# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JULY 18 and 25, 1930.\*

## I.-GENERAL; PLANT; MACHINERY.

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Heat transmission. III. Flow of fluids at low velocities. A. P. COLBURN and O. A. HOUGEN (Ind. Eng. Chem., 1930, 22, 534—539).—When heat enters a slowly moving stream of liquid, convection currents are established and the fluid is defined as moving in thermal flow. As the effect of convection increases, "transitional" and then "turbulent" flow results. For flow of fluid vertically at low velocities heat-transmission coefficients are not affected by the velocity of the stream, but are influenced considerably by the temperature fall, viscosity, density, and coefficient of expansion of the fluid. This type of flow has been designated as "thermal turbulent." Empirical equations for the upward and downward vertical flow of water and general equations are deduced.

## C. A. KING.

Fuel economisers : their construction, materials, and recent developments. O. KUBALEK (J. Inst. Fuel, 1930, 3, 264—278).—The development of fuel economisers is outlined and the factors influencing economy are discussed. It is shown that high gas velocities, with suitable other conditions, tend to higher heat transference. It is found that gilled tubes with gills 1 in. high and of root thickness  $\frac{1}{3}$ — $\frac{1}{2}$  in. give heat transference per sq. ft. of external surface equal to that of plain tubes, but tend to accumulate external deposit more readily than plain tubes. The adoption of higher gas velocities lowers the tendency to deposition. A method is outlined for the solution of problems relating to heating surface and temperature changes in economisers. The specifications of the constructional material are discussed.

## H. E. BLAYDEN.

Chemical engineering memoranda. XI. Coils for steam-heated stills. C. H. BUTCHER (Ind. Chemist, 1930, 6, 199-200).—An alcohol-rectification still of capacity 10 gals. of product per hr. may require the evaporation of 50 gals. of alcohol-water mixture per hr. The total heat requirements, allowing for 5% radiation losses, will be 240,000 B.Th.U./hr. This can be supplied by 248 lb. of steam per hr. at a pressure of 5 lb. A table of steam coils capable of giving the necessary heat transmission is given. C. IRWIN.

Maintenance and working of simple types of autoclaves. A. W. C. HARRISON (Ind. Chemist, 1930, 6, 185-188).—Autoclaves may be classified as of the jacketed, plain, or revolving-drum type. The lastmentioned are used for the treatment of solids with gases under pressure and require special design for even heat distribution. Heating by steam or oil jackets is essential where the inside of the autoclave is enamelled or homogeneously lead-lined, but in many cases, with care in design, good results can be obtained from the simpler unjacketed type. Lengthy processes require larger units. If either the raw material or the product is difficult to keep in suspension the autoclave should be shallow. In any case the diameter should not exceed 3 ft. 6 in., owing to the stress on the lid joints. For higher temperatures and pressures cast steel rather than cast iron is desirable. A number of practical details in design are given, and a method of installing is described. A leather belt or chain drive is preferred. Types of autoclave suitable for use in the production of dye intermediates are indicated. C. IRWIN.

Conduction of heat. G. GREEN (Phil. Mag., 1930, [vii], 9, 241—260).—The methods of treating problems in the conduction of heat, and the results obtained therefrom, previously described (*ibid.*, 1927, [vii], 7, 784; 1928, [vii], 8, 701) are applied to the solution of definite problems. A. E. MITCHELL.

High-sensitivity absolute-humidity recorder. C. Z. ROSECRANS (Ind. Eng. Chem. [Anal.], 1930, 2, 129-134).—An apparatus is described in which the humidity of the air can be measured. Its mode of operation is based on the fact that the thermal conductivities of dry and moist air are different. By heating two wires, forming part of a Wheatstone bridge, one of which is surrounded by the sample of humid air, while the other is enclosed in a standard sample of air, an unbalanced condition is brought about. The arrangement which has been developed is suitable for recording humidities up to saturation within the temperature range -100° to 200°. At 25° between 0% and 10% the accuracy is  $\pm$  0.05% R.H. A commercial modification of the apparatus which gives continuous readings is in operation, but it has not yet been applied for general plant use.

H. INGLESON.

Preparation of air of known humidity and its application to the calibration of an absolutehumidity recorder. A. C. WALKER and E. J. ERNST, JUN. (Ind. Eng. Chem. [Anal.], 1930, 2, 134-138).— An apparatus is described in which air of constant humidity can be passed at a constant rate through an absolute-humidity recorder (cf. preceding abstract) over very long periods with variations of not more than 0.001% by vol. in the moisture content. The method adopted is to mix dry air in fixed proportions with nearly saturated air. In the drying of the air prior to mixing the use of magnesium perchlorate trihydrate is recommended in preference to phosphorus pentoxide, since it does not become gelatinous on absorbing relatively smal amounts of water. H. INGLESON.

\* The remainder of this set of Abstracts will appear in next week's issue.

Stability of technical emulsions. R. AUERBACH (Kolloid-Z., 1930, 51, 176—179).—Observations were made on 126 emulsions of different composition, and their separation on being set aside for a period of 5 months was observed. A method for preparing the emulsions is described. Fish train-oil emulsions in water stabilised with soda were found to have a maximal stability at medium concentrations of the alkali. The optimal concentration of alkali increases with the concentration of the emulsion. E. S. HEDGES.

Prevention of nuisance from fumes and dust from power stations. M. W. TRAVERS (J. Inst. Fuel, 1930, 3, 292—299).—The problems relating to the prevention of atmospheric pollution by fumes and dust are discussed. It is suggested that the solution of the problems depends on (a) pretreatment of fuel for ash removal, (b) the design of coal-burning appliances for burning coal in the minimum and controllable amount of air. H. E. BLAYDEN.

Coal burning and steam production. Wylde. Submerged combustion. HAMMOND.—See II. Organic fluorides as refrigerants. Midgley and Henne.— See III. Continuous methods of causticising. STEWART.—See VII. Consistometer. Bulkley and Bitner.—See XII. Equation of plastic flow. Williamson.—See XIII.

See also A., June, 688, Collodion films and filtration (ELFORD). 690, Sedimentry (BLOM). 717, Photothermometry (HENCKY and NEUBERT). 729, Thermoregulators (WING.).

#### PATENTS.

Heat-treatment furnaces [for enamel ware etc.]. CARBORUNDUM CO., LTD. (B.P. 308,613, 22.3.29. U.S., 23.3.28).—A furnace suitable for coating enamel ware comprises preheating and firing zones within one furnace structure and a drying zone preferably in another structure, but so situated that the goods may be conveyed through it by the return run of the conveyor from the other chambers. The goods are fired by heat from radiating combustion chambers in the sides and bottom of the furnace, separated from the goods by walls of silicon carbide or other good conductor ; the flow of gases therefrom is preferably under the preheating chamber and then to the dryer. B. M. VENABLES.

Drying ovens. S. L. GROOM (B.P. 328,624, 24.1.29).— A<sup>\*</sup><sub>A</sub> conveyor for plate-like objects which can only be handled on one side is described; provision is made for a 180° horizontal turn to be made so that both runs of the conveyor may be used for the same objects and the dryer thus made more compact.

## B. M. VENABLES.

Supplying heat required for effecting chemical reactions. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,635, 24.10.28).—Substances are liquefied and/ or kept liquid by secondary electric currents in the mass of the material which are induced from a primary circuit through a non-conducting envelope. Metallurgical processes and glass manufacture are excluded, but good conducting masses (metals, carbon, etc.) may be embedded in the mass and lifted out, if desired, when the melting is complete. [Stat. ref.] B. M. VENABLES. Dehydration [of solids]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,032, 15.2.29).—Solid substances containing water as water of hydration or crystallisation, or hydrogels or similar substances containing absorbed or adsorbed water, or solid substances with adhering water, are dehydrated by treatment with gaseous ammonia substantially at ordinary temperature, or while cooling. If necessary, the dehydrated product is heated in order to expel any ammonia that may have combined with it. S. K. TWEEDY.

Heating of liquids. L. L. RANSOM (B.P. 328,548, 24.12.28).—A tank system for the supply of liquid (e.g., hot water to laundries) intermittently and at constant temperature while taking a steady supply of heat is described. [Stat. ref.] B. M. VENABLES.

Evaporation of salt, sugar, and other solutions and the generation of steam. W. RUSIECKI (B.P. 304,670, 23.1.29).—Several forms of evaporator are claimed in which ebullition takes place in the upper part of a column of liquid which is tall enough to prevent ebullition in the heating zone in the lower part of the column. Forced circulation is not used.

B. M. VENABLES.

Apparatus for the transfer of heat. F. HERWORTH (B.P. 328,718, 21.2.29).—A heat exchanger of the bundle of tubes type is constructed so that the bundle may be removed transversely without disturbing the connexions for either fluid. The packing round the edges of the tube plates permits expansion, and is double with a vent to atmosphere, so that any leakage of one liquid is prevented from entering the other.

B. M. VENABLES.

Heat exchangers. W. H. OWEN (B.P. 328,230, 22.1.29).—A tubular heat exchanger is constructed in such a form that a number of them may be attached to the wall of an existing flue in tiers or ranks.

B. M. VENABLES.

Heat-exchange tubes. O. Y. IMRAY. From INTER-NAT. COMBUSTION ENG. CORP. (B.P. 328,557, 24.1.29).— The casting of tubes with heat-receiving extensions on one side, suitable for the walls of boiler combustion spaces, is described. B. M. VENABLES.

Increasing the heat exchange between gases and solids or molten materials. C. STILL (B.P. 328,640, 27.12.28).-In view of the fact that at temperatures above 500° the effect of radiation becomes important compared with convection, and that above 1000° it is of dominating importance, also of the fact that the relative radiating and absorbing powers of carbon dioxide and water vapour are high and of nitrogen and oxygen low, therefore to the air entering a heat exchanger, e.g., a regenerator for coke ovens, is added a small quantity of highly concentrated carbon dioxide. If the proportion of carbon dioxide is raised from the normal 0.03 to 1% and of water from 1 to 3%, the exchange of heat between the chequerwork and the gas at the hot end where the temperature may be 1200-1300° is raised threefold. The addition must not be overdone or the bad effects of dilution will cancel the benefits. Rich carbon dioxide may often be obtained from an adjacent B. M. VENABLES. by-product plant.

Regulating and maintaining heat-transfer [e.g., during vulcanisation]. GEN. CARBONIC CO. (B.P. 305,577, 24.1.29. U.S., 7.2.28).—The heat for vulcanisation (or other similar process) is supplied by a mixture of carbon dioxide (or other inert gas) and steam (or other heating fluid); the temperature attained corresponds to the partial pressure of the latter and will remain automatically constant if a definite mass of the former be first admitted, as determined by the pressure when cold. Examples are given of the use of the process for the vulcanisation of rubber tubes at 127° where internal pressures of 200 and of 100 lb./in.<sup>2</sup> are employed, and steam is assumed to be available at those pressures. B. M. VENABLES.

Cooling or freezing cream or other liquids. C. W. VOGT (B.P. 328,434, 23.4.29).-The liquid is sprayed upon the surface of a conical lump of refrigerant that evaporates without melting, e.g., solid carbon dioxide, the freezing being effected under a minor pressure in a closed chamber so that the atmosphere also becomes chilled and will be entrained in the solid particles produced. The major pressure to operate the spraying nozzle is generated by the evaporation of another mass of carbon dioxide above the fluid contained in a feed chamber, and the valve which permits fluid to pass through the spray is operated intermittently by a device similar to a windscreen wiper, which in turn is operated by gas from the refrigerating chamber. The intermittent spraying is stated to prevent the formation of a film of ice upon the refrigerant. B. M. VENABLES.

Refrigerants for use in vacuum refrigerating machines. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 310,546, 27.4.29. Ger., 27.4.28. Addn. to B.P. 310,474).—The refrigerants claimed are such as will dissolve in the motive liquid (*e.g.*, water) to a sufficient extent to lower the f.p. to about —10°. Examples are : ethyl ether or chloride, pentane. [Staf. ref.]

B. M. VENABLES.

Cooling of grinding mills. M. TRESCHOW (B.P. 328,634, 1.2.29).—The material is caused to come in contact, inside or outside the mill, with coils or jackets cooled by a fluid which is under suction to prevent harm due to any leakage. The cooling device rotates with the mill. B. M. VENABLES.

Mixing apparatus. A. B. and C. R. SMITH (B.P. 328,401, 22.3.29).—A pan which has a stirring shaft going through the bottom is provided with an upturned lip round the central hole, and any material which gets over the lip falls on to an umbrella-like guard forming the top of the chamber containing the gearing. The top of the non-rotating umbrella is spigoted into a skirt formed as a downward extension of the rotating boss carrying the stirrer. B. M. VENABLES.

Pneumatic separators. ALPINE A.-G. EISEN-GIESSEREI U. MASCHINENFABR., Assees. of ALPINE MASCHINEN-A.-G., and A. KUHR (B.P. 309,378, 8.4.29. Ger., 7.4.28).—In a separator of the type in which the fine dust is drawn upwards through a fan, the coarse being left behind, a supplementary separation of grits is effected after passing the fan in an annular space between two walls surrounding the fan. B. M. VENABLES. Separation of materials [by air]. I. L. BRAMWELL, C. W. H. HOLMES, and BIRTLEY IRON CO., LTD. (B.P. 328,283, 23.1.29).—The material (coal, ores, etc.) is allowed to slide down an adjustable staircase having the treads sloping sufficiently to prevent undue accumulation of material on them. A slot is left under the edge of each thread between it and the riser, and a current of air carries the finer material through these slots.

B. M. VENABLES.

Centrifugal dust separators or extractors. Η. SIMON, LTD., and G. WATTS (B.P. 328,652-3, 2.2.29).-The devices described comprise a number of small, conical cyclones situated in the middle one of three parallel compartments, the middle or inlet chamber serving also as a preliminary dust settler. The outer compartments receive cleaned air and concentrated dust, respectively, and it is necessary for satisfactory operation for the air pressure to be kept equal in these two; to effect this the ratio of the diameters of the axial air and dust outlets of the cyclones is 10:7 when the cyclones are 6-12 in. in diam. at the large end. In (A) the cyclones have vertical axes and the dust is removed by reciprocating grids in the chambers; in (B) the axes are horizontal and the dust falls out by gravity.

B. M. VENABLES.

Separation of liquids by distillation. DISTILLA-TION DYNAMQUE (B.P. 308,736, 27.3.29. Fr., 27.3.28).— The vapour from the top of a rectifying column is compressed, then condensed in a coil at the bottom of the column, thus supplying most of the heat for operation. Supplementary heat and regulation of the pressure are provided for by a minor supply of heat at the bottom or of cold at the top from external sources. Permanent gases are separated from the condensate in a settling chamber with regulating float and are withdrawn through a condenser, any condensed vapour from which and part also of the main condensate being returned to the tower as reflux liquid. When several constituents are to be separated a number of towers operating at different pressures are connected in series.

B. M. VENABLES.

[Tunnel] kilns. H. M. ROBERTSON (B.P. 306,813, 23.1.29. U.S., 25.2.28).—See U.S.P. 1,737,540; B., 1930, 222.

Mercury boiler. A. J. NERAD, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,759,133, 20.5.30. Appl., 11.7.27). —See B.P. 293,756; B., 1929, 495.

Pulverising apparatus. R. S. RILEY and O. CRAIG, ASSTS. to RILEY STOKER CORP. (U.S.P. 1,759,134, 20.5.30. Appl., 7.11.25).—See B.P. 260,952; B., 1928, 72.

Bearing-protecting devices for centrifugal separators. RAMESOHL & SCHMIDT A.-G., and C. SCHMITZ (B.P. 329,548, 20.6.29).

Mixing valves for liquids. F. P. BARBER (B.P. 329,839, 18.5.29).

Steam and gas mixture of high pressure (B.P. 328,339).—See II. Tunnel kilns (B.P. 311,316).— See VIII. Tunnel kilns (B.P. 328,265).—See IX. Porous bodies, filters, etc. (B.P. 328,273—4).—See XIV. Solidified globules from gelatinous substances (U.S.P. 1,746,543).—See XV.

## II.—FUEL; GAS; TAR; MINERAL OILS.

Fuels of Italy. M. G. LEVI (Chim. et Ind., 1930, 23, 811—824).—An outline is given of the work that is being carried out at the Institute of Industrial Chemistry at Milan on the utilisation of Italy's fuel resources. This includes a survey of the coal fields, and a study of the products obtained by the low-temperature carbonisation and by the hydrogenation of the different varieties of coal available. Some account is given of the occurrence and utilisation of petroleum, natural gas, and asphaltic and bituminous rocks in Italy. A. B. MANNING.

X-Ray stereoscopic examination of coal. П. A. N. WILSON (J. Inst. Fuel, 1930, 3, 342-346; cf. B., 1930, 540).—X-Ray stereoscopic methods applied to the localisation and detection of mineral layers and particles in coal are described. A double exposure is made with the X-ray bulb in two positions separated by a distance, D, symmetrical about the path of the central ray (which is made to pass through the centre of the fixed specimen and film) and on a line parallel to the film. If d is the distance between the two resultant images of any particle and H is the height of the bulb above the film, then the height of the particle above the film is h = Hd/(D-d). The use of stereoradiographs to indicate the probable manner of fracture of the coal due to mechanical treatment such as screening or drycleaning is discussed. H. E. BLAYDEN.

Developments and problems in coal cleaning. F. S. SINNATT and D. T. DAVIES (J. Inst. Fuel, 1930, 3, 326-336).-Statistics are given showing the development in coal cleaning by washing, dry-cleaning, and froth-flotation methods during 1927-1928 in Great Britain. There has been a rapid growth of dry-cleaning during the last seven years. Lessing's process (B., 1927, 866; 1928, 356) can now be applied for the separation of middlings of greater sp. gr. than the separating liquid, and can be modified for the efficient treatment of moist coals. Experiments have been made on the automatic control of the dirt bed in a British Baum washer capable of treating 21 tons of coal per hr. The optimum conditions of the dirt bed could be maintained for long periods despite alterations in feed and coal of varying dirt content. Slack (21.3% of ash ; 29.5% of sink in liquid of d 1.45) gave 3.7%of sink material in the clean coal and a loss of 0.6% of float material in the dirt. It is suggested that attention be paid to the flotation aspect of jig-washing and the size distribution of coal in the feed. The treatment and utilisation of dust are discussed with reference to blending and pulverised fuel combustion. It has been found that addition of reagents (lime, aluminium sulphate, sodium al uminate, sodium silicate, casein, glue, gelatin, or albumin) to washery waters facilitates settlement. Gelatin was very efficient and 0.2 lb. per ton of recovered solid was required. It was found possible to control flocculation and fractionally to precipitate the coal and dirt. Problems arising from the variation of coal properties in a given seam are discussed with reference to the effects of screening and cleaning on the properties of the cleaned coal and its ash. Samples for float-and-sink tests should not be crushed before examination. If the bulk sample is a screened grade

it may be reduced after mixing, but not crushed. The weights recommended for samples are 50, 30, 10, 1 lb. for maximum particle size of  $1\frac{1}{2}$ ,  $\frac{3}{4}$ ,  $\frac{3}{8}$ ,  $\frac{1}{8}$  in., respectively. Run-of-mine coal or screened slack is usually separated into one or more screen grades and the appropriate quantities quoted are used for float-and-sink tests. It is considered inadvisable to carry out ash analysis on the coal after float-and-sink tests. The elimination of sulphur from coal and the recovery of pyrites, for commercial use, from the washery rejects are discussed.

H. E. BLAYDEN. Blending [of coals for coke manufacture]. C. B. MARSON (Gas World, 1930, 92, Coking Sect., 47-50). -Experiments are described in which typical Durham coking, Northumberland high-volatile "non-coking," Scottish coking, Northumberland low-volatile coking, and Cumberland coking coals have been blended, by pairs, in varying proportions, and the mixtures coked. Examination of these coals showed the following results. (i) The Scottish and Cumberland coking and Northumberland "non-coking" coals, compared with the Durham coking coals, were characterised by greater amounts of moisture, volatile matter, oxygen, and hydrogen, by smaller amounts of carbon, and by lower agglutinating values ; attention is drawn to the low volatile and carbon contents of a Northumberland coking coal. (ii) When tested in the Gray-King assay at 600°, the Durham and Northumberland coking coals, compared with the other coals examined, gave greater coke and lower tar yields. Again, when the "straight " coals, together with the various blends, were coked in an experimental oven, taking a charge of 3 cwt., and with an average coking temperature of 920°, it was found that (a) the mechanical strength of a Durham coke as judged by the shatter index was considerably diminished by the addition of 40% of a Northumberland "non-coking" coal, but greater proportions of the latter did not give a proportionate lowering of the shatter index; (b) small additions of a Scottish to a Durham coking coal decreased considerably the shatter index of the Durham coke, but increasing additions effected an improvement, and the blend containing 80% of the Scottish and 20% of the Durham coal possessed a shatter index of the same order as the 100% Durham coke; (c) addition of a poor quality Cumberland coking to a Northumberland coking coal gave results similar to those of the Scottish-Durham series, an optimum blend being obtained with the 80% Cumberland-20% Northumberland blend; and (d) the blending of a second Northumberland "non-coking" coal with a Durham coking coal showed that 20% of the former may be added to the latter without causing a marked decrease in the shatter index. C. B. MARSON.

Design, construction, and operation of a modern coke-oven and by-product recovery plant, equipped for the manufacture of town gas. D. V. HOLLING-WORTH (Gas J., 1930, 190, 432-435).—The coke ovens and recovery plant at the Altham works of the Lancashire Foundry Coke Co. are described.

C. B. MARSON.

Metallurgical coke. G. T. PURVES (Fuel, 1930, 9, 229-233).—Some factors of importance in the produc-

tion of metallurgical coke are discussed. By suitable control of the quenching process the coke can be delivered at the blast furnace with an average moisture content of less than 1.5%. In general, the shatter index of the coke increases with an increase in the width of the oven up to a width of about 18 in., but decreases again if wider ovens are used. Compressing the charge lowers the shatter index. Rapid carbonising in very narrow ovens does not yield a good metallurgical coke. Fine grinding of the coal produces a marked improvement in the shatter index of the coke. In order to withstand crushing and abrading in the blast furnace a certain minimum strength is necessary. Little is known, however, of the factors which control the behaviour of the coke when it reaches the hearth, and opinion is divided as to whether a reactive or a hard-burnt coke is to be desired. A. B. MANNING.

Determination of the reducing power [of coke etc.] by Agde and Schmitt's method. W. J. MÜLLER and W. COURARD (Brennstoff-Chem., 1930, 11, 125—128; cf. Agde and Schmitt, B., 1927, 833, 834).—Some minor improvements have been made in the method, particularly in the production and measurement of the constant current of carbon dioxide and in the arrangement of the combustion tube, whereby greater accuracy has been attained. Successive determinations of the reducing power of active charcoal showed only slight variations. Larger fluctuations were observed with cokes, and the values tended to fall with continued passage of the carbon dioxide. A. B. MANNING.

Reactivity of carbon materials. Y. OSHIMA and Y. FURUDA (Fuel, 1930, 9, 200-212).-The rate of reduction of carbon dioxide by coke, charcoal, or graphite has been determined by passing a current of the gas over a sample of the material maintained at 900°, and the effect of varying the temperature, the area of the external surface of the sample, and the rate of passage of the gas has been studied. In most of the determinations the exit gas was analysed by an interferometer method. The initial temperature of reaction with coke and charcoal was dependent only on the maximum temperature of carbonisation. From a comparison of the reactivity of a cylinder of hightemperature coke with that of the same material finely powdered it is concluded that the surface layer of such a coke is very inactive compared with the underlying carbon ; similar experiments with a semi-coke, however, showed this to be nearly homogeneous. The higher reactivity of charcoal as compared with coke is ascribed mainly to its larger surface area per unit volume. The reactivity of a graphite rod increased as the surface became etched by the reaction. A decrease in reactivity during the measurement may be due to loss of total carbon surface, accumulation of ash, or, in the case of a semi-coke, shrinkage of the test piece. A relationship has been deduced between the effective surface areas of two cokes (A, A') and the rates of flow of carbon dioxide (V, V') necessary to give a definite amount of carbon monoxide in the resulting gaseous product, expressed by A' = AV'/V. If A and V are determined for a graphite rod of known surface area, then A' for a coke can be calculated from V'.

A. B. MANNING.

Coal burning [applied to steam production]. W. D. WYLDE (J. Inst. Fuel, 1930, 3, 241—255).—The principles of efficient combustion of solid and pulverised fuels for steam production are discussed. In the firing of slack, preheated air and the introduction of secondary air for the combustion of volatile matter tend to give increased efficiency. A suitable method for the addition of secondary air is to pass this air through channels in the brickwork of the arch of the furnace to the position of liberation of the volatiles. Ample combustion space is necessary for efficient combustion of high-volatile coals. The interpretation of the carbon dioxide content of exit gases with respect to losses due to excess air is discussed and represented graphically. H. E. BLAYDEN.

Influence of metallic and other substances on coal during carbonisation. J. G. KING and L. T. EDGCOMBE (Fuel, 1930, 9, 213-218).—The influence of the addition of about 5% by vol. of various metals or compounds on the carbonisation of a medium-caking bituminous coal has been studied. The Gray-King assay apparatus (B., 1929, 308) was used. Coke, carbon, or silica had no measurable effect on the yields or quality of the products. Aluminium, chromium, or stainless steel had only a very slight effect, and would therefore be suitable metals for the construction of apparatus for the study of carbonisation reactions up to 600°. Copper, nickel, cast iron, and steel became sulphided by the hydrogen sulphide evolved. Iron and copper promoted the formation of hydrogen at the expense of the saturated hydrocarbons. Pyrites and marcasite reduced both the strength of the coke and the yields of tar and gas. Sulphided cast iron, copper sulphide, and nickel sulphide had effects similar to pyrites and marcasite. Sulphided iron reduced the A. B. MANNING. tar yield by 17%.

Peat. III. Low-temperature carbonisation of peat. J. T. DONNELLY and J. REILLY. IV. Low-temperature carbonisation under various conditions. C. O'SULLIVAN and J. REILLY (Sci. Proc. Roy. Dublin Soc., 1930, 19, 365-376; 441-446; cf. B., 1927, 769). -III. Samples of air-dried Irish peat were carbonised at 550° and the resulting tars examined by the method of selective solvent extraction. The carbonisations were carried out in a current of coal gas in a retort holding a charge of 500 g. and similar in design to that described in B.P. 241,659 (B., 1926, 4), but heated electrically. The tar was collected in two receivers connected by a water-cooled condenser; the first receiver, which included a glass-wool trap for retaining tar fog, was maintained at 120°, and the second at -20°. A yield of 16.64% of tar (calc. on dry material) was obtained from a black peat. The tar contained  $5 \cdot 4\%$  of phenols. Higher acids of the nature of waxes were also present. The "resinols" amounted to 4.41% of the tar, and the "resinamines" to 0.88% (cf. Morgan and others, B., 1929, 156). The yields of acetic acid and ammonia (calc. as sulphate) were 22.6 lb. and 17.4 lb., respectively, per ton of air-dried peat. The yield of gas amounted to 8.61% of the dry peat, but was of poor quality. The black peat contained 10.47% of wax extractable by acetone. Carbonisation of a sample of brown peat gave similar results, but the yield of tar was somewhat smaller (14.41% on the dry peat).

IV. The addition of 11% of calcium carbonate to a sample of black peat had no effect on the yield of tar obtained by low-temperature carbonisation. Separate carbonisation of the wax, which amounted to about 10% of the peat, and of the wax-free peat, gave a total yield of  $15 \cdot 08\%$  of heavy tar and  $1 \cdot 06\%$  of light oil, compared with the values  $13 \cdot 82\%$  and  $1 \cdot 27\%$ , respectively, obtained by direct carbonisation of the original air-dried peat. More than 40% of the total tar obtained by the separate carbonisation of the constituents was derived from the wax, whilst the whole of the light oil was derived from the wax-free peat. A. B. MANNING.

Hydrogen cyanide in the gaseous products from the propagation of a zone of combustion through powdered coal. H. E. NEWALL, W. J. SKILLING, and F. S. SINNATT (Fuel, 1930, 9, 234-236).- A current of air was aspirated over a conical heap of powdered coal which had been lighted at the apex (cf. B., 1927, 802). Hydrogen cyanide was detected in the gaseous products of combustion of the coal, and was determined quantitatively by passing the aspirated gases through dilute aqueous potassium hydroxide and converting the potassium cyanide into Prussian-blue, the solution of which was compared with a standard. The amount of hydrogen cyanide formed varied from nil to 160 pts. per million pts. of air, the highest concentration corresponding with a total amount equal to 0.11% by wt. of the coal. Attention is directed to the possible danger from this source when fires occur in the goaf, in coal A. B. MANNING. stores, etc.

