

Polyhydroxy-[organo-]mercury compound. H. GÜNZLER and O. NEUBERT, ASSIS. to WINTHROP CHEM. Co., INC. (U.S.P. 1,677,823, 17.7.28. Appl., 5.5.27. Ger., 20.1.26).—Colourless, odourless, stable products having insecticidal and fungicidal properties are obtained by dissolving in water a mixture of a polyhydroxybenzene (resorcinol, 2 : 6-dihydroxytoluene, pyrogallol) and mercuric chloride. C. HOLLINS.

Mixed crystals containing potassium and ammonium (B.P. 299,984). Fertiliser (U.S.P. 1,686,873).—See VII.

#### XVII.—SUGARS; STARCHES; GUMS.

Frothing of [sugar] juice during carbonatation in the beet sugar factory. V. STANĚK and J. VONDRÁK (Z. Zuckerind. Czechoslov., 1928, 53, 81–98).—Various vegetable and mineral oils were found to exert a varying effect on the abatement of froth in a miniature carbonatation apparatus designed closely to imitate the conditions of practice. It was shown that the optimum effect is obtained with such substances in extremely small amount, viz., only 0.2 mg. per litre of juice. Rape oil in the form of emulsion (0.1-5%) gave the same result as its direct application. J. P. OGILVIE.

Final saturation of [sugar] thin-juice. O. SPENGLER and C. BRENDEL (Z. Ver. deut. Zuckerind., 1928, 637-643). —To obtain juice low in calcium the second carbonatation is carried out so as to saturate the natural alkalinity, after which follow successively heating, filtration, sulphitation almost to neutrality at high temperature, and a second filtration. At this stage sodium carbonate is added in amount corresponding to the content in calcium present, the juice being lastly boiled and filtered. If at the syrup stage the alkalinity is too high it can be reduced by sulphiting. J. P. OGILVIE.

Automatic control of the carbonatation process of beet-sugar manufacture. R. T. BALCH and J. C. KEANE (Ind. Eng. Chem., 1928, 20, 1148—1151).— In automatically controlling second carbonatation, use was made of (1) a standard saturated calomel half-cell, contact with the juice being made through a porous cup filled with potassium chloride, and (2) a bare tungsten wire electrode. The electrodes were suitably arranged in a glass flow-chamber through which a continuous sample of the juice was passed. A temperature compensator, the purpose of which was to correct automatically for the changes in  $p_{\rm H}$  caused by temperature fluctuations, was also inserted in the flow-chamber. A potentiometer was in combination with a device for controlling the reversing motor operating the gas valve of the tank, this being gradually opened when the reaction of the juice was above the selected  $p_{\rm H}$  and gradually closed when falling below. Given means for delivering sulphur dioxide to a tank under as constant conditions as in the carbonatation system, sulphitation could be subjected to a similar automatic control.

#### J. P. OGILVIE.

Incrustations in the carbon dioxide piping [of beet sugar factories]. O. SPENGLER and C. BRENDEL (Z. Ver. deut. Zuckerind., 1928, 644—645).—Analysis of a greyish incrustation taken from the valves of the carbon dioxide pumps in two beet factories gave, respectively, organic matter 14.5, 24.4; sand and insoluble 0.2, 0.6; iron 2.5, 1.1; copper 0.6, none; potassium and sodium 35.0, 30.9; chlorine 38.3, 33.3; and  $SO_4$ 5.1, 6.3%. J. P. OGILVIE.

Determination of solubility of sucrose in beethouse syrups. R. J. BROWN, J. E. SHARP, and H. W. DAHLBERG (Ind. Eng. Chem., 1928, 20, 1230—1232).— Using a low-purity beet molasses of known composition, various solutions of known impurity-water ratios were prepared, these being saturated with sucrose at the proper temperature without any loss of water. Then the purity and sucrose contents were calculated from their dry substance values, and the results plotted. A table is given showing the concentration of saturated beet syrups from 40° to 80°, and with impurity-water ratios from 0 to 1.378. J. P. OGILVIE.

Cane sugar manufacture in Java. Centrifugal treatment of syrups. P. HONIG and W. F. ALEWYN (Arch. Suikerind. Neder.-Indië, 1928, 36, 330—370).— Experiments were made to ascertain at which stage centrifugal clarification could be applied, a De Laval machine (No. 300), 16.5 cm., being used. It was concluded not to be practicable to work with the juice, raw or clarified, as later, owing to evaporation, a further precipitate is formed. Good results, however, were obtained at the syrup stage, solids to the amount of 15 g./litre being separated. Molasses was also treated to advantage, and also the wash-liquor from the whitesugar centrifugals. J. P. OGILVIE.

Application of clarification after instead of before evaporation. B. J. VAN SANTEN (Arch. Suikerind. Neder.-Indië, 1928, 36, 579-581).—Unclarified sugar juice was evaporated to syrup (62° Brix) and clarified in this concentrated state. If generally practicable on the large scale, this procedure would be preferable to the present procedure, since working would be more rapid, scaling in the evaporators would probably be avoided, and a better product by crystallisation from a clearer syrup might result. J. P. OGILVIE.

