

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DEC. 21, 1928.



I.—GENERAL; PLANT; MACHINERY.

Milling material. Distribution of particle size in the ground product. A. H. M. ANDREASEN (Kolloidchem. Beih., 1928, 27, 349—458).—Mainly a theoretical and mathematical investigation of the possible methods of characterising a finely-ground substance. It is considered that a single numerical value, representing, for example, the increase in surface per kg. of substance, does not suffice to characterise the material, and it is essential to consider the distribution of the various sizes of particles. This may be investigated by sieving, by flotation, or by sedimentation. Several definitions of the new ideas involved are given, and the relations between these are worked out mathematically. A number of mills are described, and with their aid the characteristics of the new terms have been investigated experimentally on felspar, flint, glass, brick, barytes, iron glance, and quartz sand. It is concluded that no general relation exists for the distribution of particle size in a milled material, the results varying with the substance examined.

E. S. HEDGES.

Measurement of high temperatures by means of a tungsten-molybdenum thermocouple. D. BINNIE (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 35—36, 42).—A tungsten-molybdenum thermocouple may be used for temperature measurements up to 2000° provided that oxidation is avoided by passing a current of hydrogen through the furnace or through the protecting tube of the couple. The thermo-electric curve is almost a parabola with the equation $y = 0.151x^{2.125}$, the neutral temperature occurs at 618° when the *E.M.F.* of the junction is 1.36 millivolts, and the reversal point at 1218° using a cold junction at 18°. Above the reversal point tungsten is the electropositive metal. The tungsten wire should not exceed 0.4 mm. in diam. nor the molybdenum wire 0.64 mm.

A. R. POWELL.

PATENTS.

Maintaining an even temperature in chambers in which exothermic reactions are carried out. SOC. CHIM. DE LA GRANDE PAROISSE, AZOTE & PROD. CHIM. (F.P. 626,501, 1.4.26).—Heat is transferred from the hot end of the catalyst to the cooler end by a suitable heat-interchange system or by passing the gases, before they enter the reaction chamber, over the hotter end of the chamber to preheat them before they pass to the cooler part of the catalyst.

A. R. POWELL.

Mercury vapour generators. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of B. L. NEWKIRK (B.P. 295,021, 24.7.28. U.S., 4.8.27).—A mercury vapour generator for removing the heat from the walls of furnaces comprises a series of tubes 10 ft. long and 0.4 in. internal

diam. communicating at their lower ends with a supply pipe and heater which generates the mercury vapour in such a way that the vapour is charged with 2—3% of its volume of finely-divided globules of liquid mercury. This vapour rises through a helical spiral inside the tubes and acts as a more efficient heat-interchange substance than mercury vapour free from liquid.

A. R. POWELL.

Separation of substances of varying sp. gr. A. K. and A. K. DOBREV and D. K. ICONOMOV (F.P. 622,309, 30.9.26).—Constituents of different sp. gr. may be separated from mineral mixtures by allowing the mixture to flow down a sloping trough up which a current of liquid is pumped, whereby the lighter particles are carried upwards while the heavier particles sink to the bottom of the trough.

A. R. POWELL.

Apparatus for ascertaining the characteristics of flowing liquids. V. HENNY and J. D. SEGUY, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,688,811, 23.10.28. Appl., 5.7.23. Renewed 2.7.28).—The liquid flows into a constant-level container communicating with a second container of the same height in which a stagnant sample of the liquid collects.

L. A. COLES.

Apparatus for mechanical treatment of a mixture of gas and liquid. W. L. J. SPOOR (F.P. 624,383, 9.11.26. Holl., 10.11.25).—The gas mixture is passed through a centrifugal or screw valve in such a way that the valve opposes the flow of gas, whereby the suspended particles of liquid are deposited on the walls of the valve by the centrifugal force.

A. R. POWELL.

Purification of gases. W. NEU (F.P. 624,542, 13.11.26. Ger., 14.11.25).—The gas stream is first given a high velocity and then the rate of flow is rapidly retarded, whereby the solid impurities are deposited or carried along in the original direction of flow whilst the purified gas is taken out of the circuit through side tubes.

A. R. POWELL.

Rotary kiln and furnace. J. H. BENTLEY (U.S.P. 1,690,048, 30.10.28. Appl., 9.9.26. U.K., 8.1.26).—See B.P. 263,630; B., 1927, 175.

Heat exchanger. C. H. POTTS, Assr. to VICKERS, LTD. (U.S.P. 1,690,501, 6.11.28. Appl., 11.1.28. U.K., 8.10.26).—See B.P. 285,524; B., 1928, 506.

Minute disintegration of substances. Disintegrating device. Production of suspensions. F. J. E. CHINA (U.S.P. 1,690,667—9, 6.11.28. Appl., [A] 10.12.23, [B] 12.3.26, [C] 25.2.28. U.K., [A—C] 8.3.22).—See B.P. 193,693; B., 1923, 386 A.

Apparatus for mixing liquids with solid or semi-solid substances or with other liquids. N. BENDIXEN, Assr. to J. G. Y. D. MORGAN (U.S.P. 1,689,103, 23.10.28.

Appl., 27.11.26. U.K., 25.5.26).—See B.P. 266,950; B., 1927, 320.

Separation of liquids. H. S. HELE-SHAW and A. BEALE (U.S.P. 1,690,537, 6.11.28. Appl., 19.7.26. U.K., 22.7.25).—See B.P. 260,045; B., 1927, 1.

Drum for centrifugal separating apparatus. K. J. SVENSSON and K. A. P. NORLING (U.S.P. 1,688,838, 23.10.28. Appl., 11.12.26. Swed., 29.12.25).—See B.P. 263,443; B., 1927, 128.

Process for [rotary] filtering elements. H. A. VALLEZ (U.S.P. 1,691,001, 6.11.28. Appl., 19.5.22).—See B.P. 201,822; B., 1923, 961 A.

Gas burners for heating furnaces etc. E. E. LUCAS (B.P. 299,125, 12.4.27).

Refrigerating systems. U. R. and H. R. LORANGER (B.P. 298,498, 9.5.27).

[Whisking] machines for mixing, aerating, and like treatment of various materials [e.g., cakes, confectionery]. BAKER PERKINS, LTD., L. S. HARBER, and J. E. POINTON (B.P. 298,723, 9.8.27).

Gas cartridges for fire extinguishers. G. SCHWORETZKY, and EXCELSIOR FEUERLÖSCHGERÄTE A.-G. (B.P. 299,096, 22.7.27).

Gas purifier (F.P. 623,660).—See II. Purification of gases (B.P. 298,726).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

X-Ray examination of coal sections. C. N. KEMP (Proc. Roy. Soc. Edin., 1928, 48, 167—176).—The technique of preparing coal sections for X-ray examination is described in detail and illustrated photographically. The block of coal is placed in a wooden box and embedded in plaster of Paris. The whole is then cut into sections by means of a band-saw or, if necessary, a reciprocating power-saw. If the cut surface is chipped it may require subsequent milling or grinding. Sections $\frac{1}{2}$ in. in thickness appear to be the most suitable for giving details of structure and ash distribution. The interpretation of the photographs is briefly discussed.

A. B. MANNING.

Origin of fusain. H. BODE (Mitt. Abt. Gesteins-, Kohle-, u. Salz-Untersuch., 1927, 12—22; Fuel, 1928, 7, 487—492).—The theories of Stach and of Petrascheck on the origin of fusain are adversely criticised. A study of the occurrence of fusain in coal balls and in the sphärosiderites of brown coal proves that wood charcoal was already present in the primeval peat in the same form in which it is now found as a constituent of bituminous and brown coal, and supports the forest-fire theory of its origin.

A. B. MANNING.

Swelling of coal. G. LAMBRI (Brennstoff-Chem., 1928, 9, 341—346).—Existing tests for the swelling power of coals, such as those of Muck, Kronig, Korten, and others, are all open to the objection that they do not allow the maximum possible expansion, and the results of different investigators cannot be compared owing to lack of uniform conditions. The formation of a film of coke on the outside of the test coal will suppress expansion as it becomes rigid, and swelling will stop when the coke layer is complete; it will reach a maximum only

when the coal particles remain movable for some time after they are heated. This is achieved by putting a layer of kieselguhr at the bottom of a crucible, with powdered coal above, and heating at 900° without a cover; the coke film quickly formed on the top acts as an insulator, but does not prevent expansion as the coal is heated further only from the sides. Compared with cokes produced by other methods, the volume per unit volume of coal is more than twice as great, the apparent density is very low, the porosity high, and the yield lower. Other materials which might be used to form the layer in the crucible are decreasingly useful in the order of their increasing heat conductivities.

R. H. GRIFFITH.

Comparative action of bromine on cellulose, lignin, wood, and coals. W. FUCHS (Brennstoff-Chem., 1928, 9, 348—350).—When wood is treated with bromine, the cellulose is unaffected but the lignin reacts slowly and a definite proportion of the halogen can then be displaced by the action of aqueous acetate solution. The substance (2 g.) to be examined is suspended in 25 c.c. of carbon tetrachloride, and 25 c.c. of a 4% solution of bromine in the same solvent are added, the whole is boiled for 20 min., filtered, and washed. Bromine is determined by heating with a soda-potash mixture, and the removable bromine by boiling with 50 times its weight of 5% acetate solution; in the case of coal, calcium acetate is used. Air-dried wood takes up about 10% of bromine, most of which is removable as hydrogen bromide, and lignin reacts even when quite dry. Coals are extracted with benzene-alcohol mixtures before treatment, and are found to react readily; after removal of part of the bromine with acetate solution the residue will add on more halogen than it took up originally.

R. H. GRIFFITH.

Absorption of pyridine vapour by Upper Silesian coals. W. SWIENTOSLAWSKI and M. CHORAŻY (Przemysl Chem., 1928, 12, 526—534).—Vitrain on being kept in a desiccator containing pyridine will absorb 70% of its dry, ash-free weight of pyridine vapour, durain 54%, and fusain 12%. Under the same conditions benzene, aniline, dimethylaniline, and quinoline vapours are absorbed only to a negligible extent. A pyridine extract of coking coal will absorb over 100% of its weight of pyridine vapour. A convenient basis is thus afforded for determining the coking qualities of a sample of coal.

R. TRUSZKOWSKI.

Coke formation. II. R. A. MOTT and T. SHIMMURA (Fuel, 1928, 7, 472—486; cf. B., 1927, 353).—A number of Yorkshire and Durham coals have been heated, both in the lump and in the powdered form, to 900°, at a uniform rate of either 1° or 5° per min. With the slower rate of heating the swelling was roughly proportional to the carbon content of the coals, which varied from 82 to 85%. No correlation could be observed between the degree of swelling and the rate of gas evolution during the plastic period. The influence of the rate of tar evolution on the coking process was studied by comparing the cokes produced from coals which had been preheated to various temperatures (390—450°), before and after extraction of the preheated coal with ether. It is suggested that the first stage of coke forma-

tion from crushed coal is the liquation of tar to the surfaces of the particles until these are completely wetted, and the second stage is the loss of identity of the coal particles with the formation of pore structure due to swelling.

A. B. MANNING.

Oxidation of hydrocarbons. DUMANOIS and MONDAIN-MONVAL (Compt. rend., 1928, 187, 892—894).—The temperature-pressure curves of pentane-air mixtures show that for less than 5—10% of the theoretical amount of pentane required for combustion no ignition occurs below 300°, though at 250° there is a slight increase in pressure (cf. Lewis, A., 1927, 851). For larger quantities of pentane the graph is linear but shows a change of direction, corresponding with a relative increase in pressure, at 120°, whilst 10—15° below the spontaneous ignition point, at a temperature which decreases as the proportion of pentane increases, the pressure mounts very rapidly. Pentane-nitrogen mixtures give a steeper line with no angular point. In the presence of 0.001% of lead tetraethyl more than 10% of the theoretical amount of pentane is required for spontaneous combustion below 300°, the change in direction is less marked, and the final rise in pressure occurs at a slightly higher temperature. J. GRANT.

Excess air in gas heating. H. MÜLLER (Gas- u. Wasserfach, 1928, 71, 971—975, 995—1000, 1017—1020, 1042—1046).—The theory of combustion as applied to the domestic gas fire has been studied in detail. In addition, an experimental study has been made of the relationship between the rates of supply of gas and air to the fire, the composition and temperature of the flue gases, and the amount of water which condenses in the flue. An air supply 40% in excess of the theoretical is considered to be the practical minimum. Owing to faulty installations much higher values are common. To attain complete combustion with only 40% excess air, correctly designed burners are essential. To limit the necessary heating surface the flue-gas temperature should not be too low; it should also be possible to lower the dew point of the flue gas by the addition of secondary air without unduly lowering the temperature of the mixed gases. The volume of flue gas produced per unit volume of combustible gas supplied is roughly inversely proportional to the hourly gas consumption. It therefore appears impossible to avoid high values for the excess air with very low rates of gas consumption. Satisfactory control of the excess air at normal rates of gas consumption can be attained by the use of suitable dampers in the flue. A constant value for the excess air with a varying gas consumption has been attained in the past only by subdivision of the heating units. It appears possible that such control may be achieved to some extent by regulation of the primary air to the burners.

A. B. MANNING.

Primary tar oils. R. WEISSGERBER (Ber., 1928, 61, 2111—2119).—Fractions of the oil, b.p. 187—207°, are shaken with 2% of concentrated sulphuric acid, whereby easily resinifiable indenenes and coumarones are removed, leaving a portion, b.p. 193—201°, which is treated with 90% sulphuric acid at 15—20°. The olefines are converted thereby into alkyl hydrogen sulphates, which remain in the oil, from which they are

removed by water and then converted into their sodium salts. The acid layer retains ketonic oils (see later). The sodium salts are decomposed with steam, thus yielding methylnonylcarbinol, further identified by conversion into the oxime of methyl nonyl ketone, m.p. 45—46°. The presence of undecylene in the oil is thus established. Its amount cannot be deduced from these experiments, but the occurrence of olefines in the oil accounts for the difficulty of purifying the latter by the customary technical methods. The oil fraction (see above) contains normal alkyl sulphates in such quantity that it cannot be distilled under reduced pressure. Treatment of it with steam at 150° yields a distillate with the properties of a technically pure hydrocarbon residue and a large proportion of dark yellow resin from which sulphuric acid is removed by aniline at 170° in small amount; the production of the resin is mainly due to polymerisation of olefines. The oil dissolved by the 90% sulphuric acid (see above) is distilled with steam, thereby leaving a small amount of resin. About one third of the distillate consists of ketones which are removed as hydrazones, leaving a mixture of nitriles (from the hydrolytic product of which *o*-toluic acid is isolated) and polysubstituted thiophens, chiefly tetramethylthiophen. The latter compounds do not afford additive compounds with mercuric chloride, lose sulphur as sulphuric acid when oxidised, and give uninviting products with halogens. Their nature is established by partial dealkylation by passage through a tinned iron tube at 650—675°, whereby thiophen, thiotolen, 2:3-dimethyl- and trimethyl-thiophen are produced. 2:4-Dimethylthiophen at 675—700° similarly yields thiophen and thiotolen. 2:5-Dimethylthiophen is converted by successive acetylation and reduction according to Clemmensen into 2:5-dimethyl-3-ethylthiophen, which, by repetition of the processes, affords 2:5-dimethyl-3:4-diethylthiophen, b.p. 214—217°, d^{15} 0.9573; the compound dissolves unchanged in 80% sulphuric acid. Comparison of the behaviour of the primary tar-oil fraction with that of a coke-oven tar fraction of similar b.p. shows that the exposure to the higher temperature greatly diminishes the amounts of olefines, ketones, and homologous thiophens partly by gasification and partly by dealkylation. The apparent appreciable increase of coumarones, indenenes, and nitriles is due to their remarkable thermostability at 900—1000°. In the chemical character of their groups the tars are identical. The quantitative distribution of these groups and the predominance of homologues in primary tar impart distinctive character to the products. H. WREN.

Ammonia recovery from steamed vertical-retort gas. C. SINGLETON and J. E. STANIER (Gas J., 1928, 184, 405—407).—In vertical retorts where the yield of ammoniacal liquor per ton of coal depends largely on the amount of steam added to the retorts, the recovery of the ammonia in the highest concentration possible is of great importance. At Stockport gas from 48 retorts with a capacity of 7 tons per day each passes through water-cooled condensers, exhausters, washers, and tar extractors, and finally to an acid washer where the solution is worked up to about 35—38% ammonium sulphate containing 4—5% of free acid. A series of tests carried out showed that weak liquors free from

tar could be advantageously used to wash the gas, and that the introduction of the acid-catch washer increased the strength of the virgin liquor by $\frac{3}{4}$ oz.; no trouble was experienced with entrained acid. Figures were also obtained showing the decrease in the total oxygen absorption of the spent-liquor effluent and in the total volume of liquor to be handled. R. H. GRIFFITH.

Evaluation of used transformer and switch oils for further use. K. TYPKE (Brennstoff-Chem., 1928, 9, 346—348).—Physical and chemical tests can be applied in examination of oils which have been used; for cases where no great heating occurs changes will not be large. The sp. gr. at 20° should not be above 0.895, and in a light oil separation of water and suspended solids will take place more readily. Viscosity may be between 10° and 11° Engler, the solidifying point should be below -40°, and the dielectric strength not less than 80 kv. per cm. Among chemical tests, the ash content, which is related to the amount of soap present, should be below 0.2%, the acid value 1.0, and the saponif. value 4.0. Examination with caustic soda or sulphuric acid does not give any important information, but miscibility with other oils should be considered; this is determined by mixing, keeping over-night, and measuring the sludge obtained on centrifuging. R. H. GRIFFITH.

Artificial ageing of turbine oils. R. SCHMIDT (Z. angew. Chem., 1928, 41, 1197—1201).—In order to predict the stability of turbine oils under technical conditions it is essential that any artificial ageing (oxidation) experiments be conducted in the presence of metallic catalysts, as the presence of metals is an important factor revealing differences of behaviour of apparently similar oils. The following test is advocated: 125 g. of oil are oxidised by a slow stream of oxygen (2 bubbles per sec.) in a 250 c.c. flask immersed in a boiling water-bath; the catalyst is introduced in the form of a piece of copper foil (40 × 50 × 0.5 mm.) superficially oxidised and bent to the form of a cylinder. After 70 hrs. oxidation the oil is tested for acidity and tarry and asphaltic matter. E. LEWKOWITSCH.

Separations of emulsions. PAVLIKOVSKI.—See XI.
Lamp-blacks for rubber mixtures. KIRCHHOF.—See XIV.

PATENTS.

Retorts for distilling coal, shale, and other fragmentary solid material. AMER. HYDROCARBON Co., INC. (B.P. 270,662, 5.4.27. U.S., 4.5.26).—A horizontal or slightly inclined annular retort for the low-temperature distillation of coal etc. has both the inner and outer walls rotatable, either together or independently. The outer wall is conical, so that the cross-section of the annular chamber increases towards the discharge end. The walls are provided with inwardly projecting longitudinal fins. Both walls are heated and are jacketed, the interspaces containing a suitable heat-distributing material, e.g., molten lead. The inner wall is movable longitudinally in such a way that the fins attached to it may be brought into contact with the outer wall for detaching any material adhering thereto. A. B. MANNING.

Low-temperature distillation apparatus. H. G. LYKKEN (U.S.P. 1,685,496, 25.9.28. Appl., 23.5.24).—The material to be distilled moves along a heated pan of annular shape provided with inlet and outlet ports for the material. C. O. HARVEY.

Utilisation of heat contained in coke discharged from coke ovens, gas retorts, etc. N. V. SILICA EN OVENBOUW MIJ., and W. HIBY (B.P. 298,547, 30.6.27).—The sensible heat of the coke is utilised for the distillation, at comparatively low temperatures, of coal, shale, etc., by charging the two materials together into a chamber to which is connected condensing plant for the recovery of the volatile carbonisation products. The mixed coke and semi-coke may be used as a whole, e.g., as a domestic fuel, or may be separated by hand or by screening. The heat remaining in the coke mixture may be used to dry a further quantity of moist coal by feeding the latter into the lower part of the chamber, and subsequently separating it again by screening. A. B. MANNING.

Recovery of sulphur [from furnace gases]. W. H. HOWARD (U.S.P. 1,685,231, 25.9.28. Appl., 20.8.27).—The gases are scrubbed with water, the resulting acid wash-water is aerated, and the mixture of air and sulphur dioxide is passed over incandescent carbonaceous material. F. G. CLARKE.

Flotation processes for the treatment of coal, ores, etc. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 298,736, 19.8.27).—The products obtained by oxidising montan wax or paraffin wax at not above 200° and completely or partially neutralising with ammonia or other bases are employed as flotation agents. H. S. GARLICK.

Production of adsorption agents from carbonaceous materials. O. SCHÖBER (B.P. 298,546, 9.6.27).—Highly active carbons, characterised by containing at least 10% of free carbon and over 20% of acid-soluble ash, are produced by treating carbonaceous material at 500—800° with activating gases in which the oxygen partial pressure lies between 8 and 115 mm., until the loss of weight amounts to more than 40%. The material may be given a short after-treatment at about 1000° in an oxygen-free atmosphere. A form of apparatus is used in which the waste gases from the activation and from the heating chamber are used to preheat the raw material and the activating gases or vapours. In order to free the product from sulphur and phosphorus the hot material may be given a limited acid treatment and subsequently washed, or iron in some suitable form may be added to the raw material and the product subjected to magnetic separation in a field of small intensity. Further treatment in magnetic fields of greater intensity may be used to separate the product into fractions of varying activity and ash content. A. B. MANNING.

Gas purifier. G. VIGREUX, and SOC. ANON. DES ANC. ÉTABL. GÉPÉA (F.P. 623,660, 25.10.26).—A gas-purification apparatus comprises a metal container divided into two sections through which the gas passes in succession. The first section is filled with a liquid solvent and is provided with a series of perforated

shelves through which the gas stream must pass, whereby it is continually broken up into small bubbles. The second section is packed with a suitable dry purifying agent, which acts as a filter for solid and liquid particles.