Comparison of fused silica, gold, and platinum linings for calorimeter bombs. O. K. BATES (Ind. Eng. Chem. [Anal.], 1930, 2, 162-164).-The heating values of a number of different coals as determined by the Emerson bomb lined with vitreosil agreed closely with those determined when using a platinum lining. The values obtained with the bomb lined with gold backed with silver agreed satisfactorily with those found when a vitreosil lining was used, for samples of low sulphur content, but were higher than these latter when the coals had a high content of sulphur. This additional heat is evolved by reactions in which the lining plays an important part. The sulphur content of coals determined from the bomb washing was lower in all three series than the value obtained by the peroxide method, but that found when vitreosil was employed was nearest to the correct value. The deficiency in all cases may be due to the production of sulphonic acids which are not determined as barium salts in the subsequent analysis. H. INGLESON.

Modern methods of tar distillation. E. WEISE (Petroleum, 1930, 26, 499-504, 577-582).—A survey of processes for intermittent, semi-continuous, and continuous distillation. Representative types of plant and their mode of operation are described with the aid of diagrams. Patent references are given of a selection of modern processes not described in the text; the majority of these are concerned with continuous distillation. D. G. MURDOCH.

Fractionation by means of solvents, and chemical study, of a commercial primary tar. I. UBALDINI

(Annali Chim. Appl., 1930, 20, 118—138).—Fractionation of primary tar by means of solvents is an indispensable preliminary to the chemical investigation. Ether and ordinary benzine allow of the subdivision of the tars into groups of substances with clearly-defined characteristics, and benzene and alcohol permit of further subsequent fractionation. Treatment with benzine may be used with advantage on the industrial scale to separate the complex resinous products from chemically simpler products, and may replace the initial distillation which, even when carried out in a current of steam and under reduced pressure, often gives rise to a large amount of residual pitch. T. H. POPE.

Trichloroethylene as a solvent for bituminous materials in routine analysis. J. B. HYATT (New Zealand J. Sci. Tech., 1930, 11, 418—419).—Trichloroethylene is a suitable solvent, being non-inflammable and less toxic than carbon disulphide. Hydrochloric acid produced by decomposition in sunlight is easily removed by shaking with water. C. W. GIBBY.

Determination of phenol in effluent waters. H. A. J. PIETERS (Chem. Weekblad, 1930, 27, 334—337). —The methods of removing substances which would interfere with the phenol determination are discussed for different cases. For a phenol content of about 1 g. per litre, the bromine method, in which excess of bromine is determined iodometrically, is very accurate; for concentrations down to 0.15 g. per litre, nephelometric determination of the tribromophenol formed is most satisfactory, whilst for lower concentrations, colorimetric determination, based on the Folin and Denis reagent, is best. S. I. LEVY.

Viscosity of pitch. H. A. J. PIETERS (Chem. Weekblad, 1930, 27, 287-288).—A series of determinations of viscosities at various temperatures by the penetration and rate of flow methods indicates that the viscosity and flow point are closely related; it is concluded that determination of one value, preferably the flow point, sufficiently characterises the other.

S. I. LEVY.

Behaviour of [wood] creosote at low temperatures. W. GRUNDMANN (Chem.-Ztg., 1930, 54, 353—354). —Six's maximum and minimum thermometers, in which beech-wood creosote is used, were found to be unreliable at temperatures below —30° owing to the frequent separation of gas bubbles. A series of samples of creosote were examined and tested for viscosity at low temperatures. In most cases this rapidly increased below —20° and only mixtures of creosote and alcohol retained fluidity well. The separation of gas at low temperatures was confirmed by cooling sealed ampoules of creosote to —40°. This also occurred with alcohol mixtures. C. IRWIN.

Influence of cylinder design on pinking. G. B. MAXWELL and R. V. WHEELER (Fuel, 1930, 9, 225-229). —It has been suggested that "pinking" in an internalcombustion engine is due to the initiation of a stationary pressure-wave in the burnt and burning gases, followed by a shock-wave when the accelerated flame impinges on the wall of the combustion space (cf. B., 1929, 878). This hypothesis has been confirmed by experiments on

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the influence of the shape and dimensions of the combustion space, and the position of the source of ignition, on the explosion of pentane-air mixtures. The formation of a stationary wave appears to depend mainly on the character of the reactions proceeding in the wake of the flame, which, with a given fuel, depends in turn on the mean flame temperature. These reactions, however, are modified by turbulence, or by the addition of traces of a "pro-knock," e.g., amyl nitrite. An "anti-knock," e.g., lead tetraethyl, seems to affect the establishment, not of the stationary wave, but of the subsequent shockwave. The stationary wave is modified by the shape of the reflecting surfaces at the end of the cylinder, a concave plate being the most and a convex plate the least effective in preventing the formation of the wave. The flame vibrations and acceleration which mark the setting up of the stationary wave begin when the flame has travelled about 75% of its full distance. A restriction, e.g., a wire gauze diaphragm or an annulus, introduced at this point in the cylinder either stopped or considerably diminished the pinking. A. B. MANNING.

Connexion between the I.P.T. gravity temperature-correction constants and the coefficient of expansion of petroleum oils. I. A. J. DUFF (J. Inst. Petroleum Tech., 1930, 16, 501-502).—The I.P.T. figure for gravity temperature-correction is not the correct one for use as a volumetric expansion coefficient. The real figure is obtained by dividing the sp. gr. temperature-correction figure by the sp. gr. and changing the sign. The mathematical derivation is given, and also the values for a number of sp. gr. at 60° F.

H. S. GARLICK.

Variation with temperature of the sp. heat of typical crude oils and their residues when topped to 100°. H. R. LANG and R. JESSEL (J. Inst. Petroleum Tech., 1930, 16, 476-500).—The sp. heats of a number of topped and crude oils were determined by the continuous-flow electrical method over a wide range of temperatures. With Pennsylvanian crude and topped oils there is a minimum at about 16°, the results agreeing closely below 20°. Above 25° the values for the topped oil lie on a straight line, the slope of which is 0.0095 per 1°. With Persian oil the deviation between the two curves is more marked above the minimum point (18°), owing to the greater volatility of the lighter fractions in the untopped oil. Above 25° the values lie on a straight line with a slope of 0.00089 per 1°. Below 15° no consistent results could be obtained, owing apparently to the unstable state of the wax in the oil. With Californian crude oil all values lie on a straight line the slope of which is 0.00095 per 1°. With Mid-Continent oil the sp. heat falls linearly with temperature down to 11°, with a sudden increase until 9° is reached, after which it continues to rise slowly. The curve for the topped oil is the same as with the crude oil above 24°, the slope of the straight part of the line being 0.00085per 1°. Tables of the total heat content of the oils are given, together with a formula for calculating the sp. heats, which gives values agreeing more closely with experimental results than those of previous observers.

H. S. GARLICK. Ageing of mineral oils. J. MARCUSSON and W. BAUERSCHÄFER (Chem.-Ztg., 1930, 54, 401).—An examination of the non-acidic products formed in turbine and transformer oils during long use is described. An old transformer oil freed from acidic substances  $(2 \cdot 4\%)$ contained  $84 \cdot 3\%$  C,  $12 \cdot 4\%$  H,  $0 \cdot 37\%$  S, and  $2 \cdot 6\%$  O. Hydroxyl and carbonyl compounds were detected and 6% of petroleum resin (mean mol. wt. 293, acetyl value  $40 \cdot 2$ ) was isolated. An oil heated for a long time at  $120^{\circ}$  was transformed into a hard asphalt, m.p. 56°, containing  $0 \cdot 9\%$  of acid,  $4 \cdot 9\%$  of petroleum resin, and  $31 \cdot 2\%$  of asphaltene. The acetyl value,  $1 \cdot 4$ , was increased to  $8 \cdot 0$  after reduction of the carbonyl compounds; a process for this is described which gives a measure of the acidic and neutral decomposition products of an oil. E. H. SHARPLES.

Determination of the coumarone resin content of the solvent naphtha fraction. I. I. BURDA (Ukraine Chem. J., 1929, 4, [Tech.], 257—258).—To 100 c.c. of solvent naphtha in a long separating funnel 2 c.c. of concentrated sulphuric acid are added drop by drop with shaking. The acid layer is then run off and the remainder is steam-distilled, when coumarone resins remain chiefly in the residue; the residue obtained by redistilling the distillate is added to the first residue, and, after dehydration, the yield of coumarone resins is weighed. R. TRUSZKOWSKI.

Higher hydrocarbons from water-gas. BERL and JÜNGLING. Synthetic alcohols from water-gas. NATTA and STRADA. Solvents from natural gas. CLARK.—See III. Pressure-synthesis operations. ANON. Synthetic ammonia. CLAUDE.—See VII. Determination of montan wax in beeswax. BUCHNER.—See XII. Coumarone resins. BURDA.— See XIII. Egg-killing washes. STANILAND and others.—See XVI. Coal mine explosives. NAOUM and BERTHMANN.—See XXII.

See also A., June, 715, Catalytic decomposition of methane (FISCHER and BAHR). Equilibrium  $6CO_2 + C_6H_6 = 12CO + 3H_2$  (BAHR). 735, Catalytic oxidation of methane by sulphuric acid (FISCHER and BAHR). 786, Piperidine derivatives (BAILEY and MCELVAIN). 788, Bases in the kerosene distillate of California petroleum (POTH and others).

## PATENTS.

Briquetting of fuel. C. A. BURNE (B.P. 328,082, 7.3.29).—Coal dust is mixed with a relatively small quantity  $(2-2\frac{1}{2}\%)$  of gluten or gluten-bearing material, *e.g.*, ground rice or meal offals, and  $\frac{3}{4}$ —1% of sodium silicate, in the form of a dilute aqueous solution, and the mixture is briquetted. The briquettes are dried by being heated at 65° for about 6 hrs. A. B. MANNING.

Gas generators. HUMPHREYS & GLASGOW, LTD., Assees. of J. S. HAUG (B.P. 312,162, 14.5.29. U.S., 21.5.28).—A cylindrical producer is provided with feeding means which deposit the finer fuel at the periphery and the coarser fuel at the centre, the air and/or steam being admitted only to an annular zone at the bottom of the fuel bed, whence sufficient finds its way to the centre through the coarser particles to secure the desired rate of combustion there. The rotating grate has a flattened, imperforate top provided with curved blades which displace the ash outwardly towards the 650

peripheral part of the bottom of the fuel bed, where it is discharged. A. B. MANNING.

Producing a steam and gas mixture of high pressure. N. A. KALABIN (B.P. 328,339, 1.2.29).— Powdered or liquid fuel is supplied through a minor chamber into a larger chamber containing water. Only enough air (under pressure) is supplied to the minor chamber to effect gasification and to cause the walls to glow, the secondary air being added to the water chamber. The incoming water may be preheated or the outgoing mixture superheated in passages surrounding the minor chamber. Means are provided for the regulation and agitation of the water. B. M. VENABLES.

Washing cyanide compounds, ammonia, and sulphuretted hydrogen from gases. A. C. BECKER and W. BERTELSMANN (B.P. 328,738, 18.3.29).—In such washing processes in which solutions containing ferrous oxide are employed, additional ammonia, obtained by melting ammonium sulphate, is used for washing out the hydrogen sulphide. The ammonium bisulphate produced is used for treating the used washing lye. W. J. WRIGHT.

Extraction of hydrocarbons from gases. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,008, 15.1.29).—Coal gases etc. are treated with sulphuric acid of at least 70% concentration, and preferably in two or more stages with acid of successively higher concentrations (up to 95%), and the benzene hydrocarbons are absorbed from the purified and dry gas in known manner, *e.g.*, by means of activated charcoal.

A. B. MANNING.

Production of aqueous [bitumen pitch] dispersions. L. KIRSCHBRAUN (U.S.P. 1,734,437, 5.11.29. Appl., 22.7.27).—Solid or semi-solid bitumen pitch or asphalt is dispersed by agitation in a suspension of bentonite or ball clay stabilised by the addition of a protective colloid, *e.g.*, alkaline casein solution. The resulting dispersion is unbroken by electrolytes and, on drying, yields a product having an irreversible film.

A. R. POWELL.

Manufacture of lubricating oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,721, 10.12.28).— The flash point and viscosity of lubricating oils, especially used lubricating oils, are raised by treating the oils at high temperatures, under vacuum, with inert gases or with the vapours of liquids of low b.p. Wet steam or carbon dioxide containing benzene vapour is suitable.

T. A. SMITH.

Treating and purifying hydrocarbon lubricating oils. W. M. STRATFORD, Assr. to TEXAS Co. (U.S.P. 1,738,330, 3.12.29. Appl., 14.6.26).—Lubricating oil fractions are agitated at normal temperature with sulphuric acid and after separation of the sludge and neutralisation are distilled under vacuum in the presence of alkaline material. Caustic soda may be added as a solution or solid, or anhydrous soda ash may be used. T. A. SMITH.

Treatment of hydrocarbon gases. ANGLO-PERSIAN OIL Co., LTD., A. E. DUNSTAN, and R. V. WHEELER (B.P. 327,715, 5.10.28).—Paraffin-containing gases, such as natural gas, wild gases from stills, and gases from cracking plants and low-temperature retorts, are gradually heated to a temperature below that at which decomposition of the olefines and paraffins takes place. The temperature is then raised to 550—1250°, according to the nature of the gases used, and the gases are expanded and cooled. The free carbon produced is deposited and the aromatic substances formed are condensed. The reaction may be carried out in the presence of catalysts, such as nickel powder. The residual gas may be further decomposed to produce carbon-black, or mixed with oxygen-containing gases, such as carbon monoxide or dioxide, and used for the production of oxygenated hydrocarbons.

Т. А. Ѕмітн.

Apparatus for production of active carbon. J. N. A. SAUER, Assr. to N.V. ALGEM. NORIT MAATSCH. (U.S.P. 1,759,138, 20.5.30. Appl., 6.4.26. U.K., 9.8.23). —See B.P. 206,862; B., 1924, 548.

Process and apparatus for destructive distillation. R. H. CROZIER (U.S.P. 1,759,821, 20.5.30. Appl., 15.7.29. U.K., 18.5.28).—See B.P. 319,224; B., 1930, 179.

Purification of hydrocarbon compounds. Process and apparatus for treating hydrocarbons. T. T. GRAY, ASST. to GRAY PROCESSES CORP. (U.S.P. 1,759,812 --3, 20.5.30. Appl., [A] 26.9.23, [B] 24.3.25. Can., [B] 18.9.24).—See B.P. 222,481 and 249,871; B., 1926, 230; 1927, 290.

Jig washers [for washing coal etc.]. W., R. H., and W. REID (B.P. 329,488, 1.5.29).

Burners for pulverised or gaseous fuel. Soc. ANON. DES APPAREILS DE MANUTENTION ET FOURS STEIN (B.P. 316,667, 19.7.29. Fr., 2.8.28).

Diffuser for obtaining gaseous combustible mixtures. J. W. PENICUD (B.P. 328,493, 18.6.29).

Acetylene generator [for use with internalcombustion engines]. C. DE LA ROCHETTE (B.P. 307,401, 6.3.29. Fr., 6.3.28).

Heat exchange between gases and solids. (B.P. 328,640). Separation of materials (B.P. 328,283).— See I. Modifying isocolloid materials (B.P. 321,689, 321,691—4, 321,699, 321,722—7).—See III. Ammonium sulphate (B.P. 327,488). Heating hydrogen for hydrogenation (B.P. 329,045).—See VII. Cellular aggregate (U.S.P. 1,746,860). Bituminous masses (B.P. 310,923). Stones, road surfaces, etc. (B.P. 328,969). Road-making (B.P. 307,751).—See IX.

## **III.—ORGANIC INTERMEDIATES.**

Synthesis of the higher hydrocarbons from water-gas at atmospheric pressure. E. BERL and K. JÜNGLING (Z. angew. Chem., 1930, 43, 435—440).— The authors have repeated Fischer's experiments on the synthesis of hydrocarbons from water-gas, using various catalysts. These were prepared by reduction of various mixtures of oxides of iron, copper, zinc, manganese, cobalt, and copper, with, in some cases, addition of potassium carbonate. The temperature was usually 290°. Details of yields are given with elementary analyses of the more and the less volatile liquid products. Iron catalysts were found to oxidise carbon monoxide preferentially, cobalt catalysts to oxidise hydrogen first; cobalt catalysts are the more active, and the presence of copper is advantageous. The activity of the catalyst varies greatly with the method of preparation. It is best to prepare the catalyst by reduction of nitrates with diluted hydrogen or water-gas at as low a temperature as possible. Cobalt can be regenerated by heating in hydrogen, but iron catalysts should be heated in air to 400° and then treated with water-gas at the working temperature. The degree of saturation of the hydrocarbons produced increases with the mol. wt. The greater the hydrogenating activity of the catalyst the less is the yield of unsaturated compounds, aldehydes, and acids. C. IRWIN.

Synthesis from water-gas of alcohols higher than methyl alcohol. G. NATTA and M. STRADA (Giorn. Chim. Ind. Appl., 1930, 12, 169-174).-Previous processes for the high-pressure synthesis of oxygenated organic products from the components of water-gas by means of catalysts containing strong alkalis yield highly complex mixtures containing, besides methyl and other alcohols, considerable quantities of hydrocarbons, acids, aldehydes, ketones, esters, etc. The experiments now described show that, in presence of catalysts containing zinc oxide (obtained in a very active form by calcining certain smithsonite minerals at low temperatures) and certain alkali-metal (potassium, rubidium, cæsium) compounds, it is possible, at about 400° and under high pressures, to convert mixtures of carbon monoxide and hydrogen completely into mixtures of liquid, oxygenated organic compounds consisting principally of saturated aliphatic alcohols. Contrary to what has been stated previously, the formation of alcohols higher than methyl alcohol does not require the presence of free metals and oxides of the groups IV-VIII of the periodic system. The complete absence of such metals and oxides is, indeed, advisable in order that formation of methane and other undesirable secondary reactions may be avoided. The formation of the higher alcohols takes place through the successive formation of methyl alcohol and alkali methoxide, and the addition of carbon monoxide with formation of alkali salts of fatty acids, with subsequent decomposition of these and hydrogenation of the resulting products. The intermediate alkali salts of the fatty acids have been identified on the catalysts and may with advantage be added to the latter, as they confer high catalytic activity, which remains constant over T. H. POPE. ong periods.

High-boiling solvents from natural-gas pentanes. L. H. CLARK (Ind. Eng. Chem., 1930, 22, 439–443).— Amyl acetate for use as lacquer solvent is manufactured from pentane derived from natural gas. A fraction, 95% of which distils between 28° and 39°, consists chiefly of *n*- and *iso*-pentane. It is dried, and the vapour mixed with chlorine and passed to a pipe-still followed by four fractionating columns. These serve to separate hydrochloric acid, unchlorinated pentanes, and the mono- and di-chloro-derivatives. A mixture of six isomeric monochloropentanes is hydrolysed by digestion with 30% caustic soda and a catalyst. The vapour from the digesters consisting of amyl alcohols, amyl chlorides, and amylenes is fractionated and the wet alcohols are dehydrated. The low-boiling tertiary alcohol is removed and the mixture of the other five isomerides acetylated in a kettle at 125°. The crude ester is neutralised with soda ash and rectified. The main difficulty in the development of this process has been the design of the coolers for chlorinated pentanes. These are now of iron, which is not corroded so long as the gas is perfectly dry and cooled by open watersprays. Amongst the by-products of the process are amylenes, diamylene, and amyl ether. C. IRWIN.

Detection of small quantities of methyl alcohol in ethyl alcohol. O. MOHR (Mikrochem., 1930, 8, 154-158).-The alcohol is diluted with water so that the mixture contains about 5% of alcohol, and 5 c.c. of the mixture are treated with 0.3 c.c. of 85% phosphoric acid and 2 c.c. of 3% permanganate solution. After 10 min. 1 c.c. of 10% oxalic acid solution and, after a further 2 min., 10-12 drops of sulphuric acid  $(d \ 1.84)$ are added and the resulting aldehydes distilled into a test-tube cooled in ice and containing 5 c.c. of a 2.5% solution of ammonium chloride. After 30 min. the chloride solution, which contains hexamethylenetetramine corresponding with the methyl alcohol in the original alcohol mixture, is evaporated to dryness to expel acetaldehyde. The residue is dissolved in 5 c.c. of water, a few drops of sulphuric acid are added, and the liberated formaldehyde is distilled into 0.5-1 c.c. of a 0.5% solution of dimethyldihydroresorcinol (methone) made feebly alkaline with 1-2 drops of 10% sodium hydroxide solution. The distillate is then rendered acid with acetic acid and a drop placed on a microscope slide, which is set aside for some time; the formation of characteristic needles of methylenedimethone indicates the presence of methyl alcohol in the original alcohol. The test is sensitive to 0.05% of methyl alcohol. A. R. POWELL.

Industrial solvents : ethylene dichloride, dichloroethyl ether, and isopropyl ether. H. R. FIFE and E. W. REID (Ind. Eng. Chem., 1930, 22, 513—515).—The physical properties of these three solvents are tabulated and graphs are given showing their sp. gr. at various temperatures, vapour pressures, and comparative rates of evaporation in still and in circulating air. The solubilities of some fats, oils, waxes, resins, balsams, and dyes in the solvents are tabulated. H. INGLESON.

Organic fluorides as refrigerants. T. MIDGLEY, JUN., and A. L. HENNE (Ind. Eng. Chem., 1930, 22, 542-545).-Dichlorodifluoromethane, b.p. -30°, is quite stable at 175°, at which temperature when pure and dry it does not corrode steel, aluminium, copper, or tin. The liquid is non-corrosive to all constructional metals at atmospheric temperature. Experiments with animals show the gas to be, for practical purposes, non-toxic; it is also non-inflammable. It is prepared in 88% yield by bringing pure and dry antimony trifluoride into contact with carbon tetrachloride in the presence of a small amount of antimony pentachloride. It is economical to work at 60 lb. pressure. If any of the monofluoro-compound is formed, this may be reworked and the yield of the diffuoro-compound raised to 98%. The plant necessary consists of an autoclave, fractionating column, and dephlegmator. C. IRWIN.

[Methyl alcohol] pressure-synthesis. ANON.— See VII. Xylose. EMLEY.—See XVII. Determination of aldehydes in alcohol. WOJCIECHOWSKA-STRUSZYŃSKA.—See XVIII.

See also A., June, 680, Physical methods of separating constant-boiling mixtures (SUNIER and ROSEN-BLUM). 698, Vapour-phase esterification of acetic acid by ethyl alcohol (FROLICH and others). 711, Coupling of diazonium salts with phenols (CONANT and PETERSON). 788, Bases in the kerosene distillate of California petroleum (POTH and others).

## PATENTS.

Modifying the physical properties of (A) fatty oils, (B) organic isocolloids, containing unsaturated high-molecular acids. (c) Manufacture of bases for water-fast varnishes and lacquers. (D) Production of rubber substitutes. (E) Modifying the physical properties of resins. L. AUER (B.P. [A] 321,689 and (B-E) 321,724-7, 4.5.28. [A] Addn. to B.P. 287,943; B., 1928, 398).-(A) The process of the prior patent gives particularly useful products from fatty oils containing high-molecular unsaturated acids or esters, e.g., tung oil, linseed oil, castor oil, or fish oil. The best "modifying" agents are those which by Frumkin's method (A., 1924, ii, 462) impart a negative surface charge to water in which they are dispersed (e.g., sodium hydrogen sulphite). (B) The "modifying" effect is observed also with all substances comprising within the molecule an organic residue and an acidic inorganic residue (e.g., trichloroacetic acid, triphenylmethyl chloride, 2:5-dichlorobenzenesulphonic acid, triphenyl phosphate, diphenylamine hydrochloride, p-nitroacetanilide); these may be used in conjunction with peroxides, carbonates, sulphites, sulphides, and other agents which generate gas during the process, and preferably the modifying agents are introduced in the colloidal condition. (c) For the production of water-fast varnish bases a "modifying" agent insoluble in water is used with linseed oil or other organic isocolloids containing unsaturated high-molecular acids; the agent may be produced in situ, and the product may be vulcanised. Resins may be used alone or with the fatty oil. (D) For the production of rubber substitutes the "modifying" agents should be such as develop during the process gases other than oxygen, e.g., carbonates, sulphites, sulphides, etc., and may be produced in situ; the starting material is preferably a drying oil, castor oil, or a fish oil. (E) American, French, or Spanish rosins are converted into liquids suitable for lacquers by heating under reduced pressure with "modifying" agents, particularly ammonium iodide, zinc carbonate, magnesium sulphate, or chloride, sodium sulphide, barium thiocyanate, etc. with the addition of a little formic acid. C. HOLLINS.

Modifying the physical properties of organic isocolloid materials, by treatment of their emulsions. L. AUER (B.P. 321,692, 9.5.28).—The starting material (linseed oil, tung oil, rape oil) is emulsified in water and treated with "modifying" agents (cf. B.P. 287,943; B., 1928, 398; also preceding abstracts), with or without a gas-treatment. Preferably the emulsion is maintained alkaline by addition of ammonia as required. The products during or after formation may be vulcanised. C. HOLLINS.

Vulcanisation or sulphurisation of organic isocolloids. L. AUER and N. STRACHOVSKY (B.P. 321,699, 13.6.28).—Organic isocolloids other than rubber are mixed with the usual vulcanising agents and are then vulcanised by treatment with steam, with or without the application of heat and pressure. The process is also applicable to rubber and other isocolloids which have been treated with "modifying" agents (cf. B.P. 287,943; B., 1928, 398; and preceding abstracts). C. HOLLINS.

Vulcanisation or sulphurisation of substances capable of vulcanisation or sulphurisation. Sulphurisation of organic isocolloids which have been treated with modifying agents. P. STAMBERGER and L. AUER (B.P. 321,693 and Addn. B.P. 321,694, 10.5.28).—The starting materials, which in (B) are organic unsaturated isocolloids modified, *e.g.*, by the process of B.P. 287,943 (B., 1928, 398), are vulcanised or sulphurised to the degree necessary to complete the first stage of vulcanisation, *i.e.*, distribution of the sulphur (probably as an additive compound) evenly through the mass, and the process is then completed by treatment with water, preferably by emulsification. Accelerators may be used. C. HOLLINS.

Manufacture of new saponaceous materials from organic isocolloids containing unsaturated high-molecular acids ; vulcanisation of the products. L. AUER (B.P. 321,691, 321,722-3, 8.5.28).-(A) Alkali metals or their oxides or hydroxides are dispersed at 250-300° in substantially anhydrous isocolloids containing unsaturated high-molecular acids or their esters (linseed oil, fish oil, sunflower oil, rosin, castor oil, olive oil) to give soaps suitable also for use as binders in varnishes and linoleums, and as rubber substitutes, which can be vulcanised. The glycerin is apparently retained and acts as plasticiser. Barium peroxide is added with advantage. (c) The process is performed under pressure. (B) The products are vulcanised. C. HOLLINS.

Manufacture of acetaldehyde. W. O. HERRMANN and H. DEUTSCH, Assis. to Consort. F. ELEKTROCHEM. IND. (U.S.P. 1,714,783, 28.5.29. Appl., 3.4.28. Ger., 4.4.27).—See B.P. 288,213; B., 1930, 275.

Preparation of cyclic ketonic compounds. H. GREUNE, ASST. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,111, 20.5.30. Appl., 5.7.27. Ger., 8.7.26).—See B.P. 274,095; B., 1928, 922.

Preparation of 6-chloro-2-nitro-1-methylbenzene-4-sulphonic acid. F. HENLE and B. VOSSEN, ASSIS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,554, 20.5.30. Appl., 24.8.25. Ger., 1.9.24).—See F.P. 434,402; B., 1927, 276.

Production of 1[3]-phenylbenzanthrone compounds. W. TRAUTNER, B. STEIN, and R. BERLINER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,519, 4.3.30. Appl., 11.7.27. Ger., 23.11.25).—See B.P. 297,129; B., 1928, 847.

## IV.—DYESTUFFS.

Azotriphenylcarbinol dyes. R. N. SEN and (the late) A. K. SEN (J. Indian Chem. Soc., 1930, 7, 151-156. Cf. Green and Sen, B., 1912, 579; Sen and Sett, B., 1924, 211).-The leuco-azotriphenylmethane dyes obtained by condensing p-azobenzaldehyde-oo'-disulphonic acid or p'-salicylazobenzaldehyde-o-sulphonic acid with phenols are lighter in shade when dyed on wool than the azobenzaldehydes themselves, but the azotriphenylcarbinols produced by oxidation are slightly deeper. A methyl group meta to the hydroxyl group decreases the colour and dyeing properties ; an additional hydroxyl group para to the azo group (salicylazotriphenylcarbinols) is almost without influence. The following are described [shades given are on wool (i) before and (ii) after chroming]. From azobenzaldehydedisulphonic acid and salicylic acid, methane, (i) vellow, (ii) violet-black, carbinol, (i) red; 2-hydroxy-3-toluic acid, methane, (i) pink, (ii) black, carbinol, (i) bluish-red; 3-hydroxy-4-toluic acid, methane, (i) yellow, (ii) black, carbinol, (i) light chocolate; from salicylazobenzaldehydesulphonic acid: 2-hydroxy-3-toluic acid, methane, (i) yellow, (ii) black, carbinol, (i) red; 3-hydroxy-4-toluic acid, methane, (i) yellow, (ii) black, carbinol, (i) chocolate. H. A. PIGGOTT.

