

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 27, 1928.

I.—GENERAL; PLANT; MACHINERY.

Adhesion. II. (Sir) W. B. HARDY and (Miss) M. E. NOTTAGE (Proc. Roy. Soc., 1928, A, 118, 209—229).—Measurements of the tensile strength of a solid joint between a steel or copper cylinder and plate are described. The joint was formed by melting solid paraffins, fatty acids, or alcohols on the plate, allowing some of the liquid to be drawn under the cylinder by capillary attraction, and then freezing it. A microscopical examination of the disc of "lubricant" found on either the plate or cylinder on breaking the joint supports the view that it is composed of a central plate of flat crystals between two primary layers, one on each of the enclosing solids. The break appears to occur at one or both of the surfaces between the primary film and the plate of crystals. The effect of added impurity on the strength of the joint and the position of the surface of break was investigated, and the relation of the adhesion to the temperature, pressure, chemical composition of the lubricant, and composition of the cylinder and plate is discussed. As in the case of static friction, the adhesion varies linearly with the mol. wt. of the lubricant for a given chemical series. The distance between the surfaces of the cylinder and plate, measured by means of a specially constructed spherometer, was found to be independent, within the limits of error, of the nature of the lubricant and of the enclosing solids. A few measurements are recorded of the tangential pull required to break a joint. The fracture occurs at the same surface as with a normal force. L. L. BIRCUMSHAW.

Effect of thermometric lag in fractional distillation. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petroleum Tech., 1928, 14, 94—105).—Using mercury thermometers in distillations from a flask fitted with an eight-bulb Young "evaporator" still-head, it was found that a constant, R , called the "speed" of the thermometer, representing the number of degrees the thermometer would rise per second if the bath were maintained always 1° F. above the thermometer reading, depends on the diameter of the bulb and the thickness of its walls, and for the same thermometer depends on the latent heat of condensation of the vapour and the speed at which it passes the bulb. In a liquid bath, R depends on the rate of stirring. In general, the initial lag of the mercurial thermometer depends on the thickness of the layer of condensed liquid on the bulb, or, in the case of a liquid bath, on the layer of cooled liquid around the bulb. Measurements showed that the thickness of film required to produce the observed lag is of the order of 0.25 mm. In accurate distillations the use of a thermocouple is preferable. H. S. GARLICK.

Steam still for volatile acids. D. H. CAMERON (J. Amer. Leather Chem. Assoc., 1928, 23, 42—45).—

A Hortvet tube (J. Ind. Eng. Chem., 1909, 1, 31) is permanently mounted in a glass steam jacket by means of a rubber stopper, into which is also fitted a tube for a steam vent. Discharge of steam from the jacket and through this tube is regulated by means of a piece of rubber tubing and screw clip fitted over the end of the vent tube. Steam is generated from distilled water, free from carbon dioxide, in a 500 c.c. extraction flask, which is fitted to the lower end of the steam jacket. The Hortvet tube is fitted with a Kjeldahl bulb-form spray-trap, and a hole is blown in the vertical tube just below the stopper in the Hortvet tube. 10 c.c. of the liquor under examination are pipetted into the Hortvet tube, the apparatus is assembled, and the Kjeldahl delivery tube fitted to a condenser, and the distillate collected in 0.1N-alkali. D. WOODROFFE.

Technical reaction vessel. E. HOCHBERGER (Chem.-Ztg., 1928, 52, 130—131).—An apparatus suitable for reactions between solids and liquids with evolution of gas consists of a rotating drum mounted with the axis horizontal and provided with a tightly fitting lid on the cylindrical surface. The bearings at both ends are mounted on hollow shafts, which may permit of connexion to two concentric pipes or one single pipe to allow of introduction of liquids or gases and escape of a gaseous product etc. The whole may be made sufficiently gastight to permit of reactions being carried out under pressure. S. I. LEVY.

PATENTS.

Coal-dust furnaces. SIEMENS-SCHUCKERTWERKE G.M.B.H. (B.P. 282,333, 26.4.27. Ger., 17.12.26. Addn. to B.P. 270,295; B., 1928, 215).—In modification of the prior patent, the secondary jets are so numerous that their action on the flame may be considered as continuous; their direction of entry gradually approaches the axis of the flame and their velocity gradually decreases in the general direction of the flame, which is usually downwards. B. M. VENABLES.

Sublimation apparatus. C. A. LYFORD, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,662,070, 13.3.28. Appl., 2.4.20).—Means are provided for introducing steam both above and below the material to be sublimed, placed in a container, and for condensing sublimed material. J. S. G. THOMAS.

Drying of crystals. H. EBEL (G.P. 445,821, 1.4.25).—The crystals at the bottom of the crystallising pan are sucked into a closed vacuum filter until they reach a definite height, when the cover is removed and the mother-liquor passed through the filter and sucked into a collecting vessel, whence after removal of the vacuum it falls by gravity back to the crystallising pan. The vacuum filter is provided with a three-way cock

making connexion with the trough of the crystallising pan and with the vacuum pump. A. R. POWELL.

Recovery of volatile solvents by means of inert gases. R. OERTEL (G.P. 445,564, 26.7.25. Addn. to G.P. 444,913; B., 1928, 111).—The gas carrying the vapour of the solvent is passed from the evaporating chamber through a secondary circuit containing washing vessels and filters to remove oxygen. A. R. POWELL.

Hydrogen-ion comparator. A. HYUGA (U.S.P. 1,659,529, 14.2.28. Appl., 19.4.26).—The colour of light passed through tubes of untreated standard solution and of solution under test treated with an indicator, is compared with that of light passed through standard solution treated with the indicator and untreated test solution. T. S. WHEELER.

Colloidal dispersion of solids. S. CABOT, Assr. to S. CABOT, INC. (U.S.P. 1,662,999, 20.3.28. Appl., 31.1.25. Renewed 28.7.27).—See B.P. 246,874; B., 1927, 800.

Filtering apparatus. J. B. VERNAY (U.S.P. 1,662,726, 13.3.28. Appl., 16.2.27. Fr., 24.2.26).—See B.P. 266,719; B., 1927, 801.

Apparatus for concentrating aqueous liquids. P. J. DE S. A. MEYNARDIE (U.S.P. 1,661,489, 6.3.28. Appl., 6.4.25. Fr., 16.4.24).—See B.P. 232,589; B., 1926, 31.

Gas-analysing apparatus. O. RODHE, Assr. to SVENSKA AKTIEBOLAGET MONO (U.S.P. 1,661,627, 6.3.28. Appl., 19.8.26. Ger., 28.8.25).—See B.P. 257,609; B., 1927, 592.

Furnace or combustion chamber walls. ÉTABL. F. LABESSE (B.P. 280,561, 2.5.27. Fr., 4.5.26).

Process and apparatus for extinguishing fires. EXCELSIOR FEUERLÖSCHGERÄTE A.-G., and H. BURMEISTER (B.P. 285,934, 19.10.26).

[Nozzle for] spraying liquids. BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX Co. (B.P. 286,114, 19.4.27.)

II.—FUEL; GAS; TAR; MINERAL OILS.

Banded coal. H. WINTER (Fuel, 1928, 7, 52—63; Glückauf, 1927, 63, 14).—The work of previous investigators on banded bituminous coal is summarised and compared. It is concluded that fusain and durain must be regarded as well-defined petrological constituents of coal, which is much less the case for vitrain and clarain, together forming the "bright" coal. As a result of the discovery of plant structure in vitrain, the Stopes definition (B., 1919, 452 A), which was based essentially on the absence of structure in this ingredient, has become untenable. A. B. MANNING.

Replaceable bases in the roofs of bituminous coal seams of carboniferous age. E. MCK. TAYLOR (Fuel, 1928, 7, 66—71).—The p_H values and amounts of the replaceable bases present in a large number of rocks forming the roofs of coal seams in Great Britain have been determined. The majority are alkaline in reaction and have the replaceable sodium considerably in excess of the replaceable calcium. Where the rocks differ from this general type there is evidence that the original roof has been removed by denudation and the

present one deposited subsequently. It is concluded that the roofs of these bituminous coal seams have been deposited under such conditions that, either during or subsequent to deposition, they have been in contact with solutions of sodium chloride, and that the base-exchange product has been subjected to hydrolysis in fresh water, yielding an alkaline clay which formed the roof of the seam during coal formation. Such an alkaline medium existing under anaerobic conditions would favour bacterial decomposition of organic matter (cf. B., 1927, 691). A. B. MANNING.

Dry-cleaning of coal for carbonisation. C. W. H. HOLMES (Gas World, 1928, 88, Coking Sect., 32—38; cf. B., 1927, 641).—The influence of the relationship between density, particle size, and ash content on the theoretical possibilities of coal cleaning is discussed with reference to typical coking slacks. Pyritic sulphur is generally readily removable by cleaning, but organic sulphur is not. Representative layouts for the treatment of coking slacks by pneumatic separation are briefly described, and discussed with particular reference to the necessity for sizing, the removal and utilisation of the dust, the effect of moisture in the coal, and the running costs of the process. A. B. MANNING.

Influence of various factors on the products of the carbonisation of coal. H. H. THOMAS (J.S.C.I., 1928, 47, 77—83 T).—Increasing the temperature of carbonisation caused an increase in the yield of gas of reduced calorific value. At 950° the yield per ton was 11,110 cb. ft. of 649 B.Th.U. per cb. ft., and at 1000°, 13,340 cb. ft. at 590 B.Th.U., the net result being an increase in the gaseous therm yield from 72.1 therms per ton at 950° to 78.7 therms at 1000°. These results are presumed to be due to the increased degradation of the tar vapours at the higher temperatures. With the coal screened to pass 1 in. mesh and remain on $\frac{3}{4}$ in. mesh, 11,200 cb. ft. of gas per ton, of 635 B.Th.U. per cub. ft., were obtained (=71.1 therms per ton). By reducing the size of particle the gas yield was increased so that with a particle size through $\frac{1}{4}$ in. on $\frac{1}{8}$ in. 12,000 cb. ft. of gas (616 B.Th.U.) were obtained, equivalent to 73.9 therms per ton. Further results show the effect of the addition of inert material (coke and non-coking coal) to the coal before carbonisation, the object being to obtain a more reactive and combustible coke. Considerable importance is attached to the necessity for the fine grinding of the coals forming a blend, in order that intimate mixing and an even structure in the resultant coke be obtained. The addition of 20% of coke gave an increased yield of gas of reduced calorific value, the therms per ton of coal being the same as obtained from the coal carbonised alone. The reactivity of the coke from the mixture was only slightly increased. A blend of 70% of coking gas slack with 30% of a highly oxygenated non-coking coal gave a coke of considerably increased reactivity. The thermal yield from the blend was approximately equivalent to the sum of the yields of the two coals when carbonised separately. The rate of carbonisation in this case was not greatly increased on that of the gas slack alone; this may result in the output of a given plant being considerably reduced as the non-coking coal has only a low gas-making value.

It is suggested that the charge be removed from the retort before carbonisation is complete, after which time no appreciable loss of gaseous therms would occur. In this way the output of the plant could be maintained and the benefit of the increased reactivity in the coke obtained. The experiments were carried out in a small 1/1000-ton test plant which is described.

Low-temperature distillation of coal briquettes. P. WEISS (Chim. et Ind., 1928, 19, 195—204).—The solid fuel obtained by carbonising briquetted coal at low temperatures varies with the type of coal used, the rate of heating, the fineness of the coal, the pressure to which the briquettes have been submitted, and the proportion of binder. The influence of these factors has been investigated, the carbonisations being carried out in an internally-heated retort in which the heating medium passes successively through a number of chambers, its point of entry being moved periodically from one chamber to the next so that the contents of each chamber in turn are subjected to higher and higher temperatures until carbonisation is complete. Briquettes made from anthracitic coals with the correct proportion of pitch as binder can be satisfactorily carbonised at a rate of heating of 3° or more per min., in either internally- or externally-heated retorts. Swelling coals require a careful regulation of the rate of heating, especially over certain critical temperature ranges, when the rate of heating may have to be reduced below 0.1° per min. in order to produce briquettes of the required density and strength. With a suitable rate of heating a variation in the amount of pitch from 6 to 8% makes little difference to the briquettes. The density and strength of the carbonised briquettes increase at first with the pressure under which the briquettes are formed, pass through a maximum, and then fall off; the position of the maximum, however, depends on the rate of heating employed, which should therefore be so chosen that the maximum is just reached for the working pressure used. In general, better briquettes are obtained if the coal is more finely ground, but there is evidence of an optimum particle size for each coal. For non-coking coals and lignites a binder containing relatively light oils is necessary; the strength of the briquettes is markedly increased by the addition of a small proportion of finely-powdered sulphur to the mixture of coal and binder.
A. B. MANNING.

Combustion of powdered coal. B. MOORE (Trans. Inst. Chem. Eng., Advance Proofs, March, 1928, 12—24).—The degree of fineness of powdered coal affects considerably its rate of combustion, ignition properties, and "combustible capacity," as determined by the method previously described (B., 1925, 486; 1927, 130). The rate of combustion increases and the "glow point temperature" falls with increasing degree of fineness of the coal. The results indicate that there is a critical temperature and a critical degree of fineness at which the combustion of the particles becomes almost instantaneous.
A. B. MANNING.

Natural bitumens, with special reference to bituminous shale. W. ZWIEG (Petroleum, 1928, 24, 243—246).—In parts of north Germany a type of peat

resembling cannel coal is found which contains considerable quantities of bituminous substances. Whilst ordinary peat is classed with brown coal, this shale-peat is more suitably grouped with oil-shale; in the first class the hydrogen content per 100 pts. of carbon varies from 8.3 to 2.1, but in the second the ratio is constant at 12 pts. of hydrogen. The dry, ash-free peat contains 82.7% of volatile matter, being almost identical in this respect with an Australian shale; the yield of dry bitumen obtained on distillation is 25% by wt. Gas-making tests carried out by the Geipert method are compared with those given by carbonisation of Australian shale in horizontal retorts; 52.6% of the potential heat in the peat can be obtained in the gas.

R. H. GRIFFITH.

Preparation, purification, and analysis of pyro-ligneous oils. R. HUERRE (J. Pharm. Chim., 1928, [viii], 7, 58—69).—A description and discussion of the treatment and analysis of oils obtained by the dry distillation of various woods, particularly with regard to the nature and recovery of the phenolic substances.

E. H. SHARPLES.

Relation of the micelle to the medium in asphalt. F. J. NELLENSTEYN (J. Inst. Petroleum Tech., 1928, 14, 134—138).—Asphalt consists of a medium and a disperse phase, the asphalt micelle containing a lyophile part (protective substances) and a lyophobe part (elementary carbon). Flocculative and peptising reactions cause changes in the stability of the system depending on the relation between the micelles and the oily medium. Interfacial tension of the micelle is changed by adding a liquid which changes the surface tension of the medium. Of liquids miscible with the asphalt medium those of low surface tension are flocculating reagents, whereas those possessing high surface tension are peptising reagents. The higher the surface tension the better is the solvent power. The surface tensions of petroleum residues and blown asphalts range from 23.1—25.2 dynes/cm., and for coal tar 32.0 dynes/cm. Precipitation is never complete with liquids of low surface tension, e.g., benzene. Only by adding a liquid not completely miscible with the medium or the protective substances, such as ether-alcohol mixture, can this be achieved, but true flocculation no longer occurs and the "soft asphalt" obtained contains a large proportion of oily compounds.

H. S. GARLICK.

Diesel-engine performance on oils obtained from the low-temperature carbonisation of coal. J. S. BROWN (J. Roy. Tech. Coll. Glasgow, 1927, [4], 76—84).—The performance of a Diesel engine when three oils obtained from coal by low-temperature carbonisation processes are used as fuel has been investigated. In each case the engine was worked up to full load and normal jacket temperature on a normal petroleum oil, and the fuel was then gradually changed to that under test. In one case excessive viscosity caused an air-lock in the feed, but the performance on the other two oils was entirely satisfactory with the engine under full load. In the case of one oil only was it possible to avoid pre-ignition when the engine was running at no load. In neither case was it possible to start the engine without preheating the jackets. Two of the three oils gave a small amount of deposit when mixed with Shell-

Mex Diesel grade oil. These oils are preferable to the creosote oils previously obtained from coal, and it is considered that a little further improvement in the quality would give them all the properties of a petroleum oil. L. M. CLARK.

Spreading of lubricants on solid surfaces. P. WOOD (Compt. rend., 1928, 186, 71—73).—The spreading properties of typical animal, vegetable, and saturated neutral mineral oils on various metallic surfaces have been investigated at 100°. Whilst the animal and vegetable oils spread rapidly at low temperatures over all the surfaces tested, the mineral oil (d^{15} 0.8647, η (abs.) 0.0785 at 50°, and 0.0024 at 100°) usually required a higher temperature and a coating of stearic acid over the metallic surface in order to produce rapid spreading. The stearic acid neutralises the surface by the production of a layer of active oriented molecules. The phenomenon is most marked for cold-beaten surfaces. J. GRANT.

Regeneration of used [lubricating and insulating] oils. K. TYPKE (Gas- u. Wasserfach, 1927, 70, 1289—1291).—The removal of suspended matter from used oils is not sufficient purification, and treatment with fuller's earth is adequate only for slightly altered oils. Stirring with sulphuric acid and, in some cases, with sodium hydroxide is necessary, and may, with advantage, be followed by treatment with fuller's earth (stirring with powdered earth and filtering, or allowing the oil to percolate through a bed of granular earth). Used oils containing light constituents of low flash point should not be mixed with oils of high flash point, otherwise the former must be removed, by some method of evaporation or distillation, prior to refining. The regenerated oils should satisfy the same requirements as a fresh oil, and an important test of the adequacy of regeneration is to note the extent of tar formation when oxygen is passed for 7 hrs. through the oil at 120°.

W. T. K. BRAUNHOLTZ.

Petrol engine lubricants and lubrication. C. I. KELLY (J. Inst. Petroleum Tech., 1928, 14, 115—133).—A general discussion of the theory of lubrication and its application to automobile conditions.

H. S. GARLICK.

Liquid fuels other than petroleum. A. E. DUNSTAN and H. G. SHATWELL (J. Inst. Petroleum Tech., 1928, 14, 64—77).—A summary of contemporary information regarding the production and properties of motor fuel derived from the low-temperature carbonisation and hydrogenation of coal, and sundry gas reactions resulting in methyl alcohol, synthol, and synthetic petroleum.

H. S. GARLICK.

Blasting in tunnels. GARDNER and others.—See XXII.

PATENTS.

Ovens for treating fuels or fuel-containing materials by heat. O. HELLMANN (B.P. 272,493, 1.6.27. Ger., 8.6.26).—An oven for the heat treatment of fuels has a movable annular platform carrying transverse, evenly spaced heating chambers, between which are spaces open towards the edges of the platform and designed to receive the raw material. The platform and heating chambers are enclosed within stationary walls, inclined at the natural angle of repose of the material,

and provided with opposite openings at one point through which the product is discharged by a pusher head.

A. B. MANNING.

Activation of carbon or carbonaceous substances with simultaneous production of combustible gases. SOC. DE RECHERCHES & D'EXPLOIT. PETROLIFÈRES (B.P. 269,477, 23.11.26. Fr., 15.4.26).—Carbon to be activated, or carbonaceous material to be converted into active charcoal, is enclosed in chambers with walls which are permeable to gas, and is subjected to the action at high temperatures of gases containing no free oxygen, *e.g.*, combustion gases rich in carbon dioxide and steam, which reach the material by diffusion through the walls. The speed of activation is controlled by regulating the gaseous pressure within the chambers. The combustible gases formed in the chambers are withdrawn and used for heating purposes. The chambers or retorts are preferably of a shape, *e.g.*, narrow and flat, which possesses a large surface area. A. B. MANNING.

Drying material [coal] by means of fumes evolved in a subsequent operation. KOHLENVEREDLUNG A.-G. (G.P. 446,042, 19.11.22).—In the drying of wet substances, such as coal, the heat of the fumes or smoke evolved in the last stages of the drying operation is utilised by passing the gases through a secondary dryer arranged before or after the main dryer. Previous to passing to this apparatus the fumes may be superheated by passing them over glowing material to cool it below the ignition point.

A. R. POWELL.

[Smokeless] fuel. T. NAGEL (U.S.P. 1,658,814, 14.2.28. Appl., 15.11.26).—A low-volatile bituminous coal is mixed with a binder of the type described in U.S.P. 1,507,675—6 (B., 1924, 1003), and the mixture is briquetted and heated at 400°.

T. S. WHEELER.

Utilisation of subdivided coal. N. TESTRUP, O. SODERLUND, T. GRAM, and TECHNO-CHEMICAL LABORATORIES, LTD. (B.P. 284,405, 28.10.26).—To coal slurry containing a high percentage of water, dried powdered coal is added in such proportion that a friable mixture is produced, and the latter is then dried in a current of air, the water content being reduced until the powder will flow easily, but at the same time retain sufficient water to prevent the formation of dust. The drying may be continued further, but in that case the dust-laden air is conveyed to a furnace where the dust is burnt, or the dust may be recovered by suitable means.

A. B. MANNING.

Apparatus for the production of carbon black. W. F. FULTON, ASS. to PALMER CORP. (U.S.P. 1,658,676, 7.2.28. Appl., 31.5.27).—Apparatus for the production of carbon black comprises a rotary collector cylinder communicating with a gas supply and for heating the gas, manifolds for receiving the hot gas and delivering it to passages in arches between which a horizontal combustion tube designed to direct decomposed gas substantially normally against the rotary collector is arranged.

J. S. G. THOMAS.

Preparation of carbon black. HARTSTOFF-METALL A.-G. (HAMETAG), Assees. of E. PODSZUS (G.P. 445,498, 1.10.24).—For the grinding of carbon to colloidal fineness a mill with steel balls of 1—2 mm. diameter is used. The carbon powder mixed with the balls may be subjected

to a high-speed counter-current of gas or air to increase the efficiency of the grinding. A very even product with large surface is obtained.

C. HOLLINS.

Production of liquid hydrocarbons and other products by the destructive hydrogenation of coal, tars, and like carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 256,965, 10.8.26. Ger., 14.8.25).—Coal, tars, etc. are converted into valuable liquid hydrocarbons by being submitted, in a gaseous or finely-divided state, to the action of hydrogen or gases containing or giving rise to hydrogen, under a pressure of at least 50 atm. and at high temperatures, in the presence or absence of catalysts. The gases may be recirculated for the treatment of fresh material, and a mixture of hydrogen and water vapour or water vapour alone added to make up for the hydrogen consumed. No coke is produced in the process, and the rate of throughput is higher than when working without atomising the material.

A. B. MANNING.

Manufacture of liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of coal, tars, etc. I. G. FARBENIND. A.-G. (B.P. 257,910, 12.8.26. Ger., 2.9.25).—Coal, tars, or other carbonaceous materials are hydrogenated under pressures of at least 50 atm. and at high temperatures in a current of the hydrogenating gases; the starting material is employed in a moist state, or water may be added during the process. The formation of coke is avoided, and the process can be carried out continuously so that the material is practically completely consumed.

A. B. MANNING.

Manufacture of liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of tars, mineral oils, resins, asphalts, etc. I. G. FARBENIND. A.-G. (B.P. 282,814, 10.8.26. Ger., 14.8.25. Addn. to B.P. 256,964; B., 1928, 221).—The procedure of the main patent has been extended to the hydrogenation of liquids or materials which are liquid under the working conditions; the addition of 5–30% of brown coal or peat to the starting material facilitates the hydrogenation and prevents coking.