A. R. POWELL.

Distillation of coal tar. C. WESSEL (B.P. 299,290, 24.7.28. Addn. to B.P. 273,675; B., 1928, 778).—Tar is heated to 180° to drive off water and light oils. Heating is then reduced so that when slightly superheated steam is driven into the tar the temperature does not rise above 215°, and practically oil-free naphthalene is recovered. For continuous working, the tar may be preheated to 180° in a separate container, passed to the boiler where it is steam-treated, and continuously passed out naphthalene-free.

H. S. GARLICK.

Bituminous compositions for use as supports exposed to high pressures. R. STEIN (B.P. 299,208, 8.8.27).—Epure (natural Trinidad asphalt freed from earthy impurities), goudron (a distillation product of Trinidad asphalt), and pitch, in the proportions 1 : 2 : 2, are raised to the b.p. and mixed with 1 pt. of asbestos fibres. The composition is preferably used in conjunction with one or more layers of wire.

H. S. GARLICK.

Purification of montan wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,133, 20.8.27).—An aqueous solution or suspension of montan wax is subjected to the anodic action of an electric current in the presence of electrolytes and oxygen transferers, such as water-soluble compounds of chromium, cerium, vanadium, and manganese, and, if desired, an organic solvent.

H. S. GARLICK.

Oxidising paraffins, waxes, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 298,704, 22.7.27).—The employment of an inorganic catalyst associated with an organic nitrogenous base (*e.g.*, a manganese soap and hexamethylenetetramine) considerably accelerates the oxidation of paraffin hydrocarbons by gases containing oxygen.

H. S. GARLICK.

Continuous transformation into light hydrocarbons of the heavy hydrocarbons from petroleum and the like. H. CARROLL (B.P. 284,345, 28.1.28. Fr., 28.1.27).—Crude oil is distilled in a tubular boiler connected to a thermostatically controlled dephlegmator from which the products remaining liquid pass to a cracking apparatus. Cracking is carried out in an atmosphere rich in free hydrogen, and is obtained by causing the oil to flow in thin layers over the surfaces of metallic baths arranged in the form of steps, successively larger or wider and raised to a sufficiently higher temperature to cause the liquid to assume the spheroidal state, in order to obtain a superheating of the gaseous phase without corresponding superheating of the liquid phase.

H. S. GARLICK.

Converting or cracking heavy oils into lighter oils. E. C. R. MARKS. From PETROLEUM PROCESS CO. (B.P. 299,347, 13.6.27).—In a treating chamber preheated oil is sprayed through nozzles against baffles and falls through gratings counter-current to superheated steam. The mixed vapours are reheated and pass to a chamber similar to the first where fresh oil, admitted at

the top, flows counter-current to the vapours admitted at the bottom. Reheating of the vapours and contact with fresh oil may be repeatedly carried out before passing them to the coolers and condensers.

H. S. GARLICK.

Conversion of hydrocarbon oils into lighter oils. C. ARNOLD. From STANDARD DEVELOPMENT CO. (B.P. 299,683, 22.9.27).—In the process of cracking oil by passing it through a heated coil and digestion drums, so arranged that one drum may be used while another drum is being cleaned, the drum to be brought into use is filled with heated oil previous to diverting from the drum in use the oil from the heater coil. Preferably a portion of the oil heated in the coil is passed into the digestion drum prior to the diversion of the main stream of heated oil thereto.

H. S. GARLICK.

Manufacture of viscous [mineral or tar] oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,086, 21.7.27, 19.9.27, and 18.4.28).—Oils of high viscosity and flash point are produced by passing gases rich in lower olefines into hydrocarbon mixtures which are liquid at ordinary temperatures, in the presence of catalysts of the Friedel-Crafts type and at temperatures up to 200°. As starting materials olefines obtained by subjecting tar oils or mineral oils, or higher fractions of these, to a cracking process may be used.

H. S. GARLICK.

Reconditioning of mineral oils. C. J. RODMAN and M. HECHT (U.S.P. 1,685,681, 25.9.28. Appl., 11.11.25).—Electrical insulating mineral oils are reconditioned by evacuation, treatment with an evacuated adsorbent, and filtration, the whole operation being conducted out of contact with atmospheric gases.

C. O. HARVEY.

Refining or fractionating of oils. A.-G. F. KOHLENSÄURE-IND., and E. B. AUERBACH (B.P. 285,064, 23.8.27. Ger., 12.2.27. Addn. to B.P. 277,946; B., 1928, 843).—The treatment of oils as previously described is extended to mixtures of mineral oils with fatty oils, fats, terpenes, tar oils, resin oils, ketone oils, and essential oils, or to oils containing no mineral oil.

H. S. GARLICK.

Manufacture of [rubber-like] hydrocarbons of high mol. wt. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 298,584, 9.6.27).—Products, consisting substantially of 4-carbon olefines having at least two, preferably conjugated, double linkings, obtained from the destructive hydrogenation of coal, oils, etc., are polymerised to rubber-like compounds by treatment with metallic sodium, stannic chloride, carbon dioxide, etc., or by prolonged heating. Examples of suitable materials are the fraction, b.p. about 100°, from hydrogenated mineral oil; isoprene from a hydrogenated coal fraction, b.p. 30–40°, which has been passed over chromium oxide at 500°, converted into amyl chloride by hydrochloric acid, passed at reduced pressure over barium chloride at 400° to give β -isomylene, which is chlorinated and passed again over hot barium chloride; and butadiene prepared by cracking a hydrogenated coal fraction of b.p. 65–75°.

C. HOLLINS.

Active carbo-mineral product [mineralised charcoal]. E. URBAIN, AssT. to URBAIN CORP. (U.S.P.

1,689,647, 30.10.28. Appl., 5.5.26. Fr., 25.6.25).—See B.P. 254,262; B., 1926, 1003.

Treating aqueous emulsions of tar. B. JOHNSON (U.S.P. 1,689,728, 30.10.28. Appl., 6.3.25. Swed., 12.3.24).—See B.P. 284,401; B., 1928, 220.

Improving the properties of montan wax. W. PUNGS and M. JAHRSTORFER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,690,876, 6.11.28. Appl., 10.9.27. Ger., 13.9.26).—See B.P. 289,621; B., 1928, 473.

Cracking of liquid hydrocarbons. O. D. LUCAS and E. L. LOMAX, Assrs. to V. L. OIL PROCESSES, LTD. (U.S.P. 1,690,416, 6.11.28. Appl., 23.7.25. U.K., 9.12.24).—See B.P. 248,115; B., 1926, 396.

[Rotary] washing apparatus for coal or the like. A. P. DENBY (B.P. 299,148, 7.9.27).

Apparatus for cooling of coke. ILLINGWORTH CARBONIZATION Co., LTD., and S. R. ILLINGWORTH (B.P. 298,658, 15.6.27. Addn. to B.P. 248,078; B., 1926, 395).

Scavenging [and removing solid] combustion products in internal-combustion engines. J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 299,139, 27.8.27).

Apparatus for burning liquid fuel. J. J. KERMODE (B.P. 299,486, 21.6. and 9.7.27).

Hydrocarbon oil burner. W. TEALE and H. F. MCLAUTHLIN (B.P. 299,348, 23.6.27).

III.—ORGANIC INTERMEDIATES.

Partial oxidation of methane and ethane in the presence of catalysts. T. E. LAYNG and R. SOUKUP (Ind. Eng. Chem., 1928, 20, 1052—1055).—The catalytic partial oxidation of mixtures of methane and ethane has been studied at atmospheric pressure by the dynamic method within the temperature range 100—700°. Some experiments have also been made with natural gas under similar conditions. For the production of alcoholic or aldehydic intermediates, solid catalysts were unsatisfactory from the point of view of the low yield of the required end-products. The addition of 0.3—5% of nitrogen tetroxide, however, exerted considerable promoting action on the oxidation of the hydrocarbon-oxygen mixtures, and yields of 15—30% by vol. of the oxygenated derivatives were obtained. A temperature of 600° and a heating time of 0.5 sec. were found necessary. The effect of using auxiliary catalysts, such as activated charcoal or platinum oxide, in conjunction with nitrogen tetroxide resulted in an increased yield of hydrogen, oxides of carbon, and steam, but a decrease in the amount of oxygenated compound formed. Methyl nitrite also acts as a catalyst probably owing to its thermal decomposition into nitric oxide or nitrogen tetroxide and a carbon-hydrogen-oxygen residue, the former only having a catalytic action. G. CLAXTON.

Synthesis of methanol [methyl alcohol]. E. AUDIBERT and A. RAINEAU (Ind. Eng. Chem., 1928, 20, 1105—1110).—The results of the action of various substances in catalysing the reaction $\text{CO} + 2\text{H}_2 = \text{CH}_3\cdot\text{OH} + 27,000 \text{ g-cal.}$ are given.

The following oxides: alumina, silica, molybdenum oxide (Mo_2O_7), vanadium oxide (V_2O_5), blue tungsten oxide (W_2O_5), thoria, titania, magnesia, lime, barium oxide, and strontia were inactive. Cerium oxide (Ce_2O_3), uranium oxide (UO_2), beryllia, and zirconia gave less than 2% of methyl alcohol at 150 atm. Manganous oxide, chromium sesquioxide, and zinc oxide, prepared by precipitation from the corresponding nitrates, converted 2—17.5% of carbon monoxide into methyl alcohol, the highest conversion being for zinc oxide at 350°. Metallic copper was found to be a good catalyst if prepared at a low temperature by reduction of copper oxides obtained by precipitation of the nitrate or by the thermal decomposition of copper salts of organic acids. Copper catalysts prepared from the fused oxide or from copper oxide obtained by precipitation of solutions of the chloride or sulphate were inactive. Nickel and iron catalysts favoured the production of carbon dioxide. Single-component catalysts were sensitive to heat, high temperatures impairing their activity. Mixed catalysts were more effective than single-component catalysts. A mixture of cerium oxide and copper was very effective, and the activity reached a maximum at a Ce:Cu ratio of 30:100 with a 92% conversion of carbon monoxide. A mixture of beryllia and copper with a Be:Cu ratio of 6:100 was almost as good, and showed the greatest heat resistance of the whole series studied. G. CLAXTON.

Values obtained in synthesis of methanol [methyl alcohol]. A. C. FIELDNER and R. L. BROWN (Ind. Eng. Chem., 1928, 20, 1110—1112; cf. Brown and Galloway, B., 1928, 780).—A comparison of the values obtained at 400° by the authors with those obtained by Audibert and Raineau (preceding abstract) and by Lewis and Frolich (B., 1928, 359) has been made. Using a development of the Nernst approximation formula, K_p has been calculated from the results of different workers, assuming no departure from the perfect gas laws. The values of K_p obtained are of the same order and vary as might be expected from the activities of the catalyst employed and the relative space velocities used. Taking into account deviations from the perfect gas laws, the fugacities (effective pressures) and the partial fugacities of the components have been calculated from the results of the three sets of investigators. From the partial fugacities a new set of K values, designated K_f , is calculated; the values, as for K_p , are of the same magnitude. Space-time yields are also discussed. G. CLAXTON.

Iron oxide colours. BUDNIKOV.—See XIII.

PATENTS.

Transformation into water-soluble form of organic substances which are *per se* insoluble in water. A. L. MOND. From I. G. FARBENIND A.-G. (B.P. 298,823, 11.1.28).—Insoluble substances, such as tetrachloroethylene, eucalyptus oil, menthol, etc., give concentrated "solutions" in solvent mixtures consisting of Turkey-red oil (40 pts.), potassium ricinoleate (10 pts.), paraffin oil (5—10 pts.), an alkali salt of an alkylnaphthalenesulphonic acid (3—5 pts.), and water (10—20 pts.), especially in presence of a little acid. An 82% solution of tetrachloroethylene, or 4% of eucalyptus oil, in this

mixture may be diluted without the precipitation which occurs in the absence of paraffin. C. HOLLINS.

Chlorination of saturated hydrocarbons with a graphite catalyst. VER. F. CHEM. U. MET. PROD. (F.P. 605,950, 12.11.25).—Chlorine and hydrocarbon vapour are passed separately into a mixing vessel filled with graphite from which the mixture passes to the reaction chamber. Alternatively, the mixed gases are passed directly through a hot chamber packed with a graphite catalyst. The reaction mixture is strongly cooled to condense the greater part of the chlorinated hydrocarbon, and the remainder is separated by washing out the hydrogen chloride in a stream of water.

A. R. POWELL.

Recovery of concentrated from dilute acetic acid. H. SUIDA (B.P. 280,501, 22.7.27. Austr., 9.11.26).—In the process of B.P. 230,447 (B., 1925, 827) aliphatic esters of the hydroxylated products are substituted for the latter as solvents, e.g., acetates of glycol, glycerol, etc., amyl acetate, amyl butyrate, hexyl acetate. The acetates may be produced *in situ*. C. HOLLINS.

Manufacture of acetaldehyde from acetylene. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 299,234, 2.1.28).—To prevent corrosion by mercuric sulphate solution the reaction vessel is made of or lined with a chromium-nickel-molybdenum steel, e.g., 70% Fe, 19–20% Cr, 7–8% Ni, and 2–3% Mo.

C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. BRIT. CELANESE, LTD., H. DREYFUS, and C. I. HANEY (B.P. 298,667, 14.7.27).—Acetic anhydride is extracted from vapours produced by the pyrogenic decomposition of acetic acid at 600–1000° by means of a light petroleum and ether mixture. The vapours are mixed with solvent vapour and passed into cooled solvent, water being drawn off from the bottom of the extracting vessel and acetic anhydride solution from the top; fresh solvent enters the vessel from below through a spray and removes from the water layer any acid or anhydride. C. HOLLINS.

Manufacture of aliphatic acid anhydrides. BRIT. CELANESE, LTD., H. DREYFUS, and W. BADER (B.P. 299,342, 23.7.27).—An acetate or acetic acid is heated with an alkali or alkaline-earth salt of meta- or pyrophosphoric acid at 150–180°, with or without the addition of inert diluents such as kieselguhr. E.g., a dry mixture of sodium metaphosphate, sodium acetate, and kieselguhr is passed through a reaction vessel at 150–180°, liquid glacial acetic acid being added at suitable intervals along its course; acetic anhydride (and acid) distils off, and the exhausted mixture, consisting of sodium orthophosphate etc., is regenerated by acidification and dehydration, and returned to the process.

C. HOLLINS.

Manufacture of crotonaldehyde and its homologues. DISTILLERIES DES DEUX SÈVRES (B.P. 274,488, 14.7.27. Belg., 14.7.26).—Aqueous acetaldehyde (50%) is aldolised at 15° or below in presence of alkali, the mixture is acidified (with phosphoric acid), and distilled, the crotonaldehyde being obtained in the form of its constant-boiling mixture with water, b.p. 84–85°. Butaldehyde under the same conditions gives Δ^{α} - α -ethylhexenaldehyde. C. HOLLINS.

Manufacture of dimethylolurea [bishydroxymethylcarbamide]. POLLOPAS, LTD., E. C. C. and E. J. BALY (B.P. 299,487, 24.6. and 11.11.27).—Formaldehyde and carbamide are condensed below 35° in aqueous sodium hydroxide, the alkalinity being adjusted to about 0.01*N* so as to avoid rise of temperature. A bishydroxymethylcarbamide of m.p. 138–140° is readily obtained. C. HOLLINS.

Manufacture of compounds of the perylene series. I. G. FARBENIND. A.-G. (B.P. 272,528, 9.6.27. Ger., 9.6.26).—Perylenetetracarboxylic acid is decarboxylated in stages by heating it in neutral or alkaline aqueous suspension at 200–260°. The neutral potassium salt gives *perylene-3:9(or 10)-dicarboxylic acid*; in presence of potassium hydroxide a mixture of this and *perylene-3-carboxylic acid*, m.p. above 300°, results, whilst with larger excess of alkali perylene is obtained. The monoimide similarly yields with 7–8 equivalents of alkali *perylene-3:4-dicarboxylimide*, and with 2 equivalents perylene-3-carboxylic acid. C. HOLLINS.

Manufacture of highly-chlorinated perylenes. F. BENZA (B.P. 288,171, 1.3.28. Austr., 2.4.27).—In the presence of a chlorine carrier, such as aluminium chloride or antimony pentachloride, 7–10 atoms of chlorine may be introduced into perylene, e.g., by chlorination in nitrobenzene at 100–140°. C. HOLLINS.

Manufacture of sulphamic acids of aromatic secondary bases. O. Y. IMRAY. FROM I. G. FARBENIND. A.-G. (B.P. 298,550, 8.7.27).—Secondary arylamines (including diarylamines and carbazole) or mixtures containing them are treated with chlorosulphonic esters, sulphur trioxide, or pyrosulphuric acid in presence of an acid-binding agent (pyridine bases, sodium carbonate) with or without a solvent or diluent (pyridine bases, chlorobenzene) to give *N*-sulphonic acids, which may be purified and hydrolysed to regenerate the pure secondary base. C. HOLLINS.

Manufacture of phenylthioglycolic acids containing halogen and alkyl groups in the nucleus. I. G. FARBENIND. A.-G. (B.P. 281,290, 22.11.27. Ger., 24.11.26).—A halogenated toluene or xylene is treated with chlorosulphonic acid and the thiophenol obtained by reduction of the resulting sulphonyl chloride is condensed with chloroacetic acid. *m*-Chlorotoluene gives a 6-sulphonyl chloride, m.p. 54°, 5-chloro-*o*-thiocresol, and 5-chloro-*o*-tolylthioglycolic acid, m.p. 127°. 2:4-Dichlorotoluene-5-sulphonyl chloride, m.p. 71°, 4:6-dichloro-*m*-thiocresol, and 4:6-dichloro-*m*-tolylthioglycolic acid, m.p. 112°, are similarly obtained from 2:4-dichlorotoluene; 2:6-dichlorotoluene-3-sulphonyl chloride, m.p. 60°, the thiocresol, and 2:6-dichloro-*m*-tolylthioglycolic acid, m.p. 100°, from 2:6-dichlorotoluene; 2-chloro-*p*-xylene-5-sulphonyl chloride, m.p. 50°, 2-chloro-5-thio-*p*-xylenol, and 2-chloro-5-*p*-xylylthioglycolic acid, m.p. 96°, from 2-chloro-*p*-xylene. C. HOLLINS.

New intermediates and dyes therefrom. BRIT. DYESTUFFS CORP., LTD., and M. MENDOZA (B.P. 299,501, 27.7.27).—Amino- and diamino-diaryl sulphides are prepared by condensing 3-thiolsalicylic acids carrying a 5-substituent (e.g., methyl or chlorine) or a 5:6-benzo-group (as in 2-hydroxy-1-thiol-3-naphthoic acid)

with reactive halogenated aromatic nitro- or dinitro-compounds. Dyes showing little change in shade on chroming are obtained from the products (cf. B.P. 297,855; B., 1928, 887). 5-Chloro-3-thiolsalicylic acid, m.p. 198—200°, 5-thio-p-cresotic acid, m.p. 198°, and 2-hydroxy-1-thiol-3-naphthoic acid, decomp. 160°, are obtained by reduction of the corresponding disulphides, m.p. 250—252°, 249—250°, and 280° (decomp.), respectively, prepared from the sulphinic acids or sulphonyl chlorides. The dyes described are: 2-amino-2'-hydroxy-4-sulpho-3'-carboxy-5'-methyldiphenyl sulphide \rightarrow β -naphthol (scarlet), γ -acid (acid-coupled, blue-red), or 1-(2:5-dichloro-4-sulphophenyl)-3-methylpyrazolone (yellow); naphthionic acid \rightarrow 2:4-diamino-2'-hydroxy-3'-carboxy-5'-methyldiphenyl sulphide [red; the diamine, m.p. 178—180°, is prepared from the dinitro-compound, m.p. 27° (decomp.)]; benzidine = 2 mols. of the same diamine (deep brown); 2-amino-4-acetamido-2'-hydroxy-3'-carboxy-5'-methyldiphenyl sulphide \rightarrow γ -acid (red), R-acid (red-orange), or 1-(2:5-dichloro-4-sulphophenyl)-3-methylpyrazolone (yellow); 2-amino-2'-hydroxy-4-sulpho-3'-carboxy-5'-methyldiphenyl sulphide, or the 5'-chloro-compound, or 2-amino-4-sulphophenyl 2-hydroxy-3-carboxynaphthyl sulphide \rightarrow cresidine \rightarrow phenyl-J-acid (red-violet). C. HOLLINS.

Manufacture of alkylisrosindulinesulphonic acids. J. R. GEIGY A.-G. (B.P. 282,803, 28.12.27. Ger., 27.12.26).—By heating a 1-chlorodialkylisrosinduline acetate (from phenyl- β -naphthylamine and 3-chloro-4-nitrosodialkylaniline) with sodium hydrogen sulphite the chlorine is replaced by a sulphonic group and a second sulphonic group enters position 7, *para* to the nitrogen. The products are useful intermediates for naphthasafranine dyes. C. HOLLINS.

Manufacture of cyclic ketones. I. G. FARBENIND. A.-G. (B.P. 274,095, 7.7.27. Ger., 8.7.26. Addn. to B.P. 273,321; B., 1928, 887).—A hydrocarbon with free *peri*-positions is condensed with succinic anhydride or its mono- or *s*-dichloro- or *s*-dibromo-derivatives. Acenaphthene with succinic anhydride gives according to conditions 5- β -carboxypropionylacenaphthene, m.p. 206°, or the cyclic diketone, m.p. 180°. C. HOLLINS.

Manufacture of naphthalene-1:4:5:8-tetracarboxylic acid. I. G. FARBENIND. A.-G. (B.P. 274,103, 8.7.27. Ger., 9.7.26).—The cyclic diketone, m.p. 180°, of B.P. 274,095 (preceding) is oxidised with chromic-acetic or chromic-sulphuric acid. C. HOLLINS.

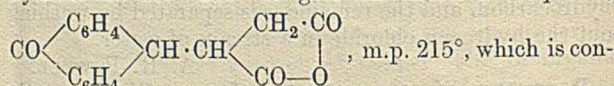
Intermediate compounds for sulphide dyes and dyes therefrom. IMPERIAL CHEM. INDUSTRIES, R. W. POPE, and M. WYLER (B.P. 299,152, 8.9.27).—Phenazines, naphthaphenazines, and dinaphthazines carrying a nuclear hydroxyl group are carboxylated with carbon dioxide or a bicarbonate; the products give on thionation sulphide dyes of increased redness and brightness. C. HOLLINS.