Black, chromable disazo dye derived from aminosulphosalicylic acid. V. G. GULINOV (Ukraine Chem. J., 1929, 4, [Tech.], 267-273).-A black dye for wool is prepared in the following way. 4-Nitroso-3-sulphosalicylic acid is reduced by sulphite to yield a mixture containing 62% of 4-amino-3-sulphosalicylic acid, together with, amongst other substances, 4-amino-3:5-disulphosalicylic acid. The crude product is diazotised and coupled with x-naphthylamine, and the disazo compound is again diazotised and coupled with β-naphthol. The final product is a mixture of two black dyes, the one containing disulphosalicylic acid being the more soluble in water and separable from the other by extraction with boiling water. These dyes should be treated with chromium solution after deposition on wool. Tables giving the fastness of these dyes to various factors are given. R. TRUSZKOWSKI.

## Solvents. FIFE and REID.—See III.

See also A., June, 694, Colloid-chemical colour changes in organic dyes (OSTWALD and RUDOLPH). 780, Colouring matter from saffron (KARRER and HELFENSTEIN). 793, Formation of pyrrole-blacks (QUILICO and FRERI).

#### PATENTS.

Manufacture of sulphur dyes. G. KALISCHER and H. RITTER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,261, 20.5.30. Appl., 26.6.28. Ger., 11.7.27).-See B.P. 319,860; B., 1930, 136.

Sulphur dyes. E. KRAMER, B. BOLLWEG, and L. ZEH, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,264, 20.5.30. Appl., 21.6.28. Ger., 31.8.27).-See B.P. 317,139; B., 1929, 890.

Dyes of the triphenylmethane series. K. SCHMIDT, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,113, 4.3.30. Appl., 20.10.28. Ger., 21.4.27).—See B.P. 324,966; B., 1930, 411.

Vat dyes. H. NERESHEIMER and W. SCHNEIDER, Assrs. to Gen. Aniline Works, Inc. (U.S.P. 1,749,501, 4.3.30. Appl., 9.11.27. Ger., 15.11.26).—See B.P. 280,492; B., 1928, 667.

Preparation of vat dyes of the anthracene series containing nitrogen. K. WILKE and J. STOCK, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,249, 20.5.30. Appl., 21.6.26. Ger., 2.7.25).—See B.P. 254,742; B., 1927, 550.

Acylaminodibenzanthrones and products resulting therefrom by treatment with alkylating agents. P. NAWIASKY, ASST. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,272, 20.5.30. Appl., 10.7.25. Ger., 11.8.24).-See F.P. 598,752; B., 1926, 311.

Manufacture of azo dyes. H. WAGNER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,681, 4.3.30. Appl., 23.12.25. Ger., 24.2.22).—See B.P. 201,712; B., 1923, 968 A.

Production of trisazo dyes. H. Schweitzer, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,513, 4.3.30. Appl., 4.2.27. Ger., 26.2.26).—See B.P. 287,232; B., 1928, 399.

Aminodiarylsulphonepyrazolone-azo dyes. H. SCHWEITZER and W. NEELMEIER, Assrs. to GEN. ANIL-INE WORKS, INC. (U.S.P. 1,749,677, 4.3.30. Appl., 30.3.27. Ger., 8.1.25).—See B.P. 245,765; B., 1926, 734.

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

Study of the fibroin from silk in the isoelectric region. M. HARRIS and T. B. JOHNSON (Ind. Eng. Chem., 1930, 22, 539-542).-Stable colloidal solutions containing up to 5% of fibroin were prepared by treating highly purified, degummed silk with 50% lithium bromide or 70% calcium thiocyanate solutions at 80° and dialysing for about two weeks, after addition of a few drops of toluene to prevent bacterial action. Using a 1 or 2% solution, determinations of the isoelectric point by means of measurements of solubility, viscosity, electrophoresis, and precipitation of the dyed fibroin gave results varying between  $p_{\rm H} 2.0$  and 2.3.

F. R. Ennos.

Manufacture of chemical cotton. W. D. MUNSON (Ind. Eng. Chem., 1930, 22, 467-471).-The purification of cotton linters and its conversion into cotton cellulose are described. The value of linters for production of chemical cotton depends not only on the percentage of pure cellulose present, but also on the quantity and character of the foreign matter. Most of the chemical cotton is now produced from second-cut linters, corresponding to U.S. Standard grade Nos. 6 and 7, which are made by de-linting the seeds after removal of the longer F. R. ENNOS. fibre in the first cut.

Synthetic fibre industry of America. C. E. MULLIN (Ind. Eng. Chem., 1930, 22, 461-467).-A short description of the methods of manufacture of the different types of synthetic fibre, with statistics of their production F. R. Ennos. in the United States.

Cellulose as it is completely revealed by X-rays. Special application to growth and classification of cotton, structure of wood, and manufacture of 654

**rayon.** G. L. CLARK (Ind. Eng. Chem., 1930, 22, 474– 487).—An account is given of the fundamental structure of cellulose as deduced from X-ray diffraction spectra, employing certain improvements in technique. The method is also applied to show the various stages in the growth of cotton fibres, as a means of classifying the different grades of cotton, to obtain information as to wood structure, and to serve as a control in the manufacturing processes for viscose silk. F. R. ENNOS.

Determination of the degree of purity of cellulose fibres. M. FREIBERGER [with Z. VON VASS and L. HÖNIG] (J. Soc. Dyers and Col., 1930, 46, 111-118).-The reducing power of cellulose is utilised as a means of determining its purity or degree of degradation, and is determined by extracting the soluble reducing constituents with caustic soda ( $d \ 1.0375$ ) and oxidising the extract with potassium ferricyanide, or, preferably, by digesting the cellulose directly with a caustic alkaline solution of potassium ferricyanide and determining the ferrocyanide formed. In the first method several successive extractions with caustic soda must be made to ensure complete extraction of the reducing constituents. The reducing power of the cellulose is termed the ferricyanide number (=g. of potassium ferricyanide reduced by 100 g. of the cellulose); the oxygen number is the equivalent quantity (in g.) of oxygen per 100,000 g. of the cellulose. In the first method 1 g. of cellulose cut up into small pieces is boiled for 1/2 hr. with 150 c.c. of caustic soda ( $d \ 1.0375$ ) in an Erlenmeyer flask provided with a ball stopper. The contents are then cooled, filtered through a Jena fritted-glass filter (No. 1), the filtrate is diluted to 250 c.c., and the residue similarly extracted until the extract is free from reducing properties. 10 C.c. of each extract are then heated together with 9 c.c. of 0.005N-potassium ferricyanide in a testtube immersed in a boiling water-bath for 10 min., 2 c.c. of 30% acetic acid are added, the mixture is cooled, a few crystals of potassium iodide are dissolved therein, 10 c.c. of a zinc sulphate solution (50 g. of zinc sulphate crystals and 250 g. of sodium chloride per litre) are added, and the iodine liberated by the residual ferricyanide is titrated with 0.005N-thiosulphate. In the second method 0.2 g. of cellulose is heated as above with 10 c.c. of 0.005N-potassium ferricyanide and 10 c.c. of 10% caustic soda, and the residual ferricyanide then determined also as in the first method. The ferricyanide number obtained by the first method is usually about 14% higher than that given by the second, which is not attributable to oxidation during the extraction. Sizing impurities in the cellulose affect the ferricyanide number and must be removed before its determination. A. J. HALL.

Dispersion of cellulose and cellulose derivatives. S. E. SHEPPARD (J. Physical Chem., 1930, 34, 1041-1052).—Cellulose in the form of cotton fibres or wood pulp can be dispersed in inert organic liquids to give finely-divided suspensions by wet grinding in pebble or ball mills. A small fraction becomes colloidally dispersed. Different rates of dispersion obtain with different organic liquids. The viscosities of the solutions in Schweitzer's reagent fall with increasing subdivision, and the higher the initial viscosity of the cellulose the longer is the time require for disintegration to a given degree of dispersion. When grinding and drying are carried out in an atmosphere of carbon dioxide the copper number remains unchanged. The dispersions produced by mechanical disintegration in organic liquids behave almost identically with the hydrocellulose produced by the action of mineral acids on native cellulose. The modified properties include (i) diminished cuprammonium viscosity, (ii) increased solubility in 10% potassium hydroxide solution, (iii) increased reactivity on esterification, and (iv) increased adsorption of basic dyes. Small differences in reactivity, however, can be detected in the degraded celluloses of the same degree of mechanical dispersion produced by the action of acids in water or glacial acetic acid or by the action of organic liquids in the mill.

L. S. THEOBALD.

Manufacture and properties of a cellulose product (maizolith) from cornstalks and corncobs. C. E. HARTFORD (U.S. Bur. Stand. Misc. Publ. No. 108, 1930, 10 pp.).-Maize stalks or corncobs are digested for 3 hrs. under 40 lb. steam pressure with 10% of their weight of caustic soda diluted to a 1% solution, and, after being washed free from alkali, the cooked pulp is diluted with water to a consistency of 4%, beaten for 2 hrs. to yield a gel, which is dried in moulds at 70°. The product is a dense, hard, bone-like substance, which is practically unaffected by oil, but softens on prolonged soaking in water; it is somewhat heavier and harder than the hardwoods and is a good electrical insulator. Possible cheaper sources of maizolith are the fine fibres or pith particles rejected in the insulating wall-board industry, and the soft residue from the process of extracting adhesive material from corncobs. F. R. ENNOS.

Decomposition of [rye and] maize straw with nitric acid. H. SUIDA, H. SADLER, and F. Noss (Papier-Fabr., 1930, 28, 345—346, 363—365; cf. B., 1929, 675).—The results are given, mainly in tabular form, of experimental digestions of straw with nitric acid. In the case of rye straw, digestion at  $60-70^{\circ}$  with acid of  $5-10^{\circ}$  concentration gives yields of  $50-60^{\circ}$ , the resulting pulps being of normal bleachability. Lower concentrations of acid give higher yields, but the pulps are difficult to bleach. Maize straw gives lower yields of pulp, optimum results being obtained with  $4^{\circ}$  acid at  $80^{\circ}$ ; digestion for 90 min. gives a yield of  $42^{\circ}$ , the pulp being difficult to bleach. The effects of varying conditions of digestion on the parenchymal cells, knots, and pith have been investigated, and the results are illustrated by means of photomicrographs.

T. T. Potts.

[Manufacture of wood-pulp board etc. by] the Masonite process. R. M. BOEHM (Ind. Eng. Chem., 1930, 22, 493-497).—Wood chips are disintegrated without the use of chemicals by heating in a "gun" for 30-40 sec. with steam at a pressure of 350 lb./in.<sup>2</sup>, which is afterwards increased to 1000 lb./in.<sup>2</sup> and maintained thereat for 5 sec. On discharging from the "gun," the chips explode immediately owing to the high internal pressure, yielding a mass of long-fibre bundles, which, after suitable treatment, are formed into

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board for insulating, panelling, and generally as a substitute for wood in many directions.

F. R. ENNOS.

Production of naval stores from waste wood. R. C. PALMER (Chem. Met. Eng., 1930, 37, 289—292).— The preparation of turpentine, pine oil, and rosin is described. All the turpentine and part of the pine oil are isolated by steam-distillation of the chipped wood, which is then extracted with petroleum naphtha, b.p. 100—175°, to give an extract from which pure rosin is subsequently obtained. Refining of the above products, the preparation of abietic acid, and uses of the extracted wood are discussed. E. H. SHARPLES.

Influence of experimental conditions on results of pulp-strength tests. E. GRUND (Papier-Fabr., 1930, 28, 329-337).-The effect of varying the successive operations in the preparation of test-sheets is discussed. Variation of the substance of the sheets gives a maximum breaking length with sheets of 80 g./m.<sup>2</sup> By allowing the fibre suspension to settle for 1 min., a reduction in breaking length of 15% is effected; longer settling times have no further influence. Dilution beyond 1:750 has no influence on strength, but less dilution gives lower strengths. Variation of couching pressure shows that maximum strength is obtained at 120-150 kg./cm.<sup>2</sup>, and for times of application over 2 min. constant results are obtained. Increasing the drying temperature causes a reduction of strength. The effect of using a shorter strip in the Schopper tensile tester is to increase the breaking length. The Schopper-Riegler beating tester is used throughout to control the beating degree of the pulps. T. T. Potts.

Viscosimeter. Kämpf.—See I. Causticising plant. STEWART.—See VII. Xylose. EmLey.—See XVII.

See also A., June, 695, Swelling of cellulose fibres (HESS and others). 749, Cellulose chemistry (Welt-ZIEN). Acetolysis of cellulose (WEBBER and others). 751, Cellulose xanthate (GEIGER). 814, Enzymic hydrolysis of viscose silk (PRINGSHEIM and THILO).

## PATENTS.

Manufacture of textile packages. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 328,944, 5.2.29).—In order that the yarn package may be removed from the bobbin after winding, an adhesive (wax or gelatin) is applied to the threads while travelling to the package or to the package itself during its formation.

F. R. Ennos.

Manufacture of material capable of being marked by electrolytic decomposition. I. G. FARBENIND. A.-G. (B.P. 329,258, 16.10.29. Ger., 16.10.28).—Paper, textile fabric, etc. is impregnated with an acid solution which has been used as a photographic fixing bath, and is then dried; alternatively the silver in the impregnating solution may be displaced by a non-precious heavy metal, e.g., lead, before use. F. R. ENNOS.

Production of high- $\alpha$ -cellulose fibre for the manufacture of cellulose derivatives. G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,741,540, 31.12.29. Appl., 16.12.25).—Raw pulp is treated with caustic alkali, hot or cold, to remove non- $\alpha$ -cellulose constituents, bleached, and again treated with a more dilute alkali. either hot

or cold, to dissolve oxycelluloses formed during bleaching. F. R. ENNOS.

Manufacture of ammoniacal copper cellulose solutions. K. HESS and C. TROGUS (B.P. 301,752, 3.12.28. Ger., 3.12.27).—Cellulose is immersed in cuprammonium solution containing caustic alkali, in which the proportion of copper present to cellulose treated is at least  $1: 2\cdot 6$ , until the fibrous product has the composition  $Cu(C_6H_{10}O_5)_{2,x}NaOH$ ; it is then dissolved in water, aqueous ammonia, or dilute cuprammonium solution. F. R. ENNOS.

Manufacture of organic esters of cellulose. BRIT. CELANESE, LTD. (B.P. 300,140, 5.11.28. U.S., 5.11.27).— Cellulose acetate is partially hydrolysed in two stages. First a mild hydrolysis is produced by allowing the ester to remain for a time with sufficient water, which may contain acetic acid or hydrogen peroxide, and, after addition of excess of water, the ripening of the precipitated ester is completed in the presence of dilute acid.

F. R. ENNOS.

Manufacture of cellulosic esters. KODAK, LTD., Assees. of H. T. CLARKE and C. J. MALM (B.P. 304,279, 4.1.29. U.S., 18.1.28).—Cellulose esters of halogensubstituted fatty acids are produced by treating cellulosic material with a halogen-substituted fatty acid containing more than five carbon atoms (e.g. stearic or hexoic acid) and a halogen- or alkoxy-substituted fatty acid anhydride containing less than 10 carbon atoms, preferably chloroacetic anhydride, in the presence of catalysts, e.g., perchlorates, zinc chloride, or red phosphorus, if desired. F. R. ENNOS.

**Preparation of cellulose esters.** G. B. ELLIS. From Soc. DES USINES CHIM. RHÔNE-POULENC (B.P. 328,588, 22.12.28).—For the production of crotonyl esters, cellulose is pretreated with crotonic acid and subsequently esterified with crotonic anhydride in the presence of a catalyst (sulphuric acid, sulphonic acids, zinc chloride, etc.), with or without diluents (benzene, toluene, or melted crotonic acid). F. R. ENNOS.

Cellulose esters and ethers and their uses. G. P. DAVIES, W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 328,657, 25.10.28).-To avoid the high cost of the "vehicle" in the production of cellulose ester enamels, emulsions of the ester of the water-in-oil type, which give tough and transparent films, may be prepared by dispersing water, to an extent such that it is substantially absorbed in the emulsion, in a solution of the ester in small quantities of a solvent (cyclohexanone or its methyl derivatives) which is only slightly miscible with water, is sufficiently volatile to prevent undue prolongation of the drying period, and, under the conditions of drying, is less volatile than water, so that excess of solvent is present during drying of the film ; besides pigments, gums and plasti-F. R. Ennos. cisers may also be added.

Manufacture of artificial threads by the dryspinning method. A. CARPMAEL. From I. G. FARB-ENIND. A.-G. (B.P. 327,423, 3.11.28).—Solutions of cellulose ethers or esters containing benzene and alcohol in addition to the usual solvents are heated before spinning to near the point at which they begin to boil 656

within the nozzle, and are extruded into a chamber where the threads produced meet a countercurrent of air containing the vaporised solvent, if desired, at a lower temperature. F. R. ENNOS.

Manufacture of filaments, films, and the like from cellulose derivatives. H. DREYFUS (B.P. 328,636, 29.10.28).—Filaments of low denier, *c.g.*, below 1.5, are produced by spinning solutions of cellulose acetate in aqueous acetone (containing upwards of 10% of water) which have a low viscosity compared with solutions of the same cellulose acetate in commercial acetone, and drawing out the resulting filaments to the desired denier. F. R. ENNOS.

Manufacture of artificial silk from cellulose esters or ethers. J. Y. JOHNSON. From I. G. FARBEN-IND. A.-G. (B.P. 327,420, 4.10.28).—On leaving the spinning nozzles, liquid threads consisting of a cellulose derivative in a volatile solvent, with or without a swelling agent, are treated for a short time with a volatile liquid or vapour capable of swelling the cellulose derivative without completely coagulating it, while being stretched, and are then solidified by evaporation of the volatile material, with or without continued stretching.

F. R. ENNOS.

Uniting [cellulosic] sheets or articles. BRIT. CELANESE, LTD. (B.P. 305,992, 12.2.29. U.S., 13.2.28).— Organic derivatives of cellulose (cellulose acetate), which have been ground with plasticisers to a finely-divided state in the absence of volatile solvents as described in B.P. 282,723 (B., 1928, 853), with or without the addition of fillers, pigments, etc., are applied between the surfaces of the articles to be united; these are then subjected to heat and pressure. F. R. ENNOS.

[Cellulose binding agent for] joining wood, metal, glass, and the like together or to other materials. COMP. FRANÇ. D'EXPLOIT. DES PROC. PLINA-TUS, Assees. of W. PLINATUS (B.P. 303,855, 1.1.29. Ger., 11.1.28. Addn. to B.P. 302,324; B., 1930, 504). —Leather meal or sawdust is added to the cellulose ester adhesive specified in the prior patent.

A.-R. POWELL.

Sizing of paper. E. LECOCQ (B.P. 313,171, 6.6.29. Belg., 8.6.28).—The pulp containing an agglutinant such as a resin soap is treated first with sodium bisulphate and then with aluminium sulphate, the acidity being afterwards neutralised by the addition of powdered chalk. F. R. ENNOS.

Smoothing of paper. I. G. FARBENIND. A.-G. (B.P. 327,635, 6.6.29. Ger., 19.10.28. Addn. to B.P. 310,340; B., 1930, 505).—In modification of the prior patent, the paper is first coated with a substance, such as glue, gelatin, cellulose esters or ethers, which is impermeable to the smoothing agent. F. R. ENNOS.

Manufacture of cellular products [from pulp]. M. GRUNBAUM (U.S.P. 1,746,648, 11.2.30. Appl., 8.5.28). —Paper, paper board, wall-paper, etc. may be rendered insect-, germ-, and fungus-proof by incorporating a cinchona derivative (0.04% of cinchonine) in the pulp, fillers, or printing ink during process of manufacture. F. R. ENNOS.

Production of highly glazed, insulating pasteboard. M. M. SEREBRIANY (B.P. 328,962, 30.1.29).-- Bark or wood material, worked up in the mill and in the form of dry fibre, is mixed with boiled linseed oil, raw turpentine, etc., with addition, if necessary, of solvents such as benzene, turpentine, petroleum, etc., and with or without resins dissolved therein ; the paper mass so obtained is worked up into pasteboard on suitable machines. F. R. ENNOS.

Treatment of residual liquors etc. [from woodpulp manufacture]. L. BRADLEY and E. P. MCKEEFF, Assrs. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,747,047, 11.2.30. Appl., 28.3.21).—The cyclic process described consists in cooking fibrous material in a suitable reagent, e.g., alkaline sulphite, neutralising the residual liquor, e.g., with sulphite, to precipitate organic matter, which is then separated, and adding acid sulphite to adapt the liquor for use as a cooking liquor. A further quantity of fibrous material is then cooked in this acid sulphite, and the residual liquor is neutralised, e.g., with calcium hydroxide, separated from the precipitated organic matter, and treated so as to adapt it for use as a cooking liquor in the first process. F. R. ENNOS.

Manufacture of vegetable wool. J. L. LAMBRECTH (U.S.P. 1,759,752, 20.5.30. Appl., 31.5.29. Fr., 9.6.28). —See B.P. 313,169; B., 1930, 236.

[Apparatus for] opening and cleaning of fibrous materials. TEXTILE & FILATURE Soc. ANON., and G. PLACQUET (B.P. 304,666, 23.1.29. Fr., 23.1.28).

Manufacture of [composite] textile yarn or thread. O. RASCH (B.P. 328,989, 9.2.29).

Method and apparatus for spinning fibrous material. BRIT. COTTON INDUSTRY RES. ASSOC., PLATT BROS. & CO., LTD., and A. E. OXLEY (B.P. 329,457, 6.4.29).

Manufacture or [stretching] treatment of [travelling] artificial threads or filaments. BRIT. CELAN-ESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 329,620, 20.12.28).

Acid-resisting materials (U.S.P. 1,732,140).—See VII. Stratified bodies (B.P. 328,919).—See VIII. Insulating material (B.P. 329,604).—See XI. Grease from wool-scouring waters (B.P. 328,606).—See XII. Plastic masses (B.P. 304,612 and 305,238).—See XIII. Cellulose films (B.P. 305,653).—See XXI.

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

Absorption of tannic acid by cellulose acetate silk. P. E. KING and A. R. SAHASRANAM (J. Soc. Dyers and Col., 1930, 46, 118-121).-The observation of Ermen (B., 1929, 850) that cellulose acetate silk absorbs tannic acid similarly to viscose silk is confirmed, and tabulated results show the comparative affinities of these two types of artificial silks as obtained by steeping the silks in aqueous tannic acid and determining the nonabsorbed portion by Loewenthal's method. Viscose silk absorbs about 40% more tannic acid than does cellulose acetate silk under comparable conditions. The tannic acid absorbed by cellulose acetate silk is most satisfactorily fixed by further steeping the silk in a solution containing an amount of tartar emetic equal to 25% of the weight of tannic acid originally applied; 50% of the weight is usual in similarly fixing cotton mordanted with tannic acid. Cellulose acetate silk mordanted with tannin-tartar emetic has an increased affinity for the majority of the basic dyes examined, but not for Malachite Green. Rhodamine B and 6G give fluorescent shades on non-mordanted cellulose acetate silk, but on mordanted silk no fluorescence appears. A. J. HALL.

Fading of dyed textiles. I. P. W. CUNLIFFE (J. Soc. Dyers and Col., 1930, 46, 108-111).—The results obtained by exposing dyed textile materials to sunlight and artificial light under various conditions are summarised. Fading due to exposure to strong sunlight during the midday hours is fairly reproducible in different parts of the world except with certain dyes known to be sensitive to humidity or other agents such as sulphur dioxide. Dyed wools and silks behave more regularly than other fibres during fading; this is ascribed to their smaller sensitivity to humidity. Changes of hue which the unexposed parts of the samples frequently show in outdoor tests at Leeds are generally of the same type as is produced by a little sulphur dioxide acting in the dark. Tables are given showing, semi-quantitatively, the reaction of dyed wool, silk, cotton, linen, and viscose towards light and moisture as determined in the laboratory. Cotton dyes show wide differences in their sensitivity to moisture, but basic and azoic dyes are the most sensitive. Dyes are rather more sensitive to moisture on linen than on cotton; they are least sensitive on wool and silk. Lissamine Fast Red B, Erio Fast Floxine BL, and Naphthol Green B are appreciably more fugitive in the presence of sulphur dioxide than in a pure atmosphere. A. J. HALL.

Submerged combustion. HAMMOND.—See II. Tanning processes. Schindler and Klanfer.— See XV.

See also A., June, 749, Cellulose chemistry (WELT-ZIEN). Action of sodium hydroxide on cellulose (RUMBOLD). 779, Theory of dyeing (PFEIFFER and others).

#### PATENTS.

Bleaching of [organic] materials. L. MELLERSH-JACKSON. From PILOT LAB., INC. (B.P. 328,544, 3.10.28). —Mixtures of mutually activating organic or organic and inorganic peroxides (e.g., a mixture of benzoyl peroxide with 5—10% of butyryl, hexoyl, or decoyl peroxide) are used for bleaching flour, soaps, oils, etc. [Stat. ref.] L. A. COLES.

Transfer of colour designs to fibrous materials. S. J. Cox (B.P. 328,909, 2.11.28).—The design is first produced in printing ink on a temporary support of flexible material such as sized paper, which has been coated with an emulsion of soft resins, ordinary rosin, and water-soluble gums. The coated surface of the support, after dusting with powdered resin, is then applied to the surface of the material to be treated, and the back of the support is subjected to the action of steam and pressure, whereby the colouring matter is intimately mixed with the fused coating and carried into the fibres of the material, giving a clean, sharp outline. F. R. Ennos.

Mercerisation of cotton for the production of a silk-like appearance and handle. E. LANG (F.P.

637,921, 23.11.26).—Cotton is kier-boiled, treated for 6—12 hrs. in a liquor containing 2—4% of an alkali, 0.5% of rosin soap, 5—20% of starch, and (optionally) a protective colloid, washed, mercerised in caustic soda of  $d \cdot 1.115$  containing 1% of sodium hyposulphite or other reducing agent, soured, and bleached. The starch may be replaced by dextrin, flour, glue, or gelatin.

A. J. HALL. Waterproofing of fabrics. M. and R. WALRAVE (F.P. 638,374—5, 3.12.26).—(A) Fabric is impregnated with a mixture containing 8 kg. of flour dissolved in 100 litres of dilute countie sode to which are added 15 kg.

100 litres of dilute caustic soda to which are added 15 kg. of white soap (2% solution) and a mixture of 3 kg. of beeswax, 4 kg. of linseed oil, and  $3 \cdot 5$  kg. of paraffin; it is then dried and treated with a solution containing 10 pts. of blood albumin, 35 pts. of aluminium acetate, and 55 pts of water. (B) Fabric is impregnated with a solution consisting of 6 kg. of Marseilles soap, 8 kg. of blood albumin, and 100 litres of water, then dried, treated with a solution consisting of 47 litres of aluminium acetate ( $d 1 \cdot 115$ ), 11 litres of formaldehyde, and 50 litres of water, heated to 60°, and again passed through this solution. A. J. HALL.

Preparation of dyeings on the fibre. W. HENT-RICH, M. HARDTMANN, and E. TIETZE, ASSIS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,759,258, 20.5.30. Appl., 20.12.28. Ger., 28.12.27).—See B.P. 321,737; B., 1930, 320.

Dyeing of acetate silk. R. S. HORSFALL, L. G. LAWRIE, and J. A. R. HENDERSON, ASSIS. to BRIT. DYE-STUFFS CORP., LTD. (U.S.P. 1,759,010, 20.5.30. Appl., 19.5.26. U.K., 29.5.25).—See B.P. 257,654; B., 1926, 976.

Treatment of fabrics, cloths, and similar material [to remove shininess]. A. Ross (B.P. 329,003, 10.1.29).

Apparatus for liquid treatment of textile materials. M. FREIBERGER (B.P., 329,526, 5.6.29).

Drying machines for fabrics. Machines for treating fabrics by drying, dry-cleaning, dyeing, or washing. H. PIERCE (B.P. 329,347-8, 15.12.28. U.S., 7.5.28).

Heating of liquids (B.P. 328,548).--See I.

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

Pressure-synthesis operations of the Du Pont Ammonia Corporation. ANON. (Ind. Eng. Chem., 1930, 22, 433-437) .- A description is given of the ammonia and methyl alcohol synthesis of the Du Pont Ammonia Corporation at Belle, W.Va. Coal is carbonised in beehive coke ovens and water-gas is produced from the coke thus obtained. The coke-oven gas is not used in hydrogen production. The crude watergas, containing about 50% H<sub>2</sub> and 40% CO, is passed through scrubbers to low-pressure holders. The watergas generators have a capacity of 70 million cub. ft. per day. The correct mixture is made in these holders and compressed at 25 atm., the pressure of the gas being further raised to 1000 atm. in the following stages by hypercompressors : inlet 25 atm., second stage 85 atm., third stage 320 atm. Carbon monoxide is removed in

methyl alcohol converters. After the purification step the gas is converted into ammonia, which is drawn off and stored as a liquid. Uncondensed gas is scrubbed with condensed steam. Tanks for the transport of ammonia have a shell thickness of  $\frac{3}{4}$  in. H. INGLESON.