A. B. MANNING.

Manufacture of unsaturated gaseous hydrocarbons and mixtures of carbon monoxide and hydrogen. I. G. FARBENIND. A.-G. (B.P. 269,547, 12.4.27. Ger., 15.4.26).—Gas mixtures containing saturated hydrocarbons are subjected to a process, e.g., passage through an electric arc, or incomplete combustion, partially converting them into unsaturated hydrocarbons. These are removed by some suitable process (acetylene, for example, by conversion into benzene, and absorption of the latter in active charcoal), and the residual gas is then treated with steam or oxygen or both, either in the presence or absence of catalysts, to give a mixture of hydrogen and carbon monoxide.

A. B. MANNING.

Conversion of heavy hydrocarbons into light and stable hydrocarbons. P. GRENE (B.P. 285,668, 21.3.27).—Tars, crude oils, etc. are cracked and converted into hydrogenated products by passage through heated tubes containing layers of different catalysts. The oils to be treated are subjected to a preliminary

mild cracking process, wherein coke and gas production is reduced to a minimum, and the vapours are diluted with a constant concentration of suitable neutral gases, in opposition to the production of gaseous products, before passage through the heated catalyst tubes. These contain (1) a porous carbonaceous substance, such as coke, which promotes cracking, prevents carbon formation, and promotes the decomposition of organic sulphur compounds; (2) a layer of manganese dioxide which removes sulphur compounds, and may be regenerated by means of an air blast; (3) a further layer of a carbonaceous catalyst capable of causing hydrogen to be generated from the water vapour present; and (4) a hydrogenation catalyst such as reduced nickel.

C. O. HARVEY.

Distillation of hydrocarbons, and fuel produced thereby. L. T. BATES (U.S.P. 1,657,815, 31.1.28. Appl., 5.6.26).—Coal, mixed with liquid hydrocarbons, is pulverised at a temperature sufficiently high to vaporise the volatile matter present in the mixture.

C. O. HARVEY.

Controlling the destructive distillation of petroleum oil. R. W. HANNA, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,658,241, 7.2.28. Appl., 12.2.23).—The oil is cracked and vaporised and the evolved vapours are passed (under the pressure of the cracking reaction) through a three-stage separating system. In the first stage the vapours are scrubbed with a descending shower of the original feed oil mixed with the reflux descending from the second stage; in the second stage the reflux condensate from the third stage is used as the scrubbing liquid, and in the third the vapours are cooled to the desired discharge temperature by means of a liquid other than that undergoing treatment. The combined reflux condensates, admixed with feed oil, are passed to the cracking chamber.

C. O. HARVEY.

Oil-shale distillation apparatus. C. D. TRIPP (U.S.P. 1,658,143, 7.2.28. Appl., 12.7.24).—The shale is introduced at one end of a horizontal retort, the bottom of which is covered with molten metal, and the other end of which is fitted with an inverted weir dipping below the surface of the metal bath. Means are provided for mechanically propelling the shale towards the weir, and for delivering the gangue beneath the weir to a point of discharge. The products of distillation pass out from the upper portion of the retort.

C. O. HARVEY.

Treatment of hydrocarbon oils. H. W. CAMP, Assr. to DOHERTY RES. Co. (U.S.P. 1,657,524, 31.1.28. Appl., 29.11.22).—The temperature gradient in a dephlegmating column is controlled by allowing a part of the chilled condensate to flow from a point below the top of the column in a downward direction, the vapours from the still passing upwards. The other portion of the condensate from the column is mixed with a further quantity of the original oil and injected into the still.

C. O. HARVEY.

Treatment of [hydrocarbon] oils. R. J. DEARBORN, Assr. to TEXAS Co. (U.S.P. 1,658,077, 7.2.28. Appl., 18.4.22).—In a process for the production of gasoline, a continuous stream of oil is raised, by passage through a tubular still, to a temperature adequate only for cracking when aluminium chloride is present. The

oil without further heating comes in contact with aluminium chloride in a separate reaction chamber, and the vapours produced are partially condensed, the condensate passing back to the still for recirculation.

C. O. HARVEY.

Apparatus for treating oils. G. D. SIMS, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,658,420, 7.2.28. Appl., 17.6.21. Renewed 10.3.27).—The level of the oil in a vaporising chamber which is connected with a pressure still is indicated by means of a gauge which communicates with the base of the chamber via a cooling device. The vapour space above the oil in the gauge communicates with the receiver for condensate, whereby equal pressure is maintained on the oil in the gauge and that in the chamber.

C. O. HARVEY.

Preparation of hydrogen and hydrogenation of hydrocarbons. A. MALEVILLE (F.P. 621,902, 23.9.26).—Molten metals, *e.g.*, zinc, and water are used. The metallic oxides formed must be capable of reduction by carbon below a red heat. The hydrogen so produced is allowed to act on the coal or hydrocarbons in the bath itself.

A. B. MANNING.

Extraction of petroleum products from bituminous material. P. DVORKOVITZ (B.P. 285,564, 17.11.26).—The yield of the products obtained by the destructive distillation of bituminous materials (*e.g.*, coal) is augmented by introducing into the charged retort, preheated to a temperature (500°) sufficiently high to effect the removal of a portion of the organic matter, a solvent in liquid or vapour form capable of removing a further portion of the organic matter. The solvent (*e.g.*, petroleum or gas tar) is subsequently recovered for re-use.

C. O. HARVEY.

Oil composition suitable as a power-transmitting agent in hydraulic brakes for automobiles etc. E. A. EVANS, and C. C. WAKEFIELD & Co., LTD. (B.P. 285,144, 10.11.26).—Castor oil is mixed with an alcohol or mixture of alcohols of mol. wt. higher than that of ethyl alcohol, *e.g.*, benzyl alcohol and cyclohexanol.

A. B. MANNING.

Lubricating composition. E. A. NILL, Assr. to F. C. ACKER and J. R. DALL (U.S.P. 1,659,149, 14.2.28. Appl., 4.5.23. Cf. U.S.P. 1,659,150; B., 1928, 293).—A mixture of a mineral oil and a fatty-acid anilide, *e.g.*, stearanilide, is claimed.

T. S. WHEELER.

Reclamation of used crank-case oil. W. H. ELLERINGTON (U.S.P. 1,662,701, 13.3.28. Appl., 23.1.26).—Fine particles are removed from lubricating oil by intimately mixing the latter with soap, sodium silicate, and talc.

H. ROYAL-DAWSON.

Use of heavy oils in internal-combustion engines. SOC. ANON. LE CARBONE (B.P. 262,407, 10.11.26. Fr., 5.12.25).—The mixture of air and atomised fuel is subjected to the action of active carbon at 300–400° immediately before admission to the engine, a part of the fuel being thereby transformed into light oil vapours. The catalyst, which may either be interposed between the carburettor and the engine inlet or contained in the carburettor itself, consists of a block of activated wood charcoal preferably provided with oblique passages and so mounted that the proportions of air and fuel mixture which pass respectively through the body of the charcoal

and through the passages can be varied to suit the running of the engine. The tube containing the charcoal block is jacketed so that by the use of a suitable heating medium its temperature can be maintained above 250°.

A. B. MANNING.

Prevention of pinking or knock in internal-combustion engines. ASIATIC PETROLEUM CO., LTD., and A. C. EGERTON (B.P. 279,560, 29.7.26).—Thallium vapour or the vapour of a thallium compound, *e.g.*, thallium ethyl, ethoxide, oleate, etc., is introduced into the cylinder.

H. ROYAL-DAWSON.

Bonding of activated carbon. A. B. RAY, Assr. to CARBIDE & CARBON CHEMICALS CORP. (Re-issue 16,896, 6.3.28, of U.S.P. 1,565,328, 15.12.25).—See B., 1926, 119.

Dry-cooling of coke. R. WUSSOW (U.S.P. 1,661,211, 6.3.28. Appl., 19.10.26. Ger., 17.1.23).—See B.P. 251,530; B., 1926, 573.

Prevention of [coal] mine explosions. W. E. TRENT, Assr. to TRENT PROCESS CORP. (Re-issue 16,914, 20.3.28, of U.S.P. 1,642,912, 20.9.27).—See B., 1927, 900.

Liquid fuel. E. M. FLORES (U.S.P. 1,657,961, 31.1.28. Appl., 9.6.22. Argentine, 16.1.22).—See B.P. 249,577; B., 1926, 524.

Coke oven closures. H. J. LIMBERG (B.P. 276,005, 11.8.27. Ger., 11.8.26).

Machines for separating coal and for other analogous purposes. W. H. BARKER (B.P. 286,452, 1.3. and 28.6.27).

Coke quenching and loading appliances. Dr. C. OTTO & COMP., G.M.B.H. (B.P. 280,944, 18.11.27. Ger., 22.11.26).

[Automatic] apparatus for separating benzol from waste-water. W. LINNMANN, JUN. (B.P. 272,512, 8.6.27. Ger., 8.6.26).

Water-gas (B.P. 282,573).—See VII. **Paving mixtures (U.S.P. 1,662,377).** **Road binder (U.S.P. 1,662,299).**—See IX. **Resin from tar oil (U.S.P. 1,658,281).**—See XIII.

III.—ORGANIC INTERMEDIATES.

Vanillin from sulphite[*-*cellulose] waste liquors. K. KÜRSCHNER (J. pr. Chem., 1928, [ii], 118, 238–262).—The evidence from sublimation experiments in favour of the view that pine lignin is a colloidal coniferin complex (cf. A., 1925, 890, 1387; Routala and Sevón, "Über die Ligninstoffe") is reviewed. Considerable amounts of vanillin are formed when a rapid current of air is passed through boiling alkaline sulphite-cellulose waste liquors (cf. Grafe, A., 1905, i, 22). Using a neutralised liquor containing 64% of total solids and 24.8% of lignin, the optimum alkalinity, concentration, and reaction period are obtained when 10 c.c. are refluxed with 10 g. of sodium hydroxide or 10–20 g. of potassium hydroxide and 30 c.c. of water for 1.5 hrs. After neutralisation and filtration the vanillin (0.53 g. = 21–22% of the lignin) is determined colorimetrically in both solution and precipitate using phloroglucinol and hydrochloric acid or orcinol and sulphuric acid. This yield,

which is in good agreement with the proportion of aromatic derivatives obtained by other degradative methods (cf. Klason, A., 1923, i, 187; Heuser, A., 1923, i, 900), is not improved by the use of oxygen nor by adding copper dust, mercuric, cupric, or nickelic oxides, ferric chloride, or potassium nitrate, and is decreased by adding sodium peroxide or potassium ferricyanide. Extraction of the solution and the dried precipitate with ether affords 0.5—1.0 g. of crude vanillin. This is obtained crystalline by sublimation and then contains guaiacol but no pyrocatechol or oxalic acid. Experiments with decayed pine-wood and lignite are also described.

H. E. F. NOTTON.

Chemical treatment of trade waste. IV. Waste from organic ester synthesis. F. D. SNELL and C. T. SNELL (Ind. Eng. Chem., 1928, 20, 240—241).—The wastes to be treated consist of a calcium sulphate sludge from the stills which is highly contaminated and strongly acid, succeeded by wash waters of lower acidity. Neutralisation with lime followed by vacuum filtration is recommended as the best which can be done at a reasonable cost. The lime is to be added as a 20% slurry at a rate to correspond with the flow and variation in acidity of the effluent, necessitating a flexible control of the feeding system. About 240 lb. of lime are required per 1000 gals.

C. JEPSON.

Alcohol from Nipah palm. DENNETT.—See XVIII.

PATENTS.

Fusion of aromatic sulpho-compounds. C. R. DOWNS, Assr. to WEISS & DOWNS, INC. (U.S.P. 1,658,230, 7.2.28. Appl., 30.8.24).—An alkali arylsulphonate is heated in a thin layer on a metal pan in a bath of fusible metal at 300—425° with an alkali hydroxide (2—2.5 mols. for each atom of sulphur) to yield the corresponding alkali phenoxide.

T. S. WHEELER.

Manufacture of anilides. E. A. NILL, Assr. to F. C. ACKER and J. R. DALL (U.S.P. 1,659,150, 14.2.28. Appl., 10.5.24. Cf. U.S.P. 1,659,149, p. 292).—Anilides of the higher fatty acids, e.g., stearanilide, are prepared by passage of aniline vapour through the acid at about 235°.

T. S. WHEELER.

Manufacture of derivatives of acenaphthene. I. G. FARBENIND. A.-G. (B.P. 274,902, 23.7.27. Ger., 24.7.26).—When acenaphthene-5-carboxylic or -5-sulphonic acid is nitrated, single products are obtained. 6-Nitroacenaphthene-5-carboxylic acid, m.p. 235—236°, obtained by nitration in acetic acid, is reduced by ferrous sulphate and ammonia to the amine (hydrochloride, m.p. about 300°).

C. HOLLINS.

Production of indigo-white [leuco-indigo]. J. E. SEITZ, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,657,869, 31.1.28. Appl., 25.3.24. Renewed 19.8.27).—The yield of leuco-indigo from the reduction of indigo by the action of iron and sodium hydroxide solution at 65° is improved by the addition of sodium hyposulphite or sodium sulphide near the end of the reaction.

T. S. WHEELER.

Catalytic alkylation of aromatic hydrocarbons. M. G. SHEPARD, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,658,176, 7.2.28. Appl., 7.3.25).—See B.P. 259,507; B., 1926, 1028.

Preparation of p-hydroxy-o-benzoylbenzoic acid. NEWPORT Co. (B.P. 281,227, 15.8.27. U.S., 29.11.26).—See U.S.P. 1,654,289; B., 1928, 224.

Manufacture of diphenylguanidine. H. S. ADAMS and L. MEUSER, Assrs. to NAUGATUCK CHEM. Co. (U.S.P. 1,662,626, 13.3.28. Appl., 19.6.22).—See B.P. 199,354; B., 1924, 768.

Preparation of [mercaptobenz]thiazoles. L. B. SEBRELL and J. TEPPEMA, Assrs. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,662,015, 6.3.28. Appl., 16.3.27).—See B.P. 282,947; B., 1928, 152.

Oxidation of aralkyl-substituted dithiocarbamic acids to the corresponding disulphides. W. FLEMING, Assr. to SILESIA VER. CHEM. FABR. (U.S.P. 1,662,664, 13.3.28. Appl., 16.8.27. Ger., 30.10.26).—See B.P. 279,790; B., 1928, 225.

Recovery of volatile solvents. J. H. BRÉGÉAT (U.S.P. 1,663,155, 20.3.28. Appl., 1.8.25. Fr., 8.6.25).—See B.P. 251,492; B., 1926, 616.

IV.—DYESTUFFS.

Colouring matter present in the rhizomes of *Curcuma aromatica*, Salisb. B. S. RAO and V. P. SHINTRE (J.S.C.I., 1928, 47, 54 t).—The colouring matter present in the rhizomes of *C. aromatica*, Salisb., has been shown to be curcumin identical with that found in *C. longa* etc.

Light-sensitivity of dyes. II, 2. A. STEIGMANN (Kolloid-Z., 1928, 44, 249—250).—A discussion of the sensitising effect of dyes on light reactions, especially in regard to the formation of vitamins. E. S. HEDGES.

PATENTS.

Vat dyes obtained by dehalogenating polyhalogeno-violanthrones. B. MAYER and H. SIEDENBÜRGER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,658,003, 31.1.28. Appl., 28.6.26. Switz., 24.12.25).—A leuco-polyhalogeno-violanthrone or -isoviolanthrone, e.g., leuco-dichloro-violanthrone, is partially dehalogenated by treatment with alkaline reducing agents in the warm, e.g., by heating the vat to a temperature higher than is usual, to yield products which in the vat give shades similar to, but more brilliant than, those of the non-halogenated parent dyes.

T. S. WHEELER.

Manufacture of modified basic dyes. J. G. KERN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,659,145, 14.2.28. Appl., 11.6.24).—A basic dye is heated in alcoholic solution in presence of mineral acid with a hydroxy-aromatic carboxylic acid, e.g., tannic acid, and the solution is poured into water to precipitate a product which dyes unmordanted wool and cotton fast shades.

T. S. WHEELER.

Manufacture of a dye powder. O. M. BISHOP and M. S. THOMPSON, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,659,131, 14.2.28. Appl., 6.6.25).—A paste of an insoluble dye is mixed with a soap, e.g., sodium stearate, a sugar, a protective colloid, and water, and after being agitated to produce a foam, the mixture is dried to yield a product which disperses in water.

T. S. WHEELER.

Manufacture of [sulphide vat] dyes. A. CATINEAU, Assr. to Soc. Chem. Ind. in Basle (U.S.P. 1,662,415, 13.3.28. Appl., 19.4.27. Switz., 30.4.26).—See B.P. 270,348; B., 1927, 869.

Manufacture of vat dyes of the anthraquinone series containing the triazine ring. F. ACKERMANN, Assr. to Soc. Chem. Ind. in Basle (U.S.P. 1,663,474, 20.3.28. Appl., 24.9.27. Switz., 6.10.26).—See B.P. 278,728; B., 1928, 226.

Manufacture of acylaminoflavanthrones. S. THORNLEY, and Brit. Dyestuffs Corp., Ltd. (U.S.P. 1,662,872, 20.3.28. Appl., 3.9.27. U.K., 2.9.26).—See B.P. 282,852; B., 1928, 152.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Nitrogen content of natural and processed wools. J. BARRITT (J.S.C.I., 1928, 47, 69—72 τ).—The nitrogen content of natural and processed wools has been determined, using the Kjeldahl method. For normal wools the nitrogen content varies within narrow limits, 17.07% to 16.50%, the mean value for a representative series of wools being 16.72%. The effect of a normal scouring, carbonising, or chlorination on the nitrogen content is small. Excessive treatment with alkali causes an increase in nitrogen content, sulphuric acid a decrease, and chlorination up to 10% a slight decrease. Samples of black and white wool from the same fleece (Jacob's flock) show a definite difference in nitrogen content, the black wool having the lower value (cf. Gortner, B., 1913, 939). Samples of white, brown, and black alpaca exhibit a marked gradation in nitrogen content with colour, the respective values being 17.00, 16.66, and 15.85% of nitrogen. The black alpaca after treatment with 2% sodium carbonate at 50° has a markedly increased nitrogen content, and it is suggested that the hot alkali dissolves out melanin, the melanin having a considerably lower nitrogen content than normal wool.

Microscopical investigation of artificial silk fibres. L. G. LAWRIE (J. Soc. Dyers & Col., 1928, 44, 73—78).—The preparation and mounting of fibres and cross-sections for examination, and methods of illuminating them, with avoidance of glare, are described. For the examination of the fibre entire by transmitted light and dark-ground illumination, the structural markings and appearances of different kinds of silk are classified, whilst typical interference colours shown by the different varieties under the influence of polarised light are also described. Cellulose nitrate silks show the most brilliant effects, whilst the acetate silks show no interference colours. The swelling power of artificial silks in water, as measured by the percentage increase in the diameter of the fibre, is greatest for viscose and cuprammonium silks and least for acetate silks, the fully acetylated types of the latter showing negligible swelling. B. P. RIDGE.

Value of sisal hemp for the manufacture of marine cordage. ANON. (Bull. Imp. Inst., 1927, 25, 359—368).—Comparative tests on ropes made from Manila hemp "J" grade and first and second quality East African sisal hemp indicate that first-quality East African sisal is slightly superior and second-quality

East African sisal slightly inferior to Manila hemp "J" as regards resistance to the action of sea-water, the percentage loss in strength of sisal No. 1, Manila "J," and sisal No. 2 amounting to 63%, 69%, and 75%, respectively, after 12 months' exposure. Sisal hems, however, sink far more rapidly in water than does Manila, and show a far greater increase in girth, the percentage increase for No. 1 sisal, No. 2 sisal, and Manila "J" amounting to 9.26%, 6.21% and 3.60%, respectively, after exposure to sea-water for four months. D. J. NORMAN.

Hemp from Cyprus. ANON. (Bull. Imp. Inst., 1927, 25, 368—370).—Cyprus hemp is only slightly inferior in chemical composition and behaviour to commercial samples of Italian hemp, its cellulose content (calculated on the dry material) being 81.5%, as against 83.8% for Italian hemp. The length of the ultimate fibre is 6—50 mm. (average 24 mm.) and its diameter 0.010—0.051 mm. (average 0.027 mm.). D. J. NORMAN.

Lignocellulose of Victorian mountain ash (*Eucalyptus Regnans*). H. W. STRONG (J.S.C.I., 1928, 47, 87—90 τ).—In the determination of the cellulose in *Eucalyptus Regnans* it is shown that it is necessary to extract the sample with alkali hydroxide, and that this is best achieved by heating at boiling temperature for not more than 1 hr. with 1% sodium hydroxide solution. More concentrated solutions of alkali give diminished yields of total cellulose with a smaller content of α -cellulose. The amount of chlorine reacting to form hydrochloric acid is approximately twice the amount of chlorine combining with the lignone, the latter being 7.5—8% of the oven-dry sample.

Bamboos from British Guiana for papermaking. ANON. (Bull. Imp. Inst., 1927, 25, 370—374).—Of three samples of bamboo from British Guiana one, consisting entirely of stems, gave, on digestion with 20% of caustic soda for 4 hrs. at 160°, 46% of pulp (on the weight of air-dry raw material) which bleached fairly readily (yield of bleached pulp 39%) and gave a paper of excellent quality and strength. This sample compared favourably with Indian *Bambusa Tulda*, and should be suitable for the commercial production of paper pulp. Of the other two samples, one gave a pulp of similar quality though in lower yield, whilst the other required more severe treatment and gave a pulp of inferior quality. D. J. NORMAN.

Pulp and paper manufacture in North America. J. D'A. CLARK (Proc. Tech. Sect. Papermakers' Assoc., 1927, 7, 221—311).—North American pulp- and paper-making processes are described and compared with those worked in Great Britain. The advantages of using pulp in slush form are indicated, though there is no evidence that, from the point of view of strength, slush mechanical pulp is superior to baled moist mechanical pulp, or slush sulphite pulp to air-dry sulphite pulp. The relative merits of magazine grinders and pocket grinders are discussed both from the point of view of operating conditions and costs. The most satisfactory grade of pulp for newsprint is a soft well-cooked pulp made at a maximum cooking temperature of about 140°. Kraft pulp could be profitably made

in Great Britain, since a lower grade of wood can be used than is possible with the sulphite process, and higher yields of pulp are obtained. Pressures varying from 90 to 130 lb./in.² are employed, and the amount of active alkali (calculated as NaOH) may be 18–22% for kraft pulp, and about 25% for easy-bleaching sulphate pulp. The total cooking time is 4–7 hrs. with 2.5–4.5 hrs. at maximum pressure. For washing the pulp, a vacuum drum-washer, provided with a monel metal wire cloth, gives the most satisfactory results, particularly when used in conjunction with a rotary vacuum pump. Various methods which are being used for working up spent liquors are described. The monosulphite (Keebra) and semi-monosulphite (semi-Keebra) processes are in use for the treatment of wood, and their application in Great Britain for the treatment of esparto grass is suggested; a harder and stronger pulp is obtained in considerably higher yields than is given by the soda process. It is impractical to use the monosulphite process alone, owing to the unwieldy accumulation of thiosulphates in the liquor during recovery and regeneration. When, however, both processes are used, this difficulty can be avoided to a large extent by using as much as possible of the black liquor from the full monosulphite cook as a diluent for the cooking liquors for both full and semi-monosulphite cooks; further, in the recovery of the semi-monosulphite black liquor, the ash should be burnt without excessive firing in order to avoid the formation of sulphides (and hence of thiosulphates during the sulphiting operation), and the sulphite liquor should, as far as is practicable, be prepared from fresh caustic soda. Methods of testing mechanical and chemical pulps for strength are given, and modern tendencies in the design of pulp- and paper-making machinery are outlined. D. J. NORMAN.