Production of dye intermediates [of the anthraquinone series]. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 299,333, 19.4.27 and 9.1.28).—Chlorine or bromine in α -positions may be removed from halogenated β -aminoanthraquinones by reduction with alkaline glucose or hyposulphite or other agents capable

of producing a leuco-compound. *E.g.*, 1:3-dichloro-2-aminoanthraquinone is boiled with aqueous alkaline hyposulphite for 1 hr. and air-oxidised to give 3-chloro-2-aminoanthraquinone, m.p. 310°; 1-chloro-2-aminoanthraquinone yields 2-aminoanthraquinone.

C. HOLLINS.

Manufacture of benzantrones and intermediate products. I. G. FARBENIND. A.-G. (B.P. 277,342, 7.9.27. Ger., 10.9.26. Addn. to B.P. 268,830; B., 1928, 666).—Anthrone is condensed with maleic anhydride at 160—240° to give an additive compound,



verted by aluminium chloride in nitrobenzene at 100° into 3-hydroxybenzanthrone, m.p. 317°, with loss of carbon dioxide and hydrogen. By heating anthranol with maleic acid at 200° and then at 100—130° with sodium aluminium chloride, 3-hydroxydihydrobenzanthrone, readily oxidised on drying, is obtained. From anthrone and ethyl fumarate in sulphuric acid at 140° there is obtained a 3-hydroxybenzantronesulphonic acid. C. HOLLINS.

Manufacture of benzantronecarboxylic acids. I. G. FARBENIND. A.-G. (B.P. 277,670, 14.9.27. Ger., 20.9.26).—3- or 4-Methylbenzanthrone is oxidised with nitrobenzene and potassium hydroxide at 140—150° to the corresponding 3-carboxylic acid, m.p. 335°, or 4-carboxylic acid, m.p. 307—308°. C. HOLLINS.

Manufacture of derivatives of pyrazolanthrone. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 298,775, 21.10.27).—Pyrazolanthrone is condensed with an aromatic halogen derivative in presence of copper powder, potassium acetate, and a solvent (nitrobenzene, amyl alcohol) to give *N*-derivatives. The following 2-substituted pyrazolantrones are described: *phenyl*-, m.p. 211°; *o*-carboxyphenyl-, m.p. 262—265°; 3-carboxy-2-naphthyl-, m.p. 277—279° (decomp.); *o*-nitrophenyl-, m.p. 246—248°; 3-benzanthronyl-, m.p. 398—400°; 2-phenyl-3-benzanthronyl-; 9(10)-nitro-3-benzanthronyl-, m.p. 404—405° (*amine*, m.p. 417°); 4-benzanthronyl-, m.p. 398—400°; 1-anthraquinonyl-; 1-naphthyl-, m.p. above 300°. The 2-(3-benzanthronyl) derivatives of 5-methylpyrazolanthrone (m.p. 332—333°) and dipyrazolanthrone, and the product from pyrazolanthrone (2 mols.) and 1:3-dichloro-4:6-dinitrobenzene are also described. (Cf. B.P. 298,284; B., 1928, 923.) C. HOLLINS.

Production of complex metal alcoholates [ethoxides]. H. MEERWEIN, Assr. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,689,359, 30.10.26. Appl., 1.11.26. Ger., 12.11.25).—See B.P. 261,377; B., 1927, 316.

Production of alkylated aliphatic nitriles. M. BOCKMÜHL and G. EHRHART, Assrs. to WINTHROP CHEM. Co. (U.S.P. 1,690,325, 6.11.28. Appl., 17.6.26. Ger., 22.6.25).—See B.P. 253,950; B., 1927, 892.

Manufacture of dinitro-derivatives of perylene and its compounds. K. STIEGER, Assr. to F. BENSA (U.S.P. 1,688,837, 23.10.28. Appl., 5.1.26. Austr., 28.10.25).—See B.P. 260,568; B., 1927, 579.

Purification [of phthalic anhydride]. C. E. ANDREWS, AssT. to SELDEN CO. (U.S.P. 1,685,624, 25.9.28. Appl., 27.2.18).—See B.P. 142,902; B., 1920, 501 A.

Dinaphthylidicarboxylic acid and its manufacture. R. HERZ and W. ZERWECK, AssTs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,272, 11.9.28. Appl., 23.6.26. Ger., 2.7.25).—See B.P. 278,100; B., 1927, 902.

Furnace for catalytic gas reactions (F.P. 625,893).—See XI. **Phenols for germicides (B.P. 299,522).**—See XXIII.

IV.—DYESTUFFS.

Influence of sulphonic groups and other substituents on the colour of trisazo dyes. H. DINNEN (Z. angew. Chem., 1928, 41, 1078—1083; cf. Meuly, B., 1924, 86; Wanner, B., 1925, 537).—The absorption spectra of series of trisazo dyes of the Benzo Fast Blue type (sulphanilic acid \rightarrow α -naphthylamine \rightarrow Cleve acid \rightarrow J- or phenyl-J-acid) are examined by Formanek's method. In all examples the introduction of a new sulphonic group produces a bathochromic effect, which is usually smaller as more sulphonic groups are originally present. The effect is greater in *p*-position to the azo group than in *o*-position, and least in *m*-position, but is always small when the group is introduced into the first component. If α -naphthylamine is made the third component and Cleve acid the second, an appreciable bathochromic effect is observed; the position of the entering sulphonic groups often has greater effect than their number. Light-fastness is somewhat increased by the introduction of sulphonic groups; dyes having aniline-*o*-sulphonic or -2:4-disulphonic acid as first component are respectively faster than those derived from metanilic acid or aniline-2:5-disulphonic acid. If the first component is of the benzene series and the others are naphthalene derivatives, the dyes are faster to light than when the first two components are of the benzene series. The use of phenyl-J-acid in place of J-acid has a bathochromic effect, and light-fastness and affinity for cotton are increased; the solubility is diminished. The effect of the introduction of a methyl group may be batho- or hypso-chromic according to position, and is always small (positive in the first component, negative in the second); light-fastness is somewhat increased. Entry of an alkoxyl group, especially into the third component, produces a strong bathochromic effect; the difference between methoxyl and ethoxyl or propoxyl is inappreciable. The alkoxy-group improves the alkali- and acid-fastness of the disazo intermediate, but has little effect on that of the trisazo dye. C. HOLLINS.

Effect on coal-tar colours of lime and zinc white. ARNOLD.—See XIII.

PATENTS.

Manufacture of benzanthrone derivatives. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, A. SHEPHERDSON, and S. THORNLEY (B.P. 298,931, 16.7.27. Addn. to B.P. 276,766; B., 1927, 837).—The process of the prior patent is extended to simple homologues and derivatives, e.g., methyl- and chloro-benzanthrones.

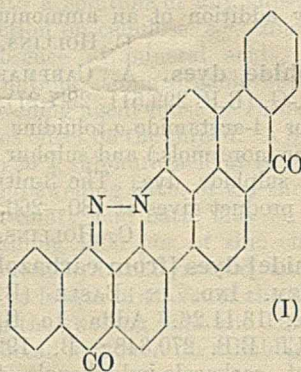
C. HOLLINS.

Manufacture of new vat dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 298,583, 12.5.27).—The products obtained from 3-phenylbenzanthrones and aluminium chloride (B.P. 297,129; B., 1928, 847) are converted by alkaline fusion into bluish-green vat dyes. C. HOLLINS.

New black and grey vat dyes. BRIT. DYESTUFFS CORP., LTD., and S. THORNLEY (B.P. 298,928, 15.7.27. Addn. to B.P. 276,767; B., 1927, 837).—The process of the prior patent is extended to synthetic benzanthrone-sulphonic acids and to homologues and derivatives, e.g., methyl- and chloro-benzanthronesulphonic acids.

C. HOLLINS.

Manufacture of vat dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 298,284, 12.7.27).—2-(3-Benzanthronyl)pyrazolanthrone (cf. B.P. 298,775; B., 1928, 922) is converted by alkaline fusion at 90—100°



into a blue vat dye (I), or at 160° into a greenish-grey to black vat dye (II). Other *N*-substituted pyrazolanthrone of B.P. 298,775 give greenish-blue to violet-blue vat dyes. Dichlorination of (I) with sulphuryl chloride or chlorine yields a violet-blue vat dye; a reddish-blue tetrachloro-compound and a greenish-blue dibromo-compound are also obtainable. Nitration of (I) for a bright green vat dye, becoming grey to black on chlorination, and of (II) for an olive-green (greyer on chlorination), is also described. The compound (I) is readily hydroxylated with sulphuric acid and manganese dioxide to a blue vat dye, which becomes greener and fast to chlorine on methylation. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 298,545, 7.6.27).—Anthraquinone-oxazoles containing as *C*-substituent an anthraquinonyl group are fast vat dyes of good affinity. Those carrying a free amino-group are deepened in shade by treatment with 65% oleum and a trace of sulphur, with or without subsequent methylation. Examples are: oxazole from 2:3-aminohydroxyanthraquinone and 2-aldehydoanthraquinone (yellow); from 2-amino-1-hydroxyanthraquinone and 1-amino-2-aldehydoanthraquinone (bluish-red); from 3-aminoalizarin and 1-amino-2-aldehydoanthraquinone, then methylated (red); from 1-amino-2-hydroxyanthraquinone and 1-aminoanthraquinone-2-carboxylic acid (bluish-red); from 1:3-dibromo-2-aminoanthraquinone and anthraquinone-2-carboxylic acid (yellow); from 2:3-bromoaminoanthraquinone and 1-aminoanthraquinone-2-carboxylic acid (bluish-red); from 2-amino-3-hydroxyanthraquinone and 1-amino-2-aldehydoanthraquinone, treated with oleum (blue) and methylated (violet); from 1:5-diamino-2:6-dihydroxyanthraquinone and 1-amino-2-aldehydoanthraquinone, treated with oleum (dark blue). C. HOLLINS.

Vat dye from dianthraquinonyldiaminophenanthraquinone. W. MIEG and A. JOB, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,327, 11.9.28. Appl., 28.6.26. Ger., 25.6.25).—2:7-Dibromophenanthraquinone is condensed with an α -aminoanthraquinone and the product is converted into a biscarbazole derivative by treatment with aluminium chloride or sulphuric acid. α -Aminoanthraquinone gives a red-brown vat dye, 1-amino-5-benzamidoanthraquinone a yellow-brown, 1-amino-4-benzamidoanthraquinone a red-brown (cf. B.P. 278,771 and 279,003; B., 1928, 9). C. HOLLINS.

Preparation of [sulphide] black colouring materials. R. VIDAL (B.P. 283,468, 9.1.28. Fr., 8.1.27).—2:4-Dinitro- or 2:4:6-trinitro-phenol or a mixture of these is first reduced with aqueous sodium sulphide, nitrosophenol or nitroso-*o*- or -*m*-cresol is added, and after reaction is complete the product is thionated with sodium polysulphide. The sulphide black is preferably precipitated by addition of an ammonium salt. C. HOLLINS.

Manufacture of sulphide dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,511, 28.7.27).—2-Nitroaceto-*p*-toluidide or 4-acetamido-*o*-toluidine is heated with benzidine (2 or more mols.) and sulphur at 200–260° for an orange sulphide dye. The 3-nitro-isomeride or its reduction product gives at 180–260° a greenish-yellow. C. HOLLINS.

Manufacture of [sulphide] dyes [from carbazole-indophenols]. SOC. CHEM. IND. IN BASLE (B.P. 280,595, 14.11.27. Switz., 13.11.26. Addn. to B.P. 199,360; B., 1924, 9. Cf. B.P. 270,348; B., 1927, 869).—The thionation of carbazole-indophenols for blue sulphide vat dyes is assisted by the addition of aliphatic compounds containing nitrogen and carbon in atomic ratio of at least 1:2, *e.g.*, carbamide, dicyanodiamide, semicarbazide, guanidine, thiocarbamide, ammonium thiocyanate, acetamide. Arylamines are not added. C. HOLLINS.

Polyarylamine dye and its manufacture. K. THIESS, B. DEICKE, and R. SCHMIDLIN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,331, 11.9.28. Appl., 10.5.26. Ger., 22.5.25).—1:3-Dichloro-2:6-dinitrobenzene is condensed with an aminodiphenylamine-sulphonic acid (2 mols.) in boiling water in presence of alkali carbonate and a little alcohol to give wool dyes. 4'-Nitro-4-aminodiphenylamine-2'-sulphonic acid gives a yellow, 4-aminodiphenylamine-2-sulphonic acid a brown-yellow (claimed specially), 4-amino-4'-methyl-diphenylamine-2-sulphonic acid an orange-yellow. 2:6-Dichloro-3:5-dinitrotoluene condenses with 4-aminodiphenylamine-2-sulphonic acid to give a brown. C. HOLLINS.

Water-soluble dye and its manufacture. O. SIEBERT, K. THIESS, B. SCHÖNER, R. SCHMIDLIN, W. BENADE, and B. DEICKE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,330, 11.9.28. Appl., 17.6.26. Ger., 5.5.23).—See B.P. 274,999; B., 1927, 772.

Manufacture of sulphurised derivatives of phenols, and of naphthols. E. KRAUS, Assr. to FABR. VAN CHEM. PROD. (U.S.P. 1,690,640—1, 6.11.28. Appl., 16.2.26. Holl., [A] 5.8.25, [B] 24.11.25).—See B.P. 269,970; B., 1927, 469.

Manufacture of vat dyes derived from naphthalene-1:4:5:8-tetracarboxylic acid and aromatic *o*-nitroamines. W. ECKERT, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,775, 6.11.28. Appl., 27.1.27. Ger., 29.1.26).—See B.P. 265,232; B., 1928, 225.

Azo dye and its manufacture. E. HOFFA and E. FISCHER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,275, 11.9.28. Appl., 27.7.26. Ger., 1.8.25).—See B.P. 256,272; B., 1927, 837.

Manufacture of diphenyl- and ditolyl-methane [mordant] azo dyes. C. METTLER, Assr. to J. R. GEIGY A.-G. (U.S.P. 1,689,739 and 1,689,740, 30.10.28. Appl., [A] 14.1.27, [B] 25.11.27. Ger., [A, B] 28.1.26).—See B.P. 265,203; B., 1928, 442.

Dyes (B.P. 299,501). Sulphide dyes (B.P. 299,152).—See III. Lakes (B.P. 299,521).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Influence of temperature on sulphite-cellulose cooking. E. HÄGGLUND (Papier-Fabr., 1928, 26, 689–695).—If the conditions of cooking (circulation in the cooker etc.) are otherwise the same, defibreable cellulose in a yield of 55% of the weight of wood taken, with a 7–8% lignin content, may be obtained equally well at a maximum cooking temperature of 120° as at 135°. At the higher temperature decomposition takes place so rapidly that it is difficult to choose the right time for stopping the cooking, whilst at 120° no such difficulty is experienced, and in this lies the advantage of the lower temperature. For the stage of cooking corresponding with a bromine value of 0.15–0.30, *i.e.*, for a 52–48% yield of cellulose, it is immaterial whether the cooking is done at a maximum temperature of 120° or 135°, since the bromine values and yields of the products are identical; also, for the same degree of mealing, papers of equal strengths are given. For the "quick-cooking" process the yield of cellulose of all qualities is about 3% lower than for the longer processes at 120–135° maximum temperature, and an inferior product is obtained. Raising the cooking temperature 10° roughly doubles the velocity of decomposition, but variation of temperature has little effect on the acidity of the solution or on the sugar and sulphuric acid formation during cooking.

B. P. RIDGE.

Relation between the carbon, hydrogen, and oxygen contents in cotton cellulose under thermal decomposition, and its loss of weight. T. AKAHIRA (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1928, 9, 165–180).—Samples of absorbent cotton were heated for different times up to 13 days at constant or varying temperatures between 200° and 300° in air or nitrogen, under normal or reduced pressures, or in a high vacuum, and the carbon, hydrogen, and oxygen contents of the carbonised residues were found. Straight-line relationships which are independent of the heating conditions obtain between the percentage of each of the three elements and the percentage loss of weight of the original material. The ratio of hydrogen to oxygen in the residues is constant at 2:1 (*i.e.*, it is the same as for water or for the original cellulose molecule), and is

independent of the loss in weight of the material. From the curves obtained it is deduced that when the hydrogen or oxygen content is reduced to zero a certain value for the final carbon residue remains which approximates to 22.2%.
B. P. RIDGE.

Behaviour of lignin and chlorolignin in the preparation of wood pulp by means of chlorine. H. WENZL (Z. angew. Chem., 1928, 41, 1008).—In criticising the work of Waentig (cf. B., 1928, 476) the author describes processes using chlorine gas (Pomilio) and those using chlorine water (De Vains) as being fundamentally different, whereas Waentig regards these as chemically the same, but modified by the differences in temperature obtaining and the resulting effect on hydrochloric acid formation.

A. G. POLLARD.

Action of bromine on cellulose etc. FUCHS.—See II.

PATENTS.

Treatment of wood with steam for the manufacture of paper. R. BLASCHKE (F.P. 624,224, 11.7.27).—Wood is heated for 1–3 hrs. in steam under 5 atm. pressure, allowed to cool slowly for 5–10 hrs., and then treated with the usual chemicals for 3–10 hrs.

A. R. POWELL.

Weighting of silk. R. J. O'BRIEN, JUN., Assr. to COLLWAY LABORATORIES, INC. (U.S.P. 1,684,286, 11.9.28. Appl., 16.12.24).—Silk threads are treated with rubber latex or solution and vulcanised. Tin oxide or zinc oxide may be added.

C. HOLLINS.

Cotton treating. A. COBB (U.S.P. 1,688,524, 23.10.28. Appl., 11.6.26).—Cotton is conditioned by being heated, while it is worked, to above 80° in a substantially dry atmosphere.

H. ROYAL-DAWSON.

Obtaining cellulose from ligno-cellulosic material. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 298,333, 30.8.27).—The comminuted raw material is treated with sufficient calcium hypochlorite solution containing not less than 2% of available chlorine (preferably about 2–8%) to oxidise the whole of the lignin. For fir wood that has been pretreated with, e.g., 0.5% caustic soda solution at 95°, about 50% of chlorine on the weight of wood is required. The temperature should not exceed 45° and the liquor is kept faintly alkaline during the process by the addition of an alkaline-earth hydroxide, preferably milk of lime; at no time, however, should the alkalinity be higher than is represented by the presence of 0.15% of free calcium hydroxide.

D. J. NORMAN.

Separating or extracting cellulose or paper pulp. (Sir) G. C. GODFREY, E. C. BENTHALL, E. S. TARTON, H. F. WHEELER [BIRD & Co.], and E. SPENCER (B.P. 291,064—5, 5. and 14.7.27. India, 28.5.27).—(A) In the semi-continuous, i.e., progressive, digestion of fibrous material such as bamboo, grasses, etc. the digesters are connected by a ring pipe provided with suitable cocks and valves so that the liquor in any one digester may be blown into any other digester in the ring. Preferably four or any larger even number of digesters are connected in a double ring, alternate digesters being connected to alternate rings so that the

digesters in one ring may be operated independently of those in the other ring. Only one ring pipe for steam and another for spent liquor need be provided for all the digesters in the system. (B) In order to avoid loss of cellulose during the final stage of progressive digestion processes, the concentration of active alkali should be highest at one of the intermediate lower-pressure stages, and the activity of digestion in the final stage should be modified by, e.g., reducing the concentration of the liquor, reducing the pressure, or replacing a part or the whole of the caustic soda in the liquor by sodium sulphide or sulphite.

D. J. NORMAN.

Manufacture of artificial [viscose] threads. L. LILIENFELD (B.P. 298,548, 6.5.27. Cf. B.P. 274,521; B., 1927, 745).—Viscose is spun into a solution, other than a strong mineral acid, which has both a coagulating effect and a swelling effect, or the viscose may be coagulated in an ordinary acid bath and then passed into a solution of any substance which has a swelling action on cellulose, e.g., zinc chloride in the presence or absence of mineral acids, cuprammonium hydroxide solution, strong organic bases, etc. The action of the swelling agent must be checked before it has any injurious effect on the thread. A 34% solution of zinc chloride in 20% sulphuric acid at 16° may be used as a coagulating and swelling bath, or the thread, after coagulation in an ordinary acid bath, may be passed into a solution containing 4200 pts. of zinc chloride, 500 pts. of concentrated hydrochloric acid, and 1800 pts. of water at 16°. The length of travel of the thread should be 10–20 cm. in the swelling bath and 120 cm. in air. The coagulated thread is wound on a bobbin the lower part of which is immersed in water.

D. J. NORMAN.

Paper pulp from cane (U.S.P. 1,688,904—5).—See XVII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Steeping and dyeing. R. LORENZ and F. MICHAEL (Z. physikal. Chem., 1928, 137, 142–175).—The tendency of wool and of proteins, such as animal membranes, to form compounds with hydrogen chloride has been determined at varying p_H values. For these substances the necessary conditions exist for the establishment of the Donnan membrane equilibrium, and by application of this theory the degree of swelling produced by the action of acids may be calculated. Indications may also be obtained of the process of adsorption of dyestuff anions, and its variation with the hydrogen-ion concentration of the bath, the presence of electrolytes, and the concentration of the dye. Experimental determinations of the optimum p_H for given dye concentrations are in good agreement with the theoretical values. The action of chromium mordants may be explained as due initially to hydrolysis of the chromium salt at the membrane; artificial loading of natural silk by steeping in stannic chloride solution depends on the same phenomenon.

H. F. GILLBE.

Effect of printing, discharging, and stripping agents on muslin. I. Rongalite groups and Decroline. II. Blankit, hyposulphite, sodium bisulphite, and steaming pressure. III. Ageing test. IV. Destruction of wool by hyposulphite groups.