Application of continuous methods of operation to causticising by the lime-soda method. R. F. STEWART (Ind. Chemist, 1930, 6, 194-198).-The balanced type of Dorr thickener consisting of a steel tank divided into two or more compartments by sloping trays is described. Each compartment has its own feed and clear-liquor overflow. This apparatus does not deal with gritty solids ; these, if present, should be previously removed in a classifier. The Dorr agitator employing a hollow shaft carrying compressed air to be distributed by the rotating arms is suitable for causticising in most cases. In kraft-pulp mills, however, air should not be used. A Dorr continuous causticising plant installed in such a mill is described. It includes a slaking mill, classifier, three agitators, and three thickeners; the last-named effect systematic washing. The units are naturally small compared with those necessary for batch-working. The advantages of continuous working are summarised. C. IRWIN.

Manufacture of hydrogen peroxide and X-ray ingestion material. J. SCHWYZER (Pharm. Ztg., 1930, 75, 594—595).—A process by which pure hydrogen peroxide solution and barium sulphate of quality suitable for administration for purposes of X-ray examination are obtained consists in treatment of barium peroxide with phosphoric acid, dissolution of the precipitated monohydrogen phosphate with further phosphoric acid, and regeneration of the latter by addition of sulphuric acid to the purified solution of the dihydrogen phosphate. S. I. LEVY.

Analysis of sodium bisulphite and of its compounds with formaldehyde. V. Morlík (Chem. Listy, 1930, 24, 179—181).—The indigo titration method for the determination of sodium bisulphite or of its formaldehyde compound is, if carried out in an inert atmosphere, such as coal gas, as exact and more convenient than the Bosshart-Grob copper-reduction method.

## R. TRUSZKOWSKI.

Conversion of superphosphate into ammonium phosphate. K. MONIKOWSKI (Przemysl Chem., 1930, 14, 217-221).—Solutions containing about  $3\cdot 3\%$  $P_2O_5$ ,  $0\cdot76\%$   $H_2SO_4$ , and  $0\cdot15\%$  Ca, suitable for use in the fermentation industries, can be cheaply obtained by treating 200 pts. of superphosphate with  $16\cdot6$  pts. of sulphuric acid and  $20\cdot6$  pts. of ammonium sulphate in 1000 pts. of water; under these conditions the greater part of the calcium present is precipitated as sulphate. The use of oxalates for the precipitation of calcium is not advantageous, owing to the high cost of recovery of oxalic acid from calcium oxalate. R. TRUSZKOWSKI.

Calcination or enrichment of phosphate rock. C. G. MEMMINGER, W. H. WAGGAMAN, and W. T. WHITNEY (Ind. Eng. Chem., 1930, 22, 443—446).— The more extensive and cheaply mined pebble deposits may be concentrated by calcination after a preliminary washing process. When calcined at 1100° in rotary kilns pebble phosphate containing  $76 \cdot 17\%$  of tricalcium phosphate may be raised to a content of  $80 \cdot 43\%$ . All combined water is expelled, calcium carbonate is decomposed, organic matter completely oxidised, and most of the sulphur trioxide eliminated. Treatment at this temperature renders the phosphate rather resistant to attack by sulphuric acid, and for certain purposes a less drastic calcination at about  $800^\circ$  is preferred. The phosphate content is increased then only by about  $2 \cdot 5\%$ . The kilns used are 140 ft. long and 8 ft. in diam., and revolve once in 70 sec. C. A. KING.

Interaction of ammonium carbonate with alcoholic potassium chloride solution. A. STERN (Z. angew. Chem., 1930, 43, 425—427).—The ammoniasoda reaction cannot be applied to the preparation of potassium carbonate in aqueous solution, the comparative solubilities of the salts causing their action to be reversed. It has been found, however, that it is practicable in alcoholic solution. Ammonium bicarbonate is at once decomposed by alcohol, but the normal salt is sufficiently stable to be used. The reaction was studied under varying conditions. The best yield, about 40%, was obtained at 5°, using 85% alcohol. The salt precipitated is a mixture of potassium carbonate and potassium chloride. The latter can be readily recovered if the final product of the process is to be caustic potash. C. IRWIN.

Conversion of sodium chromate into dichromate by the action of carbon dioxide under pressure. B. NEUMANN and C. EXSSNER (Z. angew. Chem., 1930, 43, 440-444).-This reaction was studied in a glass apparatus which permitted the precipitated sodium bicarbonate to be collected on a filter without releasing the pressure. The authors used a saturated solution of sodium chromate (16.7 g. Cr/100 c.c.) and one of half this concentration, the carbon dioxide being under pressures up to 10 atm. The conversion decreases with rise of temperature and is nil at 50°. Concordant results were difficult to obtain with the saturated solution, but with the more dilute solution conversion increased regularly with increase of pressure and reduction of temperature, rising to 70% at 0° and 10 atm. Conversions were apparently better in the saturated solution at 15-25°, but lower temperatures could not be used. Higher conversion figures than 65-75%, such as have been claimed, are not possible of attainment. It is, however, practicable to separate the sodium chromate and dichromate by evaporating in vacuo and then heating in an open pan to 145°, when the unconverted chromate is entirely precipitated. C. IRWIN.

Antiformin and antiformin substitutes. J. K. GJALDBÆK (Dansk Tidsskr. Farm., 1930, 4, 137—146).— The composition and stability of a number of technical solutions containing sodium hydroxide and sodium hypochlorite has been investigated. H. F. HARWOOD.

Analysis of calcium hypochlorite solutions.' [Chloramine-T as a volumetric reagent.] E. FABRIZI (Papier-Fabr., 1930, 28, 346-347).—A method is given which is claimed to be less expensive and easier to manipulate than the usual methods. 10 C.c. of bleaching liquor, containing about 26 g. of available chlorine per litre, are diluted with 300 c.c. of water, 75 c.c. of 0.1N-arsenite solution are added, and the excess is titrated with 0.1N-Chloramine-T solution, starch-iodide solution being used as an indicator.

T. T. Potts.

Assay of calcium carbide and Czechoslovakian standards. R. VONDRAČEK (Chem. Listy, 1930, 24, 193—197).—The provisional Czechoslovakian standards envisage the use of a modified Caro's apparatus for the assay of calcium carbide; this method is not exact, and an apparatus giving more accurate results is described. The yield of acetylene should not be given in c.c. of moist gas, as the Gay Lussac formula is only applicable to dry gases. The experimental error in the assay of carbide should not exceed 2%. R. TRUSZKOWSKI.

Manufacture of carbon dioxide and Epsom salts. W. P. HEATH (Ind. Eng. Chem., 1930, 22, 437-439).— Dolomite containing about 54% CaCO<sub>3</sub> and 45%MgCO<sub>3</sub> is ground and stirred with water and sulphuric acid. The carbon dioxide evolved is scrubbed, compressed, liquefied, and finally stored in steel cylinders. Specifications to which these cylinders are required to conform are mentioned. A solution of potassium permanganate is added to the neutral sulphates and iron and aluminium are removed. The sludge is then centrifuged and magnesium sulphate is washed out, crystallised, dried, and screened. H. INGLESON.

Titrimetric determination of magnesium. J. S. PIERCE and M. B. GEIGER (Ind. Eng. Chem. [Anal.], 1930, 2, 193-194).-Previous methods have been modified (B., 1928, 402; 1929, 284). For limestone, 0.5 g. is dissolved in 50 c.c. of 0.25N-hydrochloric acid, the solution being boiled and titrated with alkali until neutral to bromothymol-blue. The precipitate is collected and washed ; to the filtrate are added 5 drops of a 0.1% alcoholic solution of dimethylaminoazobenzene and 0.25N-hydrochloric acid until faintly pink. After the addition of 10 c.c. of a saturated alcoholic solution of trinitrobenzene, the liquid is titrated with 0.25Nsodium hydroxide (carbonate-free) until the colour is dark red. The liquid is diluted to 100 c.c. and filtered ; 50 c.c. of the filtrate are titrated with 0.25N-hydrochloric acid until neutral to an indicator having a colour change at  $p_{\rm H}$  about 4.0 easily recognisable in a reddish E. H. SHARPLES. solution (Congo-red).

Commercial preparation of phosphorescent substances. B. RHODE (Chem.-Ztg., 1930, 54, 369-371).-The phosphorescence of the alkaline-earth sulphides is associated with the crystalline state and also involves the presence of traces of activators, usually heavy-metal sulphides. These substances are prepared by treating the oxides with sulphur, a flux, and the necessary activator in a crucible at 1000°. After the sintered mass has cooled it is crushed and sieved. A full range of colour in the phosphorescence is possible by varying the The metals used, and several formulæ are given. longest duration of phosphorescence is obtained by the use of strontia and magnesia. Phosphorescent zinc sulphide is prepared by precipitating an ammoniacal zinc solution with pure hydrogen sulphide, lead being excluded from the apparatus. The zinc sulphide is settled, filtered, and washed, and then ignited with a mixture of sulphur and the chlorides of potassium,

sodium, and ammonium, with copper chloride as activator. Careful attention to the purity of all the ingredients is necessary. A mixture of zinc and cadmium sulphides gives a greenish-yellow to red phosphorescence. C. IRWIN.

Splitting strength of mica. J. W. OBREIMOV (Proc. Roy. Soc., 1930, A, 127, 290—297).—It is shown that the splitting strength of mica is a constant. A surface energy of mica can be determined which is independent of the shape of the mica sheets, and it is found that mica placed in optical contact is completely restored. Some electrical phenomena obtained by splitting mica in a high vacuum are described. The strength of mica appears to be greater in a high vacuum than in air. L. L. BIRCUMSHAW.

Manufacture of bismuth salts. E. A. MAUERS-BERGER (Chem. Weekblad, 1930, 27, 337–338).—An account is given of the preparation of various pharmaceutical bismuth compounds from the metal and its oxide ores. S. I. Levy.

Dependence of the catalytic activity of vanadium pentoxide on its mode of combination with silica. I. E. ADADUROV and G. K. BORESKOV (Ukraine Chem. J., 1929, 4, [Tech.], 259—266).—Vanadium pentoxide deposited on dehydrated silicic acid has a greater catalytic action on the oxidation of sulphur dioxide than in the absence of silica; at the optimum temperature of 400°, 97.85% oxidation is effected. If vanadium is precipitated together with silicic acid and the resultant gel is dried, 99.4% yields of sulphur trioxide are obtained, at a rate of flow of gas (containing 6.2—7.0% SO<sub>2</sub>) of 60 c.c./sec., at 400°. The second type of catalyst probably contains vanadium zeolite.

R. TRUSZKOWSKI.

Vanadium compounds as catalysts for the oxidation of sulphur dioxide. H. N. HOLMES and A. L. ELDER (Ind. Eng. Chem., 1930, 22, 471-473).-The principle in preparing a series of vanadium catalysts was to precipitate the vanadium compound, the promoter, and the supports simultaneously as far as possible in order that the catalyst should be peptised or colloidally dispersed. The best catalyst was made by mixing potassium metavanadate with an excess of calcium chloride and then neutralising the excess calcium chloride with sodium silicate. The gel should be prepared from dilute solutions. This form of catalyst showed a 98% conversion of sulphur dioxide when working on 8% mixtures. Replacement of calcium by iron also gave good results, but copper, cobalt, nickel, chromium, and aluminium were not efficient promoters. Tungsten was inefficient and carnotite proved a poor catalyst. It is calculated that 1 pt. of vanadium is required for 48 pts. of sulphur, which indicates much larger contact space than with platinum, though with an equal life the cost of preparation of vanadium catalysts would be only 1% C. A. KING. that of platinum.

Moisture content of compressed nitrogen. A. C. WALKER and E. J. ERNST, JUN. (Ind. Eng. Chem. [Anal.], 1930, 2, 139—140).—Moist nitrogen at an initial pressure of 150 atm. was allowed to discharge at a regulated rate through an instrument in which the moisture content of the free gas was continuously measured. The humidity of the gas increased as the pressure in the cylinder decreased. Calculations of the value of the R.H. of the gas expanded to atmospheric pressure from different initial pressures were made on the assumption that the gas in the cylinder remains saturated throughout the discharge. This should give results which are higher than those observed. At pressures greater than 3 atm. the observed values are greater than those calculated. H. INGLESON.

Hydrogen cyanide in gaseous products from coal. NEWALL and others.—See II. Uranium from its fused salt. DRIGGS and LILLIENDAHL.—See X. Concentration of manganese dioxide. MARTIN.—See XI.

See also A., June, 708, Explosion regions of gas mixtures (POSTHUMUS). 715, Preparation of arsine and stibine (HLASKO and MASLOWSKI). 722, Purity of electrolytic oxygen (KEFFLER). 725, Analysis of nitrating acid (Müller and Kogert).

## PATENTS.

Contact sulphuric acid process. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,741,310, 31.12.29. Appl., 3.7.28).—The catalyst comprises a complex zeolite containing vanadium in ter-, quadri-, and/or quinquevalent form with or without other metals of group VI, VII, or VIII, and with or without inert acid-resisting fillers, such as quartz, kieselguhr, or colloidal silica. Before use the substance is leached with dilute mineral or organic acids to remove the base-exchangeable alkali or alkaline earths. A. R. POWELL.

Purification of highly concentrated nitric acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,156, 14.6.29).—The acid is freed from sulphuric acid by treating it with compounds of such ter- or bivalent metals as form nitrates soluble in concentrated nitric acid, *e.g.*, aluminium, magnesium, and iron. These metals give sulphates that are insoluble in concentrated nitric acid, and these can be readily separated. W. J. WRIGHT.

Production of phosphoric acid and hydrogen. BAYERISCHE STICKSTOFFE-WERKE A.-G. (B.P. 308,598, 19.3.29. Ger., 24.3.28).-Phosphorus is treated with water, both preferably being in a state of fine subdivision, at temperatures below 600° and at increased pressures (which may be produced by operating in a closed system, and stopping, if desired, when a constant pressure is developed). Phosphine which is also formed is in turn converted by the same process, in the same or a separate system, into the same products. Iron promotes the formation of phosphine, and the vessels used. therefore, should not be made of this metal; a material which catalyses the formation of phosphoric acid, e.g., precious metals (silver), metal alloys (monel metal), or which becomes an active catalyst in the course of the process is, however, suitable. Other catalysts are certain metal (e.g., copper) phosphides and phosphates, which may be used to line the reaction vessel; these may also be produced in situ by allowing the reactants, or other phosphorus compounds, to interact with the vessel wall, which is made of a suitable metal. All the said catalysts may be used in admixture. The reaction is also favourably influenced by small quantities of bases (alkali or alkaline-earth hydroxides).

S. K. TWEEDY.

Production of chemically pure phosphoric acid. I. G. FARBENIND. A.-G. (B.P. 319,656, 25.7.29. Ger., 25.9.28).—Crude phosphoric acid is electrolysed with a cathode current density less than 1 amp./dm.<sup>2</sup> and using cathodes of copper, silver, chromium, platinum, or tantalum, at which metals or metalloids resulting from cathodic reduction of impurities are deposited without the phosphoric acid being reduced. J. S. G. THOMAS.\*\*

**Porous acid-resisting materials.** A. Z. PEDERSEN, Assr. to PEPLOID CORP. (U.S.P. 1,732,140, 15.10.29. Appl., 5.7.23).—A solution of an organic colloid in an organic solvent is thoroughly incorporated with an aqueous solution of a colloid, whereby mutual precipitation of the colloids takes place. After drying the product, it is rolled into sheets, and the water-soluble colloid is washed out in running water. *E.g.*, an acetone solution of cellulose nitrate is mixed with an aqueous solution of sodium silicate, and the precipitate is dried and rolled into a sheet which is soaked in water to remove the sodium silicate, leaving a porous celluloid sheet suitable for use in galvanic batteries. A. R. POWELL.

Catalytic ammonia synthesis. SELDEN Co., Assees. of A. O. JAEGER (B.P. 307,457, 8.2.29. U.S., 8.3.28).—A catalyst for the synthesis of ammonia from nitrogen and hydrogen comprises a zeolite derivative from which part of the exchangeable base may be leached out by treatment with dilute acid, and which may contain an admixture of active or inactive diluent. The catalyst may be prepared, for example, by fusing a mixture of 15 pts. of alumina, 11.5 pts. of zirconia, 18 pts. of potassium carbonate, and 100 pts. of magnetite, leaching the product with water, and treating the residue with dilute nitric acid. Other types of catalyst may contain tungsten, molybdenum, vanadium, and thorium in association with silica, iron oxide, and potash.

A. R. POWELL.

Synthetic manufacture of ammonia. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,079, 14.3.29).—The reacting gases from the catalyst chamber are passed through a water-cooler in which they are cooled under 200 atm. pressure to  $15^{\circ}$ , and about half their content of ammonia is removed in the liquid state. The gases are then passed into a low-temperature cooler in which they are cooled to  $-40^{\circ}$  by allowing the already condensed ammonia to expand into the jacket of this cooler. Practically all the ammonia is thus condensed and the residual gases are returned to the catalyst circuit. A. R. POWELL.

**Production of sodium sesquicarbonate.** C. SUNDSTROM and G. N. TERZIEV, ASSTS. to SOLVAY PRO-CESS CO. (U.S.P. 1,748,739, 25.2.30. Appl., 8.4.26).— A solution containing 196 g. of sodium carbonate, 66 g. of sodium hydrogen carbonate, and 71 g. of sodium chloride per litre is mixed with sodium carbonate in the form of soda ash, and the mixture is heated at 60—80° while a regulated current of carbon dioxide is passed through the liquor in two stages, whereby crystals of sodium sesquicarbonate gradually grow in the liquor. The finer crystals are continually returned from the second to the first saturating tank, and the coarser crystals are periodically removed from the second tank at the bottom, the mother-liquor being returned to the mixing tank at the beginning of the circuit.

A. R. POWELL.

Manufacture of sodium sulphate by the Hargreaves process. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 328,780, 22.4.29).—In order to enable the gases to penetrate readily, the sodium chloride is employed in the form of small, porous lumps (cf. B.P. 269,209; B., 1927, 831), and flue dust is incorporated with these to accelerate the reaction. A counterflow rotary furnace for causing the chloride to react with the gases below sintering temperature, with avoidance of incompletely formed sulphate, comprises a tubular shell with an axial tube for introducing and heating the gases, a spiral device attached to this tube and nearly in contact with the outer shell, and means for discharging and emptying. The furnace is electrically heated.

## W. J. WRIGHT.

**Production of ammonium sulphate.** O. PIETTE (B.P. 327,488, 25.1.29).—Ammonium sulphate is precipitated, in a saturator fed with a cold saturated solution thereof, by means of ammonia and the motherliquor is passed to a still, the liberated ammonia being introduced into the saturator. After cooling, the still liquor is treated with carbon dioxide, and the resulting ammonium carbonate solution mixed with finely-ground calcium sulphate. The calcium carbonate produced is removed, and the solution of ammonium sulphate returned to the saturator, the quantity of ammonia removed in the still being such that the solution of ammonium sulphate thus returned is saturated.

F. G. CLARKE.

Manufacture of commercial calcium arsenate [insecticide]. W. C. PIVER, Assr. to F. M. SIMPSON (U.S.P. 1,727,306, 3.9.29. Appl., 24.6.24).—Quicklime is slaked in a solution of calcium or sodium nitrates or in sodium hydroxide solution ( $d \ 1.02$ ) and the granular suspension of slaked lime is agitated thoroughly while a 50% solution of arsenic acid is run into the vat. The product is claimed to be free from objectionable soluble acid arsenates. A. R. POWELL.

Manufacture of calcium cyanamide or products containing the same. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,743, 21.3.29).-Calcium cyanamide is produced by heating calcium carbamate under such conditions that the partial pressures of the gases injurious to the reaction (carbon dioxide, water vapour) are kept as low as possible. Thus the heating may be effected in presence of indifferent or favourable gases such as ammonia, carbon dioxide, nitrogen, or mixtures of these, if desired at elevated pressures and containing small amounts of other gases, e.g., hydrogen sulphide, which render innocuous any impurities such as iron which may be present in the carbamate. Alternatively, the heating may be effected very rapidly to 700-800°, preferably in an inert gas (absence of air). Further, the carbamate may also be heated under reduced pressure, e.g., 20 or 100 mm.

S. K. TWEEDY.

Treatment of lyes containing copper and zinc. N. E. LENANDER and I. RYEN (B.P. 329,225, 13.7.29).— Lyes obtained, e.g., by a chloridising roasting and subsequent leaching of copper pyrites, are treated, after the addition, if necessary, of soluble chlorides, with sufficient sulphur dioxide to impart an acidity of about 5—10% HCl per litre and an excess of finelydivided ("cement") copper is added to convert dissolved copper into cuprous chloride, the greater part of which is precipitated together with silver and gold present in the solution; the solution and the precipitate, together or separately, are treated with iron to convert the cuprous chloride into copper, the residual solution being worked up to zinc by the usual method. L. A. COLES.

[Manufacture of] zinc sulphide [pigment]. C. W. FARBEE, Assr. to NEW JERSEY ZINC CO. (U.S.P. 1,742,030, 31.12.29. Appl., 16.6.27).—A solution of zinc chloride  $(d\ 1\cdot 5)$  is treated at 80° with a slight excess of a solution of barium sulphide  $(d\ 1\cdot 15)$ , avoiding vigorous agitation, the mixture is diluted with an equal volume of water, the precipitate allowed to settle, and the clear liquor decanted off. The precipitate is washed by decantation until the liquor contains less than 0.2 g. of chlorine per litre, a small quantity of baryta water is added, and the mixture dried to 4-10% moisture content. The powder is then calcined at  $650-725^\circ$ . A. R. POWELL.

Ferric alumina [for water purification]. A. R. MOBERG, Assr. to F. O. PAIGE, JUN. (U.S.P. 1,747,177, 18.2.30. Appl., 15.4.29).—A coagulant for use in purifying water over the entire range of alkalinity usually employed comprises a colloidal solution of aluminium hydroxide peptised with ferric chloride. The material is prepared by precipitation of 133 lb. of aluminium chloride in 40 gals. of water by means of ammonia and agitation of the washed precipitate with 270 lb. of ferric chloride solution  $(d \ 1 \cdot 2)$ .

A. R. POWELL.

Manufacture of aluminium fluoride. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,688, 5.2.29).— Gaseous hydrofluoric acid, or a mixture of hydrofluoric and hydrochloric acid, is passed over hydrated alumina at normal temperature, until clouds of fume are evolved, the reaction being then allowed to continue at a temperature not above 400°. A product containing 56% HF is thus obtained. W. J. WRIGHT.

Recovery of vanadium from complex solutions. G. S. TILLEY (U.S.P. 1,747,403, 18.2.30. Appl., 3.6.27).— The mother-liquors from the crystallisation of alum obtained by digestion of alunite with dilute sulphuric acid contain iron and vanadium; these two elements are separated from the remaining aluminium by neutralising the solution ( $d \ 1.25 - 1.30$ ) with milk of lime to  $p_{\rm H}$  4, and, if necessary, adding bleaching powder to oxidise ferrous and vanadyl salts. The precipitate of basic ferric sulphate contains all the vanadium; it may be smelted in the usual way to obtain ferrovanadium. The mother-liquor is returned to the alumleaching vats. A. R. POWELL.

Production of amphoteric hydrated oxides of metals by hydrolysis. (SIR) G. C. MARKS. From R. H. MONK and J. IRWIN (B.P. 329,041, 18.2.29).- 662

Amphoteric hydrated oxides of metals of group IV of the periodic table are produced by hydrolysing a solution of a salt of the metal to which a small amount of an oxide of the metal, which has been precipitated by means of an alkaline compound (ammonia, sodium carbonate), has been added, for the purpose of providing nuclei around which the hydrolysed hydrated oxide can crystallise. The product precipitates out readily, and is easily washed and separated by settling.

S. K. TWEEDY.

Detergent and its preparation. A. H. COWLES, Assr. to ELECTRIC SMELTING & ALUMINUM CO. (U.S.P. 1,745,844, 4.2.30. Appl., 6.3.26).—Mixtures of silica, aluminium silicate, and sodium (or potassium) carbonate in quantities sufficient to yield a product having the formula  $Al_2O_3$ ,  $(SiO_2)_x(Na_2O)_{x-1}$  (x>7) are heated to fusion, cooled, and pulverised; alkali hydroxides, alkali salts of weak acids, and/or emulsifying agents may be mixed with the product. L. A. COLES.

Heating hydrogen for use in destructive hydrogenation. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 329,045, 21.2.29).—The hydrogen is passed through a cooling jacket on the inside wall of the hydrogenation converter, thence to a heat interchanger, utilising the sensible heat of the vaporous reaction products, and finally over an electric heater. The latter is preferably situated within the converter. The approximate temperatures attained by the hydrogen may be 100°, 300°, and 500° in the three stages, respectively. S. K. TWEEDY.

Purification of gases. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,592, 18.1.29).-Gases, e.g., carbon monoxide, hydrogen, or combustible gases, containing phosphorus in combined (hydrides, chlorides) or elementary form are purified by mixing with oxygen, or a gas containing oxygen (e.g., air), and passing the mixture at a temperature above 100° (e.g., 150-400°, or even 800°) over mixed catalysts containing two constituents, one, (a), adapted to cause catalytic oxidation of the phosphorus impurities, and the other, (b), to bind chemically or to adsorb the phosphorus oxides formed by the action of (a). Constituent (a) comprises at least one earth or heavy metal of group I, III, IV, VII, or VIII of the periodic system, or a compound, e.g., oxide or hydroxide, of this metal; constituent (b) comprises at least one solid compound, e.g., oxide or silicate, of an element of group I, II, III, IV, or VIII. Examples are: kieselguhr or quartz moulded with copper nitrate solution to give a mass containing 5% Cu, and moulded brown coal briquette ashes. Pressures above or below atmospheric may be employed, and the adsorbed phosphorus oxides may be lixiviated from the . catalyst. S. K. TWEEDY.

Manufacture of oxygen preparations [for use in respirators]. W. ZISCH, ASST. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,745,465, 4.2.30. Appl., 31.1.29. Ger., 30.1.28. Cf. B.P. 319,393; B., 1929, 1034).—Pulverised alkali peroxides (100 pts.) are mixed at a low temperature with less than 23 pts. (preferably 10—15 pts.) of water as spray, vapour, or snow; oxides of iron, manganese, etc. may be added as activators. L. A. COLES.

[Recovery of oxides of nitrogen from waste gases obtained in the] production of nitrates [from chlorides and nitric acid]. B. UEBLER, and KALI-FORSCHUNGS-ANSTALT GES.M.B.H. (B.P. 329,200, 17.6.29). -The waste gases, which contain nitrosyl chloride, chlorine, nitrogen, etc., after, if desired, removal of the free chlorine by fractional distillation at  $-30^{\circ}$ , are passed at the ordinary temperature over scrap iron to yield nitric oxide and a nitrosyl chloride-ferric chloride additive product from which the nitrosyl chloride can be recovered by heating, or are passed over heated iron or lime to yield nitric oxide, or are treated with air and water or some of the original reaction liquid to yield nitric acid and chlorine. The recovered oxides of nitrogen and nitric acid are used again in the main process. L. A. COLES.

Manufacture of liquid nitrogen dioxide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,029, 13.11.28).—Undiluted lower oxides of nitrogen are obtained by converting very dilute mixtures of nitrogen oxides into compounds in which they are chemically combined, but from which they can be recovered only in lower oxidised forms. The recovered lower oxides are then converted into nitrogen dioxide by means of pure oxygen, and the product is condensed to liquid nitrogen tetroxide by cooling to 15—18°.

W. J. WRIGHT.

**Production of elemental sulphur.** R. C. BENNER and A. P. THOMPSON, ASSTS. to GEN. CHEM. CO. (U.S.P. 1,741,551, 31.12.29. Appl., 21.1.27).—Gases containing sulphur dioxide, *e.g.*, roaster or smelter gases, are passed through a layer of hot carbonaceous fuel, whereby the sulphur dioxide is reduced to sulphur, carbon oxysulphide, and hydrogen sulphide. The gases are cooled to about 120° to effect deposition of the sulphur, and the residual gas mixture is passed together with more sulphur dioxide over a catalyst, *e.g.*, bauxite or activated carbon, heated at 200—300° to effect complete conversion of all the sulphur compounds into free sulphur.

A. R. POWELL.

Recovery of sulphur from iron pyrites. R. F. BACON (U.S.P. 1,731,516, 15.10.29. Appl., 13.4.27).— Pyrites or other sulphide ore mixed with sufficient lime to combine with all the silica present is heated in steam at 400—800° in a rotary furnace, the evolved hydrogen sulphide being converted into sulphur by the Claus process or by treatment with sulphur dioxide.

A. R. POWELL.

Electrolytic production of fluorine. K. FREDEN-HAGEN (B.P. 315,768, 17.7.29. Ger., 17.7.28).—Solutions of alkali fluorides in hydrogen fluoride in a molecular ratio less than 1 : 1, but not so low that the electrodes are wetted by the melt, are electrolysed between carbon or graphite electrodes. In the case of potassium fluoride the above ratio is conveniently 5 : 9. The electrodes, not being wetted, are not destroyed, and a temperature appreciably lower than the usual may be employed.