Vanillin from sulphite-cellulose waste liquors. KÜRSCHNER.—See III.

PATENTS.

Recovery of waste fibrous material. L. KIRSCHBRAUN (U.S.P. 1,659,401, 14.2.28. Appl., 16.8.22. Renewed 30.6.26).—Waste fibrous stock containing bitumen, e.g., waste from the process described in B.P. 174,114 (B., 1922, 213 A), is agitated with bentonite and water at 95° to remove bitumen. T. S. WHEELER.

Production of cellulose by decomposition of vegetable fibres. O. C. STRECKER (U.S.P. 1,658,213, 7.2.28. Appl., 21.9.25. Ger., 30.9.24).—The fibres are heated at about 100° with a solution of an alkali salt of a homocyclic hydroxy-compound, e.g., sodium phenoxide. T. S. WHEELER.

Manufacture of cellulose compounds [viscose silk]. F. C. NIEDERHAUSER and H. B. KLINE (U.S.P. 1,655,626, 10.1.28. Appl., 21.12.26).—The addition of a small proportion (0.2%) of an alkali salt of a sulphated vegetable or animal oil to viscose solution lowers the surface tension and thus facilitates spinning. T. S. WHEELER.

Manufacture of viscose. J. M. LEAVER, Assr. to PACIFIC LUMBER Co. (U.S.P. 1,659,033, 14.2.28. Appl., 23.12.24).—The live bark of the *Sequoia* is used as a source of cellulose for artificial silk and film production. H. ROYAL-DAWSON.

Recovery of poisonous gases [hydrogen sulphide] in viscose manufacture. F. C. NIEDERHAUSER and H. B. KLINE, Assrs. to INDUSTRIAL FIBRE Co. (U.S.P. 1,655,709, 10.1.28. Appl., 9.3.27).—Evolution of hydrogen sulphide from viscose solution during spinning is inhibited by treating the concentrated solution prior to dilution and precipitation with carbon dioxide. The hydrogen sulphide and carbon disulphide evolved in the ensuing decomposition of sodium trithiocarbonate are absorbed in alkali solution. T. S. WHEELER.

Improvement of artificial fibres consisting of regenerated cellulose. HEBERLEIN & Co. A.-G. (B.P. 264,529, 15.1.27. Ger., 15.1.26).—The lustre and other properties of cellulose artificial silk are modified by treating the silk in fibre or fabric form with mercerising solutions (other than caustic alkali solution of or above 18% strength) in the absence of sulphonic acids of the benzene or naphthalene series, but optionally in the presence of restrainers such as mono- and poly-hydric alcohols, heterocyclic bases, etc. Suitable mercerising solutions are alkali hydroxide solution above d 1.075 and below d 1.21, sulphuric acid of $d > 1.41$, phosphoric acid of $d > 1.53$, hydrochloric acid of $d > 1.162$, nitric acid of $d > 1.32$, mixtures of these acids, and concentrated salt solutions. With caustic soda solution of d 1.162 at the ordinary temperature 4 sec. is a suitable time of treatment. The treated fabric shows not only a modified lustre, but also increased stiffness and a greater uniformity of structure. Mixtures of nitric and sulphuric acids containing less than 20% of water produce a non-translucent lustreless effect when nitric acid predominates, and a soft, broken-up, lustrous effect, resembling crêpe, when sulphuric acid predominates. D. J. NORMAN.

Manufacture of products having a basis of cellulose derivatives. W. A. DICKIE and J. H. ROONEY, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,655,677, 10.1.28. Appl., 21.11.25. U.K., 10.12.24).—The process of B.P. 249,946 (B., 1926, 532) is modified in that the plasticising of a coloured cellulose derivative, e.g., cellulose acetate, is performed in presence of a moulding powder comprising, e.g., cellulose acetate and monomethylxylenesulphonamide. T. S. WHEELER.

Cellulose ether composition. S. J. CARROLL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,658,369, 7.2.28. Appl., 7.10.25).—A flexible, transparent, waterproof sheet of value in the manufacture of photographic film is formed of an alkylcellulose, e.g., ethylcellulose (100 pts.), and a glyceryl acetate, e.g., triacetin (5–50 pts.). T. S. WHEELER.

Manufacture of articles of plastic material. A. LANDUCCI, Assr. to DU PONT-PATHÉ FILM MANUF. CORP. (U.S.P. 1,658,725, 7.2.28. Appl., 8.2.26. Fr., 20.2.25).—A cellulose ester or ether, e.g., cellulose acetate, dissolved in an organic solvent miscible with water, e.g., acetone, is precipitated in an aqueous bath, and the opaque product is rendered transparent by treatment with acetone or similar solvent. T. S. WHEELER.

Rejuvenation of cellulose ester plastic. J. H. SHERTS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,657,870, 31.1.28. Appl., 1.11.23. Renewed 1.8.27).—Brittle nitrocellulose plastic is rendered flexible

by immersion for about 1 min. in a mixture of a high-boiling solvent, *e.g.*, amyl acetate, and a low-boiling solvent, *e.g.*, benzene. The treated material is dried at about 38°.

T. S. WHEELER.

Reducing the viscosity characteristics of cellulose acetate. L. E. BRANCHEN and C. U. PRACHEL, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,658,368, 7.2.28. Appl., 6.10.26).—Cellulose acetate dissolved in formic or acetic acid is subjected at room temperature for about 50 hrs. to the action of ultra-violet light.

T. S. WHEELER.

Apparatus for the manufacture of artificial silk. F. W. SCHUBERT, Assr. to BRYLSILKA, LTD. (U.S.P. 1,662,478, 13.3.28. Appl., 7.7.27. U.K., 2.7.25).—See B.P. 258,371; B., 1926, 1009.

Manufacture of compositions with cellulose derivatives. H. DREYFUS (U.S.P. 1,661,169, 6.3.28. Appl., 26.5.23. U.K., 13.7.22).—See B.P. 205,195; B., 1923, 1172 A.

Machine for washing wool and other textile materials. G. HEINTZE (B.P. 271,063, 2.5.27. Ger., 15.5.26).

Nozzles for use in the production of artificial [silk] filaments. C. L. WALKER (B.P. 286,342, 19.11.26. Addn. to B.P. 253,209; B., 1926, 661).

Washing apparatus for use with parchmentising and like machines. P. ERKENS (B.P. 286,506, 9.5.27).

Treatment of colloids (B.P. 283,686).—See XIV. **Catgut substitute** (B.P. 285,752).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Treatment [weighting] of artificial silks. R. CLAVEL (B.P. 280,094, 30.3.27. Addn. to B.P. 277,602; B., 1928, 154).—Artificial silk (especially acetate silk) is given an acid reaction by immersion in an acid solution (phosphoric acid, chloroacetic acid, aluminium sulphate, etc.) with or without addition of glue or other protective colloid. The intermediate washing between impregnation and precipitation is thus rendered unnecessary, no precipitation occurring on the surface of material so treated. Alternatively, acid and/or protective colloid may be added to the impregnating bath or to the fibre before precipitation. The acid may be subsequently removed by means of a mild alkaline bath, to which hæmatein, or other dyes containing tannin, may be added.

C. HOLLINS.

Dyeing process [for fibres]. A. ESCAICH and P. J. WORMS (U.S.P. 1,661,606, 6.3.28. Appl., 20.11.23. Ger., 25.11.22).—See B.P. 227,906; B., 1925, 204.

Increasing the affinity of animal fibres for dyestuffs. E. STOCKER, Assr. to CHEM. WORKS, FORMERLY SANDOZ (U.S.P. 1,662,404, 13.3.28. Appl., 27.10.27. Ger., 20.11.26).—See B.P. 245,759; B., 1927, 475.

Dyeing [cotton] with basic dyes involving the use of arylamides of aromatic hydroxycarboxylic acids as mordants. A. GUENTHER, J. HALLER, and E. KÖSTER, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,663,451, 20.3.28. Appl., 29.1.26. Ger., 19.2.25).—See G.P. 441,326; B., 1927, 905.

Dyeing of cellulose esters or ethers. R. S. HORSFALL, L. G. LAWRIE, and J. HILL, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,662,514, 13.3.28. Appl., 2.3.27. U.K., 11.6.26).—See B.P. 275,373; B., 1927, 776.

Metallising process [for textiles]. J. VON BOSSE (U.S.P. 1,661,517, 6.3.28. Appl., 6.5.26. Ger., 27.5.25).—See B.P. 252,710; B., 1927, 820.

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

Alteration of concentrated solutions of sodium bisulphite. E. ISNARD (J. Pharm. Chim., 1927, [viii], 6, 211—212).—A concentrated solution of sodium bisulphite, prepared in 1923, when added to an aqueous sodium thiosulphate solution produced a precipitate and hydrogen sulphide was evolved. The bisulphite solution, which originally contained 38% Na₂SO₃, was found to have the following composition: water 60.7%, Na₂SO₃ 1.27%, NaHSO₄ 16.54%, Na₂SO₄ and sulphur (traces) 11.49%.

E. H. SHARPLES.

Manufacture of ammonium chloride from ammonium sulphate and common salt. E. HOCHBERGER (Chem.-Ztg., 1928, 52, 22—23).—Common salt is added to a saturated solution of ammonium sulphate at the boiling point. Sodium sulphate separates, and is washed with a solution of sodium sulphate saturated at 32.4° to remove adherent liquor and ammonium chloride. The mother-liquor is cooled to 40°. Ammonium chloride separates and is recrystallised. The liquors are used repeatedly in cycle.

S. I. LEVY.

Decomposition of alkaline-earth sulphates. L. E. BHATT and H. E. WATSON (J. Indian Inst. Sci., 1927, 10A, 117—129).—When calcium sulphate is heated with carbon, the sulphide formed begins to react with the sulphate at about 950° with evolution of sulphur dioxide, but even at 1150° this reaction is relatively slow. The amount of sulphur as dioxide and trioxide which is expelled from calcium sulphate by heating with silica increases with increase in the relative amount of the latter, and with 2 mols. of silica per mol. of sulphate decomposition is nearly complete in 4 hrs. at 1100°. Regrinding the reacting mixture after it has been heated for some time much facilitates reaction, whereas moisture has an unfavourable effect. Addition of carbon to the mixture of silica and sulphate results in a decrease in the amount of sulphate decomposed, probably because of the formation of sulphide, which reacts with silica less readily than does sulphate. At the same time, however, there is an increase in the quantity of volatile sulphur compounds formed, possibly owing to the formation of an intermediate product, perhaps the sulphite, which is more reactive towards silica than is the sulphate. The presence of iron in a reacting mixture of silica and calcium sulphate is not advantageous on account of the somewhat extensive sulphide formation, and it appears that the maximum amount of volatile sulphur compounds is secured by heating with silica only. Calcium sulphite at 950° decomposes chiefly according to the equation $4\text{CaSO}_3 = 3\text{CaSO}_4 + \text{CaS}$; silica almost completely prevents the formation of sulphide. The decomposition points of the sulphates of magnesium, strontium, and

barium when heated in air are at 740°, 1100°, and above 1100° respectively. Silica promotes the decomposition of magnesium sulphate to a considerable extent, but with barium sulphate the effect is much less, and with strontium sulphate least of all. R. CUTHILL.

Production of ammonium phosphates from secondary and tertiary calcium phosphates. C. MÜCKENBERGER (Z. anorg. Chem., 1928, 169, 81—95).—Tricalcium phosphate readily reacts at 180—220° and under 11—17 atm. pressure with phosphoric acid, only a slight excess of acid being required, but equilibrium is established when a large part of the phosphate has been converted only into dicalcium phosphate. In presence of ammonium sulphate, however, the yield of soluble phosphate is increased, owing to the reaction $\text{Ca}_3(\text{PO}_4)_2 + 3(\text{NH}_4)_2\text{SO}_4 + 4\text{H}_3\text{PO}_4 = 6\text{NH}_4\text{H}_2\text{PO}_4 + 3\text{CaSO}_4$, and with an amount of phosphoric acid about 40% more than the theoretical amount the whole of the phosphate is rendered soluble. It is suggested that a valuable artificial fertiliser might be manufactured from natural phosphate by opening it up in this way, then converting the calcium sulphate into ammonium sulphate by one of the available methods, and using part of the filtrate, after it has been heated so as to drive off some ammonia and give a strongly acid product, to open up fresh phosphate. For this purpose a considerable excess of monoammonium phosphate is required, but the temperature need not exceed 100°; diammonium phosphate has very little solvent action on tricalcium phosphate. The action of ammonia and carbon dioxide on dicalcium phosphate apparently involves a number of simultaneous reactions, an equilibrium finally being set up between an ammoniacal solution of ammonium phosphate and unchanged dicalcium phosphate, tricalcium phosphate, calcium carbonate, and a little calcium hydroxide. Under the most favourable conditions a yield of 50% of soluble phosphates is obtained. R. CUTHILL.

Arsenious oxide in solution. MILLAR.—See XX.
Oxidation of ammonia to nitric acid. FOWLER and others.—See XXXIII.

PATENTS.

[Preparation of silica gel for] the recovery of gas and vapours from gas mixtures. C. F. BOEHRINGER & SÖHNE G.M.B.H., and E. WILKE (Swiss P. 119,220—1, 20.10.25. Ger., 3.11.24).—An adsorbent silica gel is obtained by treating a solution of an alkali silicate with (A) a solution of a salt of a metal the silicate of which is only slightly soluble in water, or (B) with carbon dioxide. In either case the gelatinous precipitate is collected, washed, dried, and dehydrated at 200—500°.

A. R. POWELL.

Collection of phosphoric acid. B. G. KLUGH, Assr. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,659,146, 14.2.28. Appl., 7.12.20. Renewed 12.5.27).—The gases containing phosphoric acid obtained by processes such as those of U.S.P. 1,492,713 and 1,497,727 (B., 1924, 557,632) are freed from phosphoric acid by passage through a filter-bed of sand, from which phosphoric acid is periodically removed by treatment with a current of air or water. The dilute solution obtained by the

latter process can be concentrated by treatment with the hot gases passing to the filters. T. S. WHEELER.

Manufacture of anhydrous magnesium chloride. R. GRIESSBACH and K. RÖHRE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,661,894, 6.3.28. Appl., 22.11.26. Ger., 7.12.25).—Crystals of the chloride are treated in a gaseous mixture containing 10—25% HCl at 200—350°.

H. ROYAL-DAWSON.

Manufacture of zirconium compounds. C. J. KINZIE, Assr. to TITANIUM ALLOY MANUF. Co. (U.S.P. 1,658,807, 14.2.28. Appl., 27.5.26).—A siliceous zirconium ore is fused with potassium hydroxide at 950°, and the product is treated with dilute sulphuric acid at 65° to form a solution from which, on cooling, potassium zirconium sulphate separates. Alternatively, the ore is fused with sodium carbonate, and potassium chloride is added after treatment with sulphuric acid.

T. S. WHEELER.

Recovery of sulphur. I. G. FARBENIND. A.-G. (Swiss P. 119,973, 7.11.25. Ger., 6.7.25).—The solution of sulphur in a suitable solvent is allowed to fall through a heated vessel on to a plate heated at such a temperature that the solvent is distilled away and the separated sulphur is melted.

A. R. POWELL.

Catalytic oxidation of sulphur dioxide. A. O. JAEGER and J. A. BERTSCH, Assrs. to SELDEN Co. (U.S.P. 1,657,753—4, 31.1.28. Appl., [A] 6.2.26, [B] 15.2.26).—(A) A calcium, ferric, or other zeolite is treated with an alkali vanadate, tungstate, or chromate solution at 60° to give a product of value as a catalyst for the oxidation of sulphur dioxide. (B) Potassium vanadate solution is neutralised with dilute acid and treated with sodium silicate and hydrochloric acid at 70°, and the product is treated with copper sulphate, calcium chloride, or ferric sulphate solution.

T. S. WHEELER.

Extraction of phosphorus from its compounds by reduction. H. WITTEK (B.P. 275,145, 20.11.26. Ger., 27.7.26).—Phosphorus is produced concurrently with calcium cyanamide by mixing a phosphorus compound, other than a heavy-metal compound, with calcium or other carbide, and heating in a furnace in a current of nitrogen, the heat of formation of the cyanamide being utilised for the reduction. W. G. CAREY.

Production of water-gas and hydrogen. SYNTHETIC AMMONIA & NITRATES, LTD., and H. A. HUMPHREY (B.P. 282,573, 22.2.27).—Semi-coke in lump form is made from slack coal of high caking power by carbonising below 600° after preheating in presence of oxygen to control its caking properties. The semi-coke, which may be further heated at 800—900° to drive off volatile matter, is fed direct from the retort to a water-gas generator operated with excess of steam and having a shallow fuel bed, and the resulting gas mixture is compressed and treated with excess of steam above 800° in the presence of a catalyst, e.g., nickel, to convert methane into carbon monoxide and hydrogen. If hydrogen for ammonia synthesis is desired the carbon monoxide is converted into carbon dioxide by treatment with steam in the presence of an iron oxide catalyst at about 500°, the carbon dioxide being removed, preferably, by dissolution in water under pressure.

W. G. CAREY.

Manufacture of hydrogen peroxide. H. S. TAYLOR, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,659,382, 14.2.28. Appl., 1.2.26).—An equimolecular mixture of hydrogen and oxygen containing mercury vapour is passed through a quartz tube, in which it is subjected to the action of the rays emitted by a mercury vapour lamp. T. S. WHEELER.

Production of argon. W. DANNENBAUM, Assr. to PACIFIC NITROGEN CORP. (U.S.P. 1,658,631, 7.2.28. Appl., 13.10.23).—A mixture of argon and nitrogen is passed with excess of hydrogen under suitable conditions of temperature and pressure over a catalyst adapted to induce formation of ammonia, and the mixture of hydrogen and argon resulting is treated with excess of oxygen to form water, the argon being finally purified from oxygen by treatment with copper.

T. S. WHEELER.

Rapid evaporation to dryness of ammonium nitrate solutions. C. TONIOLO (U.S.P. 1,661,203, 6.3.28. Appl., 27.1.26. It., 7.2.25).—See B.P. 247,228; B., 1927, 408.

Purifying and bleaching of heavy spar. K. EBERS (U.S.P. 1,663,159, 20.3.28. Appl., 22.12.25. Ger., 24.12.24).—See B.P. 245,155; B., 1926, 742.

Manufacture of aluminium compounds. G. MUTH (U.S.P. 1,661,618, 6.3.23. Appl., 29.3.21. Ger., 13.5.19).—See G.P. 319,420; B., 1920, 517 A.

Hydrogen-ion comparator (U.S.P. 1,659,529).—See I. Preparation of hydrogen (F.P. 621,902).—See II. Electrolysis of water (Swiss P. 120,249). **Electrolysis of salt solutions** (G.P. 446,009).—See XI. **Base-exchange substances** (G.P. 445,377—8).—See XXIII.

VIII.—GLASS; CERAMICS.

Ceramic colours and their use in vitreous enamels. W. N. HARRISON and T. O. HARTSHORN (J. Amer. Ceram. Soc., 1927, 10, 747—760).—The production of many colouring stains, including such substances as cobalt oxides, chromic oxides, cadmium sulphide and selenide, and copper oxide as bases, and their incorporation in vitreous enamel bodies, are discussed. It is stated that removal of soluble matter from the calcined stains, especially selenium-red stains, by washing prevents a spotted appearance in the fired enamels. It was found necessary to duplicate the time and temperature of calcination of the stains, and the furnace atmosphere in order to duplicate the colour obtained with a given batch composition; to such variations, again, selenium-red stains are very sensitive. Opacifiers introduced either in the raw batch or mill batch generally tend to lighten the colour produced by a given stain. Some materials, however, probably due to chemical reaction have a counteracting tendency to darken the enamel. Cryolite and felspar in the frit have little effect on the shades obtained except a slight lightening of tone. On the other hand, sodium antimonate produces a darker tone in spite of its tendency to opacity; this effect is accentuated by the substitution of silica for felspar. Lead oxide darkens the shade of stains, especially red stains. A. T. GREEN.

Uranium oxide colours and crystals in low-temperature glaze combinations. J. R. LORAH (J. Amer. Ceram. Soc., 1927, 10, 813—820).—Glazes containing lead, sodium, and boron combined with other inorganic compounds were compounded according to recipe and fired to a definite maturing temperature in an oxidising atmosphere. Colours varying from yellow to black were obtained. It is shown that a small quantity of uranium oxide in a lead glaze produces a yellow colour, 5% a bright orange, and 10% a dark orange colour. Black spots, noted in certain glazes, are due to the reduction of the yellow uranium oxide to the black oxide. Glazes containing, additionally, boron compounds with silica give yellow glazes with a greenish tinge and show no tendency towards the formation of orange colours or crystalline structures. Lead oxide, owing to its volatility during firing, appears to be essential to the production of crystals. Two types of crystals depending on the ratio of the uranium to the lead content are noted. The effect of adding such substances as clay, calcium fluoride and carbonate, and barium carbonate appears to be to decrease the amount of lead which must be volatilised before crystal formation commences. A. T. GREEN.

Dehydration and firing behaviour of clays. R. F. GELLER and W. H. WADLEIGH (J. Amer. Ceram. Soc., 1927, 10, 929—955).—Results of laboratory studies of the drying and water-smoking behaviour of twelve clays used in the manufacture of bricks and tiles are correlated with data obtained on works where the clays are in actual use. As the result of preliminary drying tests, two equations were developed by means of which the air temperature and the relative humidity could be calculated for any given schedule. In the drying tests the maximum temperature reached was 110° and the minimum relative humidity 4%. The water-smoking tests were carried out in an electrically-heated furnace specially constructed to enable the amount of loss in weight and the temperature lag within the specimens to be observed at intervals of 15 min. In the works' tests thirteen kilns were investigated; two tests were made, one under conditions of normal practice and the other with kiln setting and firing method modified in accordance with conclusions based on the results of the first firing. The shortest time in which the drying shrinkage could be completed without injury to the specimens varied from 4 to 42 hrs.; the fire clays were much more difficult to dry than the shales and surface clays. No direct relation was found between the elimination of shrinkage and pore water and the removal of hygroscopic and chemically combined water. It is concluded that clay wares such as bricks and paving blocks could be heated with safety to 1000° if proper precautions were taken, but in practice a longer period is necessary owing to the setting of insufficiently dried ware, the limitations of kiln construction and design, and the impracticability of removing promptly all water vapour from the kiln. F. SALT.

Cyanite and diasporite refractories. E. J. VAC-HUSKA and G. A. BOLE (J. Amer. Ceram. Soc., 1927, 10, 761—773).—Mixtures containing diasporite-cyanite, di-

aspore-clay, and cyanite-clay were made, and methods for manufacturing and testing such refractories are examined. Diaspore products show a permanent shrinkage after prolonged subjection to high temperatures. Such after-shrinkage, probably due to an inversion of the diaspore to corundum, and accompanied by an increase in specific gravity (3.3—4.0), can be prevented either by the addition of 20% or more cyanite to the diaspore mixtures, or the precalcination of the diaspore to as high a temperature as possible. The addition of cyanite, however, must be carefully controlled so that the resistance to thermal shock is not impaired. Data concerning the linear shrinkage, porosity, and modulus of rupture of the fired products are given.