H. UDAKA (J. Soc. Chem. Ind., Japan, 1928, 31, 374—381, 382—386, 386—388, 458—465).—I. The tensile strength and elongation of muslin decreased as the concentration of Rongalite C with zinc oxide and the time of steaming were increased. Zinc oxide promoted the lowering of the elongation of muslin and seemed to prevent the depression of the tensile strength. Distinct differences were observed between the effect of fresh Rongalite CW and that of a sample 1 year old. In the latter case the tensile strength and elongation were seriously affected. Decroline in acetic acid solution, as distinct from its aqueous solution, did not lower the tensile strength and elongation. Rongalite C and zinc powder had less effect than Rongalite C and zinc oxide.

II. Blankit and zinc oxide, and hyposulphite and zinc oxide both greatly decreased the tensile strength and elongation of muslin, but sodium bisulphite and zinc powder had little effect. These properties of muslin that had been treated with such reagents were also influenced by the steaming pressure used in printing.

III. Ageing slightly damaged muslin treated with the stable hyposulphite groups, but scarcely affected those treated with the unstable hyposulphite groups. The elongation of the treated muslin increased generally by ageing.

IV. The effects on wool of various decomposition products of hyposulphite groups and zinc salts were examined. Thus, zinc sulphate, sodium hyposulphite, and sodium bisulphate had little influence on wool fibre; sodium bisulphite was much more active. The Allwörden reaction was observed when wool fibres were treated with sulphuric acid; it is suggested that this reaction is caused by the hydrolysis of wool protein. Y. TOMODA.

PATENTS.

Dyeing etc. of materials made with or containing cellulose esters or ethers. BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 298,699, 19.7.27).—A suitable arylamine is oxidised on the fibre with *p*-toluenesulphonchloroamide or its sodium salt in presence of acid and/or an oxidation catalyst (e.g., vanadium chloride). C. HOLLINS.

Dyeing of materials made with or containing cellulose derivatives. H. C. OLPIN, ASSR. to CELANESE CORP. OF AMERICA (U.S.P. 1,688,553, 23.10.28. Appl., 11.4.27. U.K., 9.2.27).—See B.P. 285,641; B., 1928, 364.

Dyeing, printing, or stencilling of acetyl cellulose or products made therefrom. G. H. ELLIS and W. O. GOLDTHORPE, ASSRS. to CELANESE CORP. OF AMERICA (U.S.P. 1,690,481, 6.11.28. Appl., 6.8.25. U.K., 14.8.24).—See B.P. 242,711; B., 1926, 50.

Dyeing leather with acid azo dyes. B. VOSSEN, ASSR. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,318, 6.11.28. Appl., 23.1.26. Ger., 3.2.25).—See B.P. 247,187; B., 1927, 329.

Apparatus for wet treatment of textile threads. M. SCHOENFELD (B.P. 298,721, 5.8.27).

[Supports for] fluid treatment of fabrics. BRIT. CELANESE, LTD. (B.P. 295,283, 7.8.28. U.S., 8.8.27).

Apparatus for dyeing of fabrics. C. HACKENBROICH (B.P. 295,569, 7.12.27. U.S., 13.8.27).

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

Gas analysis in an Opl tower plant. W. HANSEN (Chem.-Ztg., 1928, 52, 813—815, 830—832).—For the analysis of exit gases a Riesenfeld meter is preferred to an aspirator. In this the gas is drawn through a capillary and the volume determined by the pressure drop. The maximum volume to be tested is 100—150 litres. The absorption train consists of a glass-wool filter, 2 bottles containing 50 c.c. of 0.1*N*-caustic soda, and 2 or 3 bottles containing 50 c.c. of acidified 0.5*N*-permanganate. No difference could be detected as a result of adding hydrogen peroxide to the caustic soda. Sulphuric acid alone is not a satisfactory absorbent for oxides of nitrogen, but mixed acid containing 5% of concentrated nitric acid gave good results, hydrogen peroxide in this case being added to the alkali absorbent. An examination of Raschig's method, using iodine and sodium acetate as absorbents, gave unsatisfactory results. Even with a gas velocity of 20 litres/hr., iodine was carried forward and the sodium acetate failed to retain nitric oxide. In addition, iodine is liberated from potassium iodide. The method is, however, suitable for tower gases containing sulphur dioxide in excess. It gave good results for the gases leaving tower 2, but not for gases later in the sequence. In such cases absorption in alkali and subsequent analysis of the absorbent is necessary. C. IRWIN.

Toxicity of fluorine compounds. S. MARCOVITCH (Tenn. Agric. Exp. Sta. Bull., 1928, No. 139, 1—48).—For insects and lower organisms, sodium fluosilicate is more toxic than sodium arsenite, possibly on account of their low calcium content; toxicity values are: sodium fluosilicate 34.5, sodium arsenite 13.1, sodium arsenate 4.8, sodium fluoride 4. For man and the higher animals the arsenicals are nine times as toxic as sodium fluosilicate, and 30 times as toxic as sodium fluoride. The protoplasmic poisoning action of fluorine is ascribed to the removal of calcium from the tissues as calcium fluoride. In presence of moisture, calcium hydroxide, when used as a carrier for sodium fluosilicate, may cause foliage injury. Calcium, magnesium, and strontium fluorides are only slightly toxic; of the organic acids, salicylic was the most toxic, followed by oxalic, formic, benzoic, and acetic acids. Of the metals, mercury was the most toxic; barium, lead, zinc, and copper were slightly toxic. The arsenicals and fluorine compounds are much more effective above 39°.

CHEMICAL ABSTRACTS.

Lead sulphate. H. SCHRADER (Farben-Ztg., 1928, 34, 268—269).—Lead sulphate is produced as a by-product of the manufacture of aluminium acetate for waterproofing tarpaulins etc. It may be used as a starting point for chrome yellow, first being converted into carbonate and then treated with potassium dichromate and sulphuric acid; the process has some advantage in the cheapness of lead as sulphate. Other variants of the method are: conversion of lead carbonate into basic nitrate or chloride by treatment with acid and double decomposition with alkali dichromate; decomposition of the lead sulphate with caustic soda or lime and treatment of the basic sulphate with dichrom-

ate; production of basic sulphate by treatment of the normal salt with dilute acetic acid and then proceeding as in the previous method.

C. IRWIN.

PATENTS.

Manufacture of sulphuric acid. W. A. PATRICK and E. B. MILLER, Assrs. to SILICA GEL CORP. (U.S.P. 1,683,694, 11.9.28. Appl., 24.5.24).—For production of an active catalyst a silica gel, capable when dry of absorbing at least 21% of its weight of water vapour at 30° and under 22 mm. partial pressure, is impregnated, while still containing 30–40% of water, with a solution of ammonium chloroplatinate and heated to 360°.

C. HOLLINS.

Manufacture of sulphuric acid. PULVERFABR. SKODA-WETZLER A.-G., and M. FEIGENSOHN (Austr. P. 106,847, 18.1.26).—The gases are passed, on their way between the Glover tower and the lead chamber, through a closed vessel in which they are brought into intimate contact with an oxidising medium, such as nitrous or nitric acid, which retains the selenium and arsenic present in the gases.

A. R. POWELL.

Preparation of purified phosphoric acid. J. MAXWELL, P. C. HOFFMANN, and C. E. MCCOY, Assrs. to VIRGINIA-CAROLINA CHEM. CORP. (U.S.P. 1,688,822, 23.10.28. Appl., 22.12.25).—Impure phosphoric acid is heated to 1200° or over in the presence of an oxidising gas, and the phosphoric acid is recovered from the fumes.

W. J. BOYD.

Manufacture of phosphoric acid and hydrogen. NORSK HYDRO-ELEKTRISK KVAELSTOF-A./S. (F.P. 624,438, 10.11.26. Nor., 23.12.25).—Phosphorus vapour and carbon dioxide are caused to interact at a high temperature, the phosphorus pentoxide formed is condensed and converted into phosphoric acid, and the carbon monoxide is mixed with steam and passed over a catalyst to obtain hydrogen and carbon dioxide for use again in the process.

A. R. POWELL.

Purifying brine to be used in electrolytic processes. J. DRUCKER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,687,433, 9.10.28. Appl., 17.8.27. Ger., 29.9.26).—The brine is saturated with an alkali fluoride, whereby any calcium or magnesium present is separated as fluoride.

H. ROYAL-DAWSON.

Recovery of potassium chloride. N. M. McGRANE, Assr. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,688,873, 23.10.28. Appl., 4.5.27).—Material containing potassium and sodium chlorides and sulphates is leached with an aqueous solvent at a raised temperature until the concentrations of the sodium and sulphate ions have reached, and then decreased considerably below, their maximum values, while the concentrations of the potassium and chlorine ions have continued to increase. The solution is then separated from undissolved solid and cooled to cause deposition principally of potassium chloride crystals.

L. A. COLES.

Manufacture of potassium nitrate. WOLFF & Co., and F. FROWEIN (Austr. P. 106,983, 25.5.26. Ger., 25.5.25).—Nitric acid is neutralised with powdered dolomite and the clarified solution is treated with potassium sulphate. The heat of the reactions is

utilised to obtain hot, highly concentrated solutions from which potassium nitrate crystallises on cooling.

A. R. POWELL.

Production of nitrites. E. T. DRAKE, Assr. to CUDAHY PACKING Co. (U.S.P. 1,685,629, 25.9.28. Appl., 8.2.26).—An aqueous solution of sodium nitrate, sugar, common salt, and amino-nitrogen is inoculated with selected groups of nitrate-reducing and salt-tolerant bacilli.

C. RANKEN.

Recovery of alkali chlorides from minerals. A. J. MACDOUGALL (Can. P. 265,449, 10.11.25).—Lithium and potassium chlorides are recovered from aluminosilicates, such as lepidolite, spodumene, petalite, etc., by heating the powdered mineral mixed with carbon in a current of chlorine.

A. R. POWELL.

Preparation of pure alumina. ZAIDAN HOJIN RIKAGAKU KENKYUJO (B.P. 284,661, 29.7.27. Jap., 3.2.27).—Clay or bauxite is reduced with carbon in an electric furnace and silicon, iron, ferrosilicon, and iron oxides are removed from the resulting impure alumina by treatment with chlorine or hydrogen chloride at 200° or above, with the addition of a reducing agent if iron oxides are present.

W. G. CAREY.

Production of compounds of metal and sulphur. K. BRODOWSKI (B.P. 290,986, 21.5.28. Ger., 21.5.27).—The appropriate metal and sulphur are heated under pressure by the passage of an electric current with exclusion of oxygen, and the heated mass is forced into metal moulds lined with insulating material, e.g., paper.

W. G. CAREY.

Manufacture of hydrogen. G. A. MOURLAQUE (F.P. 624,330, 22.9.26).—Steam is generated by means of an electrical resistance heater arranged inside a porous cylinder, and is then caused to react with a suitable metal for the production of hydrogen.

A. R. POWELL.

Purification of gases. J. Y. JOHNSON, From I. G. FARBENIND. A.-G. (B.P. 298,726, 10.8.27).—Impurities injurious to catalytic reactions are removed from gases by treatment, preferably at an elevated pressure, with reactive metals dissolved or finely distributed in melts of salts, oxides, or hydroxides, or melts of other metals, to which may be added substances inert towards the melts but having a catalytic effect on the purifying reaction. Any metallic vapour may be recovered from the gas by means of active charcoal.

H. S. GARLICK.

Lead carbonate from ores (N.P. 41,124).—See X. **Barium compounds for sugar manufacture** (U.S.P. 1,688,071).—See XVII.

VIII.—GLASS; CERAMICS.

Boric oxide-silica glasses. A. COUSEN and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 169–190).—Clear boric oxide-silica glasses were prepared up to a silica content of 80%, and homogeneous samples were obtained over the range 0–61.4% SiO₂. The thermal expansion of boric oxide and of the mixtures was measured up to the softening point. Continuous diminution of the rate of expansion (which was linear up to the lower critical point) occurred with increase of silica content.

In addition, the lower and upper critical temperatures continuously increased, and the straight portion of the curve in the critical range became progressively shorter. The density of pure, annealed boric oxide was found, as a mean of two methods, to be 1.8442, the values previously given by Briscoe, Robinson, and Stephenson (B., 1926, 219) being too low, principally through lack of annealing. Densities of the silica-boric oxide glasses continuously increased with increase of silica content, the values lying on a smooth curve. A. COUSEN.

Determination of the durability of glass by the powder method. M. THOMAS (J. Soc. Glass Tech., 1928, 12, 87—92 p).—A standard method, chosen by a Committee of the German Society of Glass Technology, is a compromise, due to practical considerations, between the more desirable measurement of deep-seated as opposed to surface durability. By it, 10 g. of glass of grain size 0.30—0.49 mm. are boiled for 5 hrs. with 100 c.c. of water, in a salt-water bath at 100°. A. COUSEN.

Thermal expansion factor of aluminium oxide in glass. I. KITAIGORODSKY and S. RODIN (J. Soc. Glass Tech., 1928, 12, 164—168).—Dilatometric determinations of the thermal expansions of glasses rich in alumina gave results deviating but little from theoretical values calculated from the factors given by English and Turner (B., 1928, 262), and confirmed, in particular, the factor for alumina, *i.e.*, 0.52, as opposed to the value 5.0 previously given by Winkelmann and Schott. A. COUSEN.

Characteristics of refractory clays for the glass industry. G. GEHLHOFF, H. KALSING, K. LITZOW, and M. THOMAS (J. Soc. Glass Tech., 1928, 12, 213—279).—Results are given of the analyses and determination of properties of a number of German and Bohemian clays, some kaolins, natural sillimanite, pure alumina, and Dinas brick. Properties studied were: (1) the proportion of water needed to produce working plasticity, (2) drying and firing shrinkage, (3) bending strength, (4) porosity, (5) fusion temperature, (6) softening under load, (7) thermal resistance, (8) resistance to corrosion by batch materials, molten batch, and glass, both quiescent and in motion. Chemical resistance and stability of the clays at high temperatures increased with increase of alumina content (except to attack by saltcake). They depended also on the firing temperature, and in particular on density of burning and porosity. Grog grains under 2 mm. in diam. did not influence the softening temperature under load, but affected the resistance to chemical attack. Compounds in clays, such as iron oxide and alkalis, in certain cases injuriously affected the properties, irrespective of the alumina content. A. COUSEN.

Expansion and tensile tests on glass-house refractories. J. F. HYSLOP, R. F. PROCTOR, and H. C. BIGGS (J. Soc. Glass Tech., 1928, 12, 190—204).—From expansion tests up to 1400°, silica-alumina refractories were classified into three groups according as they showed (1) rapid and large contractions beginning at, or above, 1000° (characteristic of lightly fired ware), (2) a decreased rate occurring gradually or abruptly above 1100° (hard-burnt clays), and (3) a more or less uniform rate of expansion up to 1400° (hard-burnt

sillimanite and synthetic silica-alumina materials. Tensile tests on pot clays show the good effect of hard burning and the close relationship between the condition of the grog and the resistance to stress at high temperatures. For good and complete burning, aluminous clays are superior to siliceous, whilst the presence of iron oxide lowers the temperature of rupture. A. COUSEN.

PATENTS.

Tunnel kiln, furnace, etc. C. F. GEIGER, Assr. to CARBORUNDUM Co. (U.S.P. 1,686,083, 2.10.28. Appl., 1.2.27).—The kiln is provided with a combustion chamber which is movable along the whole length of the tunnel. A. R. POWELL.

Drying ceramic ware and apparatus therefor. T. H. RHOADS, Assr. to PROCTOR & SCHWARTZ, INC. (U.S.P. 1,689,082, 23.10.28. Appl., 13.3.24).—The articles are passed through a long drying chamber into which currents of air increasing in temperature towards the discharge end are introduced at points along the chamber, and are passed transversely across it and caused to blend with one another so as to maintain a uniform increase in temperature along the chamber. L. A. COLES.

[Porcelain] insulators. S. E. AUSCHER (F.P. 620,987, 30.9.25).—The slip used for making ordinary porcelain, which is fired at 1400°, is mixed with a proportion of finely-ground porcelain which has already been fired at its usual firing temperature. The mixture is cast into shapes, fired, and glazed as usual; the products have great strength and a high dielectric constant. A. R. POWELL.

Manufacture of rubber-bonded abrasive articles. D. E. WEBSTER, Assr. to NORTON Co. (U.S.P. 1,687,410, 9.10.28. Appl., 30.10.25).—A mixture of rubber latex, a vulcanising agent, an accelerator, and abrasive grains is coagulated and vulcanised. H. ROYAL-DAWSON.

Insulators (B.P. 299,408).—See XI.

IX.—BUILDING MATERIALS.

Effects of moisture changes on building materials. R. E. STRADLING (Dept. Sci. Ind. Res. Building Res. Bull. No. 3, 1928, 22 pp.).—The effects of continuous or intermittent wetting of building materials resulting eventually in their disintegration may be due to actual chemical combination in the material, *e.g.*, the hydration of a grain of lime near the surface, or by dissolution of constituents, *e.g.*, of lime by sulphur acids in rain with the production and destructive crystallisation of calcium sulphate. The alternation in the formation of ice crystals during winter is not considered vitally destructive in England, though serious damage has been observed in Sweden from this cause, and it is doubtful whether osmosis can exert sufficient pressure owing to the nature of the materials to cause disruption of a building. It has been shown that repointing old buildings with a strong mortar has sometimes been damaging in effect due to soluble salts being forced afterwards through the brick or stone work, whereas formerly they escaped through the softer mortar joint. The effect of "sorbed" water is unknown, though many

materials possess the property of absorbing water, causing the swelling of a gel formation. With increasing moisture content the stress-strain values of Yorkshire sandstones decreased, though corresponding values for cement concrete were less regular, due probably to the effect of internal strain in the cementing material when drying-out takes place. Some correlation has been obtained between the measurement of the adsorption of water and the adsorption of dyes. C. A. KING.

Thermal constants and strength of concrete of light aggregates. M. HAMADA (J. Fac. Eng. Tokyo Imp. Univ., 1928, 17, 201—212).—Tests on cylinders made from Japanese materials and of the size used for the standard compression test showed that for each kind of aggregates the mixture and age have comparatively little effect on the diffusivity of concrete, though the nature of the aggregate caused a wide difference, concrete composed of common sand and gravel showing the largest value. Soft "Kokaseki," a kind of liparitic pumice, and lapilli gave a diffusivity value of only about 50% of that of ordinary concrete; hard Kokaseki and cinders about 60%; and broken brick 65%. The apparent sp. gr. increased with the relative richness of cement in the mixture; soft Kokaseki had the lowest value of all aggregates. Little variation (0.2—0.3) was observed in the sp. heats of the specimens, but the lowest value for conductivity (0.0012) was obtained with Kokaseki concrete. All the light concretes showed very low strength, the decrease in strength being greater than the decrease in weight. C. A. KING.

Thermal conductivities of walls, concretes, and plasters. E. GRIFFITHS (Dept. Sci. Ind. Res. Building Res. Tech. Paper No. 6, 1928, 19 pp.).—A presentation of the methods used for determining the heat-transmission of materials commonly used for building purposes. C. A. KING.

PATENTS.

Production of porous concrete etc. AEROCRETE (FOREIGN), LTD., and C. NICOL (B.P. 299,484, 28.4.27).—Material for the production of porous concrete comprises Portland cement etc., zinc or aluminium powder, and liquid or solid grease, e.g., stearin, which is preferably mixed previously with the metal powder in the absence of air. L. A. COLES.

[Cement for] grinding stones or wheels. BIRMINGHAM SMALL ARMS CO., LTD., A. R. PAGE, and P. S. DEVEREUX (B.P. 299,633, 16.12.27).—A cement which sets without heating for filling spaces between the segments of abrasive material comprising grinding stones is made from abrasive grit 100 pts. by wt., calcined magnesium oxide 15 pts., and a saturated solution of magnesium chloride 35 pts. W. G. CAREY.

Material for damping sound and other waves. M. HAHN and K. B. EISENBERG (B.P. 299,543, 9.8.27).—Acoustic, mechanical, electrical, and thermal waves are damped by applying to walls etc. a coating comprising a non-drying oil, e.g., mineral oil, and powdered filling material, e.g., loam or clay. L. A. COLES.

Production of calcium aluminate cement. E. M. ROCHE, Assr. to U. B. VOISIN (U.S.P. 1,689,891, 30.10.28.

Appl., 4.12.24. Fr., 6.5.24).—See B.P. 233,698; B., 1925, 960.

Bituminous supports (B.P. 299,208).—See II.

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

Relation between the quantity and depth of carburisation [of iron]. G. TAKAHASHI (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 1135—1156).—The relation between the amount of carbon which diffuses into iron and the depth of the diffusion zone has been determined for various carburising reagents at temperatures between 880° and 1000°. In all cases the weight increment-depth of penetration graphs are parabolas for any given temperature. The more efficient the carburising agent the greater is the amount of carbon absorbed for a given depth of carburisation. Wood charcoal is a more efficient carburising agent than any other form of solid carbon, but its efficiency is greatly reduced by prolonged heating at 900—1000° prior to use. On the other hand its efficiency is greatly increased by admixture with alkali or alkaline-earth carbonates. With these mixtures the quantity of carbon absorbed for a given depth of penetration is the greater the higher is the temperature; with carbon monoxide or a mixture of hydrogen and ethylene or acetylene, however, the quantity of carbon absorbed at 900—1000° is the greater the lower is the temperature or the longer the time. When the carburising temperature is between 900° and the A1 point, the percentage of carbon in the innermost layer is the greater the lower is the carburising temperature. A. R. POWELL.

Corrosion of iron. H. ENDÔ (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 1111—1134).—The loss in weight of Armeo iron in distilled water is a linear function of the time, and is greater in sunlight than in the dark; the rate increases with rise of temperature to 60—70°, then decreases owing to the smaller solubility of oxygen in water at high temperatures. In nitric acid solutions the rate of dissolution increases with rise of concentration up to 30%, then falls almost to zero in 50% nitric acid; in hydrochloric acid solution the rate of dissolution increases very rapidly when the concentration exceeds about 15%, whereas in sulphuric acid solution the rate of dissolution is almost a linear function of the concentration. The rate of corrosion of iron in various salt solutions has been determined. Oxidising salts inhibit the rusting of iron when their concentration exceeds a certain limiting value; for potassium chromate and dichromate this concentration is 0.0001M, for potassium chlorate 0.01—0.003M, and for potassium permanganate 0.002—0.001M. For most other salt solutions corrosion increases rapidly to a maximum with increasing concentration, then falls much more slowly. Addition of potassium chromate to chloride solutions greatly retards their corrosive action on iron, whereas the dichromate tends to increase the action owing to the liberation of hydrochloric acid. A. R. POWELL.