S. K. TWEEDY.

[Electrolytic] production of fluorine. K. FREDEN-HAGEN (B.P. 315,769, 17.7.29. Ger., 17.7.28).—The comminuted alkali hydrogen fluoride is dried, prior to electrolysis, by passing dry indifferent gas, *e.g.*, air, through it at elevated temperature below the m.p. of the salt until the hydrogen fluoride evolved by the salt when periodically thermally decomposed has, after condensation, a specific conductivity of less than 0.02 reciprocal ohm at  $0^{\circ}$ . The drying is preferably effected in the electrolysis vessel itself, which should preferably be air-tight during the electrolysis, but it may also be effected in a separate vessel adapted to charge the cell with the dried material without the latter coming into contact with the atmosphere. The hydrogen fluoride content of the electrolyte is maintained by adding hydrofluoric acid containing only an extremely small quantity of water, *i.e.*, having the conductivity mentioned above.

#### S. K. TWEEDY.

Preparation of metallic cyanamides or mixtures containing them. H. H. FRANCK and H. HEIMANN (U.S.P. 1,745,753, 4.2.30. Appl., 19.10.27. Ger., 23.10.26).—See B.P. 279,419; B., 1929, 171.

Manufacture of iron carbonyl. A. MITTASCH and C. MÜLLER, ASSTS. to I. G. FARBENIND. A.-G. (U.S.P. 1,759,268, 20.5.30. Appl., 29.1.25. Ger., 1.2.24).—See B.P. 244,895; B., 1926, 191.

Apparatus [freezing-press] for making carbonic ice [solid carbon dioxide]. Solid Carbonic Co., Ltd. (B.P. 329,772, 8.4.29. U.S., 15.2.29).

Apparatus for the supply of chlorine or other gas for treatment of liquids. WALLACE & TIERNAN Co., INC., Assees. of G. D. PEET (B.P. 307,473, 8.3.29. U.S., 8.3.28).

Dehydration of solids (B.P. 329,032). Evaporation of solutions (B.P. 304,670).—See I. Cyanide compounds etc. from gases (B.P. 328,738).—See II. Aluminous materials (U.S.P. 1,719,131).—See VIII. Flux for welding (U.S.P. 1,748,785).—See X. Cyanogen compounds (B.P. 318,548).—See XI. Fertilisers (B.P. 299,896 and 328,620).—See XVI. Polluted liquids (U.S.P. 1,747,803).—See XXIII.

## VIII.—GLASS; CERAMICS.

Action of dust from the batch on the checker brick of glass-furnace regenerators. D. S. BELIAN-KIN and M. A. BESBORDOV (J. Amer. Ceram. Soc., 1930, 13, 346—353).—The nature of slag formations in the chambers of regenerators of two types of pot-furnaces is discussed. Microscopical examination showed the constituents of this glazed slag to be alkaline aluminosilicates (nepheline, carnegieite) and some mullite. Analyses indicate that the composition of the slag is more complex than just  $m_{0}^{\prime}$  of fireclay and  $(100 - m)_{0}^{\prime}$  of batch dust. R. J. CARTLIDGE.

Use of borax in manufacture of tank glasses. D. D. SCHURTZ (J. Amer. Ceram. Soc., 1930, 13, 341-345).—A moderate amount of borax in making common types of tank glasses tends to improve melting, reduce factory breakage, and increase lustre, durability, and strength. A glass containing about 73.5% SiO<sub>2</sub>, 2% Al<sub>2</sub>O<sub>3</sub>, 8% (Ca,Mg)O, and 1% B<sub>2</sub>O<sub>3</sub> meets most demands; the beneficial effects, however, are stated to be almost independent of the composition of the glass.

R. J. CARTLIDGE.

Disintegration of glass by means of compressed steam. C. HASSLER (Chem.-Ztg., 1930, 54, 366).—The inner surfaces and edges of Petri dishes which had been heated for 30 min. in steam at 5 atm. showed, on removal from the autoclave next day, a deposit of ice-crystal-like structure. Only traces of condensed water could be detected in the dish. The deposit was distinctly alkaline. The outer surfaces remained unattacked.

S. K. TWEEDY.

Testing of "resistance-glass" ampoules. J. PRITCHARD (Pharm. J., 1930, 124, 539-541).-A 0.02% solution of phenol-red in 60% alcohol is diluted 20 times with freshly distilled water and boiled for 8 min. immediately before use. Of the resulting solution 0.3 c.c. is sealed in a 0.5-c.c. ampoule, which latter is then placed in a Koch's steriliser for 1 hr. The colour of the indicator when cold, is compared with a set of five standards ranging in  $p_{\rm H}$  from below 6.8 to above 8.0, and the ampoule is thus assigned to one of five arbitrary classes. The heating and camparison are repeated on each of the next two days. A batch of ampoules is "good" if 90% of them after the first heating and 80% after the third have  $p_{\rm H}$  7.7 or less. Those of German origin fully conformed to this standard, but English-made ampoules did not, except a sample made from special hard borosilicate glass. The indicator does not change at all in ampoules of pyrex glass. The method of testing recommended in the German Pharmacopœia (1926) gives results in agreement with the author's method, provided the glass is powdered sufficiently to pass through a No. 10 sieve but remain on a No. 20 sieve.

## S. K. TWEEDY.

Volumetric determination of alkali metals in glass. N. A. TANANAEV and A. K. BABKO (Ukraine Chem. J., 1929, 4, 539-543).-0.5 Gram of glass is treated with 2-3 g. of oxalic acid and 4-5 c.c. of a mixture of hydrofluoric and sulphuric acids, and the mass is evaporated at 100° with occasional stirring. On the complete dissolution of the glass, 1 c.c. of 2N-barium chloride is added and evaporation is continued to dryness; then 2-3 c.c. of water are added, and the mixture is again evaporated. This operation is repeated 5-6 times, to the disappearance of acid fumes. Silica, excess hydrofluoric acid, and hydrochloric acid from the decomposed barium chloride are volatilised, and the dry residue consists of barium sulphate, a mixture of oxalates, and excess oxalic acid, the last-named being removed by sublimation at a higher temperature. The residue is extracted with hot water, and ammonia and ammonium carbonate solutions are added to precipitate aluminium and iron. The filtrate, which contains only alkali oxalates and ammonium salts, is evaporated to dryness and the residue ignited to yield carbonate. This is dissolved in water and determined by titration.

R. TRUSZKOWSKI.

Turquoise-blue glaze. G. M. PETERSEN (J. Amer. Ceram. Soc., 1930, 13, No. 5, ii, 47-49).—Trials were made to produce a turquoise-blue colour in a cone 1 alkaline glaze containing barium and calcium. Though the colour was satisfactory, all the samples crazed and had a tendency to "run." F. SALT.

Effect of different fluxing oxides on the constitution of porcelain. A. H. FESSLER and H. NAVRATIEL (J. Amer. Ceram. Soc., 1930, 13, 332—340). —On firing hard siliceous porcelain to a state approaching equilibrium, glasses having the formula  $RO, Al_2O_3, xSiO_2$ are probably obtained. Measurements of solubility and sp. gr., together with micrographs of the fused and quenched materials serve to indicate the numerical values of this formula, by determining the largest amount of silica and alumina that these fluxes (artificial felspars) will hold in solution before crystallisation takes place. R. J. CARTLIDGE.

New form of non-plastic material [for porcelain]. A. H. FESSLER (J. Amer. Ceram. Soc., 1930, 13, 325— 331).—A comparison is made of the use of semi-crystalline (unstable) and crystalline non-plastic materials in porcelain mixtures. The unstable material as produced by heat-treatment consists of a glass phase and an arrested crystalline phase, depending on the molecular ratio of alumina to silica, the fluxing oxides and impurities in the ingredients, and on their rate of cooling. During firing of porcelain the glass partially devitrifies and mullite crystals grow, forming a network of interwovem crystals and resulting in increased resistance to shock and mechanical strength of the finished product.

R. J. CARTLIDGE.

Interpretation of plant and laboratory test data [in the ceramic industry]. R. F. FERGUSON (J. Amer. Ceram. Soc., 1930, 13, 354–362).—Mathematical.

R. J. CARTLIDGE.

Manufactured abrasives. F. L. NOBES (Ind. Chemist, 1930, 6, 183-184).- A modern carborundum furnace is a rectangular firebrick structure with electrodes at opposite ends. A typical charge consists of 56% of sand, 35% of petroleum coke, 7% of sawdust, and 2% of salt. The initial resistor charge in the core of the furnace is granular coke, and a current of 10,000 amp. is used. After interaction is complete the furnace is pulled down, the outer layer of partly reduced silica removed, and the silicon carbide crystals are broken The temperature range for the reaction is 1500up. 2200° and the probable equation 3Si+2CO=2SiC+SiO2. Silicon carbide is slowly oxidised in air at 1000-1350° and reacts with chlorine at 900° and above. It is not acted on by acids, but is attacked by fused alkalis. It has d 3.12 - 3.20, very high thermal conductivity, and low coefficient of expansion. Crystalline fused alumina (alundum etc.) gives better results than carborundum for grinding materials of high tensile strength. It is prepared by treating bauxite in an electric furnace consisting of an unlined, water-cooled. cylindrical, steel shell. The impurities in the bauxite are reduced to ferrosilicon. Alundum has d 3.9, is almost as hard as carborundum, but has not so high a thermal conductivity. It is slightly soluble in hot acids. C. IRWIN.

Gypsum plaster and ceramics. Wiss and others. —See IX. Cadmium-selenium red. MARLEY.— See XIII.

See also A., June, 665, Fluorescence of praseodymium and neodymium glasses (PRINGSHEIM and SCHLIVITCH). 669, Dynamic aggregation in glass (BERGER). 690, Argillaceous suspensions (DUBRISAY and TRILLAT). Dispersion of clays (ALLAM).

## PATENTS.

[Continuous-type tunnel] kilns. H. M. ROBERT-SON (B.P. 311,316, 23.1.29. U.S., 9.5.28).—The kiln comprises zones in succession for preheating, firing, saltglazing, and cooling. Hot air is withdrawn from the cooling zone and passed through passages in the roof to points distributed in the preheating zone; this air returns in the same direction as the goods and in contact with them and is used for secondary combustion in the firing zone, the combined gases from this and the saltglazing zone being withdrawn by a fan through ports situated where the cooling zone commences.

B. M. VENABLES.

Manufacture of glass. K. KÜNZEL (B.P. 314,209, 20.6.28).—The charge is melted in a shaft furnace by means of a flame from the combustion of a liquid or gaseous fuel. The flame is forced through the mass by injector action or is drawn through it by suction applied at the top of the shaft. A. R. POWELL.

[Manufacture of] stratified bodies such as strengthened glass. TRIPLEX SAFETY GLASS Co., LTD., W. R. LYTTLETON, J. WILSON, and H. W. DICK (B.P. 328,919, 2.2., 18.3., and 27.3.29).—After coating the inner surfaces of two or more sheets of glass with gelatin and, preferably, with a cement containing a cellulose ester, these surfaces and/or the layers of strengthening material to be interposed (celluloid) are sprayed with a solvent or plasticiser capable of swelling both. The surfaces to be united are then brought together to effect a preliminary cohesion, and the united sheets are afterwards compressed in a closed vessel at an elevated temperature by means of a fluid under pressure, which may be inert (air) or have a softening action on the interposed layers and coatings (a phthalate or a glycol). F. R. ENNOS.

Rendering enamels cloudy. I. KREIDL (B.P. 313,411 and Addn. B.P. 315,708, [A] 10.6.29, [B] 9.7.29. Ger., [A] 9.6.28, [B] 16.7.28).—(A) Clouding agents such as ammonium oxalate etc. which act by liberating a gas are used in addition to the usual white clouding agents, e.g., tin oxide or zirconium oxide. The latter act as distributors or carriers for the gas-liberating agents, so that enamel batches of any composition may be used; a preliminary clouding of the batch, e.g., by adding fluorine compounds, is thus rendered unnecessary. (B) Any insoluble substance which is not a clouding agent is used as a distributor. It may be chosen so as to modify the viscosity or surface tension of the batch, and may be added to the mill or the melt.

F. G. CLARKE.

Refractories [magnesite bricks]. B. M. O'HARRA and E. A. SLAGLE, ASSTS. to AMER. SMELTING & REFINING Co. (U.S.P. 1,747,905, 18.2.30. Appl., 12.6.28).— Magnesite bricks without an iron or other foreign bond are composed of granular calcined magnesite to which has been added up to 10% of colloidal or finely-divided magnesium carbonate or basic carbonate. The bricks are moulded under high pressure and burned until the magnesium carbonate is converted into periclase.

C. A. KING.

Preparation of aluminous materials [corundum from bauxite]. R. R. RIDGWAY and J. B. GLAZE, Assrs. to Norton Co. (U.S.P. 1,719,131, 2.7.29. Appl., 15.10.26).-A mixture of 100 pts. of calcined bauxite, 5-10 pts. of coke, 5 pts. of pyrites, and 20 pts. of iron borings is fused in a carbon-lined electric furnace of the submerged-arc type and the aluminous slag is separated from the ferrosilicon and ferrotitanium alloy regulus. This slag, which should contain aluminium carbide and less than 3% of aluminium sulphide surrounding crystallised grains of alumina, is roughly broken up and caused to disintegrate by treatment with steam for several hours. The washed product is dried and heated in chlorine to decompose carbides and to volatilise iron and titanium, and the resulting alumina A. R. POWELL. is again fused.

Process and plant for casting rough plate glass. COMP. RÉUNIES DES GLACES & VERRES SPÉCIAUX DU NORD DE LA FRANCE (B.P. 317,820, 3.7.29. Fr., 23.8.28).

Methods and apparatus for colouring hollow glass articles. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of J. T. FAGAN (B.P. 329,940, 15.10.29. U.S., 15.10.28).

Enamelling furnace (B.P. 308,613).—See I. Joining glass to wood etc. (B.P. 303,855).—See V. Torch furnace for mullite (U.S.P. 1,747,756).—See XI.

## IX.—BUILDING MATERIALS.

Measuring viscosity of setting cement (immersion-filter method). K. E. DORSCH and A. DEUBEL (Kolloid-Z., 1930, 51, 180—186).—Measurements of the viscosity of cement pastes carried out by the usual methods did not give reproducible values, but a method of measurement is described which allows determinations to be made with an accuracy of about 5%. The course of setting of all the cements examined is marked by a sharp rise in viscosity during the first 60 min., then a region of constant viscosity for about 30 min., and finally by another slow rise in viscosity. E. S. HEDGES.

Gypsum plaster in the ceramic industries. J. E. WISS, T. P. CAMP, and R. B. LADOO (J. Amer. Ceram. Soc., 1930, 13, 287-314).-The qualities of set plaster are examined. Effects of varying consistency, mixing time, etc. are described and illustrated by graphs, indicating the importance of the water : plaster ratio. In a workable mix the ratio is 0.6:1; on setting, the product has a compressive strength of 2500 lb./in.<sup>2</sup>, a total absorption of 25% by wt., and a dry density of 73.5 lb./cub. ft. Such troubles as pinholes, hard spots, cracking, efflorescence, etc., met with in practice, are considered and methods for their control or prevention are indicated. Physical and chemical tests for the R. J. CARTLIDGE. raw materials are also given.

Penetration of water vapour into wood. L. M. PIDGEON and O. MAASS (Canad. J. Res., 1930, 2, 318— -326).—The rate of diffusion of water vapour through wood fibre was measured by means of a quartz spring balance completely enclosed in an atmosphere of constant vapour pressure, the process being a reversal of the natural drying of wood. Considerably higher rates of diffusion were observed for sapwood than for heartwood, and also when tested in a longitudinal direction with the fibres. At concentrations below the saturation point the movement appears to take place as vapour diffusion through the spaces of the wood structure, due to the pressure gradient caused by the difference of moisture content. C. A. KING.

Analysis of bituminous materials. HYATT.— See II. Maizolith. HARTFORD. Wood-pulp board. BOEHM. Naval stores from waste wood. PALMER.— See V.

## PATENTS.

[Continuous-type] tunnel kilns. H. M. ROBERTSON (B.P. 328,265, 23.1.29).—A kiln suitable for lime burning and similar purposes is constructed so that the preheating is effected by the products of combustion passing countercurrent to the goods, and the cooling by fresh air which is drawn through the goods then used for combustion. A form of carrier is described which comprises closely coupled trough-like trucks on which the material forms a continuous elongated heap. B. M. VENABLES.

Manufacture of material for [heat] insulation and constructional purposes. H. C. M. INGEBERG and A. VESTERLED (B.P. 327,407, 2.1.29).—A plastic mass, the solid constituents of which consist of 99—50% by wt. of defibrated straw or hay and 1—50% of chemical fibrous cellulose, *e.g.*, sulphite-cellulose, is shaped and dried. The articles may be surfaced with an aqueous suspension of cellulose or asbestos containing dyes, fillers, or reinforcing agents, and treated with the usual waterproofing, fire-resisting, and preservative materials. F. G. CLARKE.

Manufacture of cement. A. CARPMAEL. From Soc. POUR L'EXPLOIT. DES PROC. PETRUS (B.P. 305,170, 28.1.29).—The cement comprises a mixture of 5—27% of Portland cement, 2—22% of lime, and 60—80% of puzzuolana prepared from vitrified blast-furnace slag. Plaster of Paris (1—10%) may be added to control the setting. A. R. POWELL.

Cement manufacture. R. W. HYDE, Assr. to DWIGHT & LLOYD METALLURG. Co. (U.S.P. 1,746,944, 11.2.30. Appl., 26.3.27).—The finely-divided raw material for cement manufacture is mixed with pulverised fuel, sintered, and finally heated for a short time to the finishing calcining temperature. C. A. KING.

Cement compositions. H. and N. CADMAN (B.P. 329,107, 6.4.29). —Approx. 1 lb. of hair is mixed with 1 cwt. of Portland or similar cement as a dry mixture for the preparation of a cement plaster for external use. No free lime is used. C. A. KING.

Cement composition. M. E. GRUNEWALD (U.S.P. 1,748,839, 25.2.30. Appl., 4.6.28).—A grouting composition for use under very wet conditions consists of 100 pts. of Portland cement, 0.05—10% (1 pt.) of sodium carbonate, and approx. 100 pts. each of water and sand. C. A. KING.

Cement powders and mortar powders. I. G. FARBENIND. A.-G. (B.P. 328,538, 2.10.29. Ger., 2.10.28). —Sodium silicate cementing compositions are made by using pulverised sodium silicate or other silicates which contain hydrated silicic acid and which are easily soluble in water. C. A. KING. 666

Manufacture of cellular aggregate. R. L. LLOYD, Assr. to DWIGHT & LLOYD METALLURG. Co. (U.S.P. 1,746,860, 11.2.30. Appl., 18.1.27).—The content of combustible matter in waste products is increased and the mixture is spread evenly on a pervious bed and ignited. Combustion of the mass is allowed to proceed until a thin semi-plastic layer is formed which is immediately blown by the current of gas into thin-walled cells and chilled, thus forming a homogeneous, cellular, rigid cake for use as filler in concrete floors, etc.

C. A. KING.

**Production of sand-lime bricks.** H. W. CHARLTON (U.S.P. 1,745,601, 4.2.30. Appl., 18.11.26).—Subsequent to the usual hardening process and after the steam is blown off, a solution, *e.g.*, a 2-3% solution of sodium stearate, is introduced into the cylinder to impregnate the bricks, which are then withdrawn and dried.

C. A. KING.

Preparation of bituminous masses. N.V. MIJN-BOUW-EN CULTUURMAATSCHAPPIJ "BOETON" (B.P. 310,923, 9.3.29. Holl., 3.5.28).—The addition of a small quantity of water when preparing a cold-plastic bituminous mass by mixing a low-boiling solvent with an artificial or natural mixture of bitumen and a filler, *e.g.*, Boeton asphalt-limestone, temporarily reduces the adhesiveness of the bitumen and prevents clotting. The water and solvent may, if desired, be added as an emulsion. The product is applicable to moist surfaces, *e.g.*, soft concrete. F. G. CLARKE.

Treatment of stones or stony materials, cement, concrete, bricks, road surfaces, etc. C. G. Fox (B.P. 328,969, 7.2. and 19.7.29).—Previous to the coating of road aggregate with tar or bituminous emulsions, the aggregate is treated with a dilute (0.5-3%) solution of the emulsifying agent. C. A. KING.

Road-making materials and construction of roads. C. G. J. LEFEBVRE and E. E. F. BERGER (B.P. 307,751, 8.3.29. Fr., 12.3.28).—A tar filler composition for coating macadam road material or for dressing roads consists of about equal proportions of a tar binder and a mineral filler of low porosity, excluding clay and lime. The filler may be quartz sand, quartzites, clinkers, etc. pulverised to pass 300-mesh, and the viscosity of the mixture should be about the same as that of the tar. C. A. KING.

Preservation and disinfection of wood. I. G. FARBENIND. A.-G. (B.P. 305,943, 11.2.29. Ger., 9.2.28). —A mercurised aliphatic hydrocarbon, *e.g.*, mercuric ethyl bromide, is used as the active ingredient, being 10—20 times as active as the inorganic mercury compounds. Such organic compounds are sparingly soluble in water and may be converted into the colloidal state by dissolution in a mixture of water, acetone, and dextrin, or used in solution in organic solvents, *e.g.*, alcohol, phenol. Alkaline salts also increase the solubility of these compounds in water. C. A. KING.

Transfer of wood grain. MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTLICHER OBERFLÄCHEN (B.P. 303,804, 9.1.29. Ger., 9.1.28).—Light is allowed to pass through an extremely thin section of the wood on to a sensitised surface in contact with the wood, by a process similar to the ordinary light-copying process. The wood section may be stained and mounted on glass before copying. C. A. KING.

Manufacture of [heat-]insulating material [from felt]. UPSON CO., Assees. of C. A. UPSON (B.P. 306,559, 23.2.29. U.S., 23.2.28).

[Rotary apparatus for] manufacture of plates from asbestos or fibro-cement. R. BINDSCHEDLER (B.P. 329,850, 28.5.29).

Wall and ceiling coverings and the like. A. HIRSCHFIELD (B.P. 328,985, 8.2.29).

**Process and [hot-air] device for drying timber.** T. C. GROOT (B.P. 329,842, 21.5.29. Holl., 25.1.29).

Cellular products (U.S.P. 1,746,648). Joining wood to metal etc. (B.P. 303,855).—See V. Polluted liquids (U.S.P. 1,747,803).—See XXIII.

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

Heat economy in metallurgical furnaces. (SIR) R. HADFIELD and R. J. SARJANT (Fuel, 1930, 9, 219–224). —Improvement in the heat economy of furnaces can be effected (a) by suitable control of the load; (b) by control, preferably automatic, of the fuel supply; and (c) by the recovery of heat from the waste gases and the furnace structure. Instances from works' practice illustrating quantitatively the advantages of such measures are discussed, and attention is directed to the possibility of using heat-resisting steel in recuperator practice and in the construction of regenerators. A. B. MANNING.

Separation of carbide from rustless non-magnetic B. STRAUSS, H. chrome-nickel steel on heating. SCHOTTKY, and J. HINNÜBER (Z. anorg. Chem., 1930, 188, 309-324).-For the heat treatment of chromenickel steel alloys having the composition C < 0.16, Ni 8.5, Cr 18%, a temperature above 1100° is employed, followed by rapid cooling without subsequent annealing. In the hot-working of such steels a range of temperature exists, near 700°, in which the mechanical and chemical properties are profoundly altered, the metal becoming weak and oxidisable. Measurements of the tensile strength, extensibility, and electrode potential, in conjunction with photomicrographic examination, show that the change in properties is due to the separation of carbide from the initially homogeneous mixed crystals. The increased oxidisability is due partly to surface corrosion and partly to intercrystalline corrosion brought about by the formation of local galvanic elements. Although direct observation shows that the separation of carbide at 800-900° is considerably greater than at 600-700°, the modification of properties is less marked at the former temperatures. This is attributed to the fact that at the higher temperature the carbide is less F. L. USHER. highly dispersed.

Determination of chromium and vanadium after oxidation with potassium bromate; separate titration of chromate and vanadate in the same solution and application of the method in steel analysis. I. M. KOLTHOFF and E. B. SANDELL (Ind. Eng. Chem. [Anal.], 1930, 2, 140-145).—The deter-

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mination of chromium and vanadium in a steel is carried out by oxidising the solution of the steel in sulphuric acid by potassium bromate. The chromium can then be selectively reduced by means of a slight excess of standard arsenious acid and this excess determined by permanganate. After this titration the vanadium is determined by ferrous sulphate, using diphenylbenzidine as indicator. Since tungsten would interfere with this latter titration it must be removed, if present, by the method of Willard and Young (B., 1928, 643). Manganese does not interfere with the determinations. Results obtained in the analysis of steels of known composition agreed well with the known values.

H. INGLESON.

Determination of tantalum, tungsten, vanadium, and molybdenum in high-speed steel. K. Swoboda and R. HORNY (Z. anal. Chem., 1930, 80, 271-288).-The conditions for the quantitative precipitation and determination of the above elements have been studied, and the following procedure is recommended for their determination in high-speed steels. The finely-divided steel  $(1 \cdot 2 \text{ g.})$  contained in a nickel crucible is roasted in oxygen at 400-500° for 5 min.; 12 g. of potassium hydroxide are then added, and the whole is heated to redness for  $\frac{1}{2}$  hr. in a current of oxygen. The melt is dissolved in water, any manganate formed is destroyed with hydrogen peroxide, and the solution is made up to 300 c.c. After filtration, 250 c.c. of the filtrate ( $\equiv 1$  g. of steel) are neutralised with sulphuric acid, using phenolphthalein as indicator. The liquid is boiled for 3 min., 10 c.c. of ammonia are added, and the whole is kept at 100° for  $\frac{1}{2}$  hr. The precipitate of tantalic acid is filtered, washed with ammoniacal ammonium nitrate, ignited, and weighed after removal of silica with hydrofluoric and sulphuric acids. The filtrate is evaporated to dryness after addition of 20 c.c. of concentrated hydrochloric acid and 20 g. of manganese nitrate, and the residue heated until the nitrates are decomposed. The residue from this is treated with 50 c.c. of concentrated hydrochloric acid, the solution diluted, and the precipitated tungstic acid filtered off and weighed after treatment with hydrofluoric acid to remove traces of silica. The filtrate is evaporated to 300 c.c., 20 g. of ammonium chloride are added, and the liquid is neutralised with ammonia; 12 drops of ammonia in excess are then added, and the whole is set aside for 6 hrs. The precipitate containing the vanadium is washed with hot water, redissolved in hot hydrochloric acid, and the solution evaporated to fuming with excess of sulphuric acid. In the resulting diluted solution the vanadium is determined volumetrically by the usual permanganate method, using an excess. of permanganate and back-titrating with arsenious acid solution. The filtrate from the manganese vanadate and chromium hydroxide is evaporated to 100 c.c., treated in a pressure flask with 5 c.c. of concentrated sulphuric acid, diluted, and the molybdenum precipitated by treatment with hydrogen sulphide under pressure. The precipitated molybdenum sulphide is filtered off, roasted in a silica crucible at 450°, and weighed as the oxide. H. F. HARWOOD.

Separation of nickel and cobalt by means of hypochlorite. F. H. RHODES and H. J. HOSKING (Ind. Eng. Chem. [Anal.], 1930, 2, 164—166).—Nickel and cobalt can be separated by fractional precipitation with sodium hypochlorite in a hot alkaline solution. By allowing sufficient time of contact between the precipitate and the hypochlorite it is possible to precipitate most of the cobalt free from nickel and vice versa. A small intermediate fraction is also produced which contains both elements. A semi-large-scale experiment showed that the method might be suitable for industrial operation. H. INGLESON.

Corrosion and protective films. A. C. HANSON (Ind. Eng. Chem., 1930, 22, 554).—From visual observation of the progress of corrosion, caused by the contact of a drop of water saturated respectively with oxygen and carbon dioxide with polished steel, it is considered that electrolytic action begins in the presence of water with the formation of a film of ferrous hydroxide. This film is protective until carbon dioxide reacts to dissolve ferrous carbonate which later is precipitated in the form of ferric hydroxide with the recommencement of electro lytic action. C. A. KING.

Cleaning of oily metal parts on the works' scale. H. STADLINGER (Chem.-Ztg., 1930, 54, 354—355).— The preparation recommended for this purpose consists of a mixture of sodium silicate and trisodium phosphate, for use either in the form of powder or as a 3-4%aqueous solution. It has a powerful emulsifying action on grease of all kinds. It does not corrode aluminium, zinc, or their alloys, and is best applied as a hot spray. C. IRWIN.