 $p_{\rm H}$  value of sugar media and their power of

inversion. H. COLIN and A. CHAUDUN (Bull. Assoc. Chim. Sucr., 1928, 45, 626-642).—A lecture. The chief methods of determining the  $p_{\rm H}$  of media are mentioned and its importance in sugar manufacture is discussed. Although the values form a useful guide, simple determination of  $p_{\rm H}$  does not supply a solution to the difficulties of the manufacturer since the complex phenomena forming the basis of sugar technique do not depend exclusively on hydrogen-ion concentration. The optimum values in various stages of manufacture vary from week to week since the different batches of raw material (sugar beet) are not uniform. The quantities of reducing sugars formed during the heating of solutions are unimportant so long as the  $p_{\rm H}$  does not fall below 6.5; after this they increase rapidly.

H. INGLESON.

Inversion of sucrose by weak acids. V. ZAFOUK (Z. Zuckerind. Czechoslov., 1928, 53, 33-42).—Under like conditions regarding molar concentration, temperature (70°), and hydrogen-ion concentration, aspartic acid hydrolyses sucrose more rapidly than does succinic acid. Glutamic acid, on the other hand, has the same rate as succinic acid at  $p_{\rm H} 4.5$ , but this increases with rising  $p_{\rm H}$ , until at  $p_{\rm H} 5.1$  it is the same as in the case of aspartic acid. At a still higher  $p_{\rm H}$ , glutamic acid inverts more rapidly than aspartic acid. At  $p_{\rm H} 5.5$ succinic acid inverts insignificantly, and at  $p_{\rm H} 5.7$ aspartic acid also practically ceases to hydrolyse.

J. P. OGILVIE. Chemical composition and physical properties of beet molasses. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1928, 53, 1—11).—Average figures for beet molasses of the 1925—6 campaign in Czechoslovakia, France, and Germany, respectively, are : dry substance (refractometric) 78.5, 78.3, 76.9; direct polarisation 51.26, 50.45, 50.40; apparent quotient of purity 65.35, 64.50, 65.50; ash 10.14, 8.69, 8.27; total non-sugars 30.19, 27.85, 28.14; and nitrogen 1.64, 1.63, 1.44%. J. P. OGILVIE.

Testing decolorising media. LINSBAUER and VAŠÁTKO.—See II. Effect of manures on sugar cane. SANYAL. Sugar industry of Java. Development of sugar cane. Sugar-cane soils. ARRHENIUS. —See XVI.

#### PATENTS.

Oxidation of aldoses. CHEM. FABR. VORM. SANDOZ (B.P. 293,322, 2.7.28. Ger., 2.7.27. Addn. to B.P. 289,280; B., 1928, 461).—The cooling necessary in the process of the prior patent is avoided by treating dextrose in aqueous solution containing a little sodium carbonate and sodium bromide at 50—55° with chlorine, calcium carbonate being added from time to time to maintain alkalinity; calcium glutaconate crystallises out. Lactose similarly gives calcium lactobionate.

# C. HOLLINS.

Automatic apparatus for the continuous production of extracts, particularly of sugar contained in sugar plants. Soc. ANON. DES ÉTABL. A. OLIER (B.P. 286,668, 29.2.28. Fr., 8.3.27).

Extraction of solid materials (B.P. 298,501).— See I.

#### XVIII.—FERMENTATION INDUSTRIES.

Brewers' pitch [for lining casks]. F. KUTTER (Woch. Brau., 1928, 45, 473-478, 483-487, 493-497, 503-506, 517-521).-The Klinger-Lampe and Hoffmann-Herbst methods, and that depending on observing the fall of a ball through a disc of the heated material, all yield reproducible results for softening and melting points, but do not agree among themselves. A simplification of the Hoffmann-Herbst method, in which the rate of sinking of a brass rod through the heated material is measured, is recommended. The viscosity should increase slowly and uniformly as the liquid cools, and solidification should be gradual. A high coefficient of expansion favours the formation of cracks. In these respects rosin pitch is superior to paraffin. Compositions were intermediate in behaviour and showed no appreciable difference from rosin pitch when roughly tested as to their behaviour under impact. Rosin oil lowers the flash point, and both it and paraffin increase the proportion of neutral substances extractable by ether from an alkaline emulsion. The "oxidation value," (c.c. of N-permanganate reduced by 1 g. of the material) is best determined under standardised conditions on a solution in glacial acetic acid, the precipitated manganese oxides being dissolved by hydrogen peroxide, the excess of which is titrated with N-permanganate. In applying the evaporation test (cf. Brand, B., 1900, 761) the loss, though greater at first, soon becomes proportional to the time, and 5 hrs.' heating gives a sufficient indication of the changes which occur. The loss of weight and rise of m.p. are generally greater and the increase of viscosity is less in rosin pitches than in compositions containing a reasonable proportion of paraffin. An aeration test is described in which 400 g. of the material are placed in a covered nickel beaker, 8 cm. wide and 10 cm. high, which slides into a similar but slightly wider beaker heated in a paraffin bath. The cover carries a thermometer and an air inlet tube, and is provided with a hole of 1 cm.<sup>2</sup> area as an outlet for air and vapour. The tube is 6 mm. in diam., and slightly tapered at the end, which is placed 1 cm. from the bottom of the beaker. Air is passed continuously during heating, at 9-10 litres/hr. It is claimed that the changes undergone by a composition during this treatment are a good indication of those occurring in practice, whilst those which take place during heating without aeration have no comparative value. F. E. DAY.