Protection of steel against oxidation at the end of the open-hearth process, and decrease in the amount of gas occluded. V. N. TSVIBEL (Tekh.

Proizvodstvo, 1927, 36—49).—It is proposed to protect the slag from the action of the oxidising atmosphere by the use of a layer of heavy hydrocarbons.

CHEMICAL ABSTRACTS.

Properties of quenched steels. K. TAMARU (Bull. Inst. Phys. Chem. Res., Japan, 1928, 7, 1028—1034).—The magnitude of the A0 transformation was found to be large when the separated cementite was fine, and to become smaller as it grows. Pearlite cementite showed the largest transformation, and globular cementite the least. Two magnetic maxima were observed, at 120° and at 300—350°, when quenched high-carbon steel was tempered at gradually increasing temperatures, and both were considered to be due to the decomposition of β -martensite and the growth of minute cementite particles. Quenched steel contains α - and β -martensites. The former is more expanded than the latter, and is transformed into it at 100—150°; a quenched steel therefore contracts up to 200° and has a density greater than the original but smaller than annealed steels. Above this temperature the quenched steel expands to 260°, due to the decomposition of austenite into α -martensite and then into β -martensite, and above this temperature it contracts, due to the decomposition of β -martensite into troostite. F. S. HAWKINS.

Rapid determination of alloying constituents in iron by spectrographic analysis. G. SCHEIBE and A. NEUHÄUSSER (Z. angew. Chem., 1928, 41, 1218—1222).—The method of determining the percentage of manganese and silicon in iron by measuring the relative intensities of two neighbouring spectral lines, one due to iron and one to the alloying element, is described, and curves are given showing the variations in the intensity ratios Si/Fe and Mn/Fe for several pairs of lines when alloys of varying silicon and manganese content are examined. The capacity and self-inductance of the spark circuit must be standardised by comparing the intensities of the two tin lines, 3331 Å. and 3352 Å., and standardised photographic plates must be used throughout the tests. Once a series of curves is obtained for several pairs of lines by spectrographing standard alloys, the determination of silicon or manganese in iron may be made in 15—20 min. with an accuracy of $\pm 0.1\%$.

A. R. POWELL.

Influence of cobalt on determination of manganese in steel. I. WADA and S. SAITO (Bull. Inst. Phys. Chem. Res., Japan, 1928, 7, 1002—1027).—Cobaltous nitrate is oxidised by sodium bismuthate in the presence of nitric acid, and in the presence of ferric nitrate permanganates are reduced. Hence the manganese in a sample of steel cannot be determined by dissolution in nitric acid, oxidation to permanganate by sodium bismuthate, and titration with ferrous ammonium sulphate if cobalt be present. In the absence of iron, manganese can be determined by this method with cobalt present. F. S. HAWKINS.

The hydrogen-antimony-tin method for the determination of oxygen in cast iron. B. KJERMAN and L. JORDAN (U.S. Bur. Stand. Res. Paper No. 25, 1928, 20 pp.).—The method of Oberhoffer and others (cf. B., 1925, 993) has been examined, and it is shown that for precisions of the order of 0.006% of oxygen the results compare favourably with those given by the

vacuum-fusion method (cf. Jordan and Eckman, B., 1926, 94), but, on account of its greater precision, the latter method is preferred for cast iron containing 0.01—0.04% O. The method is applicable to alloys of high silicon content provided that the content of carbon is also high. In both methods lump rather than milled samples should be used, as adsorption of air and moisture and surface oxidation lead to high results with the latter. M. E. NOTTAGE.

Rapid analysis of bronze and brass without electrolysis. H. KRUG (Chem.-Ztg., 1928, 52, 842).—The alloy (5 g.) is dissolved in 40 c.c. of nitric acid (*d* 1.4), 200 c.c. of boiling water and 3 g. of ammonium nitrate are added, and the solution is filtered into a 500 c.c. graduated flask, the precipitate of metastannic acid being washed, ignited to stannic oxide, and weighed. After dilution to the mark, 100 c.c. of the filtrate are treated with sulphuric acid for the determination of lead as sulphate, a second portion of 100 c.c. is neutralised with ammonia and the manganese precipitated as dioxide by boiling with ammonium persulphate, and a third portion is treated with ammonia, the washed precipitate dissolved in sulphuric acid, the solution passed through a Jones reductor filled with cadmium filings, and the ferrous sulphate formed titrated with permanganate. Copper may be determined in the filtrate from the lead sulphate or, if the lead and iron content of the metal is low, 25 c.c. of the original solution are neutralised with ammonia, the copper and iron precipitated by addition of disodium hydrogen phosphate, and the copper is redissolved in 10 c.c. of 50% acetic acid and determined iodometrically. Zinc is determined as pyrophosphate in a further 50 c.c. of the original solution after removal of copper and lead by hydrogen sulphide and of manganese and iron by ammonium persulphate and ammonia. A. R. POWELL.

Thermal expansion of magnesium and some of its alloys. P. HIDNERT and W. T. SWEENEY (U.S. Bur. Stand. Res. Paper No. 29, 1928, 22 pp.).—Data for the linear thermal expansion of cast and extruded magnesium between -183° and 500° , for cast and extruded magnesium-aluminium alloys containing up to 10.4% Al, and for magnesium-aluminium-manganese alloys containing up to 4.1% Al and 0.9% Mn, have been obtained, three types of apparatus being used. The following average equation represents the expansion of magnesium between 20° and 500° : $L_t = L_0 [1 + (24.80t + 0.00961t^2) \times 10^{-6}]$. The coefficients of expansion for the extruded materials are slightly less than for cast materials. Practically no change in the coefficient is caused by the addition of up to 10% of aluminium to magnesium, but additions of 0.9% of manganese causes a slight increase and of 0.3% a slight decrease. M. E. NOTTAGE.

Function of oxygen in the dissolution of metals and minerals by cyanide solutions. R. HAY (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 23—28).—Experiments on the rate of dissolution of gold, silver, copper, and lead in dilute cyanide solutions indicate that the primary action is associated with the formation of a film of hydrogen on the surface of the metal, and this film retards further action unless it is removed by means of a depolariser such as atmospheric oxygen or by contact

with another metal. Thus copper does not dissolve in un-aerated cyanide solutions except when in contact with lead; dissolution then proceeds rapidly with evolution of hydrogen. In the precipitation of gold from complex cyanide solutions nascent hydrogen is the active agent, and this is formed by the action of the zinc on the free cyanide in the solution. This action is accelerated by the use of a zinc-lead or zinc-copper couple or by using zinc with ragged edges so as to reduce the overpotential of the hydrogen. A. R. POWELL.

Electrodeposition of chromium. J. G. ROBERTS (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 19—22).—A review of recent work. A. R. POWELL.

Mineral separation in a finely-divided state. R. C. EMMONS (Amer. J. Sci., 1928, 16, 423—445).—Experiments are described on the separation of minerals by means of the radiometer effect. The finely-powdered mineral was allowed to fall through a beam of light in a closed vessel containing gas at a low pressure. C. W. GIBBY.

PATENTS.

Operating shaft furnaces, particularly blast furnaces. F. KRUPP A.-G. FRIEDRICH-ALFRED-HÜTTE (B.P. 275,601, 25.7.27. Ger., 6.8.26).—When using a portion of waste furnace gases in operating a blast furnace, the blast is blown at a higher temperature to compensate for the loss of heat at the hearth or other particular zone of the furnace, whilst the coke charge is correspondingly reduced. The thermal and chemical equilibria may be compensated also by supplying oxygen or by the introduction of coal dust close to the tuyères. C. A. KING.

Operation of open-hearth furnaces. F. B. MCKUNE, Assr. to OPEN-HEARTH COMBUSTION Co. (U.S.P. 1,687,682, 16.10.28. Appl., 5.5.22).—A mixture of air and fuel is fed into a furnace chamber through a relatively constricted inlet, and additional air is supplied through another inlet; the products of combustion are delivered through these inlets and additional areas, these together forming an outlet of greater area than the combined area of the inlets. J. S. G. THOMAS.

[Annealing] furnace. A. N. OTIS and L. F. WOOLSTON, Assrs. to GEN. ELECTRIC Co. (U.S.P. 1,688,745, 23.10.28. Appl., 12.3.27).—A preheating chamber attached to the main furnace contains a vertically movable support to receive the cold charge for preheating and the heated charge for cooling. L. A. COLES.

Bright-annealing furnaces. SIEMENS-SCHUCKERTWERKE A.-G., Asses. of SIEMENS-SCHUCKERTWERKE GES. M.B.H. (B.P. 282,079, 8.12.27. Ger., 9.12.26).—The protective gas supplied to a bright-annealing furnace is preheated by being passed between the arched cover of the furnace and an internal "false" cover, and then through openings in the furnace walls. The gas supply pipe may be heated electrically. C. A. KING.

Electric [arc] furnace [for steel]. A. E. GREENE (U.S.P. 1,687,936, 16.10.28. Appl., 18.5.25).—Silica sand is added to a bath of molten steel covered with slag under an electrode on top of the slag, and a carbonaceous reducing agent is added on top of the sand. The

mixture is heated by the arc until the silica is reduced and the steel is deoxidised and finished. J. S. G. THOMAS.

Refining iron and steel. C. W. HILL (U.S.P. 1,686,087, 2.10.28. Appl., 30.11.25).—The metal is refined by means of a current of suitable gas passed into the molten metal under such conditions of temperature and pressure that the amount adsorbed by the metal may be controlled at will. A. R. POWELL.

Making malleable-iron castings. H. A. SCHWARTZ, Assr. to NAT. MALLEABLE & STEEL CASTINGS Co. (U.S.P. 1,688,438, 23.10.28. Appl., 23.12.26).—A casting of usual composition made in a sand mould is cooled rapidly from above the critical point, then heated above the critical point until cementite is broken down, and cooled. C. A. KING.

Hard solder for grey pig iron, cast steel, iron, etc. METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 285,485, 13.2.28. Ger., 18.2.27).—The solder consists of 42—60% Cu, 1—10% Ni, and up to 3% Si, the remainder being zinc. H. ROYAL-DAWSON.

Carburisation of ferrous metals. BRIT. THOMSON-HOUSTON Co., LTD., Asses. of G. W. HEGEL and G. R. BROPHY (B.P. 276,675, 25.8.27. U.S., 26.8.26).—The metal is heated in an atmosphere produced by the vaporisation of a mixture of 4 pts. of bone oil, 4 pts. of kerosene, and 1 pt. of nitrobenzene, the last-named compound acting as an energetic accelerator of carburisation. A. R. POWELL.

Rust-proofing [of iron and steel]. PARKER RUST PROOF Co., Asses. of M. GREEN and H. H. WILLARD (B.P. 270,679, 25.4.27. U.S., 10.5.26).—Iron or steel articles are rendered non-rusting by immersion in a hot solution of phosphoric acid containing ferrous and manganous dihydrogen phosphate in such proportions that the iron content is 2—3 times the manganese content of the solution. A. R. POWELL.

Apparatus for reducing oxides of metals [copper, lead, or zinc]. J. W. HORNSEY (B.P. 277,325, 5.9.27. U.S., 8.9.26).—The apparatus comprises a vertical shaft connected at its upper end with a stack and at its lower end with a dust-collecting device. A rotating, preheating cylinder opens into the lower half of the shaft and is slightly inclined downwards away from the shaft towards a second rotating cylinder, which acts as the reducing chamber, and which is connected to the preheating chamber through a stationary structure in which the two cylinders rotate on gas-sealed bearings. The hot gases from the reducing chamber are ignited in their passage into the preheating chamber so as to preheat the materials (finely-divided coal and ore) therein. A water-cooled screw conveyor serves to assist the passage of the charge through the apparatus and to keep it thoroughly mixed. A. R. POWELL.

Reduction of zinciferous materials. L. MELLERSH-JACKSON. From NEW JERSEY ZINC Co. (B.P. 298,921—4, 8.7.27).—(A) The reduction is carried out in a continuously operating vertical retort into which is charged a mixture of sintered lumps of zinciferous material and briquettes made by coking a mixture of bituminous coal and roasted zinc ore. The charge is made of such porosity

that the heat applied to the outer walls of the retort is conducted throughout the mass by the convection of currents of gas heated at the walls. (b) The retort is made of a non-scaling metal such as nickel-chromium, nickel-iron, chromium-iron, or iron-nickel-chromium alloy, and the temperature of the outer walls is maintained at about 1150°. Provision is made for the expansion and contraction of the retort and for maintaining a reducing atmosphere therein. (c) The retort is lined with refractory bricks with a carborundum base fitted together with butt joints cemented with a thin layer of refractory material. (d) The condenser is connected at its upper end with a stack for the removal of waste gases, the position and height of the stack being so arranged as to give sufficient draught through the retort to prevent the gaseous products of the reaction from escaping through the discharge opening at the lower end of the retort while the residues from the distillation are being removed from the retort. A. R. POWELL.

Reduction of zinc ores. FASTIGHETS AKTIEBOLAGET ØRESUND (N.P. 40,950, 11.3.21).—The mixture of ore and reducing agent is preheated in an electrically heated rotating furnace prior to treatment in the reducing furnace. A. R. POWELL.

Continuous manufacture of lead carbonate or of mixtures of lead carbonate and zinc oxide directly from ores. TROLLHÄTTANS ELEKTROTHERMISKA AKTIEBOLAG (N.P. 41,124, 28.7.22).—The finely-divided ore is reduced in an electric furnace at a sufficiently high temperature to cause volatilisation of the lead and zinc, and the metal vapours are treated with a mixture of oxygen and carbon dioxide. A. R. POWELL.

Treatment of cupriferos molybdenite. NORSKE MOLYBDAENPRODUKTER A./S. (N.P. 41,615, 31.3.24).—The ore is subjected to a flotation treatment and the concentrates are run over a rapidly vibrating shaking screen under a spray of high-pressure water, whereby the copper pyrite is washed through the screen, leaving the molybdenite flakes on the surface. A. R. POWELL.

Purification of metals [copper]. H. H. ALEXANDER (U.S.P. 1,687,277, 9.10.28. Appl., 28.10.25).—The copper is melted by contact with combustion gases the composition of which is continuously regulated by controlling the air and fuel supplies, in accordance with the impurities present in the copper. F. G. CLARKE.

[Silicon]-copper alloys. METALBANK U. METALLURGISCHE GES. A.-G. (B.P. 288,974, 5.4.28. Ger., 16.4.27).—Silicon-copper alloys containing 0.5–10% of one or more of the metals nickel, cobalt, manganese, iron, or chromium, together with 0.5–10% of lead, cadmium, or thallium, or mixtures of these are claimed. A. R. POWELL.

Copper-silicon-manganese alloys. AMER. BRASS Co. (G.P. 450,278, 11.10.24).—Acid-resisting copper alloys contain 92.3–96.5% Cu, 3–6.5% Si, and 0.5–1.2% Mn. A. R. POWELL.

Manufacture of white alloys. A. L. POCOCK (B.P. 299,493, 25.7.27).—An alloy of zinc with 5–15%

of an alloy of 2 pts. of copper, 4 pts. of aluminium, and 1 pt. of silver is claimed. A. R. POWELL.

Production of metal alloys [for tools etc.]. W. SCHROBSDORFF (B.P. 274,866, 19.7.27. Ger., 21.7.26).—A hard alloy for the manufacture of cutting tools comprises a predominating proportion of tantalum, or niobium, or both, a carbide of chromium, molybdenum, tungsten, or uranium, and one or more of the elements cobalt, nickel, copper, palladium, osmium, or iridium. For example, a mixture of 78.2% of tantalum or niobium, 20% of tungsten carbide (W_2C), and 1.8% of cobalt is melted in a vacuum, high-frequency, induction furnace, and the alloy cast into moulds of suitable shape. [Stat. ref.] A. R. POWELL.

Fusible electrodes for use in electric arc-welding. QUASI-ARC Co., LTD., and A. P. STROHMENGER (B.P. 299,575, 15.9.27).—The metallic core is wrapped with matted blue asbestos or similar fibrous mineral silicate containing, if desired, a powder paste or loading consisting of finely-divided iron silicate, ferrosilicon, etc. to form a flux, and the wrapping is cut with helical grooves which are filled in with a cementing material. J. S. G. THOMAS.

Manufacture of electrodes, welding rods, etc. for use in welding and depositing metal. B. TURNER, and FERRO-ARC WELDING Co., LTD. (B.P. 299,163, 24.9.27).—One or both wires to form the twin electrode are coated with a thin film of an adhesive solution of high insulating power, e.g., sodium silicate (cf. B.P. 295,514; B., 1928, 760). J. S. G. THOMAS.

Electrodes etc. for the fusion deposition [arc-welding] of alloys. B. TURNER, and FERRO-ARC WELDING Co., LTD. (B.P. 299,604, 19.10.27).—A welding rod for depositing a nickel-iron or nickel-chromium-iron alloy comprises an iron or steel rod wound with a nickel or nickel-chromium wire spiral, the spaces between the windings of which are packed with a mixture of 200 pts. of powdered chromium, 2.5 pts. of aluminium powder, 35 pts. of fluorspar, 15 pts. of limestone, and 6 pts. of sodium silicate. The whole is coated with a lime-soda-fluorspar flux. The weights of nickel and chromium in the spiral and coating paste are preferably so regulated that the deposited iron alloy has the same composition as "Staybrite" steel. A. R. POWELL.

Manufacture of electrical conductors in light aluminium alloys. COMP. DE PROD. CHIM. ET ELECTRO-MÉTALLURGIQUES ALAIS, FROGES & CAMARGUE (B.P. 286,264, 18.2.28. Fr., 2.3.27).—Age-hardening aluminium alloys are quenched from above 500°, reheated at 125–250° (preferably 150–175°), drawn down to the desired thickness, and re-annealed at 125°–250°. The wires have then a high tensile strength combined with maximum electrical conductivity, and are suitable for the manufacture of electrical conductors without a steel core. A. R. POWELL.

Regeneration of chromium-plating baths. E. LIEBREICH (F.P. 627,445, 5.1.27. Ger., 6.1.26).—Chromium-plating baths containing chromic acid, chromates, or more or less reduced hydroxides or oxides of chromium are treated with chromic hydroxide and an alkali compound, preferably an alkali hydroxide. A. R. POWELL.

Electrolytic extraction of aluminium. H. WADE. From ALUMINIUM-IND. A.-G. (B.P. 299,689, 25.10.27. Cf. B.P. 280,881; B., 1928, 644).—To assist the dense deposition of aluminium, small quantities (0.1—0.5%) of manganese, lead, cadmium, nickel, or their compounds or salts are added to the electrolytic bath previously described (*loc. cit.*). Alternatively, these metals may be alloyed with the anode or they may be used as separate auxiliary anodes. M. E. NOTTAGE.

Direct reduction of iron ores. T. LEVOZ, Assr. to N. V. HANDELSMAATSCHAPPIJ FERIRON (U.S.P. 1,689,734, 30.10.28. Appl., 16.2.24. Belg., 1.2.23).—See B.P. 210,795; B., 1925, 765.

Metallurgical product [cast iron]. E. SCHÜZ, Assr. to MEIER & WEICHEL (U.S.P. 1,689,456, 30.10.28. Appl., 3.11.25. Ger., 13.5.24).—See B.P. 234,106; B., 1926, 952.

Spraying and atomising fusible [metallic] material. "METALLISATOR BERLIN A.-G.," and E. GREISER (B.P. 298,858, 27.3.28. Addn. to B.P. 296,546).

Flotation of ores (B.P. 298,736).—See II. **Metal coating for electrodes** (F.P. 620,029).—See XI. **Lead coating of articles** (B.P. 276,643).—See XIII.

XI.—ELECTROTECHNICS.

Separation of emulsions using high-tension alternating currents. S. PAVLIKOVSKI (Przemysl Chem., 1928, 12, 501—525).—The separation of emulsions of sulphuric acid in mineral oil is most efficient when maximum deformation of the electric field is produced. Coagulation of the disperse phase takes place along the lines of force of the field. A certain optimal *P.D.* exists for each apparatus, above which secondary emulsification occurs. R. TRUSZKOWSKI.

Used transformer and switch oils. TYPKE.—See II. **Electrodeposition of chromium.** ROBERTS.—See X.

PATENTS.

Electric furnace. R. B. LINCOLN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,687,676, 16.10.28. Appl., 16.2.27).—Heating units are arranged along one of the walls of the furnace chamber, and a slot is provided in an adjacent wall to permit the successive removal of the units, the effective length of the slot being less than the combined widths of two of the units arranged below it. J. S. G. THOMAS.

Electric furnace for effecting catalytic gas reactions. G. A. DARZENS (F.P. 625,893, 19.3.26).—The gases or vapours are passed through a vessel packed with small Raschig rings or with a granulated mass which is heated by means of a variable electromagnetic field of high or low tension. The process is suitable for the conversion of alcohol into ethylene. A. R. POWELL.

[Electric induction] furnace lining. J. R. WYATT, Assr. to AJAX METAL CO. (U.S.P. 1,688,220, 16.10.28. Appl., 1.11.23).—Heat-insulating material is rammed in the furnace casing, and an inner lining adapted to hold the molten charge and of low heat-insulating character rests upon the insulator. J. S. G. THOMAS.

Heating liquids by electrical energy. G. BAUM, Assr. to NIAGARA ELECTRO CHEMICAL CO., INC. (U.S.P. 1,688,679—1,688,680, 23.10.28. Appl., [A] 15.10.25, [B] 19.10.26. Austr., [A, B] 28.10.24).—(A) Electric current is passed through an electrically conducting liquid flowing in and not completely filling channels in an electrically non-conducting structure. (B) The orifice of the duct supplying liquid is arranged above non-conducting structures in which small interstitial spaces are arranged between electric current supply mains and exit lines. J. S. G. THOMAS.