Series system of electrolytic copper refining at Nichols Copper Co. F. R. CORWIN and C. S. HARLOFF (Amer. Electrochem. Soc., May, 1930. Advance copy. 10 pp).-Details are given regarding the equip ment and operation of the series system of copper refining at the plant of the Nichols Copper Co., one of the two plants where this system is still in use (cf. Merriss, Min. and Met., June, 1925; Robie, Eng. and Min. J., Feb., 1925). Wooden electrolysis tanks are being replaced by larger cells of concrete. The anode weight has been gradually increased and has now reached 45 kg. The current density has also been raised, the limiting values during 1929 being 2.25 and 2.63 amp./ dm.<sup>2</sup> and the corresponding energy yields 138 and 123 kg./kw.-day, as compared with 109 kg./kw.-day in best practice by the multiple system. The proportion of anode scrap is only about half that in the multiple system and the amount of electrolyte to be circulated per ton of copper about a quarter of that used in the latter system. It is concluded that the plant installation cost for a given tonnage is notably lower for the series system, since less space is required, the power plant will be smaller, the copper tied up in conductors and bus-bars will be less, and the number of cells fewer. Labour costs are lower for the series system and the value of material tied up in process is less. The series system has now been developed to deal with less pure metal without lowering the quality of the product, and the precious metal loss has been controlled so that it compares favourably with that in the multiple system. H. J. T. ELLINGHAM.

Bent-cathode test for control of cyanide copper baths. W. L. PINNER and E. M. BAKER (Amer. Electrochem. Soc., May, 1930. Advance copy. 7 pp.).— The bent-cathode test previously developed as a means of controlling chromium-plating baths (B., 1929, 480) is now applied to cyanide copper-plating baths. The effects of varying the ratio of copper to free cyanide, the concentrations of sodium carbonate and thiosulphate, the temperature, and the mean cathodic current density on the appearance of the deposit on the bent cathode have been investigated. By means of the test the onset of unsatisfactory plating conditions can be anticipated and hence avoided. H. J. T. ELLINGHAM.

Electrodeposition of copper-nickel alloys. L. E. STOUT, O. G. BURCH, and A. S. LANGSDORF (Amer-Electrochem. Soc., May, 1930. Advance copy. 15 pp.). -Determinations have been made of the composition of copper-nickel alloys electrolytically deposited under various conditions from solutions of the complex cyanides of the two metals. The ratio of copper to nickel in the deposit is always greater than that in the bath. Increase in the copper content of the bath increases the copper content of the deposit, but to a much smaller extent than corresponds with the change in metal ratio of the bath. The ratio of copper to nickel in the deposit increases linearly with rise in temperature and also increases with rise in current density. Empirical formulæ representing the effects of these factors on the composition of the deposit are given. Increasing the free cyanide concentration in the bath lowers the current efficiency of deposition. H. J. T. ELLINGHAM.

Cadmium as a plating material. H. KURREIN (Chem.-Ztg., 1930, 54, 333-335).-An account is given of the properties and uses of deposited cadmium and of the methods of deposition, with a résumé of recent literature. S. I. LEVY.

Production of continuous and seamless tubing by electrodeposition. J. BILLITER (Amer. Electrochem. Soc., May, 1930. Advance copy. 8 pp.) .-The metal is deposited on a short, slightly tapered mandrel of chrome or silicon steel. At regular time intervals the deposit is pulled along the mandrel so as to expose a short section of the latter on which is formed a new deposit joined to that previously produced. Continuous tubes of copper, zinc, and iron have been made in this way. In a second method the metal is deposited on the exterior of a continuous lead tube, which is passed slowly through the bath. The lead may afterwards be melted out. H. J. T. ELLINGHAM.

Preparation of metal powders by electrolysis of fused salts. I. Ductile uranium. F. H. DRIGGS and W. C. LILLIENDAHL (Ind. Eng. Chem., 1930, 22, 516-519).-Uranyl salts are unsatisfactory for electrolytic reduction since they appear to ionise as the bivalent uranyl ion and deposit the dioxide. Salts of quadrivalent uranium, e.g., uranous chloride and potassium uranous fluoride, yield the quadrivalent uranium ion and deposit the metal. A quantity was prepared from potassium uranous fluoride (KUF5) in a molten mixture of equal parts of sodium and calcium chlorides, using a carbon crucible as anode and a molybdenum wire as cathode. As removed on the cathode the spongy mass was protected from oxidation by the cooled salts, which were dissolved, and the metal was washed and dried.

Uranium thus prepared was silver-grey in colour and only the finer particles tended to ignite spontaneously, but pellets pressed from the grains ignited more readily. The carefully fused metal drained from any impurities contained 99.9% U, 0.055% Fe, 0.06% C, and 0.015% Si. Hydrochloric acid dissolved the metal readily, sulphuric acid more slowly, whilst nitric and acetic acids appeared to have negligible effect. Properties of uranium include d 18.9 (fused) and 19.07 (rolled), m.p. 1690°, sp. resistance  $32 \cdot 1 \times 10^{-6}$  ohm per cm.<sup>3</sup>, hardness (fused) B92—94 (Rockwell). C. A. KING.

Indium : recovery by electrodeposition. L. R. WESTBROOK (Amer. Electrochem. Soc., May, 1930. Advance copy. 7 pp.).-In the purification of liquors obtained in the acid leaching of roasted zinc ores indium is precipitated with cadmium and nobler metals by treatment with zinc dust. In working up this precipitated sponge for cadmium, indium is removed with the other impurities and can be partially separated from the latter by adding specially pure zinc shavings to their sulphate solution. The crude indium was purified by successive precipitations as hydroxide, and an oxide of 99.5% purity (In<sub>2</sub>O<sub>3</sub>) was eventually obtained. 200 G. of this oxide were dissolved in 600 c.c. of water containing 120 c.c. of 96% sulphuric acid, 250 g. of sodium citrate added, and the solution was made up to 1 litre. This solution was electrolysed at room temperature, using a platinum anode and an indium or steel cathode at a cathodic current density of 2 amp./dm.<sup>2</sup> The indium deposit was compact and could be built up to 1 in. or more in thickness. Over 1 lb. of the pure metal was recovered in this way. The electrodeposition of indium from other types of bath was investigated and previous work on the subject is reviewed.

H. J. T. ELLINGHAM.

Status of electrolysis as a metallurgical process. T. H. DONAHUE (Amer. Electrochem. Soc., May, 1930. Advance copy. 20 pp.) .- Dominant and alternative metallurgical processes for the extraction and refining of the principal metals are tabulated, and statistics are collected to show the extent to which electrolysis 18 employed in the extraction of copper, zinc, and cadmium, and in the refining of copper, lead, nickel, and silver. The importance of the recovery of precious metals in the economy of electrolytic processes is discussed. Consideration is given to other processes such as treatment with chlorine, cementation on iron, and physical methods of separation, which are actual or possible competitors with the electrolytic processes, and the future of electrolysis in metallurgy is discussed in the light of these considerations. Expansion in the electrolytic extraction of zinc and refining of copper is expected, and electrolytic nickel refining is increasing rapidly in importance. Factors limiting the development of electrolytic copper extraction and lead refining are discussed, and the employment of electrolytic methods in the refining of tin, antimony, cobalt, iron, and the rarer metals is referred to. The possibility of the electrolytic production of oxides or basic salts of metals which cannot be electrodeposited in the free state 18 H. J. T. ELLINGHAM. considered.

Metallurgical coke. PURVES. Submerged com-

bustion. HAMMOND.—See II. Ajax-Wyatt induction furnace. ADAM.—See XI.

See also A., June, 669, Recrystallisation of metals (BERGER). 670, Testing of monocrystalline wires (DE HAAS and VAN ALPHEN). 680, Nickel-chromium system (MATSUNAGA). 681, System chromiumcarbon (WESTGREN and PHRAGMÉN). X-Ray analysis of iron and iron-manganese alloys (Shimura and WADA). Barium-tin alloys (RAY and THOMPSON). Silver-cadmium alloys (FRAENKEL and WOLF). System bismuth-tellurium (KÖRBER and HASCHIMOTO). 682, Diffusion of molybdenum and iron in the solid state (GRUBE and LIEBERWIRTH). System aluminiummagnesium-zinc (NISHIHARA). 707, Electrodepo-sition of copper (MARIE and GÉRARD; FOERSTER and GÄBLER). Deposition potential of alumina (DROSS-BACH). 715, Preparation of thallium (ANDRIEUX). 719, Behaviour of silver and of silver-copper alloys on ignition in oxygen (LEROUX and RAUB). Preparation of definitely oriented single crystals of metals (GLOCKER and GRAF).

## PATENTS.

Metallurgical furnace. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 316,986, 15.2.29. U.S., 11.5.28.).—The indirect heating of a reducing chamber situated above a smelting furnace is effected by completing the combustion of the furnace gases in a combustion chamber and passing the hot gases through a number of tubes which pass through the reduction chamber. Alternatively, the tubes may be lengthened and heated externally in the combustion chamber, in which case air is passed through the tubes to be later used for primary or secondary combustion.

C. A. KING. Apparatus for reducing ores. G. S. HOWELL (U.S.P. 1,747,716, 18.2.30. Appl., 14.11.28).—A reducing shaft furnace has a funnel-shaped base which acts as a bosh and into which tuyères are fitted. Smelted material flows through the bottom into a lower chamber which acts essentially as a separator for slag and metal which may be drawn off at different levels. Suitable means for condensing fumes from the smelting are provided. C. A. KING.

**Ore-reduction furnace [for mercury ores].** J. M. SMITH (U.S.P. 1,747,740, 18.2.30. Appl., 5.6.28).—The furnace consists of a lower circular chamber surmounted by a cupola. A circular table of slightly frusto-conical shape forms the roasting bed in the lower chamber and is fed from a series of superposed, hollow, conical baffles in the cupola above. During operation the roasting table rotates and discharges the hot residue down an exit pipe, which serves as an entry for air for combustion. C. A. KING.

Blast furnace. H. KOPPERS (U.S.P. 1,746,705, 11.2.30. Appl., 29.9.22. Renewed 1.3.27).—The uppermost part of a blast furnace consists of a double-walled steel cylinder in line with the refractory walls below. The jacket is water-cooled and the inner wall preferably corrugated to strengthen it against the wear of the charge. An expansion joint between this portion and the brickwork below is made by flanged plates angularly disposed and packed with asbestos. C. A. KING. Utilisation of the heat of slag. COMP. DES FORGES DE CHATILLON-COMMENTRY ET NEUVES-MAISONS (B.P. 327,732, 10.1.29. Fr., 6.12.28).—Hot slag cast into blocks is introduced into either a vertical or horizontal chamber provided also with a discharge opening, and a fluid is circulated through the chamber in countercurrent to the movement of the slag. C. A. KING.

Treatment of ferrotitanium [ilmenite] ores. G. J. BANCROFT (U.S.P. 1,745,732, 4.2.30. Appl., 19.2.29). —Titaniferous iron ores are ground with fluorspar and carbonaceous material and the mixture is heated in a reducing atmosphere to produce iron sponge and calcium fluotitanate. The reduced mass is discharged through steam into water containing a small proportion of sodium hydroxide, and the granular product is ground wet, screened to remove coarse iron, and concentrated on Wilfley tables or by means of a magnetic separator. A. R. POWELL.

Dephosphorisation of iron. P. RIES and F. BICHE-ROUX (B.P. 310,468, 11.4.29. Belg., 26.4.28).—The lime is introduced into the converter partly as lumps at the beginning of the blow and partly as powdered lime in the blast during the decarburisation stage of the blow. The lime-laden air-blast is supplied at the bottom of the converter from a chamber which is provided with a tangential entry for the air and which communicates with the bottom tuyères by way of a plate having counterbored inlets to the tuyères. The bottom of the converter is closed with a cover pressed up to a conical shape on the inner side to prevent accumulation of lime powder in the feed chamber. A. R. POWELL.

Pickling of iron and steel. VER. F. CHEM. U. MET. PROD. (B.P. 312,996, 29.5.29. Czechoslov., 4.6.28).— A small quantity of a sulphonation product of crude carbazole or an alkali or alkaline-earth sulphonate derived therefrom is added to the pickling bath as an inhibitor. A. R. POWELL.

Recovery of nickel and other metals from ores containing nickel. F. L. DUFFIELD (B.P. 328,696, 8.2.29).—The ore is roasted at 750—1000° until conversion of the iron into oxide and the nickel and copper into sulphates occurs, sodium, magnesium, or calcium chloride is then added, and the mixture heated at 650° to convert the nickel and copper into chlorides which are subsequently removed by raising the temperature to cause them to volatilise. The residual iron oxide is reduced with a gaseous reducing agent and separated from the gangue magnetically. The volatilised chlorides are dissolved in water and separated by chemical or electrolytic means. A. R. POWELL.

Carbonisation of nickel and nickel alloys. WEST-INGHOUSE ELECTRIC & MANUF. Co., Assees. of H. McK. ELSEY (B.P. 305,465, 25.1.29. U.S., 4.2.28).—Nickel or nichrome for the manufacture of radio-valve grids is oxidised by heating in the air, then carbonised by heating at 700—1000° in an atmosphere of nitrogen and a hydrocarbon gas free from hydrogen. A. R. POWELL.

Treating [lead sulphate] ores and the like. F. S. MULOCK, ASST. to U.S. SMELTING, REFINING, & MINING Co. (U.S.P. 1,745,945, 4.2.30. Appl., 8.1.24).—The ore (native lead sulphate or roasted sulphide) is leached at 100° with hot, acid brine containing sufficient calcium chloride to convert all the sulphate present into calcium sulphate. After filtration the solution is cooled to 0°, preferably after dilution with 5—25% of its volume of water, whereby pure lead chloride is obtained. This is separated, dried, and smelted with lime, sodium chloride, and carbon to produce metallic lead and calcium chloride for use again in the process. The mother-liquor from the lead chloride is treated with lime to precipitate other metals which may be present and the clarified solution is re-acidified for use again. A. R. POWELL.

[Lead bearing-metal] alloy. J. V. O. PALM, Assr. to CLEVELAND GRAPHITE BRONZE CO. (U.S.P. 1,741,733, 31.12.29. Appl., 30.9.26).—An alloy of 70-75% Pb, 5-6% Cu, 4% Hg, 4-8% Sb, and about 12% Sn is claimed; it has a fine crystal structure and increases in hardness aud strength after 24 hrs. at the ordinary temperature. A. R. POWELL.

Welding lead. W. SMITH, and EXPANDED METAL Co., LTD. (B.P. 329,023, 11.2.29. Addn. to B.P. 285,995; B., 1928, 373).—The surfaces to be joined are amalgamated and then welded together by running a fan-tailed gas flame along the joint. [Stat. ref.] A. R. POWELL.

Flux [for autogenous welding]. W. B. MILLER and A. R. LYTLE, ASSTS. to OXWELD ACETYLENE Co. (U.S.P. 1,748,785, 25.2.30. Appl., 28.8.28).—The flux, particularly adapted for use with aluminium bronzes, consists of 10—50% of a fluoride (e.g., of an alkali metal) and a borosilicate glass, preferably containing more boric acid than silica. The fluoride is added either to the powdered or the molten glass, and the mixture is pulverised after cooling. S. K. TWEEDY.

Obtaining chromium [powder]. M. N. RICH, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,741,955, 31.12.29. Appl., 9.11.27).—Chromic oxide obtained by ignition of ammonium dichromate is heated at 900° in a rapid current of hydrogen which has been rigorously purified from oxygen, nitrogen, hydrocarbons, and water vapour. The product is suitable for pressing and sintering into bars which may be swayed and drawn into wire.

A. R. POWELL. [Chromium-nickel] alloys. INTERNAT. NICKEL Co., and P. D. MERICA (B.P. 328,362, 16.5.29).—Nickel alloys containing 0.1—10% Fe, 10—20% Cr, 0.1—3% Mn, 0.03—0.5% C, and 0.1—5% Al, 0.03—0.15% Mg, or 0.03—0.15% Ca are claimed. The aluminium, magnesium, or calcium is added just prior to casting to effect deoxidation and to ensure the production of an alloy which may readily be hot-worked, rolled, or forged. A. R. POWELL.

Tungsten-tantalum alloy. J. H. RAMAGE, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,741,953, 31.12.29. Appl., 28.5.27).—Tungsten alloys with 0.5—1.5% Ta for the manufacture of lamp filaments are prepared by precipitating a mixture of potassium tungstate and tantalate with a slight excess of hydrochloric acid and heating the mixture with 0.15—0.4% of lampblack for 3 hrs. in a current of coal gas at 800°, then for 5—6 hrs. at 1200—1400° in a current of hydrogen until the carbon is completely removed. A. R. POWELL.

Silver extraction [from ores]. E. HAEHRE, Assr. to T. ROBERTS & Co. (U.S.P. 1,747,072, 11.2.30. Appl., 15.2.28. Norw., 19.9.27).—Ores containing native silver are agitated in a pulp containing an alkali sulphide and the mixture is heated with steam to convert the silver wholly or superficially into sulphide, which is then separated by flotation. A. R. POWELL.

Treatment of platinum ores, concentrates, etc. containing chromite. A. R. Powell, E. C. DEERING, and JOHNSON, MATTHEY, & CO., LTD. (B.P. 328,564, 28.1.29).—Chromite concentrates containing platinum are roasted with lime and litharge to produce lead chromate and ferric oxide, silica is added, and the roasting continued until a sintered, granular mass is obtained, and finally this mass is smelted in a lead blast-furnace to obtain platiniferous lead bullion from which the precious metals are recovered by cupellation. Alternatively the concentrates are roasted with lime and sodium carbonate and the product, with or without a previous leaching to remove soluble chromates, is smelted with matte-forming materials to produce a matte from which the platinum is recovered as described in B.P. 316,063 (B., 1929, 822). A. R. POWELL.

Flotation apparatus. J. P. RUTH, JUN. (U.S.P. 1,746,682, 11.2.30. Appl., 8.10.27).—An elongated tank having sloping sides or a concave bottom is divided in the upper part into three sections by two vertical, perforated walls. Compressed air is injected near the bottom between two L-shaped partitions extending the length of the tank below the normal pulp level and the froth is carried over into launders. C. A. KING.

Metal polishes. W. HESSELGRAVE (B.P. 327,783, 26.1.29).—Precipitated chalk and turpentine are mixed to form a semi-liquid mass, ammonia solution is added, and the whole diluted with water.

H. ROYAL-DAWSON.

Method and means for electrically heating metals. F. W. THOMPSON (B.P. 328,270, 18.10.28).— Metal articles are heated electrically at a predetermined rate to a definite temperature; the applied voltage is then automatically changed to a lower value by operation of a switch controlled by the resistance of the article, so that the heating is continued at a different rate.

J. S. G. THOMAS.

Manufacture of electrolytic iron. R. D. PIKE (U.S.P. 1,746,903, 11.2.30. Appl., 4.1.26).—Sponge iron produced by the reduction of leached pyrite cinders is dissolved in a solution of ferrous and ferric chlorides containing 8—10% Fe and the solution is purified from copper and zinc by treatment with scrap iron and hydrogen sulphide. The purified solution is then electrolysed at 70° in a divided cell to obtain electrolytic iron and an anolyte containing ferric chloride which is returned to the leaching tanks. A. R. POWELL.

Electrodeposition of a coating of zinc [on iron or steel]. Q. MARINO (B.P. 305,035, 28.1.29. Fr., 28.1.28).—The electrolyte comprises a solution of 200—250 g. of zinc sulphate, 20—25 g. of aluminium sulphate, 50—60 g. of zinc chloride, and 20—50 g. of sodium or potassium borobenzoate per litre.

A. R. POWELL.

Cleansing or scouring iron and steel by electrochemical means. Q. MARINO (B.P. 305,036, 28.1.29. Fr., 28.1.28).—The bath comprises a solution of sodium or potassium sulphate in the liquor obtained by treating calcium phosphate with sulphuric acid ( $d \ 1 \cdot 1 - 1 \cdot 2$ ). The article is made first the cathode, then the anode.

A. R. POWELL.

**Protection of cuprous materials.** I. T. BENNETT, Assr. to T. E., J. B., T. E., JUN., and J. F. MURRAY (U.S.P. 1,746,987, 11.2.30. Appl., 6.3.25).—Copper is plated first with iron, then with chromium, and finally with nickel, and the whole is heated at 850—900° for 4—5 hrs. to cause the three plated metals to diffuse into one another to make a corrosion-resistant nickelchromium-iron alloy coating on the copper.

A. R. POWELL.

Electrolytic production of insulating layers on articles of aluminium and its alloys. E. W. KÜTTNER (B.P. 329,190, 6.6.29).—The articles are subjected to anodic oxidation in a bath containing oxalic acid and a strong mineral acid, *e.g.*, hydrochloric acid, or a powerful oxidising agent, *e.g.*, potassium permanganate or dichromate or derivatives thereof.

A. R. POWELL.

Cadmium plating. C. H. HUMPHRIES (B.P. 328,574, 29.1.29).—A salt mixture for the preparation of a cadmium-plating bath consists of  $2 \cdot 5$ —3 oz. of cadmium oxide, 6 oz. of sodium hydrogen sulphate, 4 oz. of ammonium sulphate, and 1—10 g. of a sugar, preferably cane-sugar caramel. This mixture is dissolved in 1 gal. (Amer.) of water for use in the bath.

A. R. POWELL.

Manufacture of pure iron. (A—c) A. MITTASCH and C. MÜLLER, (A, C) E. LINCKH, and (B) W. SCHUBARDT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,759,658— 1,759,660, 20.5.30. Appl., [A] 21.12.25, [B] 4.3.26, [C] 2.7.27. Ger., [A] 15.12.24, [B] 23.5.25, [C] 5.7.26).— See B.P. 262,938, 269,677, and 281,963; B., 1927, 144, 527; 1928, 127.

Finely-divided metals from metal carbonyls. C. MÜLLER and W. SCHUBARDT, ASSTS. to I. G. FARB-ENIND. A.-G. (U.S.P. 1,759,661, 20.5.30. Appl., 2.7.27. Ger., 6.7.26).—See B.P. 284,087; B., 1928, 270.

Working up iron ores in a direct way. P. H. LEDEBOER, ASST. to STAALSYNDICAAT DR. LEDEBOER II (U.S.P. 1,759,753, 20.5.30. Appl., 17.3.28. Holl., 30.3.27).—See B.P. 287,925; B., 1929, 984.

Blast furnace for reduction and recovery of volatilisable metals as oxides and their operation. W. JOB, Assr. to AMER. LURGI CORP. (U.S.P. 1,759,559, 20.5.30. Appl., 29.6.26. Ger., 2.7.25).—See B.P. 268,201; B., 1927, 415.

Removal of incrustation formed on metallic surfaces. R. SELIGMAN (U.S.P. 1,759,762, 20.5.30. Appl., 1.8.27. U.K., 4.11.26).—See B.P. 284,778; B., 1928, 271.

Enamelling furnace (B.P. 308,613). Separation of materials (B.P. 328,283).—See I. Joining metal to wood etc. (B.P. 303,855).—See V. Treating mixtures of alkali stannates, arsenates, and antimonates (U.S.P. 1,747,709). Lyes containing copper and zinc (B.P. 329,225). Recovering vanadium from solutions (U.S.P. 1,747,403).—See VII. Magnesite bricks (U.S.P. 1,747,905).—See VIII. Furnace for iron ores (U.S.P. 1,748,805). Controlling electrodeposition (B.P. 328,057).—See XI.

## XI.—ELECTROTECHNICS.

The Ajax-Wyatt or vertical-ring induction furnace. W. ADAM, JUN. (Amer. Electrochem. Soc., May, 1930. Advance copy. 22 pp.).-The ideal characteristics of an electric furnace for melting non-ferrous metals and alloys are set out and the extent to which these characteristics are possessed by the vertical-ring induction furnace is discussed in detail. The electrical and thermal efficiencies may be as high as 85%. Efficient circulation of the metal produced by the combined action of the motor effect, the pinch effect, and the Joüle effect ensures homogeneity of the melt and avoids local overheating, thus keeping metal losses very low. Since the heat is produced in the metal itself, there is no heat lag. The necessity of keeping a "heel" of molten metal in the ring is a disadvantage in changing over from one alloy to another. The power factor is high, and current surges cannot occur. Operating data are given, including a typical melting cost analysis of an induction furnace working 9 hrs. per day, making pressure castings of red brass. Furnaces are available with power inputs ranging from 45 to 150 kw., and pouring capacities from 400 to 2200 lb. per heat.

H. J. T. ELLINGHAM.

Contact rectification. I. Classificiation of contact rectifiers. M. BERGSTEIN (Amer. Electrochem. Soc., May, 1930. Advance copy. 8 pp.).-The literature on contact rectifiers is reviewed. Such rectifiers may be divided into integral junctions, in which the dissimilar electrodes are formed on one another, as in the copper-cuprous oxide rectifier, and non-integral junctions, where the electrodes are brought into intimate contact by pressure, as in the cupric sulphide-magnesium rectifier. Further classification is based on the chemical character of the electrodes. In general, rectified current flows from the electronegative to the electropositive member of the junction, but in some cases the reverse is reported. For the cupric sulphide-magnesium rectifier this reversal is shown to be related to the H. J. T. ELLINGHAM. voltage across the junction.

Magnetic concentration of certain natural and artificial manganese dioxides. T. J. MARTIN (Amer. Electrochem. Soc., May, 1930. Advance copy. 13 pp.). -Manganese dioxide is weakly magnetic, and this property has been utilised in concentrating pyrolusite ores, particularly in obtaining the grades of dioxide required for dry cells. Concentration tests at various magnetic intensities have now been carried out on a number of natural and artificial manganese dioxides ground to various degrees of fineness. Highly magnetic impurities can be separated, but efficient separation of weakly magnetic impurities is not always possible at the flux densities employed. Efficient separation is less possible with finely-ground than with coarsely-ground ores. Even when a manganese dioxide product separates well, no predictions as to the composition of the fractions

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can be made. Artificial manganese dioxides differ as widely from each other in magnetic susceptibility as do the ores. The possible cause of these differences is discussed. H. J. T. ELLINGHAM.

Humidity recorder. ROSECRANS; WALKER and ERNST. Ultrafiltration funnels. ZINZADZE.—See I. Maizolith. HARTFORD.—See V. Splitting of mica. OBREIMOV.—See VII. Abrasives. NoBES.—See VIII. Strontium-tin alloys. RAY. Analysis of white metals and solder. MATTHIJSEN. Copper refining. CORWIN and HARLOFF. Control of cyanide copper baths. PINNER and BAKER. Copper-nickel alloys. STOUT and others. Cadmium plate. KURREIN. Seamless tubing. BILLITER. Uranium. DRIGGS and LILLIENDAHL. Indium. WESTBROOK. Electrolysis in metallurgy. DONAHUE.—See X.

See also A., June, 681, Electrical conductivity etc. of lead-antimony alloys (STEPHENS). 707, Electrodeposition of copper (MARIE and GÉRARD; FOERSTER and GÄBLER). Deposition potential of alumina (DROSSBACH). Mechanism of a simple voltaic cell (LATTEY and PERRIN). Mercury-basic mercury sulphate cell (VOSBURGH and LACKEY). Pyrochemical Daniell cells (OPPENHEIMER). 715, Preparation of arsine and stibine (HLASKO and MASLOWSKI). Preparation of thallium (ANDRIEUX). 725, Analysis of nitrating acid (MÜLLER and KOGERT).

## PATENTS.

Induction furnaces. P. E. BUNET, and ACIÉRIES DE GENNEVILLIERS (B.P. 306,144, 14.2.29. Fr., 17.2.28).— A very low-resistance, air- or water-cooled, primary circuit is arranged as close as possible to the secondary circuit and is fed with current at a voltage which is substantially equal to that desired in the secondary, or is a low multiple thereof. J. S. G. THOMAS.

Electric furnace [for smelting iron ores]. A. STANSFIELD (U.S.P. 1,748,805, 25.2.30. Appl., 22.3.29). —The charge is partly reduced and prepared for final treatment in the furnace, by gases given off in the furnace as the charge descends in thin layers over a series of inclined surfaces over which the gases flow in countercurrent to the charge. J. S. G. THOMAS.

Electric torch furnace [for melting ceramic materials, especially mullite]. T. S. CURTIS, ASST. to VITREFRAX CORP. (U.S.P. 1,747,756, 18.2.30. Appl., 30.7.28).—A preheated bar of ceramic material is fed in line with, but in the opposite direction to, one of a pair of electrodes arranged at right angles, and provided below with quenching means, and is fused by the arc between the electrodes. J. S. G. THOMAS.

Electric furnace for continuous production of cyanogen compounds. O. STALHANE (B.P. 318,548, 28.8.29. Swed., 5.9.28).—In a furnace constructed in accordance with B.P. 272,996 (B., 1927, 602), comprising an inner and an outer tube, insulated at one end and in metallic connexion at the other, current is supplied from a source of two- or poly-phase current to both tubes at the insulated end. One end of the inner tube is connected to the earth-connected zero-point of the current system. J. S. G. THOMAS. Current-rectifying device. W. O. SNELLING (U.S.P. 1,745,723, 4.2.30. Appl., 24.2.23).—A homogeneous mixture of copper sulphide and lead sulphide is claimed. J. S. G. THOMAS.