Defective attenuation of the mash. E. LÜHDER and B. LAMPE (Z. Spiritusind., 1928, 51, 335-336).—Many worts from mashes made with malt and potatoes show a low final percentage of attenuation at the conclusion of the fermentation. This is chiefly due to a deficiency in the content of amylase in the malt, and can be alleviated by the addition to the mash of a relatively greater quantity of malt. The deficiency in attenuative properties may also be brought about if the mash is made of too high a concentration, whilst, among other contributory causes, is the use of a yeast infected with bacteria. The presence of the bacteria results in the excessive production of acidity, which tends to inhibit the action of the amylase and to weaken the yeast.

C. RANKEN.

Conditions for the aeration of fermenting vats in the yeast industry or for the aeration of liquids in general. E. G. STICH (Chem.-Ztg., 1928, 52, 865—866). —The efficient aeration of liquids is discussed. Important factors are the ratio of the total area of the air passages in the aeration apparatus to that of the vat floor, and also of the total surface area of the bubbles to the required air content of the liquid.

F. R. Ennos.

Clarification of natural sweet wines by centrifuging. J. DUBAQUIÉ (Ann. Falsif., 1928, 21, 460– 463).—Although rapid and satisfactory clarification of Sauterne wines by centrifuging was obtained the effect was not perfect, and at the end of a year control samples were equally clear. The violent mechanical action appears to affect the colloidal condition of the product, certain chemical precipitations are accentuated, particularly that of cream of tartar, and the degree of flocculation of various ingredients is altered. The use of the centrifuge, at least for the more delicate wines, is not recommended. D. G. HEWER.

Determination of lævulose and dextrose in sweet wines. F. LUCIUS (Pharm. Zentr., 1928, 69, 725-727). --Comparison is made of results obtained by the usual methods applied to various wines with those obtained by calculation from the rotations before and after inversion. S. I. LEVY.

Effect of successive generations of yeast on the alcoholic fermentation of cider. S. C. VANDECAVEYE (J. Agric. Res., 1928, 37, 43-54).—The problem of preparing unfermentable fruit juices which can be sold as non-alcoholic beverages is one of some moment in America. By coagulating freshly pressed cider at  $45^{\circ}$  and growing three successive generations of yeast in the liquid after removing the coagulum it is possible to obtain a temporarily unfermentable liquor possessing the unchanged taste and palatability of cider and containing 0.53% of alcohol. The inhibition of fermentation is due to the removal of the nitrogen and phosphorus. H. J. G. HINES.

Sauerkraut. PREUSS and others.—See XIX. Pharmaceutical preparations. PLACERES.—See XX.

#### PATENT.

Manufacture of yeast. A. J. C. OLSEN, and INTER-NAT. YEAST CO., LTD. (B.P. 299,336, 21.6.27).—In the process three or more fermenters communicating in series are used. In each of the fermenters following the first which are traversed by the incompletely treated or unripe yeast-containing liquid, the environment of the yeast is controlled by the continuous addition of yeast nutrients, with or without added seed yeast, so as to maintain constancy of quality of the final yeast, which is withdrawn from the final fermenter at a rate sufficient to prevent the intermediate fermenters from becoming overfilled. Spare fermenters may be used to shortcircuit any of the series of fermenters for the purpose of cleansing. C. RANKEN.

# XIX.-FOODS.

Determination of fat in cocoa products. A. HEIDUSCHKA and F. MUTH (Chem.-Ztg., 1928, 52, S79). —An apparatus suitable for very rapid, accurate determinations at laboratory temperature consists of a Schott

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glass filter-crucible which, after weighing with the material, is secured by means of a rubber band in an adapter carrying a ground-glass joint-piece, by which it is fitted to a flask which has a side tube for attachment to a pump during the extraction and to a condenser during removal of the solvent. S. I. Levy.

Gas production in the making of sauerkraut. L. M. PREUSS, W. H. PETERSON, and E. B. FRED (Ind. Eng. Chem., 1928, 20, 1187—1190).—The gas evolved during the formation of sauerkraut is almost 100% carbon dioxide, and most of the gas formed is given off within 40—160 hrs. after the cabbage is packed into the container. At higher temperatures gas and acid production increase together with a rapid increase in the number of bacteria, and the conclusion is reached that the gas production is due to bacterial activity and not to yeast growth or plant-cell respiration. Washing the raw cabbage before cutting and packing evidently results in a better flavoured sauerkraut. E. H. SHARPLES.