Treating liquids with ultra-violet rays. H. SCHOLL (B.P. 283,472, 9.1.28. Ger., 7.1.27).—To prevent the formation of ozone during the irradiation process, air is removed from the liquid, before treatment, by means of a stream of gas, with which the liquid may, if desired, be saturated, the gas becoming chemically active during irradiation. J. S. G. THOMAS.

Gas-filled discharge tubes. N. V. PHILIPS' GLOEILAMPENFABR., Assees. of [A] W. DE GROOT, [B] G. L. HERTZ (Can. P. [A] 265,903 and [B] 266,112, 4.2.25).—(A) The tubes are filled with one or more of the noble gases and with mercury or cadmium vapour; the glass used contains a fluorescent constituent, preferably a uranium compound, and is of a light-diffusing nature. (B) The gas filling consists of argon, krypton, or xenon or of mixtures of these, together with mercury or cadmium vapour. The pressure in the tubes is maintained at 4 mm. A. R. POWELL.

Incandescence cathode for electron-discharge tubes. S. LOEWE (B.P. 274,509, 18.7.27. Ger., 19.7.26).—A core of iron or containing iron is covered with platinum or platinum alloy and coated with metallic oxides or other electron-emitting substances. J. S. G. THOMAS.

Modifying the colour of light in electric discharge tubes. G. CLAUDE and J. M. E. DE BEAUFORT (B.P. 279,053, 21.9.27. Fr., 18.10.26).—Means are provided for readily changing the frequency of the current supply to a discharge tube containing a mixture of gases and metallic vapours. J. S. G. THOMAS.

Vacuum tube. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 298,963, 16.6.27).—In a vacuum tube built at least partly of quartz a series of glass members having successively increasing coefficients of expansion, *e.g.*, containing aluminium oxide or calcium oxide and no alkali oxide, is sealed to the quartz, for hermetically sealing in the leads. J. S. G. THOMAS.

Neon tube. R. R. MACHLETT, Assr. to RAINBOW LIGHT, INC. (U.S.P. 1,689,146, 23.10.28. Appl., 20.10.26).—Electrodes having an area less than 1 dm.² per ampère of current used are arranged in a tube containing a conducting, monatomic, inert gas, and a mirror of an electropositive metal is deposited on the wall of the tube near the cathode. J. S. G. THOMAS.

Galvanic gas cells. R. VON DALLWITZ-WEGNER (G.P. 450,341, 12.6.24).—The pressure in the gas producer is automatically regulated by means of liquid columns so that the cell is not supplied with more gas than corresponds with the current taken from it, and

at the same time not more gas than the cell can consume is produced in the generator. With hydrochloric acid as electrolyte the cell has an *E.M.F.* about equal to that of the lead accumulator. A. R. POWELL.

Production of insulating and sealing material for electric cells and batteries. E. C. R. MARKS. FROM BATTERIEN- & ELEMENTE-FABR. SYSTEM ZEILER A.-G. (B.P. 299,381, 22.7.27).—Unsaponifiable substances, *e.g.*, paraffin, distillation pitch, are mixed with wax, montan wax, resin, etc., and incompletely saponified with basic substances, *e.g.*, soda lye. After completion of the reaction the material is heated to remove water, and fillers are added. J. S. G. THOMAS.

Metal coating on carbon electrodes for dry batteries. SOC. ANON. HEWITTIC (F.P. 620,029, 9.8.26).—Gas-carbon electrodes are coated by the Schoop spraying process with a layer of nickel-steel containing 40% Ni. The coatings adhere tightly as the coefficient of expansion of the steel is approximately the same as that of the carbon. A. R. POWELL.

Porcelain-like electric insulating substance. J. L. A. HILBRENNER (B.P. 299,408, 26.7.27).—A mixture of clay, kaolin, zirconium oxide, and aluminium oxide, to which, if desired, zinc oxide or a frit composed of magnesium oxide, zinc oxide, and aluminium oxide may be added, is ground in the wet state and quickly sintered. J. S. G. THOMAS.

Galvanic element with carbon and iron electrodes. A. J. B. MAYET (G.P. 450,338, 17.8.26. Fr., 28.7.25).—The electrolyte of a cell with iron and carbon electrodes comprises a solution of hydrochloric acid, ferric chloride, alkali chlorate, and alkali nitrate in such concentrations that a protective film of a basic iron chloride is formed on the negative electrode. The cell has an *E.M.F.* of 1 volt and a low internal resistance. A. R. POWELL.

Contact [current] rectifying device. W. O. SNELLING (U.S.P. 1,686,260, 2.10.28. Appl., 29.7.27).—A finely-divided compound of a metal with an element of the sulphur group is compressed into a coherent mass which is then heated at a temperature sufficient to cause crystal growth without sintering. A. R. POWELL.

Electric coil construction. M. LATOUR, ASSR. to LATOUR CORP. (U.S.P. 1,687,298, 9.10.28. Appl., 17.5.23. Fr., 14.6.22).—The material forming the magnetic circuit of an electrical device is composed of 33.9% Ni, 1.15% Mn, and iron. H. ROYAL-DAWSON.

Incandescence-cathode discharge tube. S. G. S. DICKER. FROM N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 299,098, 22.7.27).

Montan wax (B.P., 299,133).—See II. **Electrolytic brine** (U.S.P. 1,687,433). **Aluminium sulphate** (F.P. 627,334). **Compounds of metal and sulphur** (B.P. 290,986).—See VII. **Porcelain insulators** (F.P. 620,987).—See VIII. **Electric furnace for steel** (U.S.P. 1,687,936). **Electrodes etc. for welding** (B.P. 299,163, 299,575, and 299,604). **Conductors in aluminium alloys** (B.P. 286,264). **Chromium-plating baths** (F.P. 627,445). **Aluminium** (B.P. 299,689).—See X.

XII.—FATS; OILS; WAXES.

Tung oil. IV. Wood oils as crystalloids; "autoxy-polymerisation" during drying of wood oils. A. EIBNER and E. ROSSMANN (Chem. Umschau, 1928, 35, 241—250; cf. B., 1928, 825).—The wood oils are fundamentally distinct from other amorphous fatty oils, in that the former consist principally of, or can form, crystallisable glycerides; consequently the earlier colloid theories cannot be applied to these oils. The velocity of isomerisation of α - to β -elæostearin in tung oil under the influence of light has been studied. Working with thin films of tung oil on glass slides, exposure to sunlight or the light from a mercury-vapour lamp (10 min.) causes rapid formation of crystalline β -elæostearin; by maintaining the film at 0° precipitation occurs and the crystals can be observed under the microscope by polarised light. At room temperature the β -isomeride is soluble in the bulk of the oil to a considerable extent. Isomerisation did not occur in the absence of light, but tung oil, nevertheless, dried to a clear film when kept in the dark for 3—4 weeks. (Linseed oil was still sticky after 6 weeks.) In the filming (change to amorphous condition) of pure β -elæostearic acid (m.p. 61°) it was established that oxygen is essential to the process, and that this autoxidation is favoured by light and increase of temperature. An examination of the "autoxypolymerisation" of β -elæostearin during film-formation shows that the older theory—that drying consisted of a pure polymerisation process catalysed by the presence of oxygen—is invalidated. On exposure of β -elæostearin to air and light appreciable quantities (7—10%) of oxygen are absorbed during conversion into the amorphous condition; further, examination of the fatty acids recovered from the product indicated that considerable oxidation had occurred. True polymerisation may also take place, and it is submitted that a mixed process ("autoxypolymerisation") proceeds. Thus, if a tung oil containing β -elæostearin be exposed in a thin film, oxygen is primarily absorbed by the dissolved β -glyceride in the surface layers; the autoxypolymerisation products are at first colloiddally dispersed, and then, by rapid transition to the gel condition, constitute a first disperse phase leading to film formation. Comparative experiments on the behaviour of thin and thick films of tung oil showed that oxygen absorption continues up to the drying point, and decidedly constitutes a principal reaction. It is, however, probable that the whole of the α -elæostearin in the tung oil does not undergo isomerisation to the β -form with subsequent autoxypolymerisation; the greater part contemporaneously is autoxidised directly to a film in much the same way as the di- α -linolenic linoleic glyceride of linseed oil. E. LEWKOWITSCH.

Tung oil. V. Preliminary cracking and wrinkling of drying wood oil [films]. A. EIBNER and E. ROSSMANN (Chem. Umschau, 1928, 35, 281—290; cf. preceding abstract).—Microscopical examination of films of tung oil drying on an inert base (*e.g.*, glass) shows that the first stage of the drying process consists in the formation of a thin skin, invisible to the eye, which shrinks, producing minute cracks or splits in the film and imparting a cell-like appearance. Drying proceeds with the

absorption of oxygen, and during the process small parallel folds or wrinkles at first appear within the cells at right angles to the boundary cracks, then the pressure exerted by the wrinkling and now expanding film closes the original cracks, thus forcing the edges up into characteristic double ridges. The wrinkling may proceed to such an extent that the straight cell-boundaries become distorted and folded, and finally all cell-like appearance may be destroyed. Similar changes were observed in the drying of β -elæostearin itself. On drying in the dark clear unwrinkled films were obtained; tearing occasionally occurred in thick films in which large amounts of β -elæostearin had been formed by previous irradiation. It is considered that the β -elæostearin is responsible for the characteristic double-ridge structure, by causing the preliminary shrinking and cracking of the film, followed by expansion consequent on autopolymerisation. In brilliant sunlight a clear film may be obtained, the autoxidation of the α -elæostearin proceeding more rapidly than the change to the crack-forming β -isomeride. E. LEWKOWITSCH.

Action of selenium monochloride on fatty oils. E. H. HARVEY and H. A. SCHUETTE (J. Amer. Chem. Soc., 1928, 50, 2837—2840).—The rate of rise in temperature (*a*) and the maximum elevation (*b*) observed when sulphur monochloride or selenium monochloride is added under standard conditions (cf. Fawsitt, A., 1889, 317) to a fatty oil dissolved in xylene are tabulated for a representative series of oils. The values of (*b*) are frequently similar for both reagents, but (*a*) is usually much larger for selenium monochloride than for sulphur monochloride. With the former the heat evolution is the sum of the thermal effects of substitution, addition, and decomposition of the reagent, and is not simply related to either the physical or the chemical properties of the oil. The reaction is, therefore, of no value for the quantitative characterisation of fatty oils.

H. E. F. NOTTON.

Determination of oil in olives. S. KALOYEREAS, W. V. CRUICK, and B. E. LESLEY (J. Pharm. Chim., 1928, [viii], 8, 407—415).—The methods are based on the changes in refractive index and density, respectively, of "Halowax oil" (α -chloronaphthalene) after triturating known quantities of the oil and olives together in a mortar. The corresponding constants for the pure olive oil are determined, and from the differences between these and those of the solution of olive oil in "Halowax oil" the amount of olive oil in solution can be determined. Foreign substances from the olive which dissolve can be corrected for as they exert a constant influence. Temperature corrections are also given. A modification is described in which the olives are ground with anhydrous sodium sulphate and then with trichloroethylene, and the density of the resulting solution is determined as above.

E. H. SHARPLES.

Iodine value of Spanish paprika oil. L. C. MITCHELL and S. ALFEND (J. Assoc. Off. Agric. Chem., 1928, 11, 523—527).—The iodine values of the oil extracted from eleven authentic samples of paprika by means of chloroform varied between 133.0 and 139.7; the iodine value range specified by the official stan-

dards (125—136) is therefore not applicable to samples analysed by the chloroform extraction method.

F. R. ENNOS.

Determination of organically-combined sulphur in sulphonated oils. K. H. BAUER (Chem. Umschau, 1928, 35, 290—291).—In reply to criticism levelled by Herbig (Seifensieder-Ztg., 1928, 134), the author points out that, by hydrolysis with *N*-acid, the sulphur is split off from sulphonated oils in the form of the monobasic grouping $\cdot\text{HSO}_4$. Experiments indicate that the results of hydrolysis effected by *N*-hydrochloric and sulphuric acids are influenced by the material of the flasks employed; Jena glass is recommended and blank tests should be performed where possible (cf. B., 1928, 273).

E. LEWKOWITSCH.

Fat in buttermilk. THURSTON and PETERSEN.—See XIX.

PATENTS.

Sulphonation of fats, fatty oils, or fatty acids. H. T. BÖHME A.-G. (B.P. 284,280, 11.1.28. Ger., 27.1.27).—It is claimed that sulphonation products similar to Turkey-red oil, but with an extraordinarily high resistance to acid, lime, and magnesia salts, may be obtained by sulphonating fatty acids or their glycerides in the presence of a large excess of sulphuric acid at temperatures below zero, preferably below -10° . If necessary, the fluidity of the reaction mixture may be increased by the addition of an inert diluent such as a hydrocarbon or its halogenated product. E. HOLMES.

Manufacture of fatty acids containing hydroxy-fatty acids. C. STIEPEL (B.P. 272,198, 27.5.27. Ger., 4.6.26).—Fatty acids containing such proportions of unsaturated hydroxyfatty acids of the ricinoleic acid series that they have approximately the acetyl value and properties of castor oil may be prepared by partially chlorinating products such as Japanese train oil or bone oil, and then heating the resulting mixtures at elevated temperatures with water and caustic alkali. The resulting products have very much higher acetyl values and correspondingly lower iodine values than their original materials. E. HOLMES.

Refining of oils (B.P. 285,064).—See II. **Treating liquids with ultra-violet rays** (B.P. 283,472).—See XI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Oxidation and weathering of linseed oil paints. D'ANS (Z. angew. Chem., 1928, 41, 1193—1197).—The effect on the oxidation of linseed oil of light and moisture is discussed. The swelling of linseed oil and paint films exposed to moisture, which varies with different pigments, is recommended as a delicate criterion in the evaluation of paints. It is pointed out that, under favourable conditions of warmth and moisture, paint films, especially if recently applied, are susceptible to attack by moulds (*Penicillium*, *Aspergillus*) which may cause rapid and extensive deterioration. The development of moulds appears to be hindered by the presence of zinc oxide and certain coloured pigments: the possibility of attack by moulds should not be neglected when assessing weathering tests. E. LEWKOWITSCH.

Impermanence of many coal-tar colours to lime and zinc white [in paints]. G. ARNOLD (Chem.-Ztg., 1928, 52, 815).—Trials over a period of 12 weeks were made with paints containing, respectively, zinc white, white lead, lithopone, and blanc fixe, with linseed oil and a number of coal-tar dyes (enumerated) on small wood test pieces. Whilst the others were unaffected, the zinc white paints showed the effect of the weather in a few days, and even more rapidly under a glass plate. Similar bad results were given by dyes in conjunction with casein solution and lime.

C. IRWIN.

Testing paints. A. V. BLOM (Z. angew. Chem., 1928, 41, 1178—1181).—For testing the strength of paint films, strips of iron of known tensile strength are painted on one side and then pulled in the usual tensile machine until microscopic cracks begin to appear in the paint film. With freshly painted strips this happens at or near the breaking point of the iron; after the paint has aged somewhat, fracture occurs during the region of plastic flow of the iron, but with very old and brittle films it may occur as soon as the elastic limit is exceeded. The shape and position of the fissures in the paint film after the test provide an indication of the state of preservation of the paint, and, by observing whether the first cracks appear along the Lüder-Hartmann flow lines or whether they are evenly distributed over the surface of the strip, conclusions may be drawn as to the intensity of adherence of aged paint films. The value of the method in testing red lead paint is described; after ageing for 12 days at 80° the elongation of films of ordinary red lead paint decreased from 27 to 16.5%, and that of films of highly dispersed red lead paint from 14.7 to 0.2%. The latter therefore harden and become watertight sooner than the former, and the permanent elongation of 0.2% suffices for paint films on iron as it exceeds the usual limit of proportionality of the metal.

A. R. POWELL.

Determination of wood oil in oil paints. VON REIBNITZ (Farben-Ztg., 1928, 34, 266—268).—Marcusson's method (B., 1926, 500) for the determination of wood oil, depending on its polymerisation with iodine to a solid mass, gives results about 10% low. In the case of paints (without thinners) the extraction of linseed oil with chloroform is difficult and results are frequently high. A better approximation to the truth is obtained if the ascertained value is multiplied by the iodine value of the oil under test, and the product divided by the average of the iodine values of the boiled and unboiled oils. Small quantities of turpentine oil greatly hinder the polymerisation of wood oil and should be removed by shaking with 96% alcohol. Marcusson's method is indirect since the wood oil is determined as the difference between the original oil and the extracted linseed oil. The polymerised wood oil can, however, be saponified with 0.5*N*-alcoholic potash, the fatty acid precipitated with barium chloride, and the excess alkali titrated with hydrochloric acid. This method is described. Comparative tests between it and gravimetric analysis of pure wood oil and mixtures with linseed oil show good agreement. C. IRWIN.

Production of iron [oxide] colours. P. P. BUDNIKOV (Chem.-Ztg., 1928, 52, 846—847).—Magnetic

oxide of iron, produced as a by-product in the manufacture of aniline and α -naphthylamine, is a cheap raw material. After washing, heating for 1 hr. at 500—650°, and grinding, a brown-coloured oxide is produced of poor covering power, but if the residues are treated with 40% of their weight of waste sulphuric acid (70%) and heated for 2 hrs. at 750—800° the colour and covering capacity are greatly improved. The higher the temperature of ignition the darker is the colour. The stronger the acid used the lighter is the colour. Mixtures with gypsum or heavy spar can be used as in the usual process for the production of iron oxide colours.

C. IRWIN.

Nitrocellulose lacquers—some recent developments. S. SMITH (J. Oil & Colour Chem. Assoc., 1928, 11, 342—350).—A general treatment of nitrocellulose lacquers, special reference being made to sources of cellulose, modern solvents of the glycol and other types, and pigments.

S. S. WOOLF.

Lead sulphate. SCHRADER.—See VII.

PATENTS.

Lead coating of articles. H. J. SCHIESS, A. C. BOITEL, E. ZÄHNER, A. BOITEL, and L. SCHIESS [ZÄHNER & SCHIESS & Co.], Asses. of SUBOX A.-G. ELECTRO CHEM. FABR. METALL- u. HÜTTENPRODUKTE (B.P. 276,643, 2.8.27. Ger., 30.8.26).—Articles may be protected from atmospheric influences by coating them with a paint consisting of a finely-divided tin-antimony-lead alloy in which lead predominates, and a binder, e.g., linseed oil.

M. E. NOTTAGE.

Production of valuable coloured compounds [lakes]. I. G. FARBENIND. A.-G. (B.P. 299,521, 28.7.27. Addn. to B.P. 275,943; B., 1928, 866).—The basic compounds of the prior patent are mixed with basic dyes (or dyes containing both acidic and basic groups) before conversion into phosphotungstates, phosphotungstomolybdates, etc. Examples are: Michler's hydrol with Victoria Pure Blue BO (from tetraethyldiaminobenzophenone and ethyl- α -naphthylamine), or with Light Green SF yellowish (green-blue), and *p*-aminobenzaldehyde with Victoria Pure Blue BO (olive-green).

C. HOLLINS.

Preparation of alkyl esters of abietic acid. A. C. JOHNSTON, Assr. to HERCULES POWDER Co. (U.S.P. 1,682,280, 28.8.28. Appl., 29.7.26).—Wood rosin is dissolved in hot aqueous-alcoholic sodium hydroxide, ethyl or methyl sulphate is added, alcohol is distilled off, and the residue is heated at 145°; ethyl or methyl abietate is obtained pure by distillation in a vacuum.

C. HOLLINS.

Manufacture of condensation products of phenols. BRIT. DYESTUFFS CORP., LTD., and E. E. WALKER (B.P. 298,680, 15.7.27).—A phenol is condensed first with an aliphatic ketone and then with an aliphatic aldehyde, one or both condensations being in presence of alkali. A white crystalline product is obtained if after the second condensation acid is added to a pH value of about 6; crystallisation is accelerated by "seeding." The product from phenol, acetone, and formaldehyde has m.p. 123—127°, and may be used for the same purposes as the impure oily product hitherto obtained.

C. HOLLINS.

Preparation of phenol-formaldehyde condensation products. G. PETROV and P. SHESTAKOV (U.S.P. 1,684,142, 11.9.28. Appl., 30.12.25).—A phenol, *e.g.*, cresylic or carboic acid, is condensed with formaldehyde in presence of powdered lead or zinc. Reaction occurs at 75–85°. C. HOLLINS.

Manufacture of artificial materials. SOC. CHEM. IND. IN BASLE (B.P. 283,965, 20.1.28. Switz., 21.1.27).—The working properties and insulating capacity of condensation products such as those made from an aromatic amine and more than 1 mol. of formaldehyde are improved by compressing the porous mass first obtained at elevated temperatures. In an example compression of a mass of *d* 0.6 at 100 atm. and 150° for 1 hr. raised its density to about 1.0. E. HOLMES.

[Paints for] protecting metal, wood, etc. P. W. TURNER (U.S.P. 1,689,567, 30.10.28. Appl., 18.8.26. U.K., 1.12.25).—See B.P. 271,912; B., 1927, 609.

Manufacture of peroxide [ozonides] of hydrocarbons [turpentine substitutes]. A. S. RAMAGE (U.S.P. 1,689,599, 30.10.28. Appl., 27.4.25).—See B.P. 273,832; B., 1927, 661.

Printing ink. K. STEPHAN, Assr. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,689,368, 30.10.25. Appl., 14.4.27. Ger., 5.5.26).—See B.P. 270,671; B., 1928, 24.

Protective coatings for rubber (B.P. 299,169).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Lamp-black for rubber mixtures. F. KIRCHHOF (Gumm-Ztg., 1928, 43, 309–310).—Chemical analysis can be of distinct value in discriminating between the value of various types of amorphous carbon for rubber-compounding, and examination of a colloid-chemical character is not the only useful procedure. Data are quoted from other workers in support of this opinion. D. F. TWISS.

PATENTS.

Preservative coatings for goods of rubber or like substances. B. D. PORRITT, T. R. DAWSON, and RES. ASSOC. OF BRIT. RUBBER & TYRE MANUFRS. (B.P. 299,169, 29.9.27).—Preservative paints or coatings for application to rubber goods for protection against atmospheric influence and the effect of actinic light are made with a medium, preferably capable of swelling the rubber, together with a material, *e.g.*, mineral rubber, to increase viscosity and give some mechanical protection. There are also introduced small quantities of anti-oxidants, *e.g.*, aldol- α -naphthylamine, and of yellow or red substances such as Oil-red S. D. F. TWISS.