Alternating-current rectifying element. S. J. M. ALLEN (U.S.P. 1,746,511, 11.2.30: Appl., 26.5.27).— A copper plate, coated with copper oxide and heated to 650—705°, is quenched in an inert liquid, *e.g.*, water. J. S. G. THOMAS.

Rectifying device and its production. W. D. DOOLEY (U.S.P. 1,748,012, 18.2:30. Appl., 12.9.28).— An aluminium plate coated electrolytically with a film of oxide is heated at 150—280° and exposed to vapours, whereby a superposed coating of aluminium sulphate is formed. The plate is then heated until by decomposition a second film of oxide is formed upon the first.

J. S. G. THOMAS.

Forming copper-copper oxide rectifier plates. A. K. CROAD. From HANOVIA CHEM. & MANUF. Co. (B.P. 328,701, 11.2.29).—A prepared copper plate heated to a high temperature in an oxidising atmosphere is quenched by the vapour of an active substance, *e.g.*, quinol, in an inactive base liquid, *e.g.*, liquid paraffin wax, at between 70° and 169°. J. S. G. THOMAS.

[Controlling electrodeposition in] electrolytic processes. K. BREUSING and U. GOTTESMANN (B.P. 328,057, 30.1.29).—The direction and density of current flow are controlled by magnetic, electromagnetic, and/or electrostatic means. J. S. G. THOMAS.

[Wehnelt cathode for] electric-discharge tubes. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPEN-FABR. (B.P. 328,641, 1.1.29).—A coherent, relatively thin sheath of tungsten is formed on a core of a material, *e.g.*, molybdenum, which becomes brittle at a higher temperature than tungsten. If desired, thorium oxide or other material of high electronic emission may be incorporated in the core. J. S. G. THOMAS.

Source of light. [Glow-discharge gas-filled lamp.] T. W. CASE, Assr. to CASE RES. LAB., INC. (U.S.P. 1,747,287, 18.2.30. Appl., 19.11.27. Renewed 9.7.29). —The gas-filling of a glow-discharge lamp having a pair of spaced electrodes, the negative one being coated with an alkaline-earth metal, preferably barium oxide, consists of a mixture of hydrogen with 1—25% (preferably 5% or less) of nitrogen. J. S. G. THOMAS.

Electrical method and apparatus. [Electrical conductor.] J. KREMER (U.S.P. 1,748,927, 25.2.30. Appl., 1.12.25. Renewed 10.9.29).—Current is passed through molten sodium maintained in circulation in a conduit provided with cooling means.

J. S. G. THOMAS.

[Cooling and insulating medium for] electrical apparatus. L. R. WILDER (U.S.P. 1,746,977, 11.2.30. Appl., 6.11.26).—The use of diphenyl ether, maintained in liquid form by a heating coil, is claimed.

J. S. G. THOMAS.

Insulating material for electric condensers etc. J. E. G. LAHOUSSE (B.P. 329,604, 10.10.29. Fr., 22.10.28).—Paper is impregnated with diphenyl ether or its equivalent, e.g., benzyl oxide, either alone or containing dissolved wax, resin, resinates, amides, e.g., acetanilide, urea, etc. J. S. G. THOMAS. Electrical insulating materials. W. S. SMITH, H. J. GARNETT, J. N. DEAN, B. J. HABGOOD, and H. C. CHANNON (B.P. 328,025, 14.11.28).—For submarine telephones or cables, a mixture of dirt-free gutta-percha and/or balata, with rubber from which all proteins and other non-rubber compounds have been removed, is claimed. (Cf. B.P. 307,390; B., 1929, 400.)

## J. S. G. THOMAS.

[Electrodes for] electric accumulators. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 329,514, 22.5.29).—A combination of porous lead anodes produced by eliminating admixed metal from an alloy of lead with an alkali or alkaline-earth metal, and cathodes consisting of the usual commercial negative electrodes is claimed. J. S. G. THOMAS.

Manufacture of active masses for [alkaline] electric accumulators. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 328,096, 22.3.29).—A mixture composed of the active material and a highly viscous liquid soluble in water, *e.g.*, a mixture composed of nickel hydroxide (80 pts.), graphite (20 pts.), and glycerin (15 pts.), is pressed by a matrix having a relatively long extension orifice into the form of a band, thread, or small tube, which is subdivided, worked up into the desired shapes, and pressed directly into containers. J. S. G. THOMAS.

Salvaging of storage battery plates. C. B. WHITE (B.P. 328,123, 30.4.29).—The plates are heated so as to liquefy only the metal (antimonial lead) forming the grid frame, which is collected; the mixture of hot, active or filler material and enclosed liquid metal is then disintegrated by projection upon a rough cooler surface and collected, the metal thereby adhering to the roughened surface. J. S. G. THOMAS.

**Irradiation of substances.** E. BUHTZ (B.P. 328,410, 3.4.29).—Suspensions or fine powders in thin layers to the centre of a flat, slowly rotating disc are irradiated by a source, preferably annular in shape, arranged above the disc. The process is applicable to milk.

## J. S. G. THOMAS.

Stabilisation of non-crystalline, piezo-electric microphone plates. TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 316,628, 1.8.29. Ger., 1.8.28).—A direct-current potential is continuously applied to the electrodes of plates produced by the action of high-tension direct current upon a cooling mass of resin, wax, etc. J. S. G. THOMAS.

Manufacture of laminated magnetic bodies and the like. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 314,084, 20.6.29, Ger., 22.6.28).—Metal laminations are held together by reaction products of polyhydric alcohols and polybasic acids or their respective anhydrides.

#### J. S. G. THOMAS.

Electrode compositions for electron-discharge devices. S. DUSHMAN, ASST. to GEN. ELECTRIC CO. (U.S.P. 1,747,061-4, 11.2.30. Appl., 2.7.23).—See B.P. 218,630; B., 1925, 794.

Electric-discharge tubes. N.V. PHILIPS' GLOEI-LAMPENFABR. (B.P. 309,584, 26.3.29. Holl., 13.4.28). [Envelope for] electric-discharge tubes. M.-O. VALVE CO., LTD., and W. H. PETERS (B.P. 328,763, 9.4.29).

Electron-discharge devices. CLEARTRON (1927), LTD., and J. R. SINCLAIR (B.P. 329,802, 27.4.29).

[Electrodes for] luminous electric-discharge tubes. A. E. CHAPMAN and J. VIELLE (B.P. 329,639, 18.12.28).

Long-duration arc-lamps. SIEMENS-PLANIAWERKE A.-G. F. KOHLEFABRIKATE (B.P. 328,071, 18.2.29. Ger., 16.11.28).

[Reducing the tendency to "flashing" of] gasfilled electric incandescence lamps. GEN. ELECTRIC Co., LTD., J. T. RANDALL, and L. C. JESTY (B.P. 329,446, 26.3.29).

Construction of photo-electric cells. G. B. ELLIS. From Soc. des Usines Chim. Rhône-Poulenc (B.P. 328,451, 4.5.29).

Construction of light-sensitive [selenium] cells. A. E. JONES (B.P. 327,674, 8.10.28).

[Accumulators or other] galvanic batteries. OLDHAM & SON, LTD., and W. D. WILDE (B.P. 329,831, 15.5.29).

Pasting of grid strips for the manufacture of accumulator plates. FRIEMANN & WOLF GES.M.B.H. (B.P. 329,889, 8.7.29. Ger., 16.3.29).

Heat for chemical reactions (B.P. 328,635). Temperature regulator (B.P. 299,714).—See I. Marking material by electrodeposition (B.P. 329,258) .-See V. Phosphoric acid (B.P. 319,656). Acidresisting materials (U.S.P. 1,732,140). Treating mixtures of alkali stannates etc. (U.S.P. 1,747,709). Fluorine (B.P. 315,768-9).-See VII. Nickel etc. from ores (B.P. 328,696). Carbonisation of nickel (B.P. 305,465). Tungsten-tantalum alloy (U.S.P. 1,741,953). Heating of metals (B.P. 328,270). Electrolytic iron (U.S.P. 1,746,903). Coating zinc on iron or steel (B.P. 305,035). Cleansing iron or steel (B.P. 305,036). Protecting cuprous materials (U.S.P. 1,746,987). Insulating layers on aluminium etc. (B.P. 329,190). Cadmium plating (B.P. 328,574). -See X. Rendering victuals radioactive (B.P. 329,292).-See XIX. Removing oxygen from water (G.P. 456,459).-See XXIII.

## XII.—FATS; OILS; WAXES.

Detection of hardened fats. H. P. KAUFMANN and M. KELLER (Chem. Umschau, 1930, 37, 142-144).—By the use of Grossfeld's improved lead salt separation (B., 1930, 466) the authors were successful in detecting isooleic acid due to admixtures of 2% of hardened arachis and blubber oils, or 20% of hardened coconut oil; addition of 3% of hardened coconut oil or 5% of fully hardened maize oil (both of which contain very little isooleic acid) could not be detected by this method. The detection of nickel in milk chocolate cannot be taken as an indication of the presence of hardened fats (nickel content 0.001-0.01 mg./kg.), as 0.005-0.05 mg. of nickel per kg. was found in dried milk powder (cf. Martini, A., 1930, 565; Bertrand and Nakamura, A.,

bb

1927, 992, whose figures for the occurrence of nickel in animal and vegetable materials are collected).

E. LEWKOWITSCH.

Determination of the iodine value of oils and fats by thermometric titration. T. SOMIYA (J. Soc. Chem. Ind., Japan, 1930, 33, 174—176 B).—The thermometric titration of oils etc. by solutions of chlorine in acetic acid and carbon tetrachloride must be conducted under standard conditions, the end-point being determined graphically (cf. B., 1929, 274). The values obtained were rather below those found by the usual methods.

E. LEWKOWITSCH.

Polymerised soya-bean oil and its soap. M. HIROSE and T. SHIMOMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 169-172 B).-The characteristics of soya-bean oil polymerised by heating at 290-295° in carbon dioxide are given. The rate of decrease of iodine value is considerably smaller than in the case of fish oils, but in both cases this rate decreases with the duration of heating. The yields of hexabromide and solid tetrabromide decrease to nil as polymerisation progresses, but the amount of viscous brownish-black bromide insoluble in light petroleum (which is regarded as an unsaturated polymerised product of linolenic acid) increases to an amount nearly equal to that of the hexabromide in the original oil. The increase in mol. wt. (Rast) of the fatty acids is smaller than that of the original glycerides. The surface tension of a solution of the soap increases, and the (Hillyer) drop number and the lathering power (Stiepel) decrease with increasing polymerisation. Thus polymerisation reduces the detergent power of the soap, but as the soap does not deteriorate on keeping and has remarkable tenacity it may with advantage be added to soaps of better washing power but of a brittle nature, e.g., hydrogenated soya-bean oil soap.

E. LEWKOWITSCH.

Spectrographic analysis of the fluorescence of some vegetable oils observed under ultra-violet rays. H. MARCELET (Compt. rend., 1930, 190, 1120— 1122).—Olive, soya-bean, sesamé, maize, grape-seed, arachis, tea, cottonseed, and argan oils produce different emission and absorption spectra in ultra-violet radiation. The addition of 5% of olive-pulp oil to refined or virgin olive oil may be detected by means of the sharply defined regions characteristic of its emission spectrum. Mixtures of olive oil with 10% of soya-bean, maize, or grape-seed oil gave inconclusive results. J. GRANT.

Neutralisation of castor oils. M. T. FRANÇOIS (Compt. rend., 1930, 190, 1308—1310).—The acids present in commercial castor oils, which are detrimental to their use in aviation and to their keeping properties, can be neutralised by industrial "triethanolamine" (tri- $\beta$ -hydroxyethylamine), consisting of a mixture of the corresponding primary (0.5%), secondary (20—25%), and tertiary (75—80%) amines, using methyl-orange as an indicator. The neutralised product has a slightly higher viscosity. A. I. VOGEL.

Composition of almond oil. Comparison of almond oil and apricot-kernel oil. A. HEIDUSCHKA and C. WIESEMANN (J. pr. Chem., 1930, [ii], 124, 240–260).— The following data have been obtained, the two figures referring to almond oil and apricot kernel oil, respectively,

in each case :  $d^{15}$  0.9180, 0.9181 ; refractivity at 25°  $65 \cdot 3, 65 \cdot 9, \text{ and at } 40^{\circ} 57 \cdot 4, 58 \cdot 0; \ [\alpha]_{\rm D} - 0 \cdot 007^{\circ}, 0 \cdot 007^{\circ};$ iodine value 99.36, 101.60; saponif. value 188.80, 190.15; Hehner value 94.22, 93.07; Reichert-Meissl value 0.00 0.06; Polenske value 0.00, 0.00; acid value  $1 \cdot 24$ ,  $3 \cdot 26$ ; unsaponifiable matter  $0 \cdot 50$ ,  $0 \cdot 43\%$ ; glycerol (Shukov and Schestakov) 10.00, 10.73%; fatty acids : palmitic 3.10, 2.08; oleic 77.00, 79.39; linoleic 19.90, 18.53%; linolenic acid not detectable. A modification of Twitchell's lead salt-alcohol method for the determination of saturated acids is used in which the lead salt is crystallised three times from 95% alcohol; in this way the salts of unsaturated acids are completely removed without appreciable loss of saturated acids. The unsaturated acids are determined by bromination by Hazura's method. The behaviour of the two oils on oxidation in the air has also been examined by determining the refractivities and the iodine, acid, and oxidation values at intervals ; only minor differences are observed. There are thus no readily detectable differences between the two oils, except the slight ones in taste and appearance, and the colour reactions, which are applicable only to fresh oils. R. K. CALLOW.

New consistometer and its application to greases and to oils at low temperatures. R. BULKLEY and F. G. BITNER (J. Rheology, 1930, 1, 269-282).-A consistometer is described, speedy in operation and suitable for routine determinations, which depends for its action on the measurement of flow through a capillary tube under a variable pressure-head of mercury. The apparatus is suitable for materials the consistency of which changes with time, since for each determination the volume of flow need not exceed 0.5 c.c. The accuracy claimed is  $\pm 2\%$ . Certain oils and greases behave as thixotropic gels and, after working, require several hours at 0° to attain a constant and reproducible state. By the use of this apparatus consistency-pressure curves may be obtained for materials in the completely worked T. H. MORTON. and completely unworked condition.

Determination of the so-called refined white montan wax in beeswax compositions. G. BUCHNER (Chem. Umschau, 1930, 37, 144-145).—The so-called refined montan "wax" consists almost entirely of free fatty (montanic) acids and unsaponifiable matter, and may be determined as follows (in a mixture, e.g., of stearic acid, beeswax, montan "wax," and paraffins): The acid value, ester value, and Buchner value (cf. B., 1911, 907) of the mixture are first determined, the last-named corresponding to the amount of stearic acid present, since the montanic acids are insoluble in the 80% alcohol used. The proportion of montan "wax" (acid value may be taken as 100) is calculated from the difference. between the acid value actually found for the residue from the Buchner test and that corresponding (i.e., 1/5 of the original ester value) to the beeswax present. It is assumed that other saponifiable substances (e.g., tallow) which would influence the ester value are absent : their presence would be indicated it the separated paraffins were considerably greater in amount than that calculated (1 pt. of tallow appears by calculation as  $2 \cdot 6$  pts. of beeswax).

E. LEWKOWITSCH.

Stability of emulsions. AUERBACH.-See I. Submerged combustion. HAMMOND. Lubricating oils. DAMIAN and DIXMIER .- See II. Solvents. FIFE and REID.—See III.

See also A., June, 692, Systems soap-cresol-water (ANGELESCO and POPESCU). 693, Influence of soap solution on rate of saponification of triglycerides (MCBAIN and KAWAKAMI). 702, Soap boiling (MCBAIN and others). 803, Mixed fatty acids present in glycerides of cod-liver and other fish-liver oils (GUHA and others). 804, Insect oils and fats (TIMON-DAVID). Bull-frog oil (IWAMOTA and KISEGAWA. Saturated fatty acids of Japanese great-herring oil (UENO and IKUTA). Oils and fats of Japanese birds (UENO and IKUTA).

#### PATENTS.

Manufacture of margarine. K. P. WANKLYN (B.P. 327,519, 21.2.29).-Wheat germ is dried in a deoxidised atmosphere and ground to a powder, or is crushed to produce an extract in the form of a pulp or jelly, and the powder or pulp is incorporated in margarine. E. LEWKOWITSCH.

Preservation of fats [in raw materials]. J. A. S. VAN DEURS (B.P. 303,059, 11.12.28. Denm., 27.12.27).-The  $p_{\rm H}$  within the animal or vegetable tissues surrounding the fat is maintained at not above 5 during the period of preservation, by the addition of (aqueous) acid or acid-forming micro-organisms. E. LEWKOWITSCH.

Production of soaps possessing intensive detergent power. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 327,393, 1.1.29).-Unesterified monoalkyl ethers of the glycols or polyglycols are incorporated with soaps after or during the course of their manufacture; hydrocarbons or chlorinated hydrocarbons may be added as desired. Solid, pasty, or jelly-like soaps are formed which are almost odourless and are not injurious to the skin. E. LEWKOWITSCH.

Cooling of fatty substances in liquid condition. H. G. R. NAUMANN (B.P. 328,196, 22.12.28. Holl., 6.11.28).—The rotating cooling drum used consists of an outer and an inner (concentric) cylinder, interconnected by partitions; the enclosed annulus contains the circulating, evaporating, liquid refrigerant.

#### E. LEWKOWITSCH.

Extraction of neutral grease from wool-scouring water and treating same. E. MERTENS (B.P. 328,606, 30.1.29. Belg., 6.11.28).-The wool-washing liquors are circulated through channels in which they are churned by pairs of paddle-wheels rotating in opposite directions; the scum collected by these is washed by spraying with water from atomisers, and is treated finally in a digester at 90-120° under 2-3 atm., yielding an almost anhydrous, odourless grease. E. LEWKOWITSCH.

Apparatus for separation of oil from oil-containing substances. F. KRUPP GRUSONWERK A.-G. (B.P. 297,816, 28.7.28. Ger., 30.9.27. Addn. to B.P. 272,118). -Claim is made for an arrangement of a series of horizontal presses. A. R. POWELL.

Production of oil from palm fruit and other oil seeds. VEGETABLE OIL MACHINERY SYND., LTD., and

J. MOORES (B.P. 328,734, 14.3.29).—The fruit is sterilised by cooking out of contact with water in a boiler fitted with a false bottom and pressed, the oil obtained being heated to about 100° and filtered; if desired, the fruits may be depulped after sterilising, the mashed pulp being re-cooked before pressing. E. LEWKOWITSCH.

Decoloration of [palm] oils. Co-OPERATIVE WHOLE-SALE SOC., LTD., A. GLOVER, and C. W. COUCHE (B.P. 327,990, 14.12.28).-Palm oil is bleached by heating with the exclusion of air (in vacuo, but without atomisation, steam-injection, etc.) at 200-300° until the oil may be saponified without return of the yellow colour ; fatty acids in the oil are retained. E. LEWKOWITSCH.

Deacidification and bleaching of oils and fats. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,276, 24.12.28).-The oil, which has been mixed with a finely-divided adsorption agent, e.g., fuller's earth or carbon, is subjected to a distillation treatment at elevated temperatures with wet steam, or a mist of finely-divided liquids, e.g., alcohol or benzene, dispersed in an inert gas (cf. B.P. 213,267 and 235,792; B., 1925, 619, 680). E. LEWKOWITSCH.

Separation of waxes into their acids and mixtures of their alcohols and hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 304,150, 14.1.29. Ger., 14.1.28).-The mixture obtained by saponifying beeswax, montan wax, wool fat, etc. is atomised, and the dry powder so produced is extracted with solvents to remove the unsaponifiable matter. E. LEWKOWITSCH.

Modifying isocolloid materials (B.P. 321,689, 321,691-4, 321,699, 321,722-7).-See III. Bleaching materials (B.P. 328,544) .- See VI. Heating hydrogen for hydrogenation (B.P. 329,045).-See VII. Products resembling waxes (B.P. 328,190). Plastic masses (B.P. 304,612).—See XIII.

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

Oil of white pine seeds from Cadore. V. MASSERA (Giorn. Chim. Ind. Appl., 1930, 12, 180).-A sample of this oil, obtained in about 8% yield, gave :  $d^{15}$  0.8568,  $\alpha_{\rm D}^{20} - 82^{\circ} 30', n_{\rm D}^{20} 1.4742$ , acid value 0.429, ester value 5.103, esters as bornyl acetate 1.786%, acetyl ester value 8.342, total alcohols as C10H180 2.31%, complete solubility in 90% alcohol in the proportion 1:9; Schiff's reagent gives after about 15 min. a faint blue coloration, which deepens after 2 hrs. The oil consists mostly of d-limonene, with small proportions of a sesquiterpene (? cadinene), and of borneol, partly T. H. POPE. free and partly esterified.

J. Kot'a Qualitative reactions for pine oil. (Chem. Listy, 1930, 24, 150-153, 169-179).-The only reliable reactions for the detection of pine oil in turpentine are the Grimaldi modification of Halphen's method (B., 1910, 963) and Wolff's method (B., 1923, 730 A). R. TRUSZKOWSKI.

Ultramarine blue-white lead mixtures. Η. WAGNER and E. MARTZ (Farben-Ztg., 1930, 35, 1553-1554).-A comprehensive series of tests showed that ultramarine blue-white lead mixtures in various vehicles are quite stable, no formation of lead sulphide being recorded in oil or varnish vehicles. A slight danger

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exists in the presence of water (either from aqueous media or moisture retained in the film), but this is avoidable by good working conditions and thorough drying. It is advisable, although not essential, to avoid the use of white lead containing lead acetate. It is pointed out that Brilliant Yellow, a mixture of cadmium-yellow and Kremnitz White, has long been in use without decomposition troubles. S. S. WOOLF.

Minium. J. HERITES (Chem. Listy, 1930, 24, 218–220).—The covering power of minium depends on the fineness of division of the grains, not on the actual dioxide content. Thus three specimens of minium, containing 29.65, 29.88, and 33.55% PbO<sub>2</sub>, and the diameters of the grains of which were, respectively, 0.7—15.5, 0.8—27.9, and 1.0—24.8 $\mu$ , had a covering power of 250, 80, and 133 cm.<sup>2</sup> per g.

## R. TRUSZKOWSKI.

Cadmium pigments. SEDLACZEK (Farben-Ztg., 1930, 35, 1661—1662).—The properties, composition, and preparation of cadmium sulphide and selenide pigments are discussed, with references to the patent literature on the subject. S. S. WOOLF.

Cadmium-selenium red. H. E. MARLEY (J. Amer. Ceram. Soc., 1930, 13, No. 5, ii, 44-47).—Tests were carried out to determine the proportions of cadmium, sulphur, and selenium required to produce a brilliant red, the sources of cadmium being the carbonate, oxide, and sulphide. Cadmium carbonate produced a better red colour than either the oxide or the sulphide. The limits in composition for the production of a brilliant red were : cadmium carbonate 58-70%, sulphur flowers 21-31%, selenium metal 9-15%. F. SALT,

Slippage correction in the equation of plastic flow. R. V. WILLIAMSON (J. Rheology, 1930, 1, 283-287) .- The flow-pressure diagrams of pigmented dispersions have been determined to test the validity of Buckingham's (Proc. Amer. Soc. Test. Mat., 1921, 21, 1154) equation for plastic flow. With a plastic dispersion made by grinding 2 pts. of zinc oxide with 1 pt. of raw linseed oil, the apparent fluidity decreases rapidly to a minimum as the shearing stress is decreased and again rises slightly. At this point the medium continues to flow, but the pigment becomes flocculated and stationary. The product of the minimum fluidity and the radius of the capillary is constant. A dispersion of " Titanox " in equal parts of linseed oil and a petroleum hydrocarbon, which showed little plasticity, yielded a linear flow-pressure curve passing through the origin. The slight change in the slope of this curve for different capillary radii is in the direction opposite to that expected from the equation, which, however, represents with fair accuracy the behaviour of the zinc oxide dispension at low shearing stress down to the point where flocculation begins. The minimum on the curve corresponds to the formation, in the capillary, of a core of weak gel surrounded by liquid medium.

T. H. MORTON. **Production of coumarone resins.** I. I. BURDA (Ukraine Chem. J., 1929, 4, [Tech.], 249–255).—Coumarone resins can be prepared directly from the heavy benzol fraction of coal tar, without the necessity for any purification from phenols and pyridine compounds. The yield of resin varies with the quantity of sulphuric acid added (1-4% by vol.); with less acid the yield is smaller, but the resin is of a lighter colour and is softer. The repeated treatment of the heavy benzol fraction with 0.5-1% of acid, with distillation of volatile hydrocarbons following each addition of acid, yields at first hard resins and later soft ones. The latter can be converted into the former by heating at 150-180° for 20-90 min. R. TRUSZKOWSKI.

Artificial copals [Albertols] as raw materials for modern varnishes. ANON. and E. FONROBERT (Farben-Ztg., 1930, 35, 1506—1510, 1554—1557, 1606— 1608, 1658—1661).—A dissertation on the use of Albertols. Their general properties and the suitability of various grades for particular purposes are discussed. The introduction of driers into oil varnishes is considered at some length, the characteristics of various forms of lead, manganese, and cobalt driers being detailed. The remaining sections deal with the use of Albertols in primers, fillers, and surfacers, carriage varnishes and japans, and "4-hr." varnishes, formulations and full manufacturing instructions for 14 typical products being included. S. S. WOOLF.

Solvents from natural gas. CLARK. Solvents. FIFE and REID.—See III. Naval stores from waste wood. PALMER.—See V. Phosphorescent substances. RHODE.—See VII. Casein plastics. SIM-MONS.—See XV. [Pigment from] Lantana camara. PILLAI and others.—See XVI. Ambergris. MORRISON. —See XX.

See also A., June, 746, Formaldehyde-urethane resinification (GIUA and RACCIU). 750, X-Ray investigations of cellulose derivatives (TROGUS and others). 781, Dehydrogenation of boswellic acid (BEAU-COURT). 783, Phenolcamphorein and its derivatives (DEULOFEU).

## PATENTS.

**Coating compositions and the like.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 327,981, 3.10.28).—A mixture of rubber solution and oil with or without drier is thinned with a volatile solvent to a suitable consistency, and an inhibitor, e.g., 1—10% of quinol on the rubber content, is incorporated in order to retard the catalytic depolymerisation of the rubber. S. S. WOOLF.

Coating compositions and vehicles therefor. BRIT. CELANESE, LTD. (B.P. 299,782, 29.10.28. U.S., 31.10.27).—Cellulose derivatives, preferably organic, e.g., cellulose acetate, are dissolved in a low-boiling vehicle consisting of alcohols, e.g., ethyl alcohol, aromatic hydrocarbons, e.g., benzene, and halogen addition products of unsaturated hydrocarbons, e.g., ethylene dichloride, which in the cold are non-solvents for the cellulose derivatives. Synthetic or natural resins, high-boiling solvents, plasticisers, pigments, fillers, etc. may be incorporated. S. S. WOOLF.

Manufacture of resinous products. I. G. FARB-ENIND. A.-G. (B.P. 328,728, 5.3.29. Ger., 24.12.28).— Adipic acid, its higher homologues having a branched chain, or mixtures of these with each other or with other mono- or di-basic carboxylic acids, are condensed with polyhydric alcohols in the absence of monohydric alcohols and, if desired, in the presence of a catalyst. The products may be incorporated with other resins, *e.g.*, "glyptals," for which they act as plasticisers.

S. S. WOOLF.

Synthetic resin varnishes and synthetic resins. IMPERIAL CHEM. INDUSTRIES, LTD., H. H. MORGAN, A. A. DRUMMOND, and G. C. ATTFIELD (B.P. 327,946, 6.11.28).—A polyhydric alcohol, e.g., glycerol, a dibasic acid or its anhydride, e.g., phthalic anhydride, and a fatty oil, e.g., tung oil, are made to react under heat with aliphatic alcohols of b.p. not above 175°, e.g., butyl alcohol, cyclohexanol, alkyl monoethers of ethylene glycol, ethyl lactate. The reaction may be carried out in the presence of hydrocarbon or ester solvents, e.g., hydrogenated naphthalenes, solvent naphtha, butyl acetate, benzyl benzoate. The aliphatic alcohol may be used in quantity just sufficient to bring about the incorporation of the oil, in which case the process may occupy 20-24 hrs., or in excess, when the time taken is shortened considerably, the excess alcohol being removed by distillation after the preliminary reaction has occurred. Driers may be added at any time in the process, and dyes or pigments may be incorporated. S. S. WOOLF.

**Production of polybasic acid-polyhydric alcohol resin.** J. H. SCHMIDT, Assr. to BAKELITE CORP. (U.S.P. 1,739,771, 17.12.29. Appl., 16.3.28).—Glycerin is heated to 205° with excess of phthalic anhydride, substantially toward the point of slag formation, *e.g.*, until an extracted sample "strings" at 180—190°. The mass is then chilled to arrest the reaction by pouring into thin slabs, preferably in metal trays, and the cooled product is hardened by baking at 125—145° for 12 weeks.