Iron and manganese content of foodstuffs. J. T. SKINNER and W. H. PETERSON (J. Biol. Chem., 1928, 79, 679—687).—Figures are given for the iron and manganese content of a number of (cattle) foodstuffs; in general, the content of iron is about three times that of manganese. C. R. HARINGTON.

Nutritive value of hardened oils. UENO and others.—See XII. Vitamin content of silo juices. DUSCHEK. Red- and sheep's fescue. BÄR.—See XVI.

# PATENTS.

Recovery of alcohol [from the vapours from bakehouse ovens etc.]. N. NAVROTZKY (F.P. 672,889, 22.1.27).—The gases and vapours from ovens containing fermented dough etc. are passed through cooling coils arranged to condense alcohol and water separately, the water being returned to the ovens. The withdrawal of the gases and vapours from the ovens and the supply of water to the coolers are regulated automatically.

L. A. Coles.

Preventing the blackening of tinned foods. K. OSHIMA (U.S.P. 1,686,393, 2.10.28. Appl., 19.2.27. Jap., 1.3.26).—An acidity up to  $p_{\rm H}$  4 is maintained by adding a mixture of a weak organic acid and salt thereof, *e.g.*, a mixture of acetic acid and sodium acetate, to the food before canning. Sodium chloride is also preferably added. F. G. CLARKE.

Margarine composition. J. C. SHERMAN, Assr. to BROWN CO. (U.S.P. 1,691,087, 13.11.28. Appl., 9.12.22). —See Can.P. 259,391; B., 1927, 265.

Drying machines (B.P. 299,134).-See I.

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Preparation and stability of tincture of iodine. P. BOHRISCH (Apoth.-Ztg., 1928, 43, 436—440; Chem. Zentr., 1928, i, 2514—2515).—The directions of the German pharmacoposia (6th ed.) for the determination of potassium iodide and iodine are criticised. The stability of the tincture is unaffected by the substitution of potassium iodide by bromide, or by the use of 70% alcohol. A. A. ELDRIDGE.

Mercuric iodide determination in tablets. H. O. Moraw (J. Amer. Pharm. Assoc., 1928, 17, 1084–1086). -A weighed quantity of the finely-powdered sample equivalent to 0.25 - 0.40 g. of mercuric iodide is well mixed with about 0.2 g. of lactose, and the mixture made into a smooth paste with 2-5 c.c. of 10% sodium hydroxide solution and then an additional quantity is added to make a total of 30 c.c. About 40 c.c. of water are added, and after digesting gently over a low flame for 30-50 min. the mixture is filtered and the residue 5 c.c. of 10% silver nitrate solution are washed. added to the filtrate and then 25 c.c. of concentrated nitric acid. The mixture is gently boiled until the liquid becomes clear and the silver iodide is collected on a filter and weighed. Intimate mixing with the lactose is necessary to ensure complete reduction of the mercuric iodide and to prevent losses due to occlusion and incomplete peptisation. The limits of accuracy of E. H. SHARPLES. the method are within 1%.

Determination of chloral in syrup of chloral. P. ANDRON (J. Pharm. Chim., 1928, [viii], 8, 453-455).— A reply to the statements of François (B., 1928, 243), which are shown to be incorrect. E. H. SHARPLES.

Detection of lead and copper in citric acid, tartaric acid, and extracts. G. FRERICHS (Apoth.-Ztg., 1928, 43, 513—514; Chem. Zentr., 1928, i, 2850— 2851).—The methods of the German pharmacopœia, 6th ed., are criticised. A. A. ELDRIDGE.

Pharmaceutical preparations containing lactic acid organisms. J. PLACERES (Anal. oficina quim. prov. La Plata, 1927, 1, 71–89).—When incubated at 37° for 48 hrs. with 100 c.c. of 3% lactose milk medium, the fluid preparations (1 c.c.) produced larger amounts  $(11 \cdot 70-20 \cdot 61 \text{ g./litre})$  of lactic acid than the solid preparations ( $0 \cdot 5 \text{ g.}$ ). Formic acid ( $0 \cdot 014-0 \cdot 081\%$ ) and acetic acid ( $0 \cdot 123-0.945\%$ ) were also formed, but butyric acid, aldehydes, and acetone were not observed. CHEMICAL ABSTRACTS.

Sodium phenylethylbarbiturate. H. SANDQVIST and T. H. LINDSTRÖM (Arch. Pharm., 1928, 266, 613— 616).—Commercial samples of sodium phenylethylbarbiturate have been found to contain water and as much as 5% of alcohol, which is removed only by long heating at 140—150°. The pure substance gives a red colour with phenolphthalein but not with thymolphthalein, although the latter is coloured blue by many commercial products. The organic matter in this compound is satisfactorily determined by acidification, evaporation to dryness, and extraction with ether. The results obtained by extracting an acidified, aqueous solution are not reliable. S. COFFEY.

Chemical and pharmacological examination of the leaves of Atropa belladonna, L., and of the extract prepared from them. T. EXLER (Pharm. Weekblad, 1928, 65, 1152—1177).—Examination of the leaves by the methods of the German, United States, Swiss, and Dutch Pharmacopeias gave for alkaloid content the values 0.86, 1.13; 0.64, 0.62; 0.54, 0.56; and 0.52, 0.52%, respectively. Using the same methods, but with increased amounts of solvent for extraction, the German method gave 0.92, 0.74%; the others gave values very close to those obtained previously. Errors in the German method are pointed out. The worst error arises from the method of distilling the

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ether solution before titrating, in which the solution is evaporated to one third of its bulk; ammonia and other volatile alkaline substances remain behind. If the solution is evaporated to dryness and the residue twice damped and heated, concordant and accurate results are obtained. The American method is also criticised. The methods of preparing the extract are discussed, and the differences in pharmacological properties observed are traced to racemisation and hydrolysis of the alkaloids during extraction. Gravimetric determination of the alkaloids by the silicotungstate method gives results always lower than the titration method. Pharmacological tests were carried out quantitatively by determining the amount of alkaloid necessary to counterbalance the contraction caused by pilocarpine in the intestine of the cat and rabbit. Tropine has no effect; 2 pts. of hyoscyamine have an effect greater than that of 3 pts. but less than that of 4 pts. of atropine. Hydrolysis of the alkaloids, which produces tropine, therefore lowers the pharmacological activity as well as the rotatory power. S. I. LEVY.

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Fluid extracts. II. Fluid extract of Hydrastis canadensis. A. ALBANESE and A. PEDRONI (Annali Chim. Appl., 1928, 18, 429-447).—The qualitative reactions, identification of the active principles, and the quantitative examination of liquid extract of Hydrastis canadensis are discussed, and modifications in the details of certain analytical procedures suggested. (Cf. Belloni, B., 1919, 438 A.) T. H. POPE.

Stability of homatropine hydrobromide solution. P. S. PITTENGER and J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1928, 17, 1081—1084).—Solutions of homatropine hydrobromide of varying concentrations have been examined physically and physiologically after storage under different conditions and exposure to ultra-violet light. Sterilisation or exposure to ultraviolet radiation has no apparent deleterious effect on the mydriatic action of homatropine hydrobromide either in distilled water or in sodium chloride solution isotonic with the tear. Similar solutions preserved in ampules for 9 months showed no loss in mydriatic action. E. H. SHARPLES.

Technical isolation of scopolamine. F. CHEM-NITIUS (J. pr. Chem., 1928, [ii], 120, 221-224).—The isolation, on the technical scale, of scopolamine from the roots of various *Solanaceæ* by hydrolysis with 10% sodium hydroxide solution, extraction with ether from which the base is removed with acetic acid, isolation with potassium carbonate, and purification through its hydrobromide, is described. J. W. BAKER.

Keeping properties of opium powder. A. JERM-STAD (Pharm. Zentr., 1928, 69, 693-694).—Samples of opium powder preserved in dry, well-closed bottles for 10 years showed diminutions of morphine content of only 0.32-0.38% of the actual content. S. I. LEVY.

Denicotinised tobacco. E. M. BAILEY, O. L. NOLAN, and W. T. MATHIS (Conn. Agric. Exp. Sta. Bull., 1928, No. 295, 338–351).—" Denicotinised " tobacco varies considerably in nicotine content. Some denicotinised tobaccos contained as much nicotine as is likely to be present in ordinary tobacco. The smallest nicotine content was 0.75%; ordinary tobacco may contain

as little as 1%. Nitrate- and ammonia-nitrogen are present to the same extent in both cases.

CHEMICAL ABSTRACTS.

Seed oil of Citrus limomum. F. OCCHIPINTI (Atti II Cong. Naz. Chim. Pura Appl., 1926, 945—950; Chem. Zentr., 1928, i, 2884).—The seed oil of Citrus limomum has  $d^{15}$  0.922, f.p. — 6°,  $n_D^{\circ o}$  70.8, Tortelli value 83°, acid value 21.1, saponif. value 194.6, ester value 173.5, iodine value 103.5, Hehner value 94.5, Reichert-Meissl value 0.5, unsaponifiable matter 0.5, drying value (Livache) 5%. The fatty acids (25% solid) have m.p. 41.5°, acid value 202.0, acetyl value 33.0, iodine value 114.0, mol. wt. 277.7. The solid acids consist of stearic (30%) and palmitic (70%), and the liquid acids of oleic and linoleic acids.

A. A. ELDRIDGE.

Oil of Salvia sclarea. E. KOPP (Pharm. Zentr., 1928, 69, 677–680).—The oil should be prepared from the peduncles, the leaves containing very little; peduncles from Rumanian plants yielded 0.15% of oil. Tables of the physical and chemical properties of oils from various sources are given, and show very wide variations. The Rumanian oil contains 41.8-59.2% of linally acetate and 13.8-26.5% of the free alcohol. S. I. LEVY.

Vibration theory of odour. G. M. Dyson (Perf. Ess. Oil Rec., 1928, 19, 456-459).-The relationship between vapour pressure and odour is discussed and the following theory for the sensation of odour is propounded. The odorous molecule forms a loose absorption system with the protein molecules of the osmic cells and absorbs radiant heat energy. This causes displacement of one or more electrons from their normal orbit. When the electrons revert to their normal positions the energy released as wave motion of a definite frequency is absorbed by the sensory apparatus and leaves the system as a nervous impulse. A scale of frequencies may be associated with osmic perception, each frequency being associated with a definite osmic sensation. An odour may be simple and depend on the emission of a wave motion of definite frequency, or it may be complex and arise from a series of waves of different frequencies analogous to simple and compound colours and pure tones and chords. The improvement in odour of certain unpleasant substances on extreme dilution is probably due to the "critical threshold concentration," or the limiting concentration at which osmic frequency can be physiologically detected, differing with the frequency, and the pleasant" frequency is still observable at a dilution where the "unpleasant" frequency has ceased to be detectable. E. H. SHARPLES.

Cresol-naphthene soap solutions. Kogan.—See XII.

# PATENTS.

Manufacture of chloral. Soc. D'ÉLECTROCHIM., D'ÉLECTROMÉTALLURGIE, ET DES ACIÉRIES ELECTRIQUES D'UGINE (F.P. 612,396, 27.6.25).—Chlorine is passed briskly into a mixture, cooled at first, of 97% alcohol and acetaldehyde, which is eventually boiled. Chloral alkoxide is formed by way of trichloroacetal. After 30 hrs. aqueous acetaldehyde is added, and a slow stream of chlorine is led into the warm mixture. The product is almost entirely chloral hydrate. A ferric chloride catalyst may be used. C. HOLLINS. Preparation of N-monosubstituted ethylenediamines. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of H. SCHOTTE and H. PRIEWE (G.P. 446,547, 1.7.25).—N-Alkyl- or N-aryl-N-( $\beta$ -hydroxyethyl)guanidines are heated with concentrated hydrochloric acid at 160°. Creatinol is thus converted, by way of 1-methyl-2-ketotetrahydroglyoxaline, into N-methylethylenediamine. N-Ethyl (b.p. about 130°) and N-phenyl (b.p. 261—262°; picrate, m.p. 162°) derivatives are similarly prepared (cf. Schotte and Priewe, A., 1926, 717). The products have therapeutic value. C. HOLLINS.

Manufacture of 3 : 5-di-iodo-2-[hydr]oxypyridine. SCHERING-KAHLBAUM A.-G. (B.P. 288,133, 24.2.28. Ger., 31.3.27).—2-Pyridone is treated with iodine monochloride in dilute hydrochloric acid. C. HOLLINS.

Emetine derivatives for therapeutical purposes. E. ROTHLIN and F. MÜLLER, ASSTS. to CHEM. FABR. VORM. SANDOZ (U.S.P. 1,686,930, 9.10.28. Appl., 22.12.27. Switz., 12.1.27).—Salts of bile acids with emetine, or other cephaline alkyl ethers, have strong antiparasitic action and are soluble in alcohol and nearly insoluble in water. F. G. CLARKE.

Extraction of lupanine. E. von Ammon and K. SZOMBATHY (B.P. 288,637, 12.4.28. Ger., 14.4.27) .--Rough-ground lupin seeds are treated with a solution of a salt (especially calcium chloride) of such concentration that the conglutin remains undissolved. The liquid is treated with sulphuric acid to remove proteins, lupinine, and lupinidine, and then rendered alkaline and the lupanine extracted with benzene, whence it is recovered by crystallisation or treatment with dilute hydrochloric acid, giving the hydrochloride. The pure lupanine thus obtained has m.p. 99°. The hydrochloride is separated by treatment with chloroform into its d-form (which dissolves) and its l-form (insoluble). It is used in the treatment of intestinal diseases. The residue left after extraction of the alkaloids from the seeds as above is re-extracted, and washed with water at 78—82°. The total loss of protein is 2-4%.

B. FULLMAN.

Cleansing nicotine vapours. R. G. MEWBORNE (U.S.P. 1,684,740, 18.9.28. Appl., 19.2.24).—The vapours are scrubbed with a hot, circulating solution of caustic alkali, and the cleaned moist vapour is passed to a condenser to recover free nicotine, or is absorbed in sulphuric acid. The liquid containing free nicotine may be concentrated in a vacuum evaporator and the escaping vapour absorbed in sulphuric acid.

F. G. CLARKE.

Preparation of basic phenol ethers. I. G. FARB-ENIND. A.-G., Assees. of H. HAHL (G.P. 446,606, 8.10.25. Addn. to G.P. 433,182).—The process of the prior patent is extended to alkylated amino-derivatives of higher alkyl halides. The products have strong therapeutic action. 6-Allylguaiacol is heated with alcoholic sodium ethoxide and βγ-bisdiethylaminopropyl chloride, b.p. 87—89°/5 mm. (from the alcohol and thionyl chloride), to give the bisdiethylaminopropyl ether, b.p. 184°/12 mm. Similarly 8-hydroxy-7-allyl-5-methylquinoline reacts with α-chloro-γ-dimethylamino-β-methylbutane, b.p. 52— 54°/18 mm., to form an ether, b.p. 215—220°/10 mm., and 8-hydroxy-7-allylquinoline with ββ'-bisdimethylaminoisopropyl chloride, b.p. 83—84° 20 mm., an ether, b.p. 210—214°/10 mm. C. HOLLINS

Manufacture of musk-scented lactones. HAAR-MANN & REIMER CHEM. FABR. ZU HOLZMINDEN G.M.B.H., Assees. of M. KERSCHBAUM (G.P. 449,217, 6.3.26. Cf. Kerschbaum, A., 1927, 541).-A suitable halogenated aliphatic acid is treated with silver oxide, or a metal salt is heated in an inert solvent or distilled in a vacuum to give a lactone having at least  $C_{12}$  and preferably  $C_{15-16}$ in the ring. The w-hydroxy-acids, especially, separated from vegetable musk extracts, are converted first into w-halides and thence into lactones having an intense musk odour. Juniperic acid [w-hydroxyhexadecoic acid], m.p. 93-94°, thus yields a lactone, b.p. 170-180°/12 mm., by way of ω-iodohexadecoic acid, m.p. 76°; the lactone, b.p. 160-180°/11 mm., of  $\omega$ -hydroxypentadecoic acid, m.p. 83-84°, is similarly obtained from the  $\omega$ -bromo-acid, m.p. 65°.

C. HOLLINS.

Manufacture of water-soluble, complex organic bismuth compounds. B. HEPNER (F.P. 617,063, 4.6.26).—Freshly precipitated bismuth hydroxide, with or without addition of glycerol, is heated with 3 : 3'diamino-4 : 4'-dihydroxyarsenobenzene and mannitol; alcohol precipitates an oily complex bismuth compound, which quickly sets. The formaldehydesulphoxylate of the arsenobenzene behaves similarly, as does 4 : 4'-dihydroxyarsenobenzene. C. HOLLINS.

Production of chyle and manufacture of a medicament therefrom. TORII & Co., LTD., and H. MORI (B.P. 299,530, 2.8.27) .- Fatty substances are given to an animal per os or by a stomachal probe, and a fistula is made in its ductus thoracicus. The lymph is discharged after 2 or 3 hrs., changes to chyle, which is filtered through silk and paper, respectively, and centrifuged. The liquid obtained is sealed in glass ampoules, and kept at 58-60° for 30 min., this process being intermittently repeated for a few days. The milky-white product contains fatty substances (neutra fats and fatty acids), lipoids, and small quantities of albumin, carbohydrates, and internal secretions. It is injected for therapeutic purposes. B. FULLMAN.

Naphthacridine-ms-carboxylic acids (G.P-446,543).—See III.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Negative developers for contrast. J. Southworth (Brit. J. Phot., 1928, 75, 689-691, 706-708).-The fog produced by development may be divided into two parts, viz., intrinsic fog, which is latent in the emulsion before development and is formed during either the ripening process or the subsequent ageing, and extrinsic fog, which is caused solely by the action of the developer. The latter, in absence of bromide, tends to increase more rapidly as development is prolonged. With metol-quinol-carbonate developer, and at high values of gamma, potassium bromide depresses extrinsic fog more than intrinsic fog or the latent image. Small traces of soluble iodide assist the action of potassium bromide, and are probably the cause of the decreased fogging tendencies of used developers. The yellow or reddish stain of colloidal silver produced by contrast developers is due to alternate dissolution and reduction of the silver halide. Bad stain may be obtained with quinol-caustic soda developers if the

negative is not thoroughly rinsed before insertion in the fixing bath. The stain is readily soluble in ferricyanide and thiosulphate reducer. J. W. GLASSETT.

#### PATENTS.

Photographic developers. B. MEREJKOVSKY (B.P. 299,751, 29.6.27. Cf. B.P. 255,925; B., 1926, 805).— Addition of 0.03-0.05% of a secondary amine (piperidine, methyl-*p*-aminophenol) or a primary diamine to the developers of the prior patent counterbalances the retarding effect of the hydroxylaminesulphonic acids whilst retaining the automatic correction of over- or under-exposure. C. HOLLINS.

Manufacture of photographic developers [hydroxyethylated p-aminophenols]. I. G. FARBENIND. A.-G. (B.P. 280,873,17.10.27. Ger., 19.11.26).— p-Aminophenol is heated with ethylene chlorohydrin and sodium acetate to give di- $(\beta$ -hydroxyethyl)-p-aminophenol, m.p. 140°, or is mixed with less chlorohydrin and alcoholic alkali in much alcohol in the cold to give  $\beta$ -hydroxyethylp-aminophenol, m.p. 86—97°. Both may be used as photographic developers, and are stable in alkaline solution. C. HOLLINS.

**Production of coloured photographs.** W. F. Fox, Assr. to NATURAL COLOR PICTURES Co. (U.S.P. 1,685,281, 25.9.28. Appl., 22.6.20).—A positive and a negative image of the object are printed in alinement on a single layer of sensitive emulsion coated on a suitable base, with unaffected emulsion separating the images, the positive being undermost. Both images are toned with the same colour and the emulsion is then hardened in proportion to the depth of tone. Finally the film is treated with a second dye adapted to act most vigorously on the soft portions of the film. J. W. GLASSETT.

Colour kinematography. R. GSCHÖPF (B.P. 283,560 and Addn. B.P. 289,827, 20.7.27. Austr., [A] 15.1.27, [B] 3.5.27) .- (A) Two negative films, one sensitised to red and the other to green, are exposed respectively through a yellow filter alternating with a bluetransmitting red filter and a yellow filter alternating with a blue-transmitting green filter. Positives are printed from these on to either side of a doubly-coated film, the image from the yellow and red filter negative being dyed bluish-green and that from the yellow and green filter negative bluish-red. The dyed images are reduced by washing until cleared of colour in the half tones and are then redyed with yellow. (B) To facilitate the reducing process the positive silver images are converted into a mordant, such as vanadyl ferrocyanide, which holds the dyes very loosely. J. W. GLASSETT.

Non-static and anti-static photographic films and film bases therefore. P. C. SEEL (U.S.P. 1,687,401 -2, 9.10.28. Appl., 12.8.27).—The photographic film is coated on the rear side with an anti-static layer containing (A) *p*-toluenesulphonamide-formaldehyde resin, or (B) an ester of abietic acid. J. W. GLASSETT.

Printing with vat dyes (B.P. 279,864).-See VI.

# XXII.—EXPLOSIVES; MATCHES. Patents.

Blasting explosive. R. A. LONG, Assr. to ATLAS POWDER CO. (U.S.P. 1,689,674, 30.10.28. Appl., 13.5.25). --The explosive contains ammonium nitrate, trinitro-

toluene, a non-explosive combustible, and not more than 8% of nitroglycerin. The total content of nitroglycerin and trinitrotoluene does not exceed 12%, and the proportion of nitroglycerin always exceeds that of the trinitrotoluene. S. BINNING.

Gelatinised explosive composition. K. R. BROWN, Assr. to ATLAS POWDER Co. (U.S.P. 1,686,952, 9.10.28. Appl., 10.9.26).—In order to produce a stable, readily gelatinisable explosive containing nitrated sugar as well as nitroglycerin and nitrocellulose, a nitrated glycol, e.g., ethylene glycol dinitrate, is added. An explosive containing a high percentage of nitrated sugar is obtained by nitrating a saturated solution of sugar in a glycerin-glycol mixture. F. G. CLARKE.

Storing explosive gases (B.P. 281,718).-See II.

# XXIII.—SANITATION; WATER PURIFICATION.

**Evaluation of insecticides.** C. H. PEET (Ind. Eng. Chem., 1928, **20**, 1164—1165).—In order to obtain concordant results in the biological testing of insecticides, temperature, time, humidity, and concentration of toxic mist in the testing chamber must be predetermined. Tests with flies were carried out in a large chamber with spray ports on four sides and a fan for mixing. Non-flying insects were sprayed with a known amount of insecticide in wire cages. Plant pests were tested by spraying an infested plant set in the stopper of a bottle of water. C. IRWIN.

Hydrogen-ion control in water-softening. J. R. BAYLIS (Ind. Eng. Chem., 1928, 20, 1191-1194).-If carbon dioxide is added to water containing calcium hydroxide the minimum hardness of 13 p.p.m. is reached at  $p_{\rm H}$  9.4. On the other hand, hardness due to magnesium increases with addition of carbon dioxide from zero value at  $p_{\rm H}$  10.6. The solubility relation of both carbonates to  $p_{\rm H}$  values at 23° was redetermined, and similar curves at other temperatures up to 98° were also obtained. In both cases increase of temperature produces a decrease of solubility. Calcium carbonate shows some increase in solubility in natural waters containing magnesia. A water containing both carbonates can be reduced to minimum hardness by precipitating magnesia with lime at  $p_{\rm H}$  10.6 and recarbonating to  $p_{\rm H}$  9.4. In practice, a final figure of  $p_{\rm H}$  8.7 is better to prevent the incrustation of water-pipes. The best results are obtained by carbonating in stages, but the first stage should be to about  $p_{\rm H}$  9.0 to avoid incrustation of the sand of filter-beds. C. IRWIN.

Electro-osmotic purification of water. II. A. H. W. ATEN (Chem. Weekblad, 1928, 25, 646-648; cf. B., 1928, 626).—The extent to which water may be freed from dissolved salts by electrolysis in a cell containing two diaphragms of different permeability is mathematically examined. With an E.M.F. of 40 volts, purification of 50 litres requires about 1 kw.-hr.

S. I. LEVY.

**Protective gas masks.** E. DRAGER (B.P. 289,808, 30.4.28. Ger., 2.5.27).

Sand filters (B.P. 298,835). Air filters (B.P. 275,257).—See I. Preventing explosions (U.S.P. 1,686,051).—See XI. Polyhydroxy-compound [insecticide] (U.S.P. 1,677,823).—See XVI.