Rubber compositions. C. H. CAMPBELL, Assr. to AMER. GLUE Co. (U.S.P. 1,683,862–4, 11.9.28. Appl., 22.10.26).—Reinforcing materials for rubber are obtained by hydrolysing (A) glue, (B) keratin, for 2–6 hrs. with steam at 100 lb. pressure. (C) Both materials are used. C. HOLLINS.

Rubber-like hydrocarbons (B.P. 298,584).—See II. **Rubber-bonded abrasive** (U.S.P. 1,687,410).—See VIII.

XV.—LEATHER; GLUE.

PATENTS.

Manufacturing from gelatin articles such as threads, cords, ribbons, tubes, etc., otherwise made from animal intestinal canals or skins. P. HIRSCH (B.P. 299,105, 25.7.27).—Gelatin solutions, with or without suitable additions, in a thin layer at rest or flowing downwards, and/or the articles made therefrom, are sterilised by exposure to germ-killing rays, preferably ultra-violet rays. D. WOODROFFE.

Manufacture of casein capable of swelling. E. STERN (G.P. 445,732, 6.11.24).—Dry casein is made into a thick paste with a 10% solution of dextrin or a degradation product of starch, and the mixture is dried and ground. A. R. POWELL.

Manufacturing blocks and articles of casein products. A. ZSIGMONDY (B.P. 298,815, 22.12.27).

XVI.—AGRICULTURE.

Colloidal behaviour of soils and soil fertility. V. Distribution of soluble and colloidal iron and aluminium in soils. J. S. JOFFE and H. C. McLEAN (Soil Sci., 1928, 26, 317–325; cf. B., 1928, 723).—Except in very acid conditions, molecular iron and aluminium compounds do not exist in solution in soils. Examination of dialysed soil extracts shows very little iron or aluminium in the sol form. Most of the iron and aluminium produced during the weathering of complex minerals is distributed throughout the soil in the gel condition. Extraction of soil with barium chloride brings much iron and aluminium into solution, due to liberation of acid following base-exchange effects. The amounts of iron and aluminum so dissolved are less than the equivalent of the acid formed. Less iron than aluminium becomes soluble under these conditions. The amount of iron and aluminium thus appearing in solution may be correlated with the titratable acidity of the acid liberated as a result of base exchange.

A. G. POLLARD.

Influence of nitrogenous fertilisers on the structure of soils. H. LATHER (Z. Pflanz. Düng., 1928, 12A, 227–251).—The structure of soils treated with various fertiliser materials was examined by means of mechanical analyses. The dispersive effect of sodium nitrate on the clay particles was confirmed. On very acid soils and in normal soils where unusually small amounts of nitrate were added a coagulating effect was observed. The results of treating soil with calcium cyanamide were in the main the outcome of its lime content and were somewhat indefinite in the absence of carbon dioxide. In the presence of this latter the usual flocculation occurred. Addition of ammonium sulphate to soil resulted, in the first instance, in interaction with the soil carbonates, and the ammonium carbonate produced greater dispersion of the clay. Subsequent oxidation of the ammonium salt to nitric acid resulted in flocculation of the finer particles. A. G. POLLARD.

Determination of the lime requirement of soils on the basis of laboratory and vegetation experiments. A. KAUFMANN (Z. Pflanz. Düng., 1928, 12A, 156–175).—A comparison was made on five soils of

the Hissink, Gehring, Gedroiz, and Bobko-Askinasi methods. No relationship between the values obtained by these methods was apparent, and it was impossible to say which was the most correct. Pot experiments on these soils were then conducted, using varying amounts of chalk as determined by the titration curve and Daikuhara methods. The amounts as determined by these two methods were insufficient to attain a maximum yield. Gehring's value and a quantity equivalent to three times the value obtained from the maximum yield proved sufficient for a maximum yield. On one soil depression of yield due to over-liming was observed.

H. J. G. HINES.

Detection and determination of chlorate ion in soils. F. MACH and R. HERRMANN (*Z. Pflanz. Düng.*, 1928, **12A**, 189—198).—Although the chlorate ion can be detected and determined by treating a soil extract with potassium bromide and potassium iodide and subsequently determining the liberated iodine, the following method is less liable to error. The chlorine ion in a soil extract is first determined by Volhard's method. The chlorate is then reduced by treatment with sulphur dioxide and the chlorine ion is again determined. The amount of chlorate ion is arrived at by difference.

H. J. G. HINES.

Colorimetric process for the determination of the phosphate requirement of arable soils. A. NEMEC (*Deut. Landw. Presse*, 1928, **55**, 296, 331; *Bied. Zentr.*, 1928, **57**, 442—443).—Both phosphates and silicates may be determined in soil extracts by the colorimetric method involving the reduction of the respective molybdc acid compounds with alkaline solutions of quinol. In the examination of cropped soils it is shown that the presence of silicates in soil extracts corresponds with decreased efficiency of phosphatic fertilisers. Soluble silicates favour the re-adsorption or reversion of soluble phosphates in soils. The importance of determinations of soluble silicates in the evaluation of the phosphate requirements of soils is emphasised.

A. G. POLLARD.

Laboratory methods for determining the form in which phosphatic fertilisers should be applied to different soils. L. VON KREYBIG (*Z. Pflanz. Düng.*, 1928, **12A**, 176—188).—To suspensions of soil in water are added superphosphate and Rhenania phosphate, respectively. The phosphoric acid in the liquid phase is then determined and a determination of citric-soluble phosphate in the soil is also made. If the superphosphate loses both its water and citric acid solubility, Rhenania or some basic phosphate is likely to give better field results. Similar determinations can be made by the azotobacter method.

H. J. G. HINES.

Fixation of phosphorus by soils. A. G. WEIDEMANN (*Soil Sci.*, 1928, **26**, 281—290).—The fixation of phosphorus from monocalcium phosphate solutions by soils under varying conditions is examined. Acid muck soils show a negative fixation with higher concentrations of phosphate solutions. This is assumed to be a consequence of the rendering unfree of a portion of the soil water. Liming prevents this phenomena. Muck soils with relatively high p_H values do not exhibit negative fixation, but their positive fixation capacity is reduced

by acid treatment. If soils showing negative fixation are treated with the amount of water which they can render unfree, phosphate fixation is positive, irrespective of the concentration of the solution used.

A. G. POLLARD.

Use of sulphur and rock phosphate [as fertiliser]. A. KALUSHSKY (*Z. Pflanz. Düng.*, 1928, **12A**, 217—226).—In pot experiments with millet rock phosphate was rendered fully available to plants by use with an equal amount of sulphur. Crop yields were as great as when an equal proportion of phosphorus was applied in the form of dicalcium phosphate. The optimum ratio of sulphur to phosphorite varied with soil type, being 4:1 in some soils examined. Sulphur-phosphorite fertiliser produced a considerably increased yield of grain compared with that resulting from an equivalent amount of soluble phosphate.

A. G. POLLARD.

Oxidation of sulphur in suspensions of activated sludge and its influence on the solubilisation of mineral phosphates. C. V. R. AYYAR, T. S. S. PERUMAL, and R. V. NORRIS (*J. Indian Inst. Sci.*, 1928, **11A**, 85—90).—Sulphur was rapidly oxidised when introduced into suspensions of activated sludge. The sulphates of manganese, aluminium, and iron (ferrous) acted catalytically in increasing the production of water-soluble phosphates when mineral phosphates were introduced into the system.

H. J. G. HINES.

The "wilting coefficient" and other moisture values in soils. P. I. ANDRIANOV (*Z. Pflanz. Düng.*, 1928, **12A**, 145—155).—A simple relationship is demonstrated between the moisture values in soils.

H. J. G. HINES.

Possible relationship between soil salinity and stand in cotton. J. A. HARRIS (*J. Agric. Res.*, 1928, **37**, 213—231).—An examination of the possible relationships existing between soil concentration and the seedling stand produced in cultures of three grades of upland cotton grown in experimental fields has given inconclusive results which do not fully confirm those of previous experiments (cf. *B.*, 1926, 842).

E. H. SHARPLES.

Desirable soil-nitrate levels for certain market-garden crops. J. B. SMITH (*Soil Sci.*, 1928, **26**, 265—279).—Examination of the nitrate content of soils under crops indicates that the maintainance of a minimum nitrate concentration throughout the growing season is necessary for optimum growth. Desirable concentrations are: for celery, lettuce, and beet 10, cabbage 15, tomatoes and spinach 20 p.p.m. Accumulations of nitrogen following green manuring of soils with oats, rye, and timothy are correlated with the amount of nitrogen in the particular crop used. Oat green manure reduces the relative yield of celery to an extent increasing with the nitrogen content of the green crop. This effect is not attributable to the higher nitrogen accumulation in the soil from the decomposed oat crop.

A. G. POLLARD.

Toxic fluorine compounds. MARCOVITCH.—See VII.

PATENTS.

Treatment of plants. W. EGGERT, JUN. (U.S.P. 1,686,964, 9.10.28. Appl., 22.12.24).—Tobacco plants are treated in the blossoming season with a solution

obtained by the fermentation of leaves and petals of the same type of plants, together with a sweetening substance and a derivative of gum benzoin.

A. R. POWELL.

Treatment of seed. A. KLAGES (U.S.P. 1,688,720, 23.10.28. Appl., 17.5.26. Ger., 27.10.23).—See B.P. 274,974; B., 1927, 792.

XVII.—SUGARS; STARCHES; GUMS.

Influence of the basicity of basic lead acetate in the clarification of molasses solutions. E. SOMMER (Z. Zuckerind. Czechoslov., 1928, 53, 45—52).—The basic lead acetate prescribed for sugar analysis contains 31.9% of its lead in the form of oxide. If solutions containing a higher proportion of oxide to normal acetate are used, lower values are obtained for the polarisation of beet molasses, assuming that the same total amount of lead is used for a given weight of molasses. If the defecant contains 57% of its lead as oxide it may lower a molasses polarisation of 50 by 1—2 units. The extent of the lowering is a direct function of the original alkalinity (p_H value) of the molasses; but no alkalinity likely to occur in practice will produce any lowering when basic lead acetate of the usual composition is employed. The decolorising effect (always assuming the use of the same total amount of lead for a given weight of molasses) increases with the proportion of lead oxide until this reaches 39—52%, but beyond this limit it diminishes rather rapidly. With 48% of the total lead as oxide decolorisation is much better than with the usual proportion, and the lowering of polarisation can be prevented by previous neutralisation of the solution with acetic acid.

J. H. LANE.

Action of basic lead acetate in presence of alkali on dextrins and gums, and its use in the analysis of certain sugar products. C. PERTUSI (Annali Chim. Appl., 1928, 18, 414—420).—Dextrins and gums are precipitated by basic lead acetate in presence of sodium hydroxide, and the preparation of the solution in which the sugars present in biscuits may be determined may be carried out as follows: 20 g. of the material, made into a paste with a little water, are introduced into a 200—220 c.c. Mohr flask and mixed with about 150 c.c. of water. After some hours the mass is treated with 5—6 c.c. of basic lead acetate solution, two or three drops of alcoholic phenolphthalein solution, and, drop by drop, concentrated sodium hydroxide solution until a faint pink colour appears. After some time the liquid is made up to volume and filtered, any slight coloration in the filtrate being usually removable by addition of a drop of acetic acid solution. When sucrose is the only sugar present this method gives good results, in agreement with those furnished by Savini's method (cf. B., 1917, 96). When invert sugar also is present and Clerget's method for determining the sucrose has to be employed, the results, as is also the case with Savini's method, are not so good; determination of the invert sugar is also not so satisfactory. With gum products 20 g. are treated with about 150 c.c. of hot water in a Mohr flask, and 20 c.c. of basic lead acetate solution are added in quantities alternately with sodium hydroxide. When the precipitate collects

into a consistent mass the precipitation of the gum is practically complete. The subsequent procedure is as above.

T. H. POPE.

Significance and determination of temperature coefficients in the study of inversion. V. ZAFOUK (Z. Zuckerind. Czechoslov., 1928, 53, 73—79).—Measurements of the inverting capacity of weak acids at different p_H values showed that no inversion occurs beyond about p_H 6. The p_H values of solutions of weak acids, or of mixtures of the acids and their salts, vary to some extent with temperature, and the variations are not the same with different acids; differences up to 0.1 in p_H value were observed between 20° and 70°. Curves are given showing the p_H values and corresponding temperature coefficients for different stages of neutralisation of succinic, aspartic, acetic, glutimic, and glutamic acids. For practical purposes a factory juice which is slightly alkaline to phenolphthalein will be safe from inversion, provided it has no tendency to become more acid in course of time or on heating.

J. H. LANE.

Determination of sugars from the oxygen in the cupric oxide. M. D. HADJIEFF (Z. Unters. Lebensm., 1928, 55, 613—614).—The cuprous oxide is precipitated by the Meissl-Allihn method and collected in an unweighed Allihn asbestos filter tube. It is dried at 120—130° and oxidised by heating in a stream of dry air, and the tube and its contents are cooled in a desiccator and weighed. The cupric oxide is then reduced to copper in a stream of pure dry hydrogen, cooled in the gas, and weighed again. The difference in the two weighings multiplied by 3.9731 gives the weight of copper, from which the corresponding weight of dextrose can be found in the usual way. Concordant results are obtained agreeing well with those obtained by the usual Meissl-Allihn method.

W. J. BOYD.

Polarimetric determination of sucrose in mixtures of milk and sucrose. G. W. MONIER-WILLIAMS (Analyst, 1928, 53, 569—581).—About 350 g. of the milk and sucrose mixture (weighed to 0.1 g.) are treated with 1.5 g. of (powdered) citric acid, the coagulated milk is shaken, and to it is added a mixture of 9 g. of phosphotungstic acid and 45 g. of dry sand, gradually and with shaking. A determination of the total solids is carried out on the filtrate. Then 50 c.c. of filtrate are weighed into a 100 c.c. flask, the amount of water is calculated from the total solids determination (y in formula), 2.675 g. of dry ammonium chloride are added, and the liquid is made up to volume. After 1 hr. the solution is polarised in a 200 mm. water-jacketed tube at 18—22°. A second portion of the filtrate is measured into the same flask with the same pipette, and 10 c.c. of 5*N*-hydrochloric acid are added, the mixture being placed in a water-bath at 60° for 12 min. with shaking for the first 4 min. On cooling, 10 c.c. of 5*N*-ammonia solution are slowly added to neutralise the hydrochloric acid present, the solution is made up to volume, filtered if necessary, and polarised in the same way and as nearly as possible at the same temperature as the uninverted solution. The mean of the readings is B , and $A - B$ the change of rotation on inversion. Then % sucrose = $(A - B) / [87.9 + 0.06c - 0.3(t - 20)] \times 100x / 2y$, where c is the % concentration

of total sugars in the inverted solution as diluted for polarisation, t the temperature of invert polarisation, x the % of water by wt. in the diluted condensed milk or milk and sucrose mixture, and y the weight of water in the 50 c.c. of filtrate taken for polarisation. Notes on the method have reference to the temperature of inversion; the strength of hydrochloric acid and time of inversion; the neutralisation of inverted solution and addition of ammonium chloride to the direct rotation liquid; the divisor in the formula; the total solids determination in milk and sucrose filtrates; the ratio correction; and to a method for maintaining a constant temperature in the polarimeter.

D. G. HEWER.

Sulphide-stain method for determining small quantities of "volatile sulphur" in sugar. J. M. BRYAN (Analyst, 1928, 53, 589—590).—In a modification of Mann's sulphide-stain method (cf. Ogilvie, B., 1927, 312) a Gutzeit apparatus is used, and lead acetate test papers (6 cm. \times 4 mm.). The sugar sample is added to the flask containing 25 g. of zinc pellets (washed with dilute hydrochloric acid and air-free distilled water) and 50 c.c. of air-free distilled water; 50 c.c. of 1:1 hydrochloric acid are added, the contents are shaken, and, after 1 hr., the test paper is dipped in melted paraffin and compared with the standard stains which correspond to 0.005, 0.010, 0.015, 0.020, and 0.025 mg. of sulphur dioxide when 5 c.c. of the solutions and 10 g. of pure sucrose per 100 c.c. are used in the apparatus. A blank test must be made, and a preliminary trial. Comparison showed that 1 pt. of hydrogen sulphide was approximately equivalent colorimetrically to 2 pts. of sulphur dioxide.

D. G. HEWER.

Analysis of starch sugar degradation products by selective fermentation. T. McLACHLAN (Analyst, 1928, 53, 583—588).—The method found most satisfactory for analysing starch degradation products was selective fermentation by different yeasts. As a general routine method, 8 tubes each containing 50 c.c. of a 10% solution of the material being examined are steam-sterilised for three successive days with rapid heating on the first day to destroy any diastase present. Two tubes are inoculated with *Saccharomyces exiguus*, two with *S. Froberg*, two with *S. Saaz*, and two are kept blank, and the set is incubated at 26° for 14 days, the tubes being rotated on the 4th or 5th day to distribute the yeasts and introduce a fresh air supply. One tube from each yeast is then emptied into a separate 150 c.c. beaker, evaporated to about 15 c.c., cooled, made up to 50 c.c., and the gravity determined, the second set of tubes being kept for verification. The difference between the total solids of the blank and of the solution fermented by *S. exiguus* represents dextrose and laevulose, that between *S. exiguus* and *S. Froberg* represents maltose, and that between *S. Froberg* and *S. Saaz* gives other fermentable sugars. For determination of dextrans, the optical rotation is determined and calculated for 100% product; then % dextrans = $[\alpha]_D \times 100/180$. It is suggested that values should always be calculated on 100% total solids. Analyses are given of four samples of commercial glucose and twelve of malt extracts.

D. G. HEWER.

Continuous automatic counter-current extractor for starchy and saccharine substances with press elevator. G. MEZZADROLI and G. MAZZINI (Zymologica, 1928, 3, 109—112).—Four or six superposed, steam-jacketed, horizontal cylinders are provided with screws which are driven from the outside and have tubular axes for steam heating. The material, such as beet slices, is fed into the uppermost cylinder through a charger with hermetic seal and passes from end to end of each cylinder in turn, water, previously pumped through a heater, traversing the cylinders in the opposite direction to be discharged through a grid which is kept clear by the screw moving against it. The exhausted material is carried by a screw elevator against a pressure cone and so freed from most of the absorbed liquid. The apparatus is simpler than the diffusion battery, and requires less space and less labour, whilst the heat is more efficiently utilised and initial and maintenance costs are diminished.

T. H. POPE.

PATENTS.

Cyclic process of using barium compounds in the manufacture of sugar. H. W. DAHLBERG (U.S.P. 1,688,071, 16.10.28. Appl., 16.22.25).—Insoluble material containing barium carbonate is heated with alumina thereby producing a barium aluminate compound containing water-soluble barytic material, which is used in the treatment of molasses. The insoluble material recovered from the barium saccharate is heated again with alumina to produce water-soluble barytic material.

W. J. BOYD.

Manufacture of sugar [from molasses]. T. GASPAR Y ARNAL (B.P. 297,482, 23.5.27).—Molasses is prepared for desaccharification by treatment with soluble ferrocyanides, with or without an alcohol, to precipitate alkali and alkaline-earth metals as double ferrocyanides. (Cf. B., 1928, 725.)

J. H. LANE.

Simultaneously making sugar and paper pulp from [sugar] cane. E. A. VAZQUEZ, Assr. to VAZCANE PROCESS, INC. (U.S.P. 1,688,904—5, 23.10.28. Appl., [A] 30.8.27, [B] 3.4.28. Cuba, [A, B] 2.6.27).—Sugar cane is simultaneously extracted and reduced to fibre by contact with a moving abrasive surface in the presence of extracting liquid, and, after separation from the fibres, the solution is worked up to sugar.

L. A. COLES.

Recovering sugar values. R. C. CAMPBELL, Assr. to UNITED FILTERS CORP. (U.S.P. 1,685,118, 25.9.28. Appl., 7.5.25).—The mud is filtered and washed continuously, the wash water being collected and the cloudy filtrates returned. After each cycle of operations the filter surfaces are cleaned.

F. G. CLARKE.

Increasing the stability of solid sugar, more particularly starch sugar. H. MAUTNER (B.P. 297,338, 15.6.27).—To prevent fermentation consequent on absorption of moisture, dextrose, or other sugar is incorporated with a small proportion of alcohol, not sufficient to render it wet. *E.g.*, alcohol may be added to a very concentrated dextrose solution, which is afterwards allowed to crystallise to a solid mass.

J. H. LANE.

Clarification of cane molasses for the growing of yeast. C. HOFFMAN, C. N. FREY, and F. M. HILDE-

BRANT, Assrs. to FLEISCHMANN Co. (U.S.P. 1,687,561, 16.10.28. Appl., 8.5.23).—Sufficient sodium silicate is added to a dilute solution of molasses to give p_H 7.7—12. The liquid is then filtered while hot.

W. J. BOYD.

Treatment of molasses [for use in manufacture of yeast]. F. RANSOHOFF, Assr. to FLEISCHMANN Co. (U.S.P. 1,688,831, 23.10.28. Appl., 11.5.25).—The molasses is made alkaline with ammonia, an ammonium phosphate is added, and insoluble substances are separated.

W. J. BOYD.

[Cooling] treatment of sugar and like material. A. E. WHITE. From ACKERS, LONSDALE & Co. PROPRIETARY, LTD., and G. LONSDALE (B.P. 298,735, 18.8.27).

XVIII.—FERMENTATION INDUSTRIES.