S. S. WOOLF. Synthetic resinous compositions. IMPERIAL CHEM. INDUSTRIES, LTD., W. BAIRD, R. HILL, and E. E. WALKER (B.P. 328,003, 9.1.29).—Polyhydric alcohols are condensed with polybasic acids in the presence of monocarboxylic acids preferably of high mol. wt., *e.g.*, drying oil acids, and drying oils are incorporated before or during the condensation. S. S. WOOLF.

**Obtaining aldehyde condensation products.** M. MELAMID (U.S.P. 1,727,076, 3.9.29. Appl., 8.12.24. Ger., 4.12.23).—Hydroxy-derivatives of aromatic hydrocarbons are treated with a mixture of aldehyde and an electrolyte. *E.g.*, 100 pts. of phenol are heated on the water-bath with a mixture of 60 pts. of 30% formaldehyde solution and 300—400 pts. of 10% sulphuric acid. The product is a soluble resin, m.p. about 90°.

A. R. POWELL.

Production of artificial compositions from the condensation solutions of urea, thiourea, or their derivatives and formaldehyde. L. N. REDDIE. From KUNSTHARZFABR. DR. F. POLLAK GES.M.B.H. (B.P. 328,040, 11.12.28).—The condensation of 1 mol. of urea, thiourea, or a derivative thereof with less than 2 mols. of formaldehyde in the presence of acids is interrupted before the stage at which the resin would separate from the solution in a solid form on cooling, and the resin solution is concentrated at 40—50°, e.g., by spraying it against a warm current of air or by the use of heated rollers, vacuum drying drums, etc., until a gelatinous mass containing about 5% of water and capable of swelling in water is obtained. The mass is comminuted during or after the concentration process and moulded by hot-pressing in the presence, if desired, of ammonium sulphate or other acid salts which accelerate polymerisation. The press powder may be washed with ammonia or other reagents which fix or remove formaldehyde. S. S. WOOLF.

Melting and esterification of resins or the like. J. SOMMER (B.P. 319,651, 28.2.29. Ger., 25.9.28).— Resins, e.g., copals, are esterified with glycerin etc. in apparatus comprising a heated melting pot fitted with cover, foam distributor, stirrer, and inlets for glycerin, inert gas, and air, the vapours from the pot, with the exception of water vapour, being condensed in a tubular cooler, and led to a separator from which glycerin separating out is returned to the pot. Esterified resin is discharged by pressure. S. S. WOOLF.

Manufacture of products of the nature of resins, waxes, balsams, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,190, 20.10.28).—Resin acids, e.g., abietic acid, natural resins containing them, or artificial resins still containing free carboxylic acids are treated with alkylene monoxides, at 60—200° and preferably at elevated pressures, in the presence or absence of acid condensing agents, e.g., boric acid, zinc chloride, the products obtained being acylated if desired.

S. S. WOOLF.

Manufacture of a moulding mixture. V. H. TURKINGTON, ASST. to BAKELITE CORP. (U.S.P. 1,728,378, 17.9.29. Appl., 11.12.22).—A mixture of furfuraldehyde with hexamethylenetetramine is incorporated with wood pulp or similar filling material and the heated mass is thoroughly mixed with a soluble phenol-formaldehyde condensation product. After moulding into shape the mass is hardened by suitable heat-treatment.

A. R. POWELL.

Moulding mixtures. H. WADE. From BAKELITE CORP. (B.P. 327,969, 12.1.29).—Such mixtures comprise a resin of the urea-aldehyde type, a filling material, *e.g.*, wood flour, a cellulose ester stable up to 165°, *e.g.*, cellulose acetate, and, if desired, a plasticiser preferably containing a dialkyl ester of an organic acid.

S. S. WOOLF.

Production of plastic masses. COMP. FRANÇ. D'EX-PLOIT. DES PROC. PLINATUS, Assees. of W. PLINATUS (B.P. 305,238, 2.2.29. Ger., 2.2.28).—Moist cellulose esters, e.g., nitrocellulose containing 30% of water or cellulose acetate containing 10—120% of water, the esters comprising or containing a proportion of lowviscosity material, are converted into solid solutions by the addition of high-boiling solvent and gelatinising agents, e.g., ethyl phthalate, and the use of mechanical energy and heat, in the absence of volatile solvents.

S. S. WOOLF.

Manufacture of artificial [plastic] masses. I. G. FARBENIND. A.-G. (B.P. 304,612, 21.1.29. Ger., 20.1.28). —The use of a relatively small proportion of synthetic rubber, e.g., that obtained from dimethylerythrene, as binding agent in otherwise customary processes for the production of artificial masses resembling linoleum, ebonite, celluloid, etc. is claimed. S. S. WOOLF.

Preparation of coating materials and plastic masses. Imperial Chem. Industries, Ltd., Assees. of R. T. HUCKS (B.P. 298,914, 15.10.28. U.S., 15.10.27).-Nitrocellulose is treated with a colloiding agent, e.g., butyl phthalate, which is substantially immiscible with water, and the plastic thus produced is kneaded with water-wet pigment pulp, preferably in the presence of a small quantity of a substance which is an active solvent for nitrocellulose, but also substantially immiscible with water, e.g., ethyl acetate. After incorporation of the pigment the separated water is decanted.

S. S. WOOLF.

Manufacture of lithopone. W. C. HOOEY, Assr. to NEW JERSEY ZINC Co. (U.S.P. 1,759,115-6, 20.5.30. Appl., 15.12.25).—See B.P. 263,119—20; B., 1928, 341.

Modifying isocolloid materials (B.P. 321,689, 321,691-4, 321,699, 321,722-7).-See III. Zinc sulphide (U.S.P. 1,742,030).—See VII.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Testing of automotive rubber parts assembled under compression. I, II. F. D. ABBOTT (Ind. Eng. Chem. [Anal.], 1930, 2, 145-159).-I. The current specifications relating to the testing of automotive rubber parts are adversely criticised. It is shown that high tensile strength is no criterion of relative resistance to compression-set and that hardness is not a measure of deflectability. Deflection and permanent set under compression are influenced by the size and shape of the test piece, the method of cutting the sample, the methods of measuring it, as well as by the condition and kinds of surface in contact with the test piece.

II. An instrument is described with which tests of deflection, hysteresis, and set can be made under constant load on constant distortion. Details are also given of a method of carrying out a compression-set test.

## H. INGLESON.

See also A., June, 782, Colloidal nature of caoutchouc, gutta-percha, and balata (STAUDINGER).

## PATENTS.

Manufacture of porous bodies, filters, diaphragms, etc. consisting of porous or porous and non-porous parts made of soft to hard rubber and the like. M. WILDERMAN (B.P. 328,273-4, 20.12.28). -(A) Porous rubber articles are formed from semivulcanised particles of a soft to hard mixture, which, while being spread, are kept at a temperature such that they are maintained in a state of fine division; they are then vulcanised. The particles may be sifted before use and, if stored, the storage vessel should be provided with an effective stirrer. The heat for vulcanisation may be provided by steam, but all moisture must be kept from the rubber. Articles only partly porous may be manufactured either by forming in separate moulds and vulcanising together, or by spreading the non-porous parts and compressing, then spreading the porous parts without much compression. In (B) machines for continuous and intermittent operation are described for the above processes. It is preferred to use metal foils in the

moulds so that the articles can be formed in one press and vulcanised in another. The non-porous parts of the articles may be produced by laying strips of non-porous rubber on the spread material before vulcanising.

## B. M. VENABLES.

Applying and/or attaching or securing rubber or the like to metal [by surface beating]. DUNLOP RUBBER CO., LTD., H. C. YOUNG, and C. HEMM (B.P. 327,856, 10.4.29).

Heat-transfer for vulcanisation (B.P. 305,577) .-See I. Modifying isocolloid materials (B.P. 321,689, 321,691-4, 321,699, 321,722-7).-See III. Insulating materials (B.P. 328,025).-See XI. Coating compositions (B.P. 327,981). Plastic masses (B.P. 304,612). —See XIII.

## XV.-LEATHER; GLUE.

Action of neutral salts on the enzyme activity of tryptic bates. II. V. KUBELKA and K. DOUSA (Collegium, 1930, 66-72; cf. B., 1929, 1025).-The activity of "Standard Oropon" was determined by the Schneider-Ulcek method, using different amounts of the tryptic bate and of ammonium sulphate, respectively. Maximum activity varied with the relative proportion of neutral salt to bate and also proportionally as the amount of neutral salt. The activity of the enzyme bate at the point of maximum activity was the greater, the larger was the amount used in the determination. The maximum activity of the "Standard Oropon" on a casein substrate was three times that of the material without the addition of ammonium sulphate.

D. WOODROFFE.

Action of trypsin on the properties of collagen. R. O. and A. W. PAGE (Ind. Eng. Chem., 1930, 22, 545-547).-In addition to removing a certain amount of hide proteins in the process of bating, trypsin acts on the collagen, which has been considerably pretreated with alkali in the unhairing and liming process, to reduce its plumping, to lighten the colour and increase the mellowness of the resulting leather, and to diminish its power of combining with vegetable tanning materials. F. R. ENNOS.

Action of papayotin and papain on hide powder. W. ACKERMANN (Collegium, 1930, 74-79).-When hide powder was treated with papain and papayotin, respectively, at 37° under different conditions of acidity and alkalinity, they had a bating action similar to that of trypsin. Their action was much stronger on hide powder and pelt than was indicated by the figures obtained with the Fuld-Gross casein method of testing enzyme activity. Papayotin was stronger than papain in its action. D. WOODROFFE.

Colloid-chemical investigations of some tanning processes. I, II. W. SCHINDLER and K. KLAN-FER (Kolloidchem. Beih., 1930, 31, 100-148, 149-184). -The processes of dyeing, neutralisation, oiling, and pickling in the manufacture of chrome leather are discussed from the colloid-chemical point of view. The adsorption of dyestuffs by chrome leather has been investigated experimentally for Orange II, Patent Blue A, induline, and Diphenyl Brown BBN, and the influence of amount of adsorbent, concentration of the dye, and temperature have been studied. The relation

between the final concentration of dye and the relative adsorption is given by three exponential equations, each of which is valid for a certain range of concentration. When plotted logarithmically the curves show two breaks. The velocity of taking up acid, substantive, and basic dyes was measured; the last-named were found to be the most reactive. The values of 1/n in the adsorption isotherm are lower for the basic dyes than for the other two types. The neutralisation of the chrome leather with alkalis also proceeds in accordance with an adsorption process over a wide range. Measurements were also made of the rate at which chrome leather takes up oils emulsified by soap solution or sulphonated oils. The sulphonated oils were taken up the more rapidly. The rate of adsorption of acid in the pickling process has been investigated, and sulphuric and hydrochloric acids are taken up at the same rate, but formic acid is E. S. HEDGES. adsorbed more slowly.

Hydrogen-ion concentration and tanning effect. H. MACHON (Collegium, 1930, 49-63)-Crude and sulphited quebracho extracts were analysed by the official shake method of tannin analysis, but at different  $p_{\rm H}$  values, and maximum results for tannin were obtained at  $p_{\rm H}$  6.0 and 5.9, respectively. More tannin was combined with the hide powder in the presence of weak than of strong organic acids. Similar effects were produced by acids of similar dissociation constants. The tanning value (amount of tannin absorbed by hide powder from more concentrated solutions) varied inversely as the pH value of solutions of sulphited quebracho extract, but showed maxima at  $p_{\rm H}$  2-3 and 6.5-8.0 and a minimum at  $p_{\rm H}$  5.0. The greatest tanning value was obtained with acetic acid in solutions brought to the same  $p_{\mathbf{H}}$  value with different organic acids. The  $p_{\rm H}$  of maximum tanning value increased as the concentration of the solution decreased. Precipitation of the tan liquors was dependent on their  $p_{\rm H}$  value, and independent of the particular acid present. The amount of the precipitate was affected by the nature of the acid. Tannin analysis should be made on the natural solutions and the  $p_{\rm H}$  reported. It is shown that in the early stages of tanning at different  $p_{\rm H}$  values there is no relation between the water and tannin contents of the tanned pelt. D. WOODROFFE.

Loss of tannin in liquors due to fermentation. J. M. SELTZER and F. F. MARSHALL (J. Amer. Leather Chem. Assoc., 1930, 25, 168-173).-Liquors (d 1.035) were prepared from various tanning materials and extracts left 12 weeks, during which period they were analysed at intervals. The loss in tannin (%) after 12 weeks was as follows: wattle bark 2.91; valonia cups and beards 27.5; myrobalans 60.24; solid mangrove-bark extract 0.48; liquid quebracho extract (heavily sulphited) 4.32, (lightly sulphited) 6.13; sulphite-cellulose extract 6.33; hemlock extract 14.88; chestnut (liquid)  $17 \cdot 0$ ; oak-bark extract  $21 \cdot 44$ ; valonia extract (powder)  $40 \cdot 04$ . The acidity of the liquors increased at first and then diminished slightly in most of them. No difference was shown in the acidity of the liquors by stoppering the containing vessel. D. WOODROFFE.

Manufacture of casein plastics. W. H. SIMMONS

(Ind. Chemist, 1930, **6**, 206—208).—The combination of casein with formaldehyde with prevention of the drying out and splitting which otherwise occur is probably not capable of any simple chemical explanation. The early wet process is nowhere used commercially, having been completely replaced by the dry process. The casein used should be precipitated from skimmed milk collected under antiseptic conditions by rennet; the fat content should not exceed 2%, ash content should be 8%, and moisture 10—12%. Formaldehyde is used as a 40% solution, neutral to litmus, and should be stored in a warm place. Acid dyes are used for colouring. Water used should be free from iron, softened by zeolite or distillation, and degassed. C. IRWIN.

Maizolith. HARTFORD.—See V. Xylose. EMLEY. —See XVII.

## PATENTS.

Production of thermoplastic material [resembling synthetic horn]. P. C. CHRISTENSEN (U.S.P. 1,746,070, 4.2.30. Appl., 20.8.26).—A thick aqueous paste of (preferably) equal parts of casein and "leather gum," a product obtained by successively treating scrap leather with dilute sulphuric acid, boiling with water, and removing, cooling, and grinding the residue, is passed between rollers at about 55—100° and then extruded or moulded under pressure; the products are toughened by treatment with an aldehyde solution.

L. A. COLES.

Manufacture of glues from yeast which are stable for storing. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,645, 30.1.29).—The products comprise intimate mixtures of yeast or the products of yeast autolysis with dry alkali hydroxides or salts having an alkaline reaction (e.g., di- or tri-sodium phosphate); aldehydes or substances which liberate them (e.g., paraldehyde, bishydroxymethylcarbamide) may also be added before, during, or after the mixing operation. L. A. COLES.

Production of solidified globules ["pearl glue "] from liquid gelatinous substances. T. K. Lowry (U.S.P. 1,746,543, 11.2.30. Appl., 25.1.28).—Glue etc. liquefied by heat is forced through adjustable jets into the lower end of a vertical, water-jacketed cylinder which receives, just above the jets, a stream of a non-inflammable liquid (trichloroethylene, carbon tetrachloride), the solidified globules are subsequently screened at the top of the cylinder and the liquid collected for use again. L. A. COLES.

Manufacture of products for tanning and tawing industries. M. KAHN, E. LE BRETON, and G. SCHAEF-FER (U.S.P. 1,759,700, 20.5.30. Appl., 22.4.26. Fr., 20.11.23).—See B.P. 247,977; B., 1926, 557.

## XVI.—AGRICULTURE.

Soil survey of the Nalkantha district (Limbdi State) and its significance. C. V. R. AYYAR (J. Indian Inst. Sci., 1930, 13A, 43—56).—A method of mechanical analysis is adopted in which the usual pretreatment of the soil sample with acid and hydrogen peroxide is omitted and suspensions for the sedimentation process are prepared in distilled water without the

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addition of dispersive agents. A system of soil classification based on such analyses can be correlated with field observations of soil texture and with chemical analyses. A. G. POLLARD.

Influence of stones in soil on plant growth. E. BLANCK [with H. KEESE and F. KLANDER] (J. Landw., 1930, 78, 1—8).—The proportion of stones in soil affects plant growth only as a result of its influence on the volume-water capacity of the soil. Such effects are small, being significant when the proportion of stones approaches 40%. A. G. POLLARD.

Potent factors in soil formation. C. F. SHAW (Ecology, 1930, 11, 239—245).—The influence of climate, vegetation, parent material, and erosion on soil formation is discussed. A. G. POLLARD.

Biological Azotobacter method for determining the phosphate requirement of soils. H. NIKLAS and H. POSCHENRIEDER (Superphosphat, 1930, 6, 69—72). —Modifications are described in the technique of the Azotobacter test. Results agree well with those of the seedling method, especially where the phosphate content of the soil is small. A. G. POLLARD.

**Reaction and phosphate content of soils.** F. TERLIKOWSKI (Rocz. Nauk. Roln. i Lešn., 1929, 22; Proc. Internat. Soc. Soil Sci., 1930, 5, 21–22).—Correlation between soil reaction and the easily-soluble phosphate content is only possible on certain soil types. All weakly acid soils benefit from phosphatic fertilisers, and the failure of growth of *Azotobacter* in these soils is mainly due to phosphate deficiency. General relationships between the phosphate content of soils and their reaction are to be anticipated if the latter value is expressed in terms of the degree of saturation with bases.

A. G. POLLARD. Effects of potash and phosphorus on tip-burn and mildew of cabbage. C. CHUPP (Phytopath., 1930, 20, 307—318).—Heavy applications of superphosphate to soils increased the proportion of tip-burn in cabbage, especially on soils deficient in nitrogen and potash. Sodium nitrate was without effect on tipburn unless large amounts were applied, when the disease increased somewhat. Potassium chloride reduced the disease and is recommended as a control measure. Potassic and nitrogenous fertilisers tended to increase the proportion of downy mildew in cabbage, whilst phosphates had the reverse effect. A. G. POLLARD.

Relationship between the nitrogen and carbon metabolism of Clostridium acetobutylicum. P. W. WILSON, W. H. PETERSON, and E. B. FRED (J. Bact., 1930, 19, 231–260).—Clostridium acetobutylicum can utilise protein, peptone, or beef aminoids as sources of nitrogen with but small changes in the proportion of solvents produced during fermentation. Peptone tends slightly to increase the yield of acetone at the expense of ethyl alcohol, whilst beef aminoids favour the formation of butyl alcohol. The organism utilises part of the amino-acids as such. In the presence of aminoids there is a large increase in synthesised protein. Ammonia alone cannot be used, but in the presence of protein ammonium salts are preferentially utilised, the maximum being reached with ammonium carbonate. The ratio of solvents produced is altered by the use of ammonium salts, there being a decreased acetone yield at the expense of ethyl alcohol unless the total yield falls, in which case the proportion of acetone increases. These variations in yield are attributed to changes in  $p_{\rm H}$  of the fermenting liquor. Nitrates are not utilisable by the organism, whether alone or in the presence of undegraded protein. A. G. POLLARD.

Reduction of nitrates to nitrites by Salmonella pullorum and S. gallinarum. R. P. TITTSLER (J. Bact., 1930, 19, 261-267).—Examination of numerous strains of Salmonella pullorum and S. gallinarum shows both to be capable of reducing nitrates to nitrites. There is no appreciable difference between the strains. Wittes' peptone is unsuitable for media for this work, and "Difco" is recommended. Dimethyl- $\alpha$ -naphthylamine is preferred to  $\alpha$ -naphthylamine for the detection of nitrites. A. G. POLLARD.

Spot-plate test for nitrate-nitrogen in soil and other extracts. M. F. MORGAN (Science, 1930, 71, 343-344).—To one drop of the liquid to be tested are added 4 drops of a solution of 0.05 g. of diphenylamine in 25 c.c. of concentrated sulphuric acid and the colour is compared with a standard. The nitrate concentration of the liquid under test (as nitrogen) must be adjusted to within the range 1—25 pts. per million for the test to be approximately quantitative. L. S. THEOBALD.

The plant as a factor in the action of Bordeaux mixture as an insecticide. D. M. DE LONG, W. J. REID, JUN., and M. M. DARLEY (J. Econ. Entom., 1930, 23, 383—390).—Copper can be dissolved from Bordeaux mixture residues by solutions of sugar and by expressed plant juices, but not by distilled or rain water. The sugar content of vigorously growing plants sprayed with Bordeaux mixture decreased for about 2 days after spraying, but later rose to, and maintained, a higher concentration than in unsprayed plants.

A. G. POLLARD.

Influence of Bordeaux mixture on the efficiency of lubricating-oil emulsions in the control of the San Jose scale. B. A. PORTER and R. F. SAZAMA (J. Agric. Res., 1930, 40, 755—765).—The efficiency of lubricating oil emulsion at low concentrations in reducing San Jose scale (Aspidiotus perniciosus) is lowered by the addition of ordinary strengths of Bordeaux mixture. For dormant spraying the oil content of such mixtures should be greater than 1.5%; for summer spraying 2.0%. F. O. HowITT.

Toxicity of copper to the potato leaf hopper. D. M. DE LONG, W. J. REID, JUN., and M. M. DARLEY (J. Econ. Entom., 1930, 23, 390—394).—Copper sulphate in dilutions up to 1 in 6500 and containing 5% of sugar was toxic to the nymphs of the potato leaf hopper. The plant juice of beans the roots of which were immersed in dilute copper sulphate solutions contained copper and caused high mortality among leaf hoppers feeding on the plants. A. G. POLLARD.

Two arsenical substitutes [insecticides]. S. MARCOVITCH and W. W. STANLEY (J. Econ. Entom., 1930, 23, 370—376).—Cryolite and barium fluosilicate are highly toxic to insects, and are but little harmful to foliage. Used as a spray (1 lb. to 50 gals. of water), both substances give effective control of the Mexican bean beetle, and as a dust (five-weekly treatments) were successful against hornworm and flea-beetle on tobacco. Dusted, in the proportion of 6 lb. per acre, neither material affected bean foliage, and at 30 lb. per acre caused only moderate scorching. The addition of 25% by wt. of fish oil improved the sticking quality of these dusts, which were also successful when mixed with twice their weight of lime. A. G. POLLARD.

Fungicidal efficiency of chemical dusts containing furfuraldehyde derivatives. C. S. REDDY (Phytopath., 1930, 20, 147—168).—Numerous trials of mercury compounds of furfuraldehyde derivatives for controlling dry-rot seedling blights of maize are recorded. The most successful preparations were (1) compound from mercuric chloride and hydrofurfuramide in aqueous solution, (2) compounds formed by addition of mercuric chloride and nitrate to furfuraldehyde pretreated by the Cannizzaro reaction, (3) mercury furylacrylate. Judged by the increased yields of grain, these preparations were most efficient against *Diplodia* followed in order by *Basisporium* and *Giberella*.

## A. G. POLLARD.

Egg-killing washes at the Long Ashton Research Station. L. N. STANILAND, F. TUTIN, and C. L. WALTON (J. Pomology, 1930, 8, 129-152).-Eggs of the capsid bugs (Plesiocoris rugicollis) and of the winter moth may be destroyed by the stifling or smothering action of the oily covering applied, an action performed satisfactorily by petroleum products such as heavy medicinal paraffin; the eggs of Aphis and Psylla are more resistant, but may be killed by the definite toxic action of tar distillates. It is considered that the inefficiency of the older tar washes made from "total" distillates was due to their relatively high content of volatile material which evaporated before the time at which the insect eggs were most readily killed, and to the fact that they contained tar acids and bases which had relatively low ovicidal powers and were easily washed from the eggs by rain. The "neutral" washes are far superior. E. HOLMES.

Nature of "Kuhlerde" and its action on marsh soils. E. BLANCK and W. DÖRFELDT (J. Landw., 1930, 78, 9-30).—The amelioration of marsh soils by the surface treatment or ploughing-in of certain lower soil strata ("Kuhlerde") is examined. The physical and chemical effects of Kuhlerde depending on their sand and chalk contents are discussed.

A. G. POLLARD.

Utilisation of waste vegetation. I. Preliminary study of Lantana camara, L. T. R. N. PILLAI, P.K. DE, C. V. PARAMASIVAN, M. G. RAO, S. A. RAFAY, and T. R. SATHE (Agric. J. India, 1930, 25, 143—149).—A preliminary examination of Lantana, including ultimate chemical analyses of the roots, stems, leaves, flowers, and fruits, suggests that the stems and leaves of this plant, which thrives under poor soil conditions, are well suited for the production of synthetic farmyard manure. A hydrochloric acid-soluble pigment has been isolated from the flowers. E. HOLMES.

Manuring of paddy in Lower Burma. D.

HENDRY (Agric. J. India, 1930, 25, 126—137).—The immediate manurial requirements of rice soils in Lower Burma are nitrogen and phosphoric acid; potash has little if any effect. Experiments with various proprietary ammonium phosphate fertilisers containing N and  $P_2O_5$  in the ratios of 1:1 and 1:3 approx. indicate the economic superiority of those containing equal amounts of the two nutrients. The highest economic benefits follow the application of approx. 250 lb. per acre of fertiliser containing about 20% each of N and  $P_2O_5$ , rather than the equivalent mixtures of ammonium sulphate and superphosphate. E. HOLMES.

## Xylose. EMLEY.—See XVII.

See also A., June, 725, Colorimetric determination of phosphoric and arsenic acids (ZINZADZE). 811, Repellents against the Japanese beetle (METZGER). 823, Distribution of nickel and cobalt in plants (BERTRAND and MOKRAGNATZ). Distribution of titanium in cryptogams (BERTRAND and VORONCA-SPIRT). Rôle of chlorine in nutrition and growth of the tobacco plant (GARNER and others). 824, Determination of sulphur in plants (FREAR). Determination of nitrate in green tomato and lettuce tissues (EMMERT). 825, Evaluation of plant products (NIETHAMMER).

## PATENTS.

Conversion of ammonia into fertilisers. G. F. UHDE (B.P. 299,896, 3.11.28. Ger., 3.11.27).-Crude calcium phosphate containing chalk, preferably suspended in calcium nitrate solution, is treated with sufficient nitric acid or oxides of nitrogen, obtained by the oxidation of ammonia, to convert the carbonate alone into calcium nitrate; the enriched phosphate, after separation from the liquor, is converted by treatment with sulphuric acid into calcium sulphate and phosphoric acid, these being worked up to calcium carbonate and ammonium sulphate and to ammonium phosphate, respectively. Potassium salts etc. may be added to the liquor used in the initial nitric acid treatment so that mixed fertilisers are obtained on evaporating the solution after removal of the undissolved phosphate. L. A. COLES.

Manufacture of mixed fertilisers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,620, 29,10.28).— The fertilisers comprise isomorphous mixed potassiumammonium sulphate-nitrate crystals prepared from solutions containing ammonium nitrate and sulphate and potassium nitrate and/or sulphate, one of which may be formed *in situ* by the interaction of two other salts; other nitrogenous or potassium salts or phosphates may also be present. L. A. COLES.

Calcium arsenate (U.S.P. 1,727,306).—See VII. Treatment of agricultural residues(U.S.P.1,747,538). —See XVIII.

## XVII.—SUGARS; STARCHES; GUMS.

Economic and industrial prospects of xylose. W. E. EMLEY (Chem. Met. Eng., 1930, 37, 238–285).— Xylose is obtained by hydrolysis of xylan, which is present in cottonseed hulls (40%), corn cobs (32%), peanut shells (26%), and other agricultural wastes, 682

with dilute acid. The method of manufacture, including the removal of ash, gum, and protein material, is fully described, as are also the potential uses of xylose. These include oxidation to trihydroxyglutaric acid, which may be used as a substitute for citric acid in foods and beverages, or in place of acetic-lactic acid mixtures for the vegetable-tan bath in leather tanning; the preparation of nitro-derivatives of xylose or "xylite"; fermentation to xylonic, propionic, or lactic acid; its employment by diabetics and for the preparation of syrups for certain artificial silk spinning processes. Probable costs are outlined. E. H. SHARPLES.

Sulphur dioxide in fruit syrups. WOIDICH.— See XVIII. Determination of sugar in condensed milk. DE GRAAFF.—See XIX.

See also A., June, 720, Acid clays of Japan (OKUNO). 731, Phthaleinoscope for acidimetry in coloured media (BRUÈRE).

PATENTS.

Sugar-cane and like mills. A. McA. LANG. From S. H. LEACH (B.P. 329,755, 26.3.29).

## XVIII.—FERMENTATION INDUSTRIES.

Downy mildew of the hop in British Columbia. W. NEWTON and C. YARWOOD (Sci. Agric., 1930, 10, 508-512).—For use in conjunction with Bordeaux mixture on hops the most efficient "spreader" was found to be a potash-resin soap, prepared by dissolving 1 pt. of caustic potash and 2 pts. of resin in 3 pts. of water and used in the proportion of 1 pt. of soap to 200 pts. of Bordeaux mixture. A similarly prepared soda-resin soap was of little value. (Cf. B., 1930, 299.) A. G. POLLARD.

Source of diastase. C. H. OTIS (Science, 1930, 71, 221).—The trade product, "Pangestin," is a suitable source of starch- and sugar-free enzyme which is strongly amylolytic and proteolytic. L. S. THEOBALD.