A. T. GREEN.

Spalling and loss in compressive strength of firebrick. H. R. GOODRICH (J. Amer. Ceram. Soc., 1927, 10, 784—794).—The loss in compressive strength of a brick, which is the result of thermal shock attending repeated heat-treatments at high temperatures (1250° and 1350°) and coolings in air, is suggested as a satisfactory criterion of spalling tendency. It is indicated that the development of a vitrified structure markedly predisposes the material to spalling. Whilst a material with a high quartz content and a clay bond possesses a comparatively low initial strength, yet it retains a higher percentage of strength after thermal shock than do the fireclay products investigated. Pre-treatment at high temperatures, by inducing extra vitrification, helps the failure of certain fireclay products in the spalling test. A review of methods of testing spalling tendency is given.

A. T. GREEN.

Electrical resistance of porous materials. KNOWLER.—See XI.

PATENTS.

Manufacture of refractory products. W. A. FARISH, Assr. to BUFFALO REFRACTORY CORP. (U.S.P. 1,658,406, 7.2.28. Appl., 31.8.22).—A mixture of graphite and an electric furnace refractory product, e.g., silicon carbide, is impregnated with a carbonaceous binder, formed, carbonised, and dipped in a liquid flux, e.g., sodium silicate.

T. S. WHEELER.

Manufacture of glass in continuous sheets by flowing and rolling. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY, & CIREY (B.P. 280,582, 11.11.27. Fr., 15.11.26).

IX.—BUILDING MATERIALS.

Composition of alite. O. REBUFFAT (Giorn. Chim. Ind. Appl., 1927, 9, 520).—Objection is raised to the formula $8\text{CaO}\cdot\text{SiO}_2\cdot\text{Al}_2\text{O}_3$, proposed by Jänecke for alite, the principal constituent of anhydrous Portland cement (B., 1926, 747), mainly on the ground of the difference in composition between ordinary Portland cement and that corresponding with the above formula.

T. H. POPE.

Electrical resistance of porous materials. KNOWLER.—See XI. **Concrete for water reservoirs.** MABEE.—See XXIII.

PATENTS.

Manufacture of aluminous cement. E. C. R. MARKS. From G. POLYSIUS (B.P. 286,122, 6.5.27).—

The raw unground materials are fed into a rotary retort, the size of the pieces used being dependent on their physical and chemical nature. H. ROYAL-DAWSON.

Preservation of wood and solutions therefor. L. P. CURTIN, Assr. to CURTIN-HOWE CORP. (U.S.P. 1,659,135, 14.2.28. Appl., 15.5.26).—A mixture of zinc chloride, calcium acetate, arsenic trioxide, and dilute acetic acid is of value as an insecticide and fungicide; zinc meta-arsenite is deposited on exposure to air.

T. S. WHEELER.

Production of asphalt-paving mixtures. J. S. DOWNWARD (U.S.P. 1,662,377, 13.3.28. Appl., 11.8.23).—Separately prepared mixtures of mineral matter with blown asphalt of high and low m.p., respectively, are mixed while cold, and the mixture is heated at the place of use, whereby a mixture of standard asphalt content is produced.

J. S. G. THOMAS.

Road binder. M. F. COUGHLIN (U.S.P. 1,662,299, 13.3.28. Appl., 24.2.22).—The binder consists of a basic bituminous material, sulphite liquor, and lactic acid.

H. ROYAL-DAWSON.

Production of porous [heat- and sound-]insulating materials. E. C. BAYER (B.P. 265,968, 8.2.27. Denm., 9.2.26).—A foam of a solution of organic matter, e.g., glue, chrome-gelatin, caseinates, etc., is produced by means of soap, saponin, or similar material, or by adding to the organic matter foam previously prepared from such substances; in the latter case the strength of the foam is increased by adding gelatin or glycerol.

W. G. CAREY.

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

Chemical composition and roasting of a Siegerland spathic iron ore. W. STAHL (Chem.-Ztg., 1928, 52, 162).—In the ordinary roasting process iron pyrites is mainly oxidised, but the copper sulphide sometimes present is difficult to decompose and passes on to the blast furnace, where its presence is objectionable. Chloride and sulphide roasting, with subsequent leaching to remove copper, are suggested.

C. J. SMITHELLS.

Air-hardening of rivet steels. H. K. HERSCHMAN (U.S. Bur. Stand. Tech. Paper No. 358, 1927, 22, 141—169).—The suitability of air-hardening nickel-chromium sheets for riveting armour plate has been examined, and the effect of varying the carbon content and of the addition of manganese and molybdenum on the ballistic resistance has been determined. The values obtained in the shear test are a good indication of the ballistic and impact properties of a steel for use as rivets. The most satisfactory results are obtained with steels containing 4—6% (Ni + Cr) provided that the carbon content is within the limits 0.17—0.25%. For the steel with 3.5% Ni and 1.5% Cr the optimum impact resistance is obtained with 0.20% C. Equally good results at a slightly lower cost may be obtained with smaller quantities of nickel and chromium provided that a small proportion of molybdenum or manganese or both is added, the carbon content also being suitably adjusted. With too high a carbon content there is a tendency for long-shanked rivets to become loose owing

to the expansion caused by the slow change from austenite to martensite which takes place at relatively low temperatures in these steels.

A. R. POWELL.

Contraction of steel castings. F. KÖRBER and G. SCHITZKOWSKI (*Stahl u. Eisen*, 1928, 48, 129—135, 172—178).—In making steel castings a total shrinkage of about 2% must be allowed for; even with this allowance sound castings of complicated design are not obtained unless certain conditions are observed. During the cooling of a casting with flanges or other projecting parts, fracture usually occurs almost immediately after the casting has solidified, *i.e.*, between the f.p. of the metal and 800°. Steel with the lowest possible content of sulphur and phosphorus should be used, as these elements usually segregate to the weakest parts of the castings and almost invariably cause fracture; addition of 0.6—0.8% Mn to the alloy almost entirely removes any trouble likely to be caused by sulphide segregation.

A. R. POWELL.

Yield point in steel at various temperatures. J. MUIR (*J. Roy. Tech. Coll., Glasgow*, 1927, [4], 14—23).—The yield points at various temperatures have been determined for mild steel in the form of wire. The temperatures required were obtained by electrically heating the wires. The rate of extension at a yield point is very greatly increased by increase of temperature, whilst the phenomenon of the yield point (*i.e.*, the extension of the wire by jerks and not by creeping) disappears entirely at 250°, in opposition to the observations of Lea and Crowther (*Engineering*, 1914, 98, 487).

L. M. CLARK.

Determination of the permanent strength of steel at high temperatures. F. KÖRBER (*Z. Metallk.*, 1928, 20, 45—49).—The permanent strength of a metal at high temperatures is defined as the load per unit area that will just continue to produce an elongation when constantly applied; this value largely determines the suitability of a constructional material to withstand constant high pressures at elevated temperatures. For practical purposes the load which causes an increase of not more than 0.001% in the elongation in the period between the third and sixth hour after application is determined. To carry out this work the rate of increase of elongation is determined for a number of different loads and the results are plotted against the load. A machine and furnace suitable for the determination of small increases in the length of a specimen under constant load at high temperatures are illustrated, and examples are given of the results obtained with copper, zinc, and several steels.

A. R. POWELL.

Stepped lowering of the A1 transformation in steels. T. MURAKAMI (*Sexagint [Osaka celebration]*, Kyoto, 1927, 171—179).—The appearance of the A1 transition in steel at definite temperatures governed by the rate of cooling and the maximum temperature of heating is explained on the basis of Tammann's theory of the velocity of crystal growth.

H. F. GILLBE.

Density of hot-rolled and heat-treated carbon steels. H. C. CROSS and E. E. HILL (*U.S. Bur. Standards Sci. Paper No. 562*, 1927, 22, 451—466).—The density of commercially pure and electrolytic iron,

and of carbon steels containing 0.09—1.29% C, was determined after hot-rolling, annealing, quenching, and tempering. The density of pure iron was 7.864, and of hot-rolled carbon steels containing up to 1.3% C was 7.855—0.032*C*, where *C* is the carbon content. A similar decrease in density with increasing carbon content was found for annealed steels, for which the value of *d* was 7.860—0.04*C*. Quenching decreases the density owing to the formation of martensite, and is most marked in the 1% carbon steel. The density increases on tempering and approaches that of the annealed material when tempered above 600°.

C. J. SMITHELLS.

Practical problems of corrosion. III. Formation of rust and its consequences. IV. Corrosion of wrought iron in relation to that of steel. V. Corrosion and protection at the contacts of dissimilar metals. U. R. EVANS (*J.S.C.I.*, 1928, 47, 55—62 T, 62—69 T, 73—77 T).—III. Rust is not the direct corrosion product of iron, but a precipitate, mainly consisting of iron hydroxides; it is usually formed at a sensible distance from the site of corrosion and varies in character with the conditions of precipitation. Hard waters produce calcium and magnesium compounds on the cathodic portions of rusting specimens, and these substances obstruct the access of oxygen to the metal, thus reducing the rate of corrosion; the corrosion of steel half immersed in magnesium sulphate solution of various concentrations has been measured. The "apparent volume" of the "rust sludge" produced by numerous liquids has also been determined; it may be several hundred times the volume of the iron destroyed, thus explaining the rapid choking of pipes. The true volume of rust also exceeds that of the iron destroyed, and the expansion may sometimes cause disintegration where the rust is precipitated within the metal, or in crannies between plates. This is more likely to occur in atmospheric attack than in immersed corrosion, but, if a sufficient margin of strength has been provided, the expansion will more often tend to plug the pore or crevice with rust, thus putting an end to the action.

IV. When deeply immersed in acids, wrought iron is attacked, as a whole, quicker than steel, but the corrosion proceeds most quickly when it reaches the internal portions; the material consists of alternate resistant and corrodible layers, the latter containing obstructive flaky particles. The action of drops of dilute sulphuric acid (such as drops of rain in coal-burning districts) is much the same on wrought iron as on steel. Specimens of wrought iron, half immersed in solutions of salts, are attacked rather more slowly than specimens of steel, but the difference is small. On the other hand, wrought iron appears to have a definite advantage for buried work (*e.g.*, pipes) which has to withstand anodic attack from stray electric currents in the presence of chlorides; the total amount of metal destroyed at places where discontinuities occur in the protective tar or other covering is determined by Faraday's law, and under similar conditions will be the same for both materials; but whereas the attack on steel burrows downwards, producing smooth, rounded grooves, the attack on wrought iron will usually tend to be diverted sideways by the obstructive layers, and thus perforation is likely—

under ordinary circumstances—to be deferred for a longer time if wrought iron is employed.

V. A series of potential measurements has been made between steel and seven other metals in different liquids in presence of oxygen. Zinc and cadmium are usually anodic towards steel; copper and nickel are usually cathodic, whilst tin, lead, and aluminium are variable. Measurements of the enhancement of the corrosion of steel by contact with copper, nickel, and lead plates of different sizes indicate that copper is the most dangerous and lead the least dangerous. Contact with zinc was found to afford complete protection to steel in most of the liquids tested, but the amount of zinc consumed—although low in liquids containing calcium salts—was found, in solutions of sodium chloride or sulphate, to be actually greater than the consumption of steel which took place when zinc was not present.

Disposal of acid iron wastes from a steel mill. S. E. COBURN (*Ind. Eng. Chem.*, 1928, 20, 248—249).—These wastes consisted of spent acid liquor from pickling steel, rinse waters from washing the metal after pickling, and spent lime solution from neutralising any acid adhering to the metal after rinsing. The rinse water and lime solution could be passed into the sewer after treatment, but in the case of the spent acid it is recommended to concentrate it and, after recovering ferrous sulphate, either return the mother-liquor to the pickling vat or neutralise the free acid with scrap iron and recover the ferrous sulphate so produced. C. JEPSON.

Gravimetric determination of titanium in iron alloys. Separation [of titanium] from iron. J. CIOCHINA (*Z. anal. Chem.*, 1928, 73, 40—46).—The alloy (0.5—10 g.) is dissolved in aqua regia and the solution evaporated with 5—20 c.c. of sulphuric acid (*d* 1.8) until copious fumes are evolved. The solution is cooled, diluted with 100 c.c. of cold water, neutralised with sodium carbonate, treated with 2 c.c. of sulphuric acid (1 : 1) and 6 g. of sodium thiosulphate, and poured slowly into 400 c.c. of boiling water. After boiling for 1—2 min. the liquid is cooled, filter-pulp added, and the precipitate collected, washed with hot water, and ignited to titania. The use of an excess of thiosulphate is stated to prevent precipitation of part of the titania. A. R. POWELL.

[Iron] alloys with a high content of nickel and chromium. P. CHEVENARD (*Rev. Mét.*, 1928, 25, 14—34).—A review of the present state of our knowledge of the thermal, electrical, and mechanical properties of nickel-chromium and iron-nickel-chromium alloys, and a brief account of the valuable properties of commercial alloys in these groups. A. R. POWELL.

Anomalies in nickel-steels and their applications. C. E. GUILLAUME (*Rev. Mét.*, 1928, 25, 35—43).—A review of the author's work on iron-nickel alloys during the past 30 years, with an account of the practical application of the peculiar thermal and magnetic properties of these alloys to industrial uses. A. R. POWELL.

Rapid determination of silica, alumina, lime, and magnesia in low-manganese iron ores. R. P. HUDSON (*Chemist-Analyst*, 1927, 16, [2], 4—8).—Aluminium is precipitated as phosphate with sodium ammonium phosphate and sodium thiosulphate; lime

and magnesia are determined in an aliquot part of the filtrate. The magnesium ammonium phosphate is corrected for manganese. CHEMICAL ABSTRACTS.

Detection of cobalt in steel. J. MOIR (*J. S. African Chem. Inst.*, 1928, 11, 33—34).—The steel (10 mg.) is dissolved in 8 c.c. of aqua regia and the solution examined with a direct-vision spectroscope, using a wide slit; with 5% Co or more the red end of the spectrum is cut off entirely and two narrow absorption bands (λ 6210 and 6070) are visible in the orange. The latter are not seen with less than 5% Co, but two deep bands appear in the red at 7000 and 6610. Addition of water destroys the characteristic absorption effect of cobalt.

A. R. POWELL.

Anomaly of reheating after cold-beating shown by copper and steels. P. NICOLAU (*Compt. rend.*, 1928, 186, 696—699).—The curves relating the hardness (Brinell) and the temperature of reheating of copper bars (preheated to 700° and cold-beaten to the extent of 5—660%) show systematic anomalies after a preliminary "germination" period, during which the internal equilibrium destroyed by the cold-beating is gradually restored, corresponding with a minimum hardness. Alpha-steels show points of inflexion, and the internal tensions revealed after immersion in a 0.15% aqueous solution of mercuric chloride disappear at reheating temperatures corresponding with these points.

J. GRANT.

Electrical properties of ferronickels containing added chromium. P. CHEVENARD (*Compt. rend.*, 1928, 186, 431—433).—The author summarises the results of his researches. The allotropic transformation of iron-nickel alloys, rich in iron, is accompanied by an increase or decrease in resistivity on heating or cooling, respectively, to an extent increasing with the nickel content up to 30%, and then decreasing till it becomes zero for the compound Fe₂Ni (34.45% Ni). The transformation of alloys rich in chromium and poor in nickel from the α - to the γ -state is marked by a drop in resistivity. The reversible magnetic transformation of austenitic alloys is shown as a break on the resistivity-temperature curve, and decreases in intensity with the nickel and chromium contents. The addition of chromium lowers the temperature of the transformation. Between 500° and 550° paramagnetic chromium ferronickels show a second, imperfectly reversible transformation, the temperature of which is independent of the composition of the alloy. The curves representing the thermo-electric properties of ferronickels as a function of temperature are analogous.

J. GRANT.

Causes of the variation in volume accompanying the hardening of light aluminium-copper alloys. P. CHEVENARD and A. PORTEVIN (*Compt. rend.*, 1928, 186, 144—146).—The effect of the temperature of reheating on the change in length of an aluminium alloy containing 4.30% Cu, 0.80% Mn, and 0.38% Si has been determined, the alloy being previously tempered. The diminution in dilatibility is an approximate measure of the Al₂Cu precipitated. Above 170° the curves conform to the authors' theory (B., 1923, 391 A) of the precipitation of Al₂Cu, but below 170° another reaction, accompanied by a diminution in volume, co-exists with the first. This is attributed to the presence

of impurities rather than to the production of a state analogous to the martensitic structure of steel.

J. GRANT.

Rapid analysis of ordinary brass and red brass. W. KOLLREPP (Chem.-Ztg., 1928, 52, 183).—A technical method for the rapid analysis of brass free from manganese and phosphorus is as follows: 1 g. of turnings is dissolved in 10 c.c. of water and 15 c.c. of nitric acid, diluted with 20 c.c. of hot water, filtered, washed, and the residue of tin oxide ignited and weighed. 10 c.c. of sulphuric acid (*d* 1.84) are added to the filtrate, which is kept for 15 min. at 60° and the precipitated lead sulphate collected on a filter, ignited, and weighed. The filtrate is neutralised with ammonia, acidified with hydrochloric acid, and the boiling solution treated with hydrogen sulphide. The copper sulphide is removed by filtration and neglected, the filtrate boiled to expel hydrogen sulphide, oxidised with 5 c.c. of nitric acid (*d* 1.4), excess of ammonia added, and the whole boiled. The precipitated ferric hydroxide is collected on a filter, washed, and weighed as ferric oxide. Nickel is determined in the filtrate by the glyoxime method, and, after removal, zinc is precipitated with hydrogen sulphide and weighed as the oxide. Copper is determined by difference. C. J. SMITHELLS.

Action of hydrochloric acid on extra-pure aluminium. J. CALVET (Compt. rend., 1928, 186, 369—371).—The resistance to the action of 2.5*N*-hydrochloric acid at 18° previously recorded for extra-pure aluminium (cf. Matignon and Calvet, A., 1928, 251) is only temporary, and after a few days the metal is attacked at a rate which increases with the time and finally remains constant. The period of passivity depends on the origin of the aluminium and on the impurities it contains, and is prolonged if the metal is in a laminated condition. The rate of attack was measured by the volume of gas evolved per sq. cm. of surface. J. GRANT.

Corrosion of aluminium. W. GUERTLER (Z. Metallk., 1928, 20, 104—112).—A review of the chief factors influencing the rate of corrosion of aluminium and of the mechanism of corrosion phenomena in aluminium. Inclusions of coarse particles of silicon, FeAl₃, and non-metallic impurities cause the protective layer of oxide to break, thus allowing penetration into the metal of corroding media. During rolling or drawing these inclusions tend to break up and may cause scaling on the surface or minute surface fissures. Trouble due to this cause may be averted by annealing at 400°, followed by quenching, whereby the silicon and iron are retained in solid solution or caused to separate in a finely-divided form. Care should be taken in working and annealing the metal not to develop a coarsely crystalline structure which tends to cause a break-up of the protective skin of oxide. A. R. POWELL.

Destruction of aluminium by mercury and means for preventing it. H. RÖHRIG (Korrosion u. Metallschutz, 1927, 3, 121—123; Chem. Zentr., 1927, II, 974).—The amalgamation of aluminium vessels by mercury causes rapid destruction of the vessel. Heating the spot removes the mercury effectively, but there is great danger of the vapour condensing on other parts; treatment with concentrated nitric acid, which dissolves

the mercury without attacking the aluminium, is satisfactory for thick-walled vessels, but the safest procedure consists in rubbing the spot with a warm 10% solution of potassium dichromate which, by converting the mercury into a brown oxide or basic chromate, allows it to be readily rubbed off. A. R. POWELL.

Light metals and alloys. Aluminium. Magnesium (U.S. Bur. Stand., Circ. 346. 403 pp.).—A review of the physical properties of aluminium and magnesium and their alloys.

Determination of fluorine in zinc blende. L. FRESSENIUS, K. SCHRÖDER, and M. FROMMES (Z. anal. Chem., 1928, 73, 65—69).—The finely-powdered ore (2 g.) is fused with 10 g. of a mixture of equal parts of sodium carbonate and peroxide, the product leached with water, and the solution treated with 8 g. of ammonium carbonate, evaporated to 50 c.c., and filtered. The cold filtrate is acidified with 1:1 sulphuric acid and 10 c.c. excess acid are added, followed by 5 c.c. of 3% hydrogen peroxide and 5 c.c. of a standard titanium sulphate solution (1 c.c. = 1 mg. of titania). The mixture is then diluted to 150 c.c. in a white porcelain basin. A similar solution is prepared using the same reagents but without addition of the ore; to this solution is added a solution of sodium fluoride (1 c.c. = 1 mg. of fluorine) until the colour has faded to match that of the assay. The burette reading in c.c. gives directly the number of mg. of fluorine in 2 g. of the blende. A. R. POWELL.

Composition of old Roman lead. W. A. COWAN (Inst. Metals, March, 1928. Advance copy. 2 pp.).—A description and analysis of an old Roman lead pipe, dating from A.D. 69—79. The lead contained 0.00785% Ag, 0.0328% Bi, 0.0060% Cu, 0.0004% Fe, and a trace of antimony, but no arsenic, tin, or cadmium. The pipe was found in Rome, but is of much the same composition as that previously reported (Friend and Thorneycroft, B., 1927, 281) for a pipe found in England. W. HUME-ROTHERY.

Historical note on density changes caused by the cold-working of metals. H. O'NEILL (Inst. Metals, March, 1928. Advance copy. 3 pp.).—The decrease in density produced by the cold-working of metals was probably known to Berzelius in 1844, but a detailed paper on the changes in density of copper when reduced by hammering was published by C. O'Neill in 1861 (Mem. Manchester Phil., 1861, 1, [iii], 243). W. HUME-ROTHERY.

Analytical Commission of the Platinum Institute. I. Reception of platinum ore. II. Rapid analysis of platinum ore. S. F. SHEMTSCHUSHNI, O. E. ZVJAGINSTSEV, B. G. KARPOV, V. V. LEBEDINSKI, and N. I. PODKOPAEV. **III. Analysis of platinum ores. IV. Determination of copper and iron. V. Complete analysis of platinum ore.** A. T. GRIGORIEV, S. F. SHEMTSCHUSHNI, O. E. ZVJAGINSTSEV, B. G. KARPOV, N. S. KURNAKOV, V. V. LEBEDINSKI, and N. I. PODKOPAEV. **VI. Analysis of the first insoluble residue obtained on dissolving platinum ore with aqua regia.** S. F. SHEMTSCHUSHNI, O. E. ZVJAGINSTSEV, B. G. KARPOV, V. V. LEBEDINSKI, and N. I. PODKOPAEV (Ann. Inst. Platine, 1926, No. 4, 339, 340—

343, 343—355, 355—359).—I. The ore (2 g.) is melted with silver (7—10 g.) and a flux to remove vanadium, tungsten, iron, etc. Tungsten is extracted from another portion of the ore by digesting it in a mixture of hydrofluoric and nitric acids; the solution is evaporated to recover tungsten trioxide.

V. The mineral is dissolved in aqua regia, the liquid filtered, evaporated with hydrochloric acid, and diluted; chlorine is passed in at 40° to form iridium tetrachloride and prevent reduction of the gold, the mixture evaporated at 38—42°, diluted, and precipitated at 30° with ammonium chloride, the precipitate containing platinum and iridium. Boiling aqueous dimethylglyoxime is added to a portion of the filtrate, gold and palladium being precipitated. The filtrate from this is reduced with zinc and hydrochloric acid, with precipitation of copper, rhodium, and the remaining iridium. Iron is precipitated as hydroxide from the last filtrate after oxidation. The main portion of the filtrate from the precipitation of platinum and iridium is treated with concentrated nitric acid to decompose the ammonium chloride; the residue is dissolved in water and digested at 80—100° with alkaline sodium nitrite. The precipitate consists of gold and base metals. The filtrate, containing rhodium, iridium, ruthenium, platinum, and palladium, is evaporated with hydrochloric acid to remove the nitric acid, the palladium precipitated with mercuric cyanide, the remainder of the platinum and iridium with ammonium chloride, and then all the metals with zinc. The precipitate is redissolved in aqua regia, the iridium precipitated with ammonium chloride, and the filtrate again reduced with zinc to obtain rhodium. All iridium precipitates are reduced to the metal, which is heated in fused sodium carbonate and nitrate and the product is dissolved in hydrochloric acid, which dissolves the ruthenium, leaving a residue of iridium oxide. The ruthenium is recovered by precipitation with zinc.

VI. The precipitate is treated with ammoniacal ammonium acetate solution to remove lead sulphate and silver chloride, and then fused with borax and precipitated silver. The silver button is boiled with sulphuric acid to dissolve the silver, and the residue of osmiridium is fused with zinc in an atmosphere of hydrogen or under a mixture of potassium and sodium chlorides. The mass is dissolved in water, nitric and hydrochloric acids are added, and osmium tetroxide is removed by distillation. Excess of sodium hydroxide is added to the remaining solution, and the ruthenium distilled off in a stream of chlorine, the treatment being repeated after addition of further quantities of sodium hydroxide; the ruthenium is recovered from the distillate by treatment with magnesium. The solution in the distillation flask is acidified and filtered, the filtrate oxidised with chlorine and precipitated with ammonium chloride, and further treated as in the general method for small amounts of iridium, palladium [? platinum], and (in the final filtrate) rhodium; these are precipitated with zinc, ignited, reduced, and cooled in a stream of carbon dioxide.

CHEMICAL ABSTRACTS.

Fatigue produced [in metals] by prolonged application of a constant load. G. WELTER (Z.

Metallk., 1928, 20, 51—57).—The behaviour of bars of metals suspended at one end and loaded at the other with a weight equivalent to 10—20% less than the breaking strength of the metal has been investigated. With a load 10% below the breaking strain, aluminium broke in 3 days, copper in 1 min., brass in 26 hrs., and duralumin in 24 hrs.; the corresponding values for a load 20% below the breaking strain were 8 months, 9 months, 25 days, and infinity. The degree of bending of bars of all the above-named metals, as well as of annealed soft iron, copper, and aluminium, increases rapidly during the first few days under a load equivalent to the limit of proportionality, then proceeds at a small but more or less constant rate over a period of months. With a load equivalent to the elastic limit, no bending takes place with brass, duralumin, and soft iron; the bending-time curve of aluminium is a slightly inclined straight line during the first few days, and that of electron a smooth curve becoming a straight line. With the same load, the bending-time curve taken over a period of months rises steeply concave to the time axis for electron and for annealed copper and aluminium. These results show that iron, brass, and duralumin are safe materials for constructional purposes as long as the load to which they are subjected does not exceed the elastic limit, whereas soft aluminium, copper, and electron undergo progressive fatigue with such a load. A. R. POWELL.

Changes in the mechanical properties [of metals] caused by fatigue. J. CZOCHRAJSKI and E. HENKEL (Z. Metallk., 1928, 20, 58—62).—Test-pieces of steel, aluminium, and copper in the cold-worked and in the annealed state were subjected to fatigue induced by rotating them in a horizontal position in a machine while applying a downward pull to the bearings holding each end of the specimen. The number of reversals required to produce fracture under varying loads was determined as well as the alteration induced in specimens treated in this way with loads equal to and slightly exceeding the elastic limit. In the case of annealed aluminium, the breaking strain is practically unaffected by this treatment, whereas that of copper increases, and that of steel decreases with the load applied; in all cases, however, the elastic limit and limit of proportionality increase with the load applied in the fatigue treatment within the critical limit. With hard-drawn metals the change of mechanical properties is irregular, but not so great as with the annealed metal. With 1 million reversals the critical fatigue load of soft aluminium is 4—5 kg./mm.², of hard-drawn aluminium 6—7 kg./mm.²; the corresponding values for copper are 8—10 and 16—18 kg./mm.², and for steel 20 and 30 kg./mm.². The cylindrical test-pieces on failure in the fatigue test develop cracks on the surface at angles of 45° to the axis in both directions. The cause of these cracks is discussed at some length together with the bearing of the results obtained on the question of the suitability of a metal for constructional purposes. A. R. POWELL.

Elasticity [of metals]: static and endurance tests. W. KUNTZE, G. SACHS, and H. SIEGLERSCHMIDT (Z. Metallk., 1928, 20, 64—68).—If a metal test-piece is stretched in the testing machine and then subjected to a gradual application and removal of load nearly

up to its new elastic limit the curves showing the elongation with application and removal of load do not coincide, although there is no permanent elongation. The elongation-load curve is a straight line up to $\frac{2}{3}$ — $\frac{3}{4}$ of the elastic limit, then curves away from the load axis; for loads above this point, *i.e.*, on the curved section, the hysteresis during removal of the load is relatively great, and a small deformation remains for some considerable time after complete removal of the load. Endurance tests appear to show that this slight semi-permanent internal stress alone is not sufficient to cause the breakdown of the metal. Alternating bending tests of thick wires on sharp edges give results which seem to bear some relation to the reduction of area obtained in the ordinary tensile tests, but the values obtained for breaking similar wires by alternately bending them round two rolls bear a definite relation to the tensile strength, *i.e.*, to the resistance to deformation. A. R. POWELL.

Fatigue in single crystals [of metals]. E. SCHMID (*Z. Metallk.*, 1928, 20, 69—74).—The tensile strength of a single-crystal wire of zinc is considerably increased by subjecting the wire to a repetition of twists through a small angle, whilst the ductility and bending strength are greatly reduced. As the number of twists is increased the load required to cause the wire to stretch is rapidly increased and the inclination of the load-elongation (within the plastic range) curve becomes smaller. The metal, however, becomes exceedingly brittle, and after relatively few twists breaks along the basal plane when subjected to even a small bending stress without undergoing any plastic deformation. These results confirm the work of Gough, Hanson, and Wright on aluminium single-crystal wire (*A.*, 1926, 666, 997).

A. R. POWELL.

Quantitative measurement of the cutting power of cutlery. K. HONDA and K. TAKAHASI (*Sci. Rep. Tôhoku Imp. Univ.*, 1927, 16, 755—773).—See B., 1927, 782.

Blasting in metal-mine drifts. GARDNER and others.—See XXII.

PATENTS.

Production of cast-iron castings by a centrifugal process. NEWTON CHAMBERS & Co., LTD., and J. E. HURST (*B.P.* 285,721, 15.7.27).—The structure of centrifugal castings of cast iron is rendered sorbitic by keeping the silicon content to 1.25—2.5% when metal moulds are used and to 0.5—1.5% when sand moulds are used. In either case the castings are caused to cool rapidly by directing a blast of air, preferably moist, on their inner surface. A. R. POWELL.

Hardened cast iron. K. SIPP, Assr. to H. LANZ A.-G. (*U.S.P.* 1,658,467, 7.2.28. Appl., 3.11.25. Ger., 10.11.24).—Cast iron which has been subjected to the pearlite process (*cf.* *B.P.* 147,933 and 210,091; *B.*, 1921, 853 A; 1925, 176) is heated and quenched in the same manner as steel. T. S. WHEELER.

Manufacture of a metal-treating compound. M. L. MOYER, Assr. to M. L. MOYER, E. J. PRINDLE, and B. STOUGHTON (*U.S.P.* 1,659,214, 14.2.28. Appl., 9.10.25).—Iron is toughened by being cooled in a

mixture of soap, sperm oil, alum, mercuric chloride, zinc sulphate, lead monoxide, and carbon.

T. S. WHEELER.

Iron mould. S. M. UDALE, Assr. to E. HOLLEY (*U.S.P.* 1,658,144, 7.2.28. Appl., 23.11.25).—A cast, permanent mould for iron is formed of an alloy consisting of 34.5% Ni, 2.0% C (mostly free), 1.3% Si, 0.8% (Mn, S, and P), and the balance iron.

T. S. WHEELER.

Manufacture of [ferrous] alloys. V. B. BROWNE (*U.S.P.* 1,658,879, 14.2.28. Appl., 21.10.25).—In the manufacture of alloy steel in an arc furnace, the metal bath is subjected until free from silicon to the action of any usual oxidising slag, the slag is replaced by a protective layer of calcium oxide, and the metal freed from oxides by addition of aluminium.

T. S. WHEELER.

Recovery of metals [copper, zinc, etc.] from iron pyrites and similar ores and from liquors containing them. C. F. SCHANTZ (*B.P.* 285,662, 12.3.27).—Leach liquors containing copper and zinc derived from sulphide ores which have been subjected to a chloridising roast are treated with sulphuric acid equivalent to their chlorine content, which is then precipitated as cuprous chloride by the addition of the necessary quantity of sponge copper and cupric oxide; at the same time the whole of the lead is deposited as sulphate and the silver and gold in the metallic form, leaving a solution containing sodium and zinc sulphates. After further purification the clear solution may be used for the manufacture of lithopone, zinc white, or electrolytic zinc; in the first two cases sodium sulphate may be recovered from the filtrates, and in the third case the acid liquor obtained is used in the first stage of the process. The cuprous chloride precipitate is extracted with a concentrated alkali chloride solution and the copper recovered from the resulting solution by cementation on iron or by electrolysis; the insoluble residue is worked up for its content of lead and precious metals by known methods. The spent electrolyte from the copper cells is used for extracting further quantities of cuprous chloride sludge. Instead of using an outside source of current for the deposition of copper, this may be effected by placing a sheet of copper in the solution and joining it outside the liquor to a sheet of iron immersed in dilute sulphuric acid in a porous cell which is placed in the cuprous chloride solution; ferric sulphate is obtained by evaporating the liquor in the cell after use.

A. R. POWELL.

Zinc-base alloy. W. MCG. PEIRCE and E. A. ANDERSON, Assrs. to NEW JERSEY ZINC Co. (*U.S.P.* 1,663,215, 20.3.28. Appl., 5.1.27).—Zinc is alloyed with 1—15% Al, 0.5—4% Cu, 0.05—5% Mg, and 0.01—0.5% Ni.

F. G. CROSSE.

Refining metals [removal of zinc from lead]. E. C. R. MARKS. From AMER. SMELTING & REFINING Co. (*B.P.* 285,630, 21.1.27).—Molten lead from the Parkes process is dezincified by pumping it continuously in a fine stream through a closed cylinder filled with chlorine, in such a way that a thin layer of lead is maintained on the floor of the cylinder while the treated lead passes continuously back into the bottom of the kettle containing

the bulk of the metal. The zinc chloride formed in the early stages of the process is free of lead and may be rejected, but that from the later stages contains much lead and is therefore used in the preliminary treatment of a fresh kettle of metal. The zinc content of the lead may be rapidly reduced to less than 0.003% by this process.

A. R. POWELL.

Aluminium-base alloy. R. S. ARCHER, Assr. to ALUMINUM Co. OF AMERICA (U.S.P. 1,663,150, 20.3.28. Appl., 30.6.25).—Aluminium is alloyed with 3–15% Si, more than 0.8% Fe, and more than 0.05% of a metal of the sixth group of the periodic system having an at. wt. less than 190.

F. G. CROSSE.

Bending of section rods of magnesium alloys. I. G. FARBERIND, A.-G. (B.P. 284,313, 5.1.28. Ger., 28.1.27).—The rods are heated at 100–400° before being passed through bending rollers.

H. ROYAL-DAWSON.

Colouring of [tin] foil. W. G. GOLDEN, Assr. to U.S. FOIL Co. (U.S.P. 1,662,574, 13.3.28. Appl., 26.10.25).—A gold colour is imparted to tin foil by the direct application of heat.

H. ROYAL-DAWSON.

Soldering material. S. RUBEN (U.S.P. 1,663,141, 20.3.28. Appl., 1.10.26).—An alloy of 34% Pb, 34% Sn, 31% Zn, and 1% Mg is claimed.

F. G. CROSSE.

Cyaniding of ores containing precious metals. S. B. McCLUSKEY (U.S.P. 1,658,249, 7.2.28. Appl., 8.10.26).—The pulp mixed with cyanide solution is treated with sulphur dioxide, and the hydrogen cyanide liberated is volatilised. Air is blown through the mixture to oxidise ferrous and manganous salts, which are then precipitated by addition of calcium hydroxide and carbonate. The pulp is next treated with the hydrogen cyanide evolved in the first stage, cyaniding now proceeding smoothly in the absence of base-metal salts.

T. S. WHEELER.

Concentration of ores and minerals by flotation. W. A. DOUGLASS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,659,396, 14.2.28. Appl., 25.10.26).—An organic derivative of trithiocarbonic acid is claimed as a flotation reagent.

T. S. WHEELER.

Production of rare-metal powders. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,659,209, 14.2.28. Appl., 3.1.23).—In the process of U.S.P. 1,602,542 (B., 1927, 46) calcium chloride is added as a flux to the mixture of rare metal oxide and magnesium.

T. S. WHEELER.

Cleaning and coating of metal articles. "METALLOGEN" G.M.B.H. (B.P. 285,556, 16.11.26. Addn. to B.P. 154,235; B., 1921, 87A).—An apparatus for carrying out the process of the chief patent is described.

A. R. POWELL.

Manufacture of steel and alloys from iron sand. D. CROESE (U.S.P. 1,662,302, 13.3.28. Appl., 19.2.26. U.K., 26.2.25).—See B.P. 252,455; B., 1926, 674.

Production and treatment of a copper alloy. M. G. CORSON, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,658,186, 7.2.28. Appl., 25.9.25. Can., 21.2.25).—See B.P. 256,457; B., 1926, 885.

Bearing composition. H. M. WILLIAMS, Assr. to GEN. MOTORS RES. CORP. (U.S.P. 1,661,245, 6.3.28. Appl., 22.5.23).—See B.P. 216,484; B., 1924, 1017.

XI.—ELECTROTECHNICS.

Theory of magnetic separation. B. W. HOLMAN (Trans. Inst. Chem. Eng., Advance Proofs, March, 1928, 3–11).—Theories of magnetism are briefly discussed in relation to the operation and design of magnetic separators, particular consideration being given to the influence of particle size, the shape of the pole pieces, the design of electro-magnets, the use of flux-meters, and the determination of permeability.

A. B. MANNING.

Measurement of the electrical resistance of porous materials. A. E. KNOWLER (Proc. Physical Soc., 1928, 40, 37–40).—The resistance of a specimen of refractory material is determined by passing a known current through and measuring the potential between two wires inserted in it a known distance apart. The method avoids errors due to contact resistances.

C. J. SMITHELLS.

Electrical insulating materials. O. KRAUSE (Z. Elektrochem., 1928, 34, 49; cf. Günther-Schulze, B., 1927, 881).—Polemical.

H. J. T. ELLINGHAM.

Electro-osmosis of water. PATIN. Removal of oil from condenser water. RĂSCANU.—See XXIII.

PATENTS.

Electrolytic rectifier. A. B. MUNTNYAN, Assr. to G. E. FISCHER (U.S.P. 1,662,075, 13.3.28. Appl., 15.10.24. Holl., 19.10.23).—Aluminium anode and cathode members, previously coated with different thicknesses of metallic oxide, are immersed in an electrolyte, and, when so immersed, have a constant difference of electrical capacity per unit area from one another, possess the correct capacities in reference to the current to be rectified, and remain unaltered by operation of the rectifier.

J. S. G. THOMAS.

Electrolytic rectifier. W. H. GRIMDITCH and J. N. HUNSBERGER, JUN., Assrs. to PHILADELPHIA STORAGE BATTERY Co. (U.S.P. 1,662,383, 13.3.28. Appl., 18.2.27).—A rectifying electrode for asymmetric cells consists of aluminium containing 0.04–0.4% Cu.

J. S. G. THOMAS.

Salt for electrolytic rectifiers. F. W. BARHOFF and W. C. BROOKS, Assrs. to HARTFORD BATTERY MANUF. Co. (U.S.P. 1,658,914, 14.2.28. Appl., 26.5.27).—A mixture of ammonium sulphate, sulphuric acid, and ferrous sulphate forms a solid product which dissolves in water to yield an electrolyte suitable for use with filming electrodes of tantalum.

T. S. WHEELER.

Electrolyser resembling a filter press [for the electrolysis of water]. A. E. ZDANSKI (Swiss P. 120,249, 4.1.26. Ger., 25.3.25).—An apparatus for the decomposition of water comprises a deep cell with a series of compound electrodes arranged one above the other and resembling in structure a filter press. The potential of the electrodes increases with their height above ground.

A. R. POWELL.

Electrolysis of salt solutions. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (G.P. 446,009, 27.8.24).—

An apparatus for the purpose comprises a flat cell with an internal inclined, water-cooled false bottom acting as one electrode, and a superimposed chamber with diaphragms inside which is a second flat electrode perforated with inclined slits. A. R. POWELL.

Art of incrusting galvanoplasty. F. MAAS, Assr. to NATURAL FLOWER METALIZING CO., INC. (U.S.P. 1,662,430, 13.3.28. Appl., 26.8.25).—An object is coated, in succession, with stiffening material, with a base containing phosphorus, rubber, asphaltum, carbon disulphide, and binding material, with silver nitrate, and is then electroplated. J. S. G. THOMAS.

Metallic cores for electromagnets etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 286,167, 5.9.27. Addn. to B.P. 269,770; B., 1927, 493).—The metallic powder produced from the carbonyl is heated in an atmosphere of reducing gas (e.g., 18 hrs. at 500° in a current of hydrogen) prior to being made into cores. J. S. G. THOMAS.

Cleaning-up of residual gases [in thermionic valves]. A. E. LYLE, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,659,207, 14.2.28. Appl., 7.11.24).—Small quantities of lime and magnesium are introduced into a thermionic valve containing a filament coated with an alkaline-earth carbonate. The tube is evacuated and the filament heated to decompose the carbonate to the oxide, the carbon dioxide evolved being absorbed by the lime. The magnesium metal is then heated by induction to complete gettering. T. S. WHEELER.

Electric resistance furnace adapted for heating by nitrogenation. A. FRY, Assr. to F. KRUPP A.-G. (U.S.P. 1,661,694, 6.3.28. Appl., 2.8.26. Ger., 4.9.25).—See B.P. 257,896; B., 1927, 786.

Manufacture of a tungsten product [filament]. A. DE GRAAFF, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,662,027, 6.3.28. Appl., 24.4.24. Holl., 17.5.23).—See B.P. 216,124; B., 1925, 91.

Arrangements for feeding [electric] furnaces working with open arcs. A.-G. BROWN, BOVERI & COE. (B.P. 261,785 and 263,118, [A] 20.11.26, [B] 29.11.26. Switz., [A] 21.11.25, [B] 15.12.25).

XII.—FATS; OILS; WAXES.

Action of solutions of potassium iodate and hydrogen iodide on fats. B. M. MARGOSCHES, K. FUCHS, and B. KRAKOWETZ (J. pr. Chem., 1928, [ii], 118, 225—237).—The fat is treated with 0.2*N*-aqueous hydriodic acid (which may be preserved unchanged for some 3 weeks) and excess of potassium iodate in aqueous alcoholic solution (cf. the "rapid iodine-number method," B., 1924, 341, 639). When the mixture is immediately titrated with thiosulphate, iodine numbers are obtained which, after doubling (owing to reoxidation by the iodate of the hydriodic acid formed in the reaction, $I_2 + H_2O = HI + HIO$), agree closely with the Hübl or Hanus values. After 5 min. the mixture gives a lower value. This decrease, which continues for some 24 hrs., is probably due to reactions similar to those connected with the iodine super-number (A., 1925, i, 629, 833). When equivalent quantities of hydriodic acid and

potassium iodate are used the iodine absorption after 5 min. corresponds with that obtained by the rapid method. Castor oil and resins which liberate more than 50% of free acid in the rapid method (cf. A., 1924, i, 828) naturally give low iodine numbers in presence of excess of iodate. H. E. F. NOTTON.

Comparison of the methods for separating solid and liquid fatty acids. K. AMBERGER and E. WHEELER-HILL (Z. Unters. Lebensm., 1927, 54, 431—434).—The methods compared were (1) the insolubility of the lead salts of the solid fatty acids in ether, (2) Twitchell's process (B., 1921, 817 A), and (3) the thallium process as modified by Holde (B., 1924, 755). The first process was found unsuitable for quantitative work as the solid acids recovered still contained appreciable proportions of liquid acids. The other two processes gave quantitative separations of palmitic acid from oleic and linoleic acids. Neither process was satisfactory with erucic acid. J. R. NICHOLLS.

Fatty acids of seed oils of *Brassica* species. Composition of rape, ravison, and mustard seed oils. T. P. HILDITCH, T. RILEY, and N. L. VIDYARTHI (J.S.C.I., 1927, 46, 457—462 T).—The composition of the fatty acids present as glycerides in four representative fats of the *Brassica* family, viz., English rape, Danubian ravison, and English black and white mustard seed oils, has been investigated. The procedure consisted in effecting a partial separation of the fatty acids into three groups, according to the solubility of their lead salts in alcohol and ether, followed by systematic fractional distillation of the methyl esters of each group of acids under reduced pressure. It was found that each of the fats was composed, qualitatively, of the same acids. The main components were erucic acid (about 50% of the whole) and unsaturated acids containing 18 carbon atoms (about 45—47%); the latter consisted for the most part of ordinary $\Delta^9:10$ -oleic acid and linoleic acid (the relative proportions of these varying considerably), together with small quantities of linolenic acid. There was also present in each case a small amount of isomeric oleic acid (or acids), which separated with the least soluble portion of the lead salts, and which yielded on oxidation a dihydroxy-compound (m.p. 117—118°), which was differentiated from the other known dihydroxystearic acids by its relatively great solubility in ether, alcohol, and ethyl acetate. The remaining fatty acids present belonged to the saturated series, and included 1—2% each of palmitic and lignoceric acids, with traces of stearic and arachidic acids. The quantitative results obtained by the systematic fractionation of the esters are given in the following table:—

Oil.	English rape seed.	Danubian ravison.	English black mustard (charlock).	English white mustard.
	<i>Brassica campestris</i> .	<i>B. campestris</i> var.	<i>B. (Sinapis) nigra</i> .	<i>B. (S.) alba</i> .
Source:				
Combined fatty acids.	%	%	%	%
Palmitic	1	2	2	2
Stearic	—	—	Trace	Trace
Arachidic	—	—	Trace	1
Behenic	—	Trace	—	—
Lignoceric	1	2	2	1
Oleic	32	20.5	24.5	28
Linoleic	15	25.5	19.5	14.5
Linolenic	1	2	2	1
Erucic	50	47	50	52.5

III-defined acids of the oleic series. III. "Rapic acid" and other acids of rape and mustard seed oils. T. P. HILDITCH, T. RILEY, and N. L. VIDYARTHI (J.S.C.I., 1927, 46, 462—467 T). The fatty acids present in the four *Brassica* fats dealt with in the preceding paper (see above) have been submitted to a detailed examination, mainly in order finally to settle whether any isomerides of oleic acid are present (e.g., the "rapic acid" of Reimer and Will and of Zellner, the existence of which was denied by Grabner, Raymond, and Toyama). Quantitative oxidation of the methyl esters of the unsaturated C_{18} acids (the lead salts of which are soluble in alcohol or ether) has established that they are derived practically exclusively from acids containing an ethylenic linkage in the $\Delta^{9:10}$ position (ordinary oleic and linoleic acids). Unsaturated C_{18} acids separated in the form of insoluble lead salts constitute a relatively small proportion of the whole, and of these, probably about half is also the ordinary $\Delta^{9:10}$ -oleic acid. Consequently isomeric forms are only present to the extent of 1—2% at most of the total combined fatty acids of these oils. On the other hand, the presence of the 1—2% of isomeric oleic acid (or acids) is readily distinguished in the *Brassica* fats by the formation, from the unsaturated acids of the C_{18} series which form insoluble lead salts, of the unusually soluble dihydroxy-compound which melts at 117—118° (cf. preceding abstract). By systematic lead salt separation and fractionation of a large quantity of rapeseed oil fatty acids sufficient of the isomeric acid (still admixed with some ordinary oleic acid) was finally obtained for oxidation of the corresponding methyl ester in acetone solution with potassium permanganate. The resulting acidic products (in addition to azelaic acid and nonoic acid from ordinary oleic acid) included a solid dibasic acid (which melted at 137—138°, and corresponded with the formula $C_{10}H_{18}O_4$, but was not identical with sebacic acid, $CO_2H \cdot [CH_2]_8 \cdot CO_2H$) and a mixture of monobasic acids in which octoic and decaic acids were apparently both present. It was therefore concluded that, although Reimer and Will's "rapic acid" is certainly non-existent in the sense indicated by these workers, there is present in the fats examined a small amount of acid $C_{18}H_{34}O_2$; most of this is probably a branched-chain unsaturated acid of the general structure $C_7H_{15} \cdot CH : CH \cdot (C_8H_{16}) \cdot CO_2H$, but more than one individual acid is probably present. The compounds in question are referred to as "isoleic acids of the *Cruciferae*." The erucic acid, $C_{22}H_{42}O_2$, present in these fats was shown to be exclusively $\Delta^{13:14}$ -dodecenoic acid, and no indication was obtained of the presence of isomeric forms similar to those of the oleic acids of this group. The lower saturated acid present in the fats examined was almost entirely palmitic acid; myristic acid cannot have been present in amounts exceeding about 0.2%.

III-defined acids of the oleic series. IV. "Cheiranthic acid" of wallflower seed oil. T. P. HILDITCH and (Miss) E. E. JONES (J.S.C.I., 1927, 46, 467—469 T).—The combined fatty acids of wallflower seed oil, stated by Matthes and Boltze to consist of 65% of cheiranthic acid, $C_{18}H_{34}O_2$, 30% of linoleic acid, and 5% of linolenic acid, have been shown (by separation into three groups

of fatty acids by lead salt separation, followed by systematic fractionation of the methyl esters of each group) to consist of: palmitic (3%), lignoceric (0.5%), ordinary oleic (12.5%), linoleic (41%), linolenic (4%), and erucic (39%) acids. Wallflower seed oil is, therefore, of the same general type as rape, mustard, and other seed fats of the same botanical order, *Cruciferae*. The only marked differences are a lower content than the average of combined erucic acid, and the much less saturated nature of the acids containing 18 carbon atoms compared with those in the other *Cruciferous* fats so far examined. "Cheiranthic acid" as an individual isomeric form of ordinary oleic acid is a case of mistaken identity; it should be removed from the list of naturally-occurring unsaturated fatty acids.

Head and blubber oils of the sperm whale. I. Determinations of the mixed fatty acids present. T. P. HILDITCH and J. A. LOVERN (J.S.C.I., 1928, 47, 105—111 T).—The head and blubber oils of the Antarctic sperm whale examined consisted of a mixture of higher fatty alcohol esters and glycerides of fatty acids, the wax or ester components largely predominating (head oil, about 74% of wax; blubber oil, about 66% of wax). The head oil is less unsaturated than the blubber oil, and contains larger amounts of compounds of relatively low molecular weight, both as regards the acids and alcohols. The latter appear to consist, roughly, of (i) about 60% of C_{16} alcohols (partly unsaturated), 35% of C_{15} alcohols (mainly unsaturated), and 5% of unsaturated C_{20} alcohols (head oil), and (ii) about 20% of C_{16} alcohols (partly unsaturated), 70% of C_{18} alcohols (largely unsaturated), and 10% of unsaturated C_{20} alcohols (blubber oil); the composition of the alcohols is receiving further study. The mixed acids present have been determined to consist of the following: (i) Head oil: Capric (3.5%), lauric (16%), myristic (14%), palmitic (8%), stearic (2%); dodecenoic (4%), tetradecenoic (14%), hexadecenoic (15%), oleic (17%), and eicosenoic (6.5%); all of the unsaturated acids appear to be monoethylenic. (ii) Blubber oil: Lauric (1%), myristic (5%), palmitic (6.5%); tetradecenoic (4%), hexadecenoic (26.5%), C_{18} acids (average unsaturation — 2.1H) (37%), C_{20} acids (— 2.6H) (19%), and C_{22} acids (?) (— 3.6H) (1%). The wide differences between the fatty acids of the head and blubber oil, and between both types of sperm fatty acids and those of other whale oils and of fish oils, are emphasised.

Composition of oat oil. K. AMBERGER and E. WHEELER-HILL (Z. Unters. Lebensm., 1927, 54, 417—431).—The oil was extracted by keeping the oats for 7 days in light petroleum, and was purified by dissolution in acetone and light petroleum and by washing with water. The fatty acids were separated into palmitic acid (10.4%), α -linoleic acid (17.2%), β -linoleic acid (13.9%), and oleic acid (58.5%). Erucic and linolenic acids were not detected. Individual glycerides were sought from the hardened fat, through the elaidin reaction, and from the brominated oil, and triolein and α -palmito- α - β -distearin were isolated (cf. Amberger and Bromig, B., 1922, 675 A). J. R. NICHOLLS.

Composition of ivy seed oil. A. STEGER and J. VAN LOON (Rec. trav. chim., 1928, 47, 471—476).—Ivy seed

oil, d^{35} 0.9151, acid value 11.0, saponif. value 181.1, iodine value 102.2, setting point 13.8°, n_D^{20} 1.4670, obtained in 20% yield from the dried seeds has the following percentage composition; glycerol as C_3H_2 (4.0), unsaponifiable matter (6.6), higher saturated fatty acids (4.5), linoleic acid (11.7), 9:10-oleic acid (17), and petroselinic acid (55).
H. BURTON.

PATENTS.

Washing, wetting, and cleaning agents. P. FRIESENHALM (B.P. 269,134, 26.11.26. Ger., 6.4.26).—Hydrocarbons and other solvents are rendered water-soluble by treatment with the soaps and soap-like compounds described in G.P. 365,160 (B., 1923, 318 A), the cyclohexanols in which may be replaced by alcohols or phenols of high mol. wt., alkyl naphthols, etc. On treatment of their aqueous solutions with inorganic salts (e.g., sodium carbonate or sulphate) a solid product separates which contains the salt and the organic solvent.
B. FULLMAN.

Edible fat. H. YUDOWITCH (H. A. NEWTON). (Re-issue 16,915, 20.3.28, of U.S.P. 1,601,229, 28.9.26).—See B.P. 284,368; B., 1928, 284.

Production of emulsions. W. A. WHATMOUGH (U.S.P. 1,663,323, 20.3.28. Appl., 14.5.27. U.K., 13.5.26).—See B.P. 280,096; B., 1928, 99.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

"Accelerated testing" of paints. K. WÜRTH (Farben-Ztg., 1928, 33, 1470).—A brief account of the historical development of accelerated paint tests.
S. S. WOOLF.

Metallic lead in red lead. K. WÜRTH (Farben-Ztg., 1928, 33, 1473—1474).—Heinrich's work on the presence of metallic lead in red lead used in glass manufacture is discussed from the point of view of paint technology.
S. S. WOOLF.

PATENTS.

Production of lithopone. J. E. BOOGE, M. L. HANAHAN, and J. P. KOLLER, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,658,628, 7.2.28. Appl., 23.4.24).—The charge before calcination is screened so as to lie between specified size limits.
T. S. WHEELER.

Manufacture of pigments. G. C. LEWIS, Assr. to COLUMBIAN CARBON Co. (U.S.P. 1,659,403, 14.2.28. Appl., 9.9.26).—Humus, e.g., peat, is extracted with sodium hydroxide solution, and the solution treated with acid to precipitate a pigment of value in the manufacture of varnishes.
T. S. WHEELER.

Composition of matter [white pigment]. L. WILLIAMS (U.S.P. 1,662,625, 13.3.28. Appl., 7.5.27).—A mixture of zinc oxide (60 g.), sulphur (2 g.), sodium carbonate (1.5 g.), sodium chloride (1 g.), and ultramarine-blue (0.5.), after being ground, is heated in a suitable closed retort until all the free sulphur has disappeared, and the product is cooled and again pulverised.
H. ROYAL-DAWSON.

Preparation of resin. R. BEUTNER, Assr. to A. NOWACK A.-G. (U.S.P. 1,658,281, 7.2.28. Appl., 15.11.24).—A coal-tar oil is heated with formaldehyde, aqueous sodium hydroxide solution, and kerosene to yield a

product which separates into three layers, comprising, respectively, an oil, an aqueous solution, and a bakelite resin formed from the phenols present in the tar oil.
T. S. WHEELER.

Manufacture of derivatives of the reaction product of amines and aldehydes. C. O. NORTH, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,659,151, 14.2.28. Appl., 24.3.23).—The products obtained as described in U.S.P. 1,659,152 (see p. 309) are heated with an aldehyde, e.g., formaldehyde, in aqueous solution at about 100° for 6 hrs., and water is removed at 105° to yield resins.
T. S. WHEELER.

Paints, enamels, japans, etc. D. GARDNER (U.S.P. 1,662,741, 13.3.28. Appl., 6.8.22. U.K., 21.8.22).—See B.P. 211,516; B., 1924, 391.

Treatment of dispersed colloids (B.P. 283,686).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Chemical unsaturation of rubbers vulcanised with polynitro-compounds and benzoyl peroxide, and its possible bearing on vulcanisation. H. L. FISHER and A. E. GRAY (Ind. Eng. Chem., 1928, 20, 294—295).—Samples of rubber vulcanised with dinitrobenzene, trinitrotoluene, and benzoyl peroxide, respectively, were extracted with acetone, dried, and then examined in solution, e.g., in carbon disulphide, as to the degree of chemical unsaturation by means of iodine chloride. The alteration in the degree of unsaturation did not exceed 1%. The results suggest that vulcanisation involves an undetermined type of change in the caoutchouc hydrocarbon without change in its unsaturation.
D. F. TWISS.

Activity of certain aryl-substituted diguanides as accelerators of vulcanisation [of rubber]. G. B. L. SMITH and A. J. WEISS (Ind. Eng. Chem., 1928, 20, 298—300).—Various α -aryldiguanides are compared with several better known accelerators as to their influence on the rate of vulcanisation and the tensile properties in a mixture of rubber 100 pts., zinc oxide 100 pts., sulphur 10 pts., and accelerator 1 pt. The effect of phenyl-, *o*- and *p*-tolyl-, and 3:4-xylyldiguanides compares favourably with that of hexamethylenetetramine, and is therefore of moderate character; $\alpha\alpha$ -diphenyldiguanide is somewhat more active than the monoaryl compounds, but α -naphthyldiguanide is less effective than its monoaryl analogues of lower mol. wt.; with this exception the activity of the monoaryldiguanides increases with the mol. wt. Of the α -tolylidiguanides, the *o*-compound (which was also tested in certain technical rubber mixtures), was more powerful than its *p*-isomeride.
D. F. TWISS.

Comparison of zinc oxide, lithopone, zinc sulphide, and titanium dioxide in [rubber] films vulcanised with sulphur chloride. R. DITMAR and G. BALLOG (Gummi-Ztg., 1928, 42, 1303—1304).—Solutions of masticated pale crêpe rubber (15 pts.) in benzene (100 pts.) were mixed with zinc oxide, lithopone, zinc sulphide, and titanium oxide, respectively, each powder being of colloidal fineness. Dried articles formed by a single immersion of a mould were submitted to

vulcanisation by sulphur chloride in solution or in the vapour state, and were then examined as to physical condition, colour, and ageing in sunlight. With the presence of 8% of each powder (to 100 of rubber), which is the maximum proportion for satisfactory results with such fine ingredients, the zinc oxide was generally most advantageous.
D. F. TWISS.

Electrolytic precipitation of fresh and preserved latices and the rôle of the albumin in coagulation. P. SCHOLZ (Kautschuk, 1928, 4, 5—8).—The minimum concentration of an electrolyte, in millimols. per litre, necessary to effect coagulation of latex under standardised conditions is termed the coagulation value of the electrolyte. The coagulation value of chloride of potassium, lithium, barium, or magnesium, or of sulphate of magnesium or nickel, is little influenced by dilution of the latex, but with sulphate of copper, aluminium, or lanthanum, or with acetic acid (which are coagulants for albumin), dilution of the latex causes a marked decrease in the coagulation value; with latex which has been deprived of its natural protein the coagulation value of all these substances is practically unaffected by dilution. The relative behaviour of fresh and preserved and neutralised preserved latex is also investigated.
D. F. TWISS.

Stretching of rubber. E. A. HAUSER and P. ROSBAUD (Kautschuk, 1928, 4, 12—14).—With decrease in temperature the stress-strain curve of smooth, unvulcanised, smoked sheet rubber moves nearer to the load axis, and the final extension diminishes; at -185° the rubber is inextensible and the breaking load amounts to 5.45 kg./mm.², which is exceeded only by that of rubber stretched at the ordinary temperature and then cooled in liquid air. Above 40° stretching is accompanied by local constriction with great local extension. The course and length of the stress-strain curve are greatly influenced by the speed of stretching. The proportionality indicated earlier between the intensity of the discontinuous interference in the X-rays spectrum and the heat of extension with the degree of extension holds only for experiments with constant velocity of extension.
D. F. TWISS.

Ageing of stretched rubber. A. KELLY, B. S. TAYLOR, and W. N. JONES (Ind. Eng. Chem., 1928, 20, 296—298).—Ageing experiments have been made with various commercial grades of vulcanised rubber, in sunlight, in the Geer oven at 70° , in the Bierer bomb, and in ultra-violet light. Stretching of the test pieces accelerates deterioration, but there is no simple proportionality between the degree of extension and the initial rate of depreciation. In sunlight there is a critical elongation for each sample at which a maximum initial rate of deterioration is obtained. No direct relation is evident between the results of ageing by sunlight and by the other methods. Contrary to the effect of sunlight, ultra-violet light causes a softening of stretched samples.
D. F. TWISS.

Effects of ozone on stretched rubber. F. H. HAUSHALTER, W. N. JONES, and J. W. SCHADE (Ind. Eng. Chem., 1928, 20, 300—302; cf. preceding abstract).—Rubber strips stretched even to a small extent in an

atmosphere containing as little as 0.1% of ozone develop cracking in seconds or minutes as compared with weeks in sunlight. In the early stages of exposure, the degree of cracking in ozone, like that in sunlight, increases as the tension is increased up to a certain limit, and beyond that decreases again; in unstretched samples cracking does not occur.
D. F. TWISS.

Typical-angle abrasion machine [for rubber]. W. W. VOGT (Ind. Eng. Chem., 1928, 20, 302—306).—A machine is described in which the resistance to abrasion is measured by pressing a ring of the rubber against the flat surface of a driven abrasive wheel in such a way that the plane of the ring makes an angle with the tangent of the abrasive wheel at the point of contact; means are provided for measurement of the power consumed in abrasion. The influence of various factors such as time, speed, load, and angle of contact on the extent of abrasion is discussed, and the mechanical forces involved are analysed.
D. F. TWISS.

Theory of needle-shaped caoutchouc molecule in science and practice. E. LINDMAYER (Gummi-Ztg., 1928, 42, 1025—1028).—Various recent investigations and problems are cited, and the possible application of the theory of the needle-shape of the caoutchouc molecule is indicated.
D. F. TWISS.

Jelutong. C. D. V. GEORGI (Malayan Agric. J., 1927, 15, 400—407).—Coagulation experiments with sodium silicofluoride, acetic acid, formic acid, sulphuric acid, and potash alum showed the first-named to be superior to the remainder in yielding a product resistant to deterioration by mould development; unfortunately, the toxic properties of sodium silicofluoride prevent its adoption. No tendency to deterioration by "drying out" to a brittle mass was observed even with alcohol as coagulant. Jelutong is preferably coagulated by boiling the acidified latex; thus prepared, it appears to be less porous and to retain its moisture better.
D. F. TWISS.

PATENTS.

Treatment of aqueously dispersed colloidal substances. A. BIDDLE (B.P. 283,686, 15.11.26).—Natural or aqueous dispersions, *e.g.*, of rubber, cellulose products, or resins, with or without the addition of oils, waxes, tars, and common fillers, are treated with lime and a reagent, *e.g.*, sodium fluoride, which, by chemical action with the lime, yields an alkali; the nascent condition of the alkali results in a superior product. The lime may be introduced in the form of a compound with a hydrophilic colloid, the latter, if desired, being subsequently rendered insoluble.
D. F. TWISS.

Manufacture of the aliphatic aldehyde derivative of the condensation product of an aliphatic aldehyde and an aromatic primary amine. C. O. NORTH, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,659,152, 14.2.28. Appl., 24.3.23).—A primary arylamine, *e.g.*, aniline, is treated with an aliphatic aldehyde at above 30° to give a compound containing 2 mols. of base to 3 of aldehyde, which is dehydrated at 100° to yield a product of value as an accelerator of vulcanisation.
T. S. WHEELER.

XV.—LEATHER; GLUE.

Determination of sugars in tanning materials.

I. D. CLARKE and R. W. FREY (J. Amer. Leather Chem. Assoc., 1928, 23, 91—108).—Experiments with infusions of different tanning materials and extracts show that, in the determination of sugars, no one period of hydrolysis can be selected for all materials. Infusions of divi-divi pods, wattle bark, and algarobilla pods, respectively, showed the greatest loss of sugary matter with increasing time of hydrolysis. Each tanning material showed a maximum content of sugar after a certain period of hydrolysis. Hydrolysis for only 6 min. gave results 82—100% of the maximum. The operations of clarifying and de-leading introduced salts into the solutions which were thereby buffered and brought to a constant p_H value on the addition of acid, irrespective of the nature of the tanning material. The neutralisation of the hydrolysed solution can be conveniently omitted. Determinations were made of the sugars fermentable and non-fermentable by bakers' yeast in hydrolysed and unhydrolysed solutions. The content (%) of fermentable reducing sugars in chestnut wood, chestnut extract, divi-divi pods, myrobalans nuts, sumac leaves, valonia cups, and wattle bark, respectively, was 40, 37, 88, 85, 83, 85, and 92%; corresponding values for the fermentable, non-reducing sugars are 0, 5, 63, 0, 35, 0, and 36%. Decided differences are shown in the nature of the sugars in the materials. Hydrolysis materially increased the quantity of sugars capable of reducing Fehling's solution, but did not always result in an increase in the amount of fermentable sugars.

D. WOODROFFE.

Molybdenum tannage. J. G. NIEDERCORN (Ind. Eng. Chem., 1928, 20, 257—258).—A solution of the purple-red sulphate of tervalent molybdenum, prepared by the electrolytic reduction of a solution of molybdic anhydride in sulphuric acid, was used for tanning pelt. It tanned very rapidly at p_H 1—1.5, but no longer penetrated at p_H 2.0. The corresponding green sulphate tanned calfskin at p_H 2—2.5. Both salts yielded a dark brown leather unaltered by boiling. On exposure to air, the leather gradually lost this property, but no hydroxide could be extracted with the boiling water.

D. WOODROFFE.

Progress report on effects of acids on leather. R. C. BOWKER and E. L. WALLACE (J. Amer. Leather Chem. Assoc., 1928, 23, 82—91).—A preliminary report describing the preparation of samples of different tan-nages and their impregnation with acid solutions.

D. WOODROFFE.

Clarification of waste tannery liquors and their utilisation for agricultural purposes. V. HLAVINKA (Sbornik Ceskoslovenské Akad. Zemědělské, 1927, 2, 41—54; Chem. Zentr., 1927, II, 967).—The inorganic constituents, with the exception of compounds of chromium and arsenic, of tannery waste liquors are usually harmless, and no danger is likely to arise from bacteria unless pathogenic bacteria are present; these are only imperfectly removed by treatment with chlorine or bleaching powder. To obtain efficient sedimentation the tanning liquors should be kept separate from the lime-pit liquors. Sedimentation is carried out

in sand pits and in raking plants where the chemical reactions are allowed to complete themselves so that coagulation of suspended matter is accelerated. The slime obtained contains 98% of the inorganic and 75% of the organic solids suspended in the liquors. The clarified liquors retain only 10% of the chromium content, whilst the slime has a nitrogen content of 5—6%. After drying to a water content of 60% it may be used as a fertiliser. A. R. POWELL.

Determination of residual nitrogen for the evaluation of gelatin products. T. VON FELLEBERG (Z. Unters. Lebensm., 1927, 54, 481—483).—Bacterial action causes decomposition of proteins resulting in an increase of the non-precipitable nitrogen. The residual soluble nitrogen after precipitation with phosphotungstic acid was determined and calculated as a percentage on the total nitrogen. Good gelatin gave 0.55%, second quality gelatin 1.19%, and glues 1.56—6.53%. After being kept for 19 days at room temperature in a 1% aqueous solution, good gelatin gave 1.9%.

J. R. NICHOLLS.

A new jelly strength tester and some experiments with gelatin gels. A. F. TRACEY (J.S.C.I., 1928, 47, 94—96 T).—The apparatus described measures the load on a plunger required to break the jelly surface and also gives the depression of the jelly surface under load. The jelly strength is shown to be a surface effect and also to have a temperature coefficient which reaches a minimum about 20°.

PATENTS.

Treatment [curing] of hides and skins. H. DODGE (U.S.P. 1,663,401, 20.3.28. Appl., 8.7.27).—A mixture containing sodium chloride and hydrogen carbonate, potassium nitrate, and formaldehyde is used.

F. G. CROSSE.

Substitute for catgut for use in tennis rackets etc. JOHNSON & JOHNSON (GT. BRITAIN), LTD. From JOHNSON AND JOHNSON (B.P. 285,752, 9.9.27).—Silk strands are deprived of sericin, impregnated with glue (25—30 pts.) and a softening agent, e.g., glycerin (10—15 pts.), with or without a lubricant, e.g., castor oil (20 pts.) or lard (20—30 pts.), and water. The product may be waterproofed by exposure to aldehyde vapour.

D. WOODROFFE.

Preparation of a protective colloid. F. HEIDBERG (U.S.P. 1,658,289, 7.2.28. Appl., 31.1.21).—A solution of casein, prepared by dissolving 500 g. of casein in water with the addition of 100 c.c. of 20% sodium hydroxide solution at 40°, is fermented with 50 g. of papain, and the precipitate of digested protein is separated, washed, and redissolved in sufficient alkali to form a neutral solution.

F. R. ENNOS.

Automatic scouring machine for treating hides. TURNER TANNING MACHINERY Co., LTD. (B.P. 262,451, 3.12.26. Fr., 3.12.25).

Porous insulating materials (B.P. 265,968).—See IX.

XVI.—AGRICULTURE.

Relation between plant nutrition, plant growth, and the Mitscherlich method for determining the fertiliser requirement of soils. [O. LEMMERMANN,

P. HASSE, and W. JESSEN (Z. Pflanz. Düng., 1928, 7B, 49—98).—In the Mitscherlich method for determining fertiliser requirements of soils, the "factors" are shown to be dependent on environmental conditions and previous manuring. The nitrogen and phosphate factors decrease as other soil conditions improve and as the optimum yield increases. In pot experiments the factors obtained are usually smaller than those by field trials. This is ascribed to improved water conditions maintained in the former. Variations in the factors should be considered in conjunction with "limiting values" and optimum yields. Owing to the inconstancy of the factors, the Mitscherlich method does not give accurate information as to the assimilable food-stuffs present in soils or as to fertiliser needs, although information of considerable value in practice is obtainable.

A. G. POLLARD.

Acidity of moorland soils. B. TACKE, P. ARND, W. SIEMERS, and J. SAFFRON (Landw. Jahrb., 1927, 65, 66—103; Bied. Zentr., 1928, 57, 7—8).—By means of the Tacke-Süchting method it is shown that little or no exchange acidity exists in moor soils. From a consideration of the degree of ionisation of neutral salt solutions and of the free acids, it is concluded that actual neutral salt decomposition occurs in rich organic soils, with the formation of free acids. Liming of moor soils effects a 50% neutral salt decomposition. Sphagnum peat is shown by percolation experiments to bring about a similar decomposition of neutral salts. The ease with which neutral salts are decomposed in humus soils is influenced by the nature of both ions. Sulphates are more easily affected than nitrates, and these in turn more easily than chlorides (cf. B., 1926, 1024).

A. G. POLLARD.

Hygroscopicity and the chemical properties of soils. F. GIESECKE (Chem. Erde, 1927, 3, 98—136; Bied. Zentr., 1928, 57, 4—6).—The hygroscopicity of soil may be represented as the sum of the hygroscopic values of the various mechanical (Atterberg) fractions. Mean values for the individual fractions measured on samples dried at 105—110° are recorded. Results obtained by this process agree with those yielded by the customary direct methods. There is a close parallelism between the hygroscopicity of soils and their contents of iron and aluminium oxides. This relationship is influenced by the ratio of $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3 : \text{SiO}_2$, higher ratios corresponding to decreased hygroscopicity.

A. G. POLLARD.

Slaking of dry soils with water. K. T. HARTLEY (J. Agric. Sci., 1928, 18, 41—45).—A method is described for determining the time required for complete disintegration by water of spheres of dry soil. There appears to be an equilibrium point between colloidal content and pore space at which the time of slaking is at a maximum. Determination of the time of slaking is not, however, a reliable means of comparing soils of different types.

C. T. GIMMINGHAM.

Scientific use of nitrogen fertilisers. F. E. BEAR (Ind. Eng. Chem., 1928, 20, 65—70).—A discussion of the modifications in agricultural practice likely to follow on the introduction of cheap synthetic nitrogen in the various forms. Conditions governing the choice

between them are reviewed. It is claimed that under many circumstances "green manuring" in place of the use of fertiliser nitrogen has ceased to be economical, and attention is called to experiments on the use of nitrogen on pastures.

C. IRWIN.

Loss involved by igniting soil fractions during mechanical analysis of soils. F. J. MARTIN (J. Agric. Sci., 1928, 18, 123—130).—With many tropical soils, particularly lateritic soils, the greater part of the loss of weight on ignition of the fractions is due to combined water, not to organic matter. In the clay fraction, the greater the ratio silica:alumina, the less the amount of combined water. Lateritic material is not necessarily evenly distributed among the fractions, and, with these soils, it is considered best to express the results of mechanical analysis on an oven-dry basis, noting that the clay fraction is weighted with the organic matter.

C. T. GIMMINGHAM.

Mechanical analysis of tropical soils. J. CHARLTON (Bull. Agric. Res. Inst., Pusa, No. 172, 1927, 9 pp.).—In order completely to disperse the clay in soil suspensions, it is necessary to shake the sample with alkali in an end-over-end shaker for at least 24 hrs. at a temperature of not less than 25°. Pretreatment with hydrogen peroxide does not obviate the necessity for the above prolonged shaking. Ammonia solutions of greater strength than is customary do not hasten the dispersion. "Hyperol" (a solid compound of urea and hydrogen peroxide) is more efficient than ordinary hydrogen peroxide solutions in aiding the dispersion of clay in humus soils. Hydrogen peroxide solutions frequently contain sufficient phosphoric acid to cause appreciable losses of silica and iron sesquioxide from soil suspensions.

A. G. POLLARD.

Aphiscidal properties of tobacco dust. H. C. HUCKETT (New York Agric. Exp. Sta. Tech. Bull., 1926, No. 121, 1—29).—The toxicity of tobacco dust is dependent on the nicotine content and on the period of aphid exposure. The toxicity is increased by the addition of calcium hydroxide (1:1), particularly when the nicotine content is low. Tobacco dusts so diluted are as effective as pure tobacco dust.

CHEMICAL ABSTRACTS.

Effect of iodine manuring on the iodine content of plants. J. B. ORR, F. C. KELLY, and G. L. STUART (J. Agric. Sci., 1928, 18, 159—161).—The results of experiments with oats, mustard, peas, and other plants, in water culture and in soil, show that addition of potassium iodide affords a simple means of increasing the iodine content of food plants.

Technique of seed testing. ANON. (Landw. Versuchs-Stat., 1927, 107, 1—64).—Details are recorded of sampling methods and of methods of testing germinating power, vigour, purity (of species and strain), weight and volume, husk and milling value (of cereals).

Production of ammonium phosphates. MÜCKENBERGER.—See VII. **Tannery liquors in agriculture.** HLAVINKA.—See XV.

PATENTS.

Dehydration of vegetable substances or products of organic origin. B. J. OWEN (B.P. 285,115, 5.11.26).—Vegetable matter (e.g., sugar-beet slices) is dried in thin

layers by means of heated air. Temperature, pressure, and rate of air current are so controlled that the mass of vegetable matter does not become overheated.

A. G. POLLARD.

Herbicide. G. E. BENSEL (U.S.P. 1,657,062, 24.1.28. Appl., 28.12.25).—Arsenic trioxide is heated with hydrochloric acid to give a solution of arsenic trichloride which is mixed with ferrous sulphate and acid sludge from the treatment of petroleum.

T. S. WHEELER.

Phosphorus material. H. W. CHARLTON, Assr. to AMER. CYANAMID Co. (U.S.P. 1,662,489, 13.3.28. Appl., 2.8.23).—Phosphoric anhydride is mixed with calcium cyanamide.

H. ROYAL-DAWSON.

[Mechanical spray]-treatment of growing plants etc. J. C. SAVAGE (B.P. 285,515, 10.8.26).

XVII.—SUGARS; STARCHES; GUMS.

Treatment of beet-sugar factory effluents. O. SPENGLER (Trans. Inst. Chem. Eng., Advance Proofs, March, 1928, 25—30).—The most important of the recent developments in the methods of dealing with beet-sugar factory effluents are the Hirschfelder system of clarifying the conveying and washing water, and the double fermentation or the fermentation and putrefaction processes for the disposal of the pulp press and diffusion waste-water. In the Hirschfelder system a number of basins are arranged round a sludge pond. The raw effluent passes through the basins in succession, the sludge being deposited therein until the first basin, which receives the largest deposit, is filled; the effluent is then discharged into the next basin and those following until in turn the next basin is filled. As each basin is filled the sludge in it is discharged into the central pond, whence it is pumped into the sludge field. In the Hildesheim double fermentation process the hot effluents from the battery and the pulp presses are de-pulped and then led into the first settling pond in which auto-fermentation takes place. The water thence overflows into a second pond where sufficient lime is added to neutralise 80—90% of the acid. The lime sludge settles and a second fermentation takes place. After passing through other ponds the water is conveyed to irrigation fields, in which the acids still in the water are neutralised by the lime used for fertilisation, and the albuminous substances are precipitated. By this process it is possible to purify sugar factory effluents so well that they may be discharged without objection even into small rivers.

A. B. MANNING.

Sugars in tanning materials. CLARKE and FREY.—See XV. **Alcohol from Nipah palm.** DENNETT.—See XVIII. **Starch in hazel nuts.** GRIEBEL.—See XIX.

PATENTS.

Manufacture of dextrose from starch. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 1,661,298, 6.3.28. Appl., 25.4.24. Renewed 7.12.27).—See B.P. 232,938; B., 1925, 967.

Manufacture of starch products. E. STERN (U.S.P. 1,661,201, 6.3.28. Appl., 20.3.26. Ger., 5.8.24).—See B.P. 272,274; B., 1927, 612.

XVIII.—FERMENTATION INDUSTRIES.

Modern malt kilns. A. FERNBACH (J. Inst. Brew., 1928, 34, 119—127).—The difficulties of regulating the heating of a malt kiln with two or three floors have resulted in the development of the Winckler kiln, in which hot air from the furnace is forced by a fan through the green malt which is loaded to a depth of from 3 to 5 ft. The resulting rapid evaporation of the moisture keeps the temperature of the drying malt sufficiently low, whilst the large increase in the saturation point of the air at the high temperature also effects a very rapid drying. The drying and curing processes occupy only about 10 hrs. and no turning is necessary. C. RANKEN.

Malting barleys of 1927. J. STEWART (J. Inst. Brew., 1928, 34, 128—143).—The past season's English and foreign malting barleys are reviewed, and attention is directed to the further serious decrease in acreage in the United Kingdom, which constituted a record. The stocks in California were also the lowest on record. The finest qualities of home barleys came from the Eastern counties, but owing to adverse conditions the crop was all more or less weathered. The corresponding malts from the analytical point of view compared favourably with those of the previous year, with the exception that the soluble nitrogen was low, and there was a greater variation in diastase. Czechoslovakia had the best of the foreign barleys suited for pale ales.

C. RANKEN.

Composition and estimation of barley proteins. L. R. BISHOP (J. Inst. Brew., 1928, 34, 101—118).—A method is developed and standardised for the determination of the proteins in barley. Albumin, globulin, and degradation products are first extracted from the barley by salt solution. Hordein is extracted from the residue by hot alcohol and the nitrogen of the final residue is considered to represent glutelin. Direct determinations of glutelin confirm the results obtained with this method of difference. In samples of barley grown on different soils, and with different manuring, the total nitrogen varied from 1.2 to 2.3%. The percentages of glutelin remained constant at 36% of the total nitrogen. The percentage of hordein increased as the total nitrogen increased, rising from 26 to 40% of the total nitrogen, whilst the percentage of the salt-soluble nitrogen correspondingly decreased from 36 to 24%. External conditions alter the total amount of nitrogen in the grain, but appear to have no influence on the proportions of the proteins in the mature barley. Accordingly, varying "quality" of barleys of equal nitrogen content is not due to variations in the amounts of the individual proteins. It is indicated that hordein is the chief protein which is attacked during malting, and breaks down to give salt-soluble compounds. C. RANKEN.

Citric acid content of grape must and wine. A. HEIDUSCHKA and C. PYRIKI (Z. Unters. Lebensm., 1927, 54, 466—473).—The method used was von der Heide's modification (separation of citric acid from sugars etc. by precipitation with barium chloride in ammoniacal solution and the addition of alcohol to 15% by vol.) of Stahre's process (oxidation of citric acid to acetone-dicarboxylic acid and the latter to the difficultly soluble

pentabromoacetone). 1926 Saxony must contained from 0.126—0.221 g. of citric acid per litre. The corresponding wines showed sometimes more and sometimes less; some contained traces or none. A pure culture of wine yeast was found to have no influence on the change in citric acid, which is assumed to be due to another organism. In 15 foreign wines from different countries the citric acid varied from a trace to 0.267 g. per litre.

J. R. NICHOLLS.

Fruit-residue spirits with abnormal odour and taste. T. VON FELLEBERG (Z. Unters. Lebensm., 1927, 54, 483—484).—On three different occasions at wide intervals spirits made from apple marc were found to be contaminated with mustard oil. The abnormality was traced to the drainings from the marc of fallen, un-matured apples, and appeared to have been produced by the fermentation of a substance which may occur in unripe apples, but which disappears before maturity. Unripe apples are therefore unsuitable for the production of potable spirits. On another occasion a spirit was produced with a taste and odour of cherries and in which hydrocyanic acid and benzaldehyde could be detected. Further extractions up to twelve pressings produced similar spirits. It was found that excessive pressing of the marc to obtain as much juice as possible had crushed many apple pips, and these had given the unusual flavour.

J. R. NICHOLLS.

Nipah palm as a source of alcohol. J. H. DENNETT (Malay. Agric. J., 1927, 15, 420—432; cf. B., 1927, 263).—Juice obtained by tapping the Nipah palm contains cane sugar but no reducing sugars or alcohol. The tapping process is discussed, together with the transport of the juice to the factory. The average yield of alcohol is 10% (by vol.) of the juice and 1300 gals. of alcohol per acre per annum are estimated to be obtainable. Examination of the soil under Nipah palm shows a 40% decrease in the salt content of the top soil since 1924. Little change occurs in the sub-soil.

A. G. POLLARD.

PATENTS.

Manufacture of powdered extract of malted grains. A. W. BOSWORTH, Assr. to DOUTHITT ENGINEERING Co. (U.S.P. 1,658,027, 7.2.28. Appl., 7.2.25).—The grain, after malting, is dried, mixed with water free from carbonates of sodium, potassium, or ammonium, and subjected to diastatic fermentation at 60° to convert the starch into maltose and dextrin. The aqueous extract is drawn off from the grain, which is washed with water at 60°, and the combined unfermented liquor is diluted with water until it contains 10—15% of solids, and is desiccated by injecting it in the form of a finely-divided spray into a volume of heated air, whereby a non-caramelised powder is produced. F. R. ENNOS.

Manufacture of dihydroxyacetone. I. G. FARBEININD. A.-G. (B.P. 269,950, 26.4.27. Ger., 26.4.26).—A decoction of vegetable materials, such as straw, wood, or hay, is added to glycerin and the mixture fermented by *B. xylinum* or *Acetobacter suboxydans*. The conversion of the glycerin into dihydroxyacetone is concluded in four or five days and the process is not impeded by frothing.

C. RANKEN.

XIX.—FOODS.

Examination of the biological properties of milk. M. A. DYCHNO and O. M. BRISKIN (Z. Unters. Lebensm., 1927, 54, 438—446).—Peroxydase in milk is detected by the addition of hydrogen peroxide, potassium iodide, and starch. Raw milk gives a blue colour, milk heated above 60° a brownish-green, and heated above 80° no colour. Catalase is measured by the volume of oxygen liberated in 2 hrs. after the addition of hydrogen peroxide. The average volume of gas liberated from 100 c.c. of milk was found to be 31.9 c.c. from raw milk, 14.5 c.c. from baked (*i.e.*, heated for some time below 100°) milk, and 2.5 c.c. from boiled milk. Reductase is measured by the time of decoloration of methylene-blue, and is correlated with the bacterial content. The fluctuation found was between 10 min. for a raw milk of high bacterial content and 7½ hrs. for a boiled milk, and the higher the acidity of the milk the quicker the reduction. A table is given showing the kinds of bacteria detected. The fermentation test (which indicates gas-forming bacteria) is regarded as of great significance. The milk is kept in a closed tube for 24 hrs. at 40°. The better the quality of the milk the less change is apparent; a good fresh milk remains liquid for 12 hrs. Four classes in descending order of quality are described: (1) homogeneous compact curd without whey or gas, (2) point-like, mottled curd with whey and gas, (3) streaky curd adhering to the walls of the tube, the interstices filled with whey, and (4) flocculent cheesy curd. The usual methods of heating or boiling milk are condemned, but pasteurising for ½ hr. at 60—70°, whilst slightly altering the physical character of the milk, has little effect on the biological properties and is recommended.

J. R. NICHOLLS.

Determination of lecithin-phosphoric acid in egg liqueurs. J. GROSSFELD (Z. Unters. Lebensm., 1927, 54, 450—462).—The liqueur (5 g.) is mixed with 50 c.c. of absolute alcohol and heated just to boiling; the mixture is filtered through an extraction thimble and the filtrate is used for extracting the insoluble matter in an apparatus in which the hot solvent vapours come directly in contact with the thimble. After 1 hr. the extract is mixed with potassium hydroxide and magnesium acetate, evaporated to dryness, and ashed. The ash is dissolved in acid and the phosphoric acid precipitated with molybdate solution. The weight of the precipitate after ignition multiplied by 0.0395 gives the lecithin-P₂O₅. Alternatively, the precipitated phosphomolybdate is determined by titration.

J. R. NICHOLLS.

Loss of chlorine in ashing [of flour]. W. VON BRUCHHAUSEN (Z. Unters. Lebensm., 1927, 54, 485—487).—Samples of flour, which showed up to 0.4% Cl when an aqueous extract was tested by Volhard's process, gave practically nil after ashing. Tests were carried out by adding salt to starch, lactose, and sucrose, and ashing with and without sodium carbonate. In the absence of the latter over 99% of the chloride was lost, and even in the presence of an excess of carbonate the loss was 13—15%.

J. R. NICHOLLS.

Determination of stone-cells in cacao products. E. ALPERS (Z. Unters. Lebensm., 1927, 54, 462—466).—

The number of recognisable stone-cells in a cocoa powder depends on the grinding, as some of the cells are ruptured. Powdered Accra cacao shell gave the following mean microscopical counts for stone-cell number, *i.e.*, the number of whole cells in 1 mg. of the dry fat-free substance:—once ground 9337, twice 7498, three times 5886, and four times 3290. A number of shells from cacaos of different origin were examined for stone-cell content after once grinding with cacao fat to yield a product similar in fineness to the commercial article. The stone-cell number varied from 9337 for Accra to 3574 for Caracas. To deduce the proportion of shell from the stone-cell number of an unknown cacao product, the largest of these values should be taken to avoid an over-estimation. Mixtures made up with from 1–5% of Accra shell and shell-free nib once ground together gave stone-cell numbers corresponding very closely with those calculated. It is considered that the proportion of shell normally present in cacao nib should not exceed 1%, representing 2.2% on the dry fat-free substance. This would give a stone-cell number of 220 if the round number of 10,000 is taken for shell. Since some products may be less finely ground 300–350 is suggested as the upper limit for the stone-cell number of cacao nib.

J. R. NICHOLLS.

Determination of cacao shell. W. PLÜCKER, A. STEINRUCK, and F. STARCK (*Z. Unters. Lebensm.*, 1927, 54, 488).—The authors claim priority for the method of determining cacao shell by a count of the stone-cells.

J. R. NICHOLLS.

Determination of vaseline oil in coffee. D. MAROTTA and M. E. ALESSANDRINI (*Annali Chim. Appl.*, 1928, 18, 37–39).—Vaseline oil, which, in proportions not exceeding 0.5%, is the only polishing agent for coffee permitted in Italy, may be determined as follows: 100 g. of the roasted berries are extracted with three successive portions of 70–80 c.c. of light petroleum (b.p. 60–70°), and the residue left after complete expulsion of the solvent from the filtered extract is treated with about 1 g. of coarsely-ground sodium hydroxide and 10–12 c.c. of about 30% hydrogen peroxide solution in 2 c.c. portions, the flask being cooled if necessary to allay the frothing. When the action ceases, the mass is heated for 1½ hrs. in a reflux apparatus, with frequent shaking, with 50 c.c. of 12% alcoholic potassium hydroxide solution, and is then diluted with 50 c.c. of water and extracted with three quantities of ether, vigorous shaking being avoided. The ether layer, freed from alkali by washing with water, is treated with a little anhydrous sodium sulphate and, after about 1 hr., filtered through a dry filter. The residue of this ethereal extract is heated for 15 min. on a water-bath with 5–10 c.c. of glacial acetic acid, which dissolves the impurities but only traces of the vaseline oil. The liquid, together with the acetic acid washings of the vessel, is transferred to a graduated tube about 1 cm. wide, which is shaken and left in a bath at 60–70° for 15 min. The volume of the collected vaseline oil, read after about 1 hr., is diminished by 10% and the remainder divided by 1.12 to obtain the weight of the oil present. With ground coffee, the

extraction with light petroleum is effected in a Soxhlet extractor and is continued for 4 hrs. T. H. POPE.

Detection of apple juice in preserves of other fruits. C. ESPESO (*Anal. Fis. Quim.*, 1928, 26, 25–32).—Adulteration of jams with apple juice is generally shown by the presence of malic acid, or of excess of malic acid when the fruit, *e.g.*, peaches, normally contains this acid. Muttelet's method of determination of citric and malic acids in jams (*B.*, 1922, 726) was found to be unsatisfactory, and a modified procedure was worked out. After preparation of the solution as described by Muttelet the removal of pectin substances is completed by the addition of 2 vols. of 95% alcohol and filtration after 1 day. The precipitate of barium citrate and malate, obtained as described by Muttelet, is dissolved in the minimal amount of very dilute hydrochloric acid, made up to 100 c.c., and neutralised with sodium hydroxide. Half the volume of 95% alcohol is added and the precipitate of barium citrate collected. Since this precipitate may also contain malate, it is dissolved and reprecipitated in the same way. The combined filtrates are evaporated to 25 c.c. and 75 c.c. of 95% alcohol added, precipitating barium malate. The presence of malic acid is confirmed by Piñerúa's reagent (β -naphthol in sulphuric acid), with which it gives a green fluorescence. In cases of adulteration with residues of cider manufacture the acidity is lower and little malic acid is found. The first precipitate of pectin substances is small, but the second is greater than normal. The following results are recorded for the citric acid and malic acid contents, respectively: Red currant jams, 0.02–1.6%, 0–0.075%; red currant and apple (1:1), 0.809%, 0.257%; peach jam, 0.81%, 0.614%. R. K. CALLOW.

Detection of yeast in the presence of hydrolysed animal products. H. KRAUT (*Z. Unters. Lebensm.*, 1927, 54, 446–449).—The usual process for the detection of yeast in meat extracts (Micko's) depends finally upon the precipitation of yeast gum with alcohol. In the presence of degraded animal products a precipitate with alcohol may not necessarily be due to yeast. For the certain detection of yeast gum the precipitate is dissolved in acid and 3% sodium hydroxide added. After filtering, the addition of Fehling's solution to the filtrate causes a bright blue precipitate with yeast gum. J. R. NICHOLLS.

Starch in hazel nuts. C. GRIEBEL (*Z. Unters. Lebensm.*, 1927, 54, 477–481).—Hazel nuts are generally assumed to be essentially starch-free, but some seeds are rich in starch although a fair proportion contain only small quantities. The presence of starch-bearing seed particles in certain nut preparations has been considered indicative of peanut, but without a proper microscopical examination this may be erroneous. The starchy seeds which find application in practice for the replacement of almonds etc. are peanuts, hazel nuts, and cashew nuts. These have been examined microscopically and the chief diagnostic characteristics are recorded. It is assumed that the starch in hazel nuts is due to incomplete ripening, but no diminution of starch occurred on storing for 1 year ripe seeds containing a small quantity of starch.

In all the nuts examined the starch was either in the seed coat or cotyledons, but not in both.

J. R. NICHOLLS.

Horse radish and its preparations. K. AMBERGER and K. HEPP (Z. Unters. Lebensm., 1927, 54, 435—438).—The results of analysis of several samples of horse radish of known origin are given. In unpreserved specimens only traces of sulphur dioxide were obtained, and these are assumed to have been derived from the mustard oil.

J. R. NICHOLLS.

Light-sensitivity of dyes. STEIGMANN.—See IV. **Determination of caffeine and theobromine.** OEHLI.—See XX.

PATENTS.

Separation and purification of the three phosphorus-containing bodies derived from the proteids contained in egg yolk. S. and T. POSTERNAK (B.P. 268,806, 31.3.27. Switz., 31.3.26).—The mixture of the sodium salts of the three compounds containing phosphorus, obtained as described in B.P. 268,805 (B., 1928, 138) by extraction of egg yolk with boiling alcohol, are dissolved in water and the β -compound is precipitated by means of alcohol. From the filtrate the α -compound is precipitated by addition of a soluble alkaline-earth salt, after which the γ -compound is obtained by addition of alcohol to the filtrate or by precipitation as a compound with a heavy metal, e.g., lead or copper, which is subsequently decomposed in the usual manner. Alternatively, the α - and β -compounds may be separated from the γ -compound by addition of an alkaline-earth salt to the solution of mixed sodium salts, or by saturating this solution with common salt and acidifying with mineral acid, the mixed precipitate being converted through the heavy-metal salts into the alkali compounds from which the β -compound is precipitated by alcohol. The γ -compound is then obtained from the solution by either of the above processes.

F. R. ENNOS.

Desiccation [of fruit juices]. W. H. DICKERSON, Assr. to INDUSTRIAL WASTE PRODUCTS CORP. (U.S.P. 1,658,078, 7.2.28. Appl., 2.8.22).—A dry powder is prepared from fruit juices etc. by adding sucrose and subjecting the mixture in the form of minute liquid particles to the action of a drying gas previously heated above the critical temperature of the mixture, i.e., the temperature at which caramelisation and melting occur. The heat content of the gas and the proportion of the latter relative to the amount of substance to be desiccated are so maintained that the temperature of the dried product falls below the critical point, owing to the heat absorbed by evaporation, whilst the moisture content of the gas is adjusted so as to prevent the dried particles from coalescing.

F. R. ENNOS.

Manufacture of high-grade apple juice. E. R. DARLING, Assr. to HYDRAULIC PRESS MANUF. CO. (U.S.P. 1,659,086, 14.2.28. Appl., 10.11.26).—The pomace is treated with diastase at about 60° to break down the starch cells present and thus facilitate recovery of the juice.

T. S. WHEELER.

Extract of malted grains (U.S.P. 1,658,027).—See XVIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Arsenious oxide in neutral and alkaline solution. W. H. MILLAR (Pharm. J., 1928, 120, 214—215).—In the preparation of Liquor Arsenicalis, B.P. the amount of potassium carbonate used is in excess of that actually required to cause dissolution of the arsenious oxide. 8% of the potassium carbonate reacts to form potassium meta-arsenite and, using this amount in the preparation, dissolution is complete. A 1% neutral, stable solution of arsenious oxide can be prepared by adding potassium carbonate solution to a boiling water suspension containing 1% of arsenious oxide until the latter is dissolved. Potassium carbonate equivalent to 4% of the arsenious oxide is required and the solution is neutralised to methyl-orange with 0.1N-sulphuric acid. Addition of potassium sulphate equivalent to the potassium carbonate used does not effect dissolution.

E. H. SHARPLES.

Determination of benzaldehyde in cherry laurel water. F. MORVILLEZ and (Mlle.) DÉFOSEZ (J. Pharm. Chim., 1927, [viii], 6, 204—210).—Two methods for the determination of benzaldehyde in cherry laurel water are described. The first is a quick, accurate, "sorting" test, and depends on the absorption of potassium permanganate under definite conditions. The absorption is proportional to the benzaldehyde content and, for normal samples of cherry laurel water, ranges from 22 to 29 c.c. of 0.01N-potassium permanganate per 1 c.c. of water. The method is practically unaffected by the presence of either hydrocyanic acid or nitrobenzene but gives high results if stannous cyanide is present. A more accurate method is to oxidise the aldehyde with chromic acid after hydrolysis of the mandelonitrile. The benzoic acid is extracted with chloroform and determined gravimetrically or volumetrically. Results by these methods are compared with those obtained by precipitation of the aldehyde with "melubrin," and they show satisfactory agreement.

E. H. SHARPLES.

Microchemical determination of caffeine and theobromine in drugs. A. OEHLI (Pharm. Acta Helv., 1927, 2, 155—167; Chem. Zentr., 1927, ii, 1989).—The powdered material (0.1—0.3 g.) is extracted with chloroform in a separating funnel after addition of a few drops of ammonia solution. Chloroform and ammonia are completely removed from the united extracts, the residue is boiled with aqueous magnesia free from soda, and the wax, resin, and colouring matter are removed by filtration. The filtrate is neutralised with 1% acetic acid and treated with 1% permanganate solution, the excess of which is removed with 3% hydrogen peroxide solution containing 1% of acetic acid, filtered, and the colourless filtrate extracted several times with chloroform. After removal of the chloroform by evaporation, the caffeine in the residue is determined (a) volumetrically, by the Kjeldahl-Pregl method, or (b) gravimetrically after purification by vacuum-sublimation in a manner described. For guarana, 1—4% sulphuric acid is used instead of ammonia. For the separation of caffeine and theobromine from maté, the differing solubility in benzene is employed. For the gravimetric micro-determination of the xanthine bases in cocoa, 0.1—0.5 g. is heated with aqueous alcoholic sulphuric

acid, then water is added, and the mixture boiled with magnesia. After filtration, evaporation, extraction with water, neutralisation with 1% acetic acid, and treatment with permanganate and hydrogen peroxide, the filtrate is evaporated to a small volume, and percolated with chloroform in the presence of phenol. The residue after removal of phenol and chloroform is dried in a vacuum and sublimed at 200—210°. A. A. ELDRIDGE.

Sclerocarya caffra, Sond. M. G. BRANDWYK (Pharm. J., 1928, 120, 172, 192—193, 212).—The bark of *Sclerocarya caffra*, Sond., which has been used in the treatment of malaria, has been examined. It contains no constituents volatile in steam, and on extraction with alcohol it gives 29.3% of a solid extract, which contains about 70% of tannic acid and traces of alkaloids. Glucosides and neutral principles are absent. When injected subcutaneously into a cat, the extract had a severe local effect, but was not toxic.

E. H. SHARPLES.

Constituents of Indian essential oils. XXIII. Essential oil from fruits of *Piper Cubeba*, Linn. B. S. RAO, V. P. SHINTRE, and J. L. SIMONSEN (J.S.C.I., 1928, 47, 92—94 T).—Fruits of *Piper Cubeba* grown in Mysore have been found to yield 7.5% of an essential oil. The main constituents of the oil were shown to be *d*-sabinene, *d*- Δ^4 -carene, 1:4-cineole, *d*-1-methyl-4-isopropyl- Δ^4 -cyclohexen-4-ol, and *l*-cadinene. Attention was directed to the genetic relationship of sabinene, 1:4-cineole, and the alcohol. 1:4-Cineole has not previously been found to occur in nature.

Essential oil from the leaves of *Caesalpinia Sappan*, L. P. VAN ROMBURGH (Proc. K. Akad. Wetensch. Amsterdam, 1927, 30, 840—843).—The hydrocarbon $C_{10}H_{16}$, $d_{20}^{25} 0.813$, $n_D^{20} 1.473$, which is isolated by repeated fractional distillation of the essential oil obtained by steam distillation from the leaves of *Caesalpinia Sappan*, L., yields a nitrosite identical with the nitrosite of *d*- α -phellandrene. By reduction with sodium and boiling alcohol, a hydrocarbon, b.p. 63°/15 mm., $d_{15}^{21} 0.800$, $n_D^{15} 1.4581$, $\alpha_D +15.48^\circ$, which readily absorbs bromine to yield a product from which a small quantity of dihydro-ocimene tetrabromide, separates. Hydration of the original hydrocarbon with acetic and dilute sulphuric acids yields a viscous alcohol, b.p. 115—122°/30 mm., $d_{15}^{16} 0.890$, $n_D^{16} 1.484$ (liquid urethane). The density of both the hydrocarbon and its reduction product increases on keeping. It is concluded that the hydrocarbon is probably a mixture of *d*- α -phellandrene with an olefinic terpene. J. W. BAKER.

PATENTS.

Halogeno-tert.-alkyl carbamates. A. W. DOX and L. YODER, Assrs. to PARKE, DAVIS, & Co. (U.S.P. 1,658,231, 7.2.28. Appl., 9.10.22).—A metallic halogeno-tert.-alkoxide is treated with carbonyl chloride in presence of an inert solvent, e.g., benzene, and the resulting chloroformate is treated with ammonia to give the corresponding halogeno-tert.-alkyl carbamate, of therapeutic value as a non-toxic sedative. gem.-*Trichloro-tert.-butyl carbamate* m.p. 102°, and gem.-*dichloro-tert.-butyl carbamate*, m.p. 122°, are described. T. S. WHEELER.

3':5'-Dinitro-4'-hydroxy-*o*-benzoylbenzoic acid.
3':5'-Diamino-4'-hydroxy-*o*-benzoylbenzoic acid

1:3-Diamino-2-hydroxyanthraquinone. I. GUBELMANN, H. J. WEILAND, and O. STALLMANN, Assrs. to NEWPORT Co. (U.S.P. 1,659,358—1,659,360, 14.2.28. Appl., 29.11.26).—(A) 4'-Hydroxy-*o*-benzoylbenzoic acid is nitrated with a mixture of nitric and sulphuric acids at room temperature to 3':5'-*dinitro-4'-hydroxy-*o*-benzoylbenzoic acid*, m.p. 199°. (B) This compound on reduction yields 3':5'-*diamino-4'-hydroxy-*o*-benzoylbenzoic acid*. (C) The amino-compound (B) is condensed by treatment with concentrated sulphuric acid at 180° to 1:3-*diamino-2-hydroxyanthraquinone*, m.p. 300—305°.

T. S. WHEELER.

Manufacture of tasteless alkaloid preparations. H. HEIMANN (G.P. 444,388, 31.10.24).—Alkaloids are combined with humous substances obtainable from peat, lignite, decayed wood, or other natural sources, or synthetically from sugars, phenols, or proteins; aqueous, alcoholic, or other solutions of the bases or their salts (particularly acetates) may be utilised. The reaction is of the nature of a base-exchange, but differs from the permutit process in that it is the hydrogen ions of the humic acids which take part, liberating acid from the alkaloid salts. The products, washed with pure solvent and then, if desired, with water, are quite tasteless, water-insoluble powders, which can be made up into pastilles with addition of suitable flavouring matters. The alkaloids are regenerated from the products by the action of neutral salts (sodium chloride) or dilute acids, and hence the products have in the organism the same effect as the alkaloids themselves. By shaking an aqueous solution of caffeine with lignite humus for 19 min. at 40°, filtering, and washing with water, 60% of the caffeine is obtained as insoluble humate. Alcoholic cinchonidine with humus from crude sugar, and aqueous quinine hydrochloride with calcium humate behave similarly. C. HOLLINS.

Manufacture of water-soluble double compounds of digitoglucotannoids, the total digitalis glucosides being in their natural form. KNOLL & Co. (G.P. 444,064, 13.9.24).—Digitoglucotannoids are treated with solutions of carbamide, sodium salicylate, or sodium benzoate in water or other solvent (alcohol), giving solutions of double compounds which are used as such or are evaporated to dryness in a vacuum. The neutral solutions may be sterilised. The combination also takes place, on account of the hygroscopic nature of the tannoids, when the dry mixtures are exposed to moist air. C. HOLLINS.

Preparation of heterocyclic arsenic compounds. A. BINZ and C. RÄTH (U.S.P. 1,661,871, 6.3.28. Appl., 2.4.26. Ger., 4.11.25).—See B.P. 250,287; B., 1926, 512.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Two-colour kinematograph films. J. E. THORNTON (B.P. 285,262, 8.3.27).—A two-colour film is built up from two thin component films, each consisting of a support and a sensitive colloid layer on each support, one layer being coloured orange-red and the other blue-green before printing, the layers being printed from the back and developed in hot water to produce coloured reliefs,

and the films then being cemented together with their supports in contact. One coloured image is reversed with respect to the other, and the layers are printed through the back of the supports to which they are attached. The films may be cemented together after printing, and the relief images are protected with varnish.

W. CLARK.

Film material for the production of multi-colour transparencies. J. E. THORNTON (B.P. 285,227, 20.1.27).—For producing multi-colour positive transparencies built up by cementing in superposition the gelatin layers with no waterproof celluloid supports, various quadruple-width webs of non-sensitised material are described. A sensitising agent may be absorbed from solution into the colloid film layer after it is spread on the temporary layer, or mixed with the colloid of the film-layer. [Stat. ref. to B.P. 26,828 and 25,084 of 1912, and 26,786 of 1911.]

W. CLARK.

Manufacture of multi-colour kinematograph films. J. E. THORNTON (B.P. 285,228, 28.1.27).—A multi-colour film, consisting only of the gelatin layers with no celluloid supports, comprises four differently-coloured, sensitive, colloid film layers, each layer being printed with a component image of a four-colour picture, and the layers being cemented together in register to form a single film strip of positive pictures. A cellulose cement is used as binding agent. Exposed parts of the colloid film are protected with cellulose varnish. The compound film may also consist of three or two component colloid layers.

W. CLARK.

Production of partial negatives or diapositives for multi-colour printing. A. NEFGEN (U.S.P. 1,661,079, 28.2.28. Appl., 27.2.23. Ger., 7.3.22).—The preparation of the partial plates is carried out from a diapositive of the original which has been coloured or painted with all the basic soluble colours.

W. CLARK.

Production of a colour photograph. L. D. MANNES and L. GODOWSKY, JUN. (U.S.P. 1,659,148, 14.2.28. Appl., 4.8.23. Cf. U.S.P. 1,516,824; B., 1925, 115).—The photographic film carries two superposed emulsions, that next to the support being sensitised to record orange-red sensations, and the other being treated to record blue-green sensations, and, in addition, with a yellow dye to exclude blue and violet rays from the lower emulsion. The exposed negative is developed in metol and bleached in potassium ferricyanide solution, and the upper image is redeveloped in amidol. The lower image is toned blue with ferrous chloride solution, and the upper image is treated with cupric ferricyanide as a mordant, and with fuchsin. The negative is completed by treatment with a yellow basic dye. The positive is formed by exposure of a similar film through the negative, and is developed in the same way.

T. S. WHEELER.

Photographic medium and process. M. C. BEEBE, A. MURRAY, and H. V. HERLINGER, ASSTS. to WADSWORTH WATCH CASE CO. (U.S.P. 1,658,510, 7.2.28. Appl., 15.3.26. Cf. U.S.P. 1,587,269—1,587,274; B., 1926, 773).—A cellulose ester, *e.g.*, cellulose acetate, is sensitised with a suitable organic halogen compound, *e.g.*, iodoform (1%). The image formed on exposure to light

is developed in a silver reducing solution containing silver nitrate and ammonia.

T. S. WHEELER.

Photography. N. SULZBERGER (U.S.P. 1,658,177, 7.2.28. Appl., 4.6.23).—The use of a developer containing phenylhydroxylamine oxalate is claimed to give sepia tones.

T. S. WHEELER.

Screens for photographic purposes, photo-mechanical printing processes, etc. A. R. TRIST (B.P. 286,340, 4.11.26).

XXII.—EXPLOSIVES; MATCHES.

Gases from blasting in tunnels and metal-mine drifts. E. D. GARDNER, S. P. HOWELL, and G. W. JONES (U.S. Bur. Mines, Bull. 287, 1927, 96 pp.).—Analyses have been made of the gases produced by blasting in tunnels and underground mines in order to determine the effect of varying the explosive and the methods of drilling and blasting on the formation of toxic gases. Accurate and representative sampling was difficult in some mines, but in one series of experiments the gases were confined by brattices and were thoroughly mixed before a sample was taken. The results of the sampling indicate that the gases produced by blasting are not always the same as those obtained in the Bichel gauge. Under normal conditions the only toxic gas produced in dangerous quantities is carbon monoxide, but blasts in massive sulphides give rise to dangerous quantities of hydrogen sulphide and sulphur dioxide. Oxides of nitrogen are produced only in harmless amounts even with explosives containing an excess of 3–5% of oxygen. Less carbon monoxide is made by blasting with some gelatin dynamites than with others, and the gases from two commercial "gelatins" improved during the course of the tests. The oxygen balance of the explosive is the main factor in determining the amount of carbon monoxide produced, but its condition has also an effect; gelatin dynamite that is too insensitive to detonation for efficient work should not be used. The manner of drilling does not appear to have any definite effect on the composition of the gases, but in hard rock the use of stemming reduces the amount of carbon monoxide. Removal of the wrapper from the cartridge has the same effect, but also lowers the strength of the explosive. Less carbon monoxide appears to be formed by blasting in wet ground than in dry rock.

A. B. MANNING.

PATENTS.

Production of gelatinised explosives or smokeless powders. DYNAMIT A.-G. VORM. A. NOBEL & Co. (B.P. 282,631, 27.9.27. Ger., 21.12.26).—Gelatinous dynamites or smokeless powders are made by gelatinising with nitrocellulose a solution of one or more nitrates of solid multivalent alcohols in nitroglycerol or nitroglycol. Sorbitol and mannitol are suitable alcohols for this purpose. Diphenylamine may be added as a stabiliser and urea as an aid to gelatinisation.

S. BINNING.

Gelatin dynamite. C. D. PRATT, ASSR. to ATLAS POWDER CO. (U.S.P. 1,658,816, 14.2.28. Appl., 25.3.27).—The keeping properties of gelatin dynamite are improved by addition of dried beet pulp.

T. S. WHEELER.

Explosive priming mixture. A. E. WHITE. From REMINGTON ARMS Co. (B.P. 285,232, 24.1.27).—A non-corrosive priming mixture for ammunition contains mercury fulminate as initial explosive, an oxidiser containing a salt of an alkaline-earth metal, either alone or with another oxidising salt of a heavy metal, and a reducing agent such as a thiocyanate of a heavy metal, alone or mixed with another reducing agent. Barium nitrate with or without lead nitrate may be used as the oxidising agent and antimony sulphide as the second reducing agent. S. BINNING.

Explosive composition. W. O. SNELLING and G. A. RUPP, Assrs. to TROJAN POWDER Co. (U.S.P. 1,659,449, 14.2.28. Appl., 17.7.26).—The addition of 1–5% of ferrosilicon to explosives containing nitrostarch is claimed. T. S. WHEELER.

XXIII.—SANITATION; WATER PURIFICATION.

Intensive bacterial oxidation [of activated sludge]. Oxidation of ammonia to nitric acid. I–IV. G. J. FOWLER, Y. N. KOTWAL, R. V. NORRIS, S. RANGANATHAN, and M. B. ROY (J. Indian Inst. Sci., 1927, 10A, 97–116).—The conditions under which intensive nitrification can proceed, using the activated sludge process, have been studied. It is shown that the sludge must be acclimatised to the peculiar conditions by increasing the strength of the ammonia solution slowly as the capacity to deal with it increases, the theoretical quantity of calcium carbonate to neutralise the acid produced being added on each occasion. It was found that the maximum amount of ammoniacal nitrogen which could be oxidised under these conditions was 0.25 g./litre per day. Increasing concentrations of calcium nitrate were found to reduce progressively the rate of reaction, the maximum concentration obtainable being 7.5% when nutrient salts also were added and 5.56% when such salts were absent. The influence on the reaction of various salts was investigated, of which ferrous sulphate was found to have a beneficial effect. The optimum p_H value is shown to be between 7.0 and 8.0. C. JEPSON.

Technique and significance of the biochemical oxygen demand determination [of water]. F. W. MOHLMAN, G. P. EDWARDS, and G. SWOPE (Ind. Eng. Chem., 1928, 20, 242–246).—The various methods of carrying out the test and comparative results obtained are discussed. A description is given of the method adopted in the Sanitary District of Chicago which includes the use of a standard water consisting of distilled water and 500 p.p.m. of sodium bicarbonate. The rate of satisfaction of biological oxygen demand in sewage and polluted streams is shown to follow a two-stage curve, the first, which is satisfied in 5–12 days, representing a carbonaceous oxidation, and the second, conversion of nitrogenous material into nitrate. C. JEPSON.

Method for determining the biochemical oxygen demand [of water]. F. SIERP (Ind. Eng. Chem., 1928, 20, 247).—An apparatus is described by means of which the determination is made by direct absorption of gaseous oxygen by the undiluted sample. It is possible to read off the oxygen demand when desired, and

many errors unavoidably incurred in other methods are eliminated. C. JEPSON.

Cyanide-citrate pour-plate medium for direct determination of the colon-aërogenes content of water and sewage. R. E. NOBLE (J. Amer. Water Works' Assoc., 1928, 19, 182–192).—The medium described is a modified Ayers-Rupp medium containing two new ingredients, viz., ferric citrate and potassium ferrocyanide. It is claimed that with this medium the time required to obtain the *B. coli* index number may be reduced by 40–50%, and the results obtained are more correct than with the lactose broth tube-method in general use. It also promises to afford a ready means of distinguishing between organisms of faecal and non-faecal origin. C. JEPSON.

Colorimetric p_H determinations [of water] in a neutral atmosphere. H. F. MUER and F. E. HALE (J. Amer. Water Works' Assoc., 1928, 19, 201–206).—The effect of the laboratory atmosphere on colorimetric p_H determinations of water may be considerable. It is suggested that the test should be carried out in stoppered bottles previously filled with air neutralised by passing through potassium hydroxide solution and lime water. C. JEPSON.

Monochloroamine as a disinfectant for swimming baths. W. OLSZEWSKI (Chem.-Ztg., 1928, 52, 141–142).—Chlorine, hypochlorous acid, and chloramine form a series of bactericidal agents of which the efficiency increases in that order. The most satisfactory method for the preparation of monochloroamine is by the action of ammonia on free hypochlorous acid. L. M. CLARK.

Concrete control methods in the construction of a filtered-water reservoir. W. C. MABEE (J. Amer. Water Works' Assoc., 1928, 19, 193–200).—The ratio of water to cement when mixing concrete was made part of the contract when constructing a covered, filtered-water reservoir for the Indianapolis Water Company. A 28-day strength of not less than 2500 lb./in.² in compression was aimed at and achieved by careful control of this ratio, and by study of the conditions relative to aggregates, time of mixing, distribution, placing, puddling, etc. On test, under 14-ft. head, the tank lost only 0.3% of its volume in 24 hrs. C. JEPSON.

PATENTS.

Purification of sewage and analogous liquids. A. V. DE LAPORTE, F. A. DALLYN, and F. R. MANUEL (U.S.P. 1,658,974, 14.2.28. Appl., 12.4.26).—The sewage is agitated in presence of oxygen and chlorine with a mixture of aluminium and iron salts to precipitate and oxidise colloidal matter. T. S. WHEELER.

Manufacture of base-exchange substances for the removal of iron and manganese compounds and hardness from drinking and boiler-feed waters. W. GRADENWITZ (G.P. 445,377–8, [A] 14.9.18, [B] 19.9.18. Addn. to G.P. 403,263; cf. U.S.P. 1,493,455; B., 1924, 691).—(A) Burnt clay or brick is subjected to a short treatment with concentrated hydrochloric acid; the product is then sufficiently active as a base-exchanging material without further calcination, or (B) it may subsequently be treated with ammoniacal calcium chloride solution before use. A. R. POWELL.