Preservative principles of hops. IX. Influence of special methods of drying at low temperatures on the antiseptic properties of hops. J. J. H. HASTINGS and T. K. WALKER (J. Inst. Brew., 1928, 34, 556—565).—Hops dried under special conditions at a low temperature have an initially greater antiseptic power than those subjected to the ordinary kiln processes of drying. This additional antiseptic power is lost after six months in cold storage, and the specially dried and kiln-dried hops then possess approximately equal antiseptic values. The extra antiseptic power of green and specially dried hops cannot be ascribed to the presence of any hitherto unidentified antiseptic substance, which is destroyed by kiln drying, but is due to the presence of a large proportion of crystalline lupulon in the β -portion of the soft resin. This is partly destroyed when the hops are dried by ordinary kiln processes. Humulon and, to a greater degree, lupulon are progressively destroyed when hops are boiled in water or wort. The actual proportion of the two components destroyed depends chiefly on the time factor.

C. RANKEN.

Brewing trials with new and commercial varieties of hops. W. T. SMITH and A. J. C. COSBIE (J. Inst. Brew., 1928, 34, 565—570).—The brewing value, flavour, and preservative property of certain of the new varieties of hops raised at Wye are compared with well-known commercial varieties. Among the tabulated results of the trials a hop of Oregon and English parentage takes a high place. Retaining only to a slight extent the rank Oregon aroma of its female parent, it is superior to English hops in preservative power, and gave a beer of exceptionally high class and distinctly aromatic. In all cases hops affected with Downy Mildew imparted their undesirable flavour to the beer in which they were used.

C. RANKEN.

Small-scale brewing in the laboratory. F. E. DAY (J. Inst. Brew., 1928, 34, 570—573).—Brewing conditions are imitated as closely as possible, the duration of each operation being considered as important as the temperature or the proportion of the ingredients. The main difficulty of controlling temperatures during fermentation is overcome by the use of vacuum flasks as fermenting vessels which allows the temperature during fermentation to rise slowly from 15° to 20.5°, and at the end to fall to the original figure without sudden cooling.

C. RANKEN.

Determination of glycerol in beer. H. LACROIX and S. KROPACSY (Woch. Brau., 1928, 45, 490—491).—Zeisel and Fanto's method is applicable to beers, but certain minor modifications are advisable. Duplicate results on dark beers vary more than those on pale beers, but in any case the method is more rapid and gives better results than the lime method.

F. E. DAY.

Action of chloroform on development of yeast. M. N. MEISSEL (Woch. Brau., 1928, 45, 488—490).—Yeast cultures of various ages were spread on the surface of wort-agar in Koch dishes and exposed to the action of chloroform vapour. On removal the yeasts showed no development for 3—7 days, against 1 day for the controls. Young cultures were more affected than old, both as regards narcosis and death of the cells. From a pure culture of *Saccharomyces cerevisiae* so treated for a considerable period, cells survived which showed a permanent change in cultural characters, being asporogenous and forming intensely wrinkled colonies.

F. E. DAY.

Balsam vinegar of Modena. E. PARISI (Annali Chim. Appl., 1928, 18, 395—407).—This vinegar, which, to be of good quality should be at least 100 years old, is made by storing a mixture of boiled, concentrated must, usually of the white Trebbiano grape, with strong vinegar in casks of oak, chestnut, or mulberry wood. It is an almost syrupy, black liquid of sweet, acid taste and pleasant odour, and it emits vapours which produce stimulating effects when breathed. It is not used alone, but is mixed with good ordinary vinegar. Analysis of eight samples, five dating back to 1800—1860 and the others of unknown age, gave: d_{20}^{20} 1.1055—1.2880; extract 23.44—79.77 g. per 100 c.c.; fixed acid 2.77—4.42, and volatile acid 3.27—13.78, calculated as g. of acetic acid per 100 c.c.; reducing sugars 26.28—49.44 g. per 100 c.c.; glycerol 0.67—0.91 g. per 100 c.c. Among the components of these vinegars are acetylmethylcarbinol, acetaldehyde, alcohol, formic and acetic acids, much malic, less succinic, and little tartaric acid, and esters. A moderately good balsam vinegar is now made by adding old vinegar of good quality to boiled must.

T. H. POPE.

Analysis of starch sugar. McLACHLAN.—See XVII.

PATENTS.

Production of nitrites (U.S.P. 1,685,629).—See VII. **Molasses for yeast** (U.S.P. 1,687,561 and 1,688,831).—See XVII. **Curing meat** (U.S.P. 1,685,630).—See XIX.

XIX.—FOODS.

Behaviour of bacteria in milk. G. DEI GRISOGONO (Zymologica, 1928, 3, 113—115).—The existence of Gorini's two types of coagulation of milk (A., 1926, 1278; B., 1926, 418) is confirmed. Organisms causing the first type of coagulation do not liquefy gelatin, whereas those producing the second type liquefy gelatin distinctly and rapidly. The only strain of streptococcus isolated from milk belongs to the second class.

T. H. POPE.

Detection of added water in sour milk by determination of the refractive index. A. SCHNECK (Milch. Zentr., 1928, 57, 309—312).—During the souring of a milk the refractive index of its calcium chloride serum increases, but the specific refraction remains constant. The increase in the dry substance on souring is a linear function of the acidity, from which it may be calculated. The total dry substance of the serum may be obtained from the refraction by means of Wiegner's table, and hence the amount of dry substance and the refraction of the serum of the original milk are known. In the case of a sour milk derived from milk adulterated with water, the calculated dry substance and refraction are below the normal. F. R. ENNOS.

Lipins and sterols as sources of error in the determination of fat in buttermilk by ether extraction methods. L. M. THURSTON and W. E. PETERSEN (J. Dairy Sci., 1928, 11, 270—283).—For a product such as buttermilk, containing approximately as much lecithin as fat, Babcock's method for the determination of fat is preferred. The gravimetric method gives high results owing to the extraction of unsaponifiable matter and lecithin. CHEMICAL ABSTRACTS.

Soluble carbohydrates in rye flour. T. CHRZASZCZ and W. MICHALSKI (Przemysl Chem., 1928, 12, 389—402).—Rye flour contains normally as much dextrin as reducing sugar. After storage the dextrin content is trebled, whilst where unsuitable damp magazines are used the sugar content rises at the expense of the dextrin. Better yields of bread are obtained from brown than from white flours. The baking qualities of brown flour improve up to 1½ years of storage, but after 3 years a distinct deterioration may be observed. The temperature of milling should not rise above 40°, as otherwise destruction of diastase contained in the wheat kernel takes place. R. TRUSZKOWSKI.

Evaluation of flour by iodine solution. T. CHRZASZCZ and W. MICHALSKI (Przemysl Chem., 1928, 12, 342—349).—The quantity of iodine adsorbed in unit time by an aqueous extract of flour is in proportion to the dextrin and maltose content of the latter. Since the content of these substances depends on the condition of the grain at harvest and on the milling and storage of the flour, this method affords a convenient means of evaluating it for breadmaking. R. TRUSZKOWSKI.

Vitamin contents of Japanese foodstuffs. SHIMODA, FUJIMAKI, and S. SAIKI (Imp. Japanese Inst. Nutrition; Bull. soc. hyg. aliment., 1927, 15, 481—504, 524—551).—The following were studied: summer oranges, canned plums, lard, radish juice, *Tapes Philippinarum*, radish leaves, fermented rice, preserved radish, cucumber, and egg-plant, buckwheat flour, red kidney beans, and Japanese eels. CHEMICAL ABSTRACTS.

Proteins of Indian foodstuffs. I. Proteins of ragi (*Eleusine Coracana*): eleusin, the alcohol-soluble protein. N. NARAYANA and R. V. NORRIS (J. Indian Inst. Sci., 1928, 11A, 91—95).—An improved method of extraction consists in pouring the alcohol solution into water and subsequent precipitation by an electrolyte. A preliminary examination by the Van Slyke method indicates that eleusin is characterised by its low content of basic nitrogen. H. J. G. HINES.

Influence of malic acid in lemon juice on Warington's method of analysis. F. PERCIABOSCO (Atti II Cong. Naz. Chim. Pura Appl., 1926, 1369—1370; Chem. Zentr., 1928, i, 2191).—Addition of 10% of malic acid to lemon juice does not affect the determination of citric acid by Warington's method, calcium malate being ten times as soluble as the citrate. A. A. ELDRIDGE.

Determination of phosphoric acid in foodstuffs. W. HARTMANN (Z. Unters. Lebensm., 1928, 55, 610—613).—The following solutions are prepared: (a) 210 g. of ammonium molybdate in 300 c.c. of water and 400 c.c. of 10% ammonia solution, (b) nitric acid (*d* 1.2), (c) 1% solution of potassium nitrate. Solutions (a) and (b) are mixed shortly before use in the proportions 1:2 and 75 c.c. of this mixture are added to the phosphate solution in the cold, 0.5 g. of ammonium sulphate having been previously dissolved in the latter. The mixture is stirred with a glass rod until precipitation occurs, without touching the sides of the beaker. After about 4 hrs. it is filtered through a close "barium sulphate" paper and the precipitate is washed with 1% potassium nitrate solution until 10 c.c. of the wash liquor are neutralised to methyl-orange by 1—2 drops of 0.1*N*-soda. The filter and precipitate are placed in a beaker, covered with 1% potassium nitrate solution, and the precipitate is dissolved by gradual addition of carbonate-free *N*-soda. The excess of soda is determined by titration with *N*-sulphuric acid. Satisfactory results were obtained for a standard phosphate solution, and determinations of phosphoric acid in wine and of alcohol-soluble phosphoric acid in dried eggs gave results in agreement with those obtained by the magnesium pyrophosphate method. W. J. BOYD.

Determination of cacao shell. W. PLÜCKER, A. STEINRUCK, and F. STARCK (Z. Unters. Lebensm., 1928, 55, 622—623).—A question of priority (cf. B., 1928, 314). W. J. BOYD.

Determination of sucrose in mixtures of milk and sucrose. MONIER-WILLIAMS.—See XVII.

PATENTS.

Increasing the percentage of fat in milk, and production of artificial creams. H. BÜNING (B.P. 299,617, 7.11.27).—Earth-nut oil, with or without addition of sesame oil, is hardened to 32° and added to milk in the proportion of 10 pts. of oil to 90 pts. of milk. The mixture is homogenised at 175 atm. for enriched milk, at 150 atm. for coffee-cream, and at 50—75 atm. for whipped cream. In producing the last-named product the milk must not be pasteurised prior to addition of the oil, and should preferably be unskimmed. The homogenised mixture is skimmed and the cream brought to a fat content of 35%. By using a higher proportion of oil, a cream containing 35% of fat may be obtained directly. W. J. BOYD.

Production of chocolate-milk compositions. W. A. HEYMAN (U.S.P. 1,689,028—9, 23.10.28. Appl. 4.5.27)—(A) Cocoa powder of low fat content is mixed with a suitable liquid, the mixture is heated to break up the starch cells, homogenised, and mixed with a lactil fluid. The resultant mixture is sterilised or pasteurised by heat and dried to a powder. (B) A soluble powder is

produced consisting of acid-neutralised cocoa of low fat content with the starch cells broken up, together with milk solids. W. J. BOYD.

Production of egg products. A. K. EPSTEIN (U.S.P. 1,687,268—1,687,270, 9.10.28. Appl., 24.12.26).—(A) Egg yolk is mixed with an edible substance which forms hydrogen ions, and is then frozen. Sufficient acid-producing substance is added to reduce bacterial decomposition before freezing and during thawing, and to modify the physical condition of the thawed product. (B) A neutral, soluble, edible salt is also added to control the viscosity of the thawed product. (C) A mixture containing egg yolk, a neutral organic compound having at least one hydroxyl group and capable of lowering the freezing point, *e.g.*, sucrose, and a neutral salt, *e.g.*, sodium chloride, is frozen; an edible acid may also be present. The addition of sodium chloride and of the organic compound reduces the viscosity of the thawed acid-treated yolk; a neutral salt added to a mixture of the yolk and organic compound increases the viscosity of the thawed product. F. G. CLARKE.

Curing meat. E. T. DRAKE, Assr. to CUDAHY PACKING Co. (U.S.P. 1,685,630, 25.9.28. Appl., 8.2.26).—Meat is immersed in a pickle prepared by inoculating an aqueous solution of a nitrate, sugar, salt, and a protein material with a selected group of nitrate-reducing and salt-tolerant bacilli or spirilli. C. RANKEN.

Preparing spinach and the like for canning. W. E. THOMAS (U.S.P. 1,685,511, 25.9.28. Appl., 27.9.27).—The vegetable is wilted at the maximum temperature at which the formation of phaeophytin from the chlorophyll present would be insufficient to affect the natural colour of the vegetable. H. ROYAL-DAWSON.

Manufacture of phosphorus compound from animal proteids. S. POSTERNAK, Assr. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,690,752, 6.11.28. Appl., 21.3.27. Switz., 31.3.26).—See B.P. 268,805; B., 1928, 138.

Manufacture of chocolate goods and the like. R. HEAD, ANC. ÉTABL. A. SAVY JEANJEAN & CIE. SOC. ANON., and BAKER PERKINS, LTD. (B.P. 299,340, 22.7.27).

Manufacture of compressed edible tablets. R. SUCZEK (B.P. 299,685, 18.5.28).

Treating liquids with ultra-violet rays (B.P. 283,472).—See XI.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Detection of "plasmaquin." W. SCHULEMAN, F. SCHÖNHÖFER, and A. WINGLER (Abh. Gebiete Auslandskunde, 1928, 26, D, ii, 5 pp.; Chem. Zentr., 1928, i, 2193).—"Plasmaquin" is detected in urine-free aqueous solution at a dilution of 1 in 2×10^5 by the development of a blue colour with tetrachlorobenzoquinone. Reaction with potassium mercuri-iodide, which is not specific, is sensitive to 1 in 10^5 . A. A. ELDRIDGE.

Formula for calculating composition of mixtures of mydriatic alkaloids. J. C. MUNCH and G. S. GITTINGER (J. Assoc. Off. Agric. Chem., 1928, 11, 521—523).—The physiological effect of a mixture of

mydriatic alkaloids is the sum of that of each of the constituents; from this a formula is derived for the composition of a two-fold mixture when the total alkaloid present and the relative activity of the constituents are known. F. R. ENNOS.

PATENTS.

Production of primary stibinic acids. F. DUNNING and E. E. REID, Assrs. of HYNSON, WESTCOTT, and DUNNING (U.S.P. 1,682,269, 28.8.28. Appl., 6.1.27).—*p*-Aminoacetanilide and antimonious oxide are dissolved in hydrochloric acid and diazotised. The diazo-antimony compound is suspended in water and added to 10% sodium hydroxide solution at 90°. On acidification *p*-aminobenzenestibinic acid is precipitated. C. HOLLINS.

Manufacture of water-soluble anthraquinone glucosides. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 298,674, 14.7.27).—Extracts of drugs containing anthraquinone glucosides (cascara sagrada, frangula), made, *e.g.*, by extraction of the drug with alcohol, evaporation, and dissolution of the residue in water, are purified by treatment with metallic hydroxides (*e.g.*, of aluminium, manganese, iron). On evaporation, pure mixtures of anthraquinone glucosides are obtained. B. FULLMAN.

Process for obtaining alkaloids. H. A. GILL. From N. V. CHEM. FABR. "DELTA" (B.P. 298,787, 4.11.27).—The finely-divided parts of the plants containing alkaloid are reduced to a thin pulp with water, milk of lime, etc., and the pulp is treated, in counter-current, in a column with steam, which removes the alkaloids volatile in steam. B. FULLMAN.

Process for obtaining alkaloids. G. W. F. F. KNOTH (U.S.P. 1,686,866, 9.10.28. Appl., 17.11.27. Ger., 11.10.26).—An aqueous alkaline pulp of the finely-divided plant is passed down a column, and the alkaloid is removed by an upward current of steam. F. G. CLARKE.

Refining of tobacco. ZIGARETTENFABR. LESMONA GES.M.B.H., and E. ROSENHOCH (B.P. 293,760, 10.7.28. Ger., 11.7.27).—The rib ends of the leaves are brushed to remove insects, larvæ, etc., and moistened slightly with sterilised water; after several hours the leaves are separated from one another, treated to remove foreign matter, moistened all over with sterilised water, and, after 10—16 hrs., treated in a conveying apparatus with a counter-current of ozone, the time of treatment and speed of the ozone current increasing with increasing nicotine content. L. A. COLES.

Manufacture of physiologically-active substances from ovaries, corpus luteum, and placenta. M. HARTMANN, Assr. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,690,932, 6.11.28. Appl., 14.4.24. Switz., 8.5.23).—See B.P. 226,372; B., 1925, 149.

Manufacture of derivatives of quinolinecarboxylic acids. K. MIESCHER, Assr. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,688,469, 23.10.28. Appl., 19.4.27. Switz., 30.4.26).—See B.P. 270,339; B., 1928, 730.

Refining of oils (B.P. 285,064).—See II.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Law of blackening of the photographic plate at low densities. E. A. BAKER (Proc. Roy. Soc. Edin., 1928, 48, 106—133; cf. B., 1925, 569; 1927, 461).—A theory is developed in which each grain of a photographic emulsion is assumed to act as an absorbing and emitting system, the rate of development of the grain depending on the particular sequence of absorptions and emissions which it has undergone. Comparison of the theoretical results obtained when the proportion of grains which suffer given sequences of absorptions and emissions is small, with experimental determinations of the variation of density with exposure time, indicates that the grain requires two successive absorptions or three absorptions with one intermediate emission to render it susceptible respectively to "full" or "partial" development. The development is "partial" when it is stopped at an early stage so that the grains developed are selected from those belonging to a small group of states of very similar properties. Development sufficient to blacken grains which have been reversed by short exposure to intense light is termed "full." The results apply over the whole range of wave-length tested, from 4800 Å. to below 2000 Å. A quantitative theory of the action gives good agreement with the facts relating to pre-exposure, inefficiency of short exposure to intense light, proportions of grains affected by different exposures in single-layer plates (for small grains), and influence of development on Schwarzschild's index. In the case of the "curves of constant blackening" there is a disagreement between theory and experiment for long exposures, which indicates that the "emission," though spontaneous, does not obey the unimolecular law. A new formula for the characteristic curve of a plate with uniform grains, successful over the low and moderate density region, is derived. A. B. MANNING.

PATENTS.

Varying the tones of photographic diazo prints. KALLE & Co. A.-G. (B.P. 287,063, 12.3.28. Ger., 11.3.27. Addn. to B.P. 280,593; B., 1928, 549).—Diazo prints made with the aid of titanium salts by the process of the prior patent are toned by exposure to moisture; e.g., the yellowish-browns obtained with 1:2:4-diazonaphtholsulphonic acid, dichlororesorcinol, and potassium titanium oxalate become dark brown by treatment with water vapour, moist air, or a damp sponge. C. HOLLINS.

Production of photo-prints and photo-copies. I. G. FARBENIND. A.-G. (B.P. 289,386, 25.4.28. Ger., 25.4.27. Addn. to B.P. 286,233; B., 1928, 466).—In the process of the prior patent (cf. also B.P. 286,736; B., 1928, 549) a salt of a substituted β -naphthaquinone-4-sulphonic acid is used as the light-sensitive substance, e.g., the 6-chloro- or 6-sulpho-derivative. C. HOLLINS.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Dynamite composition. N. G. JOHNSON and S. G. BAKER, JUN., Asss. to E. I. DU PONT DE NEMOURS & Co.

(U.S.P. 1,687,023, 9.10.28. Appl., 18.7.27).—A liquid explosive is mixed with ground popped corn; the composition has a low density. F. G. CLARKE.

XXIII.—SANITATION; WATER PURIFICATION.

"Natrolite" filter for water-softening. S. TYDÉN (Svensk Kem. Tidskr., 1928, 40, 250—256).—The water-softening properties of a filter of the zeolite type are studied. Analysis of the filtering medium showed it to consist mainly of the silicates of aluminium and sodium, corresponding to a formula $RO, 2R'O_3, 9SiO_2$, where R = Ca, Mg, K₂, Na₂ and R' = Fe, Al; about 1% of titania was also present. Experiments with water of hardness 9 showed that this value could be reduced to zero by passage of the water through the filter; regeneration was carried out by means of salt solution in the usual way. H. F. HARWOOD.

Toxic fluorine compounds. MARCOVITCH.—See VII. **Sulphur-oxidation of activated sludge.** AYYAR and others.—See XVI.

PATENTS.

Treatment of the higher phenols for germicidal purposes. V. LEONARD (B.P. 299,522, 29.7.27).—A germicidal solution contains 1 pt. of hexaresorcinol in a mixture of 300 pts. of glycerol and 700 pts. of water. A. COLES.

Chemical purification of waters with a high content of lime and magnesia. E. J. H. BUNCE (F.P. 624,101, 5.11.26. Tunis, 21.6.26).—The water is treated with the calculated quantity of a mixture of disodium hydrogen phosphate and sodium hydrogen carbonate. A. R. POWELL.

Water softening. P. E. LEISS (U.S.P. 1,689,036, 23.10.28. Appl., 1.11.23).—A mixture of barium carbonate and calcium hydroxide in water is added to the water to be softened. C. A. KING.

Conditioning or treating water in boilers etc. to prevent incrustation and corrosion. J. GORDON (B.P. 299,073—4, 17.5.27).—(A) Sodium carbonate or bicarbonate, or, preferably when the gauge pressure exceeds 210 lb./in.², (B) sodium phosphate, fluoride, or arsenate, is added to the water in the boilers to precipitate calcium, magnesium, etc. as a sludge, and further quantities are added as evaporation proceeds. Portions of the water are removed continuously from the boiler and returned again after filtration. L. A. COLES.

Means for preventing boiler-scale. J. PIERON (F.P. 623,440, 3.2.26).—A solution of zinc chloride or sulphate and extract of logwood is claimed. A. R. POWELL.

Means for preventing boiler-scale. W. LAZARUS (F.P. 623,808, 29.10.26. Ger., 20.11.25).—A paste of soot and water is claimed. A. R. POWELL.

Dealing with floating sludge in digestion chambers for sewage treatment. K. IMHOFF and F. FRIES (U.S.P. 1,690,682, 6.11.28. Appl., 25.8.24. Ger., 7.5.24).—See B.P. 230,733; B., 1925, 421.

Means [pads] for disinfecting and/or purifying the atmosphere. L. SCHOENFELD (B.P. 299,380, 20.7.27).

Water-softening apparatus. H. BARON. From W. NEUMANN (B.P. 298,997, 19.7.27).

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P

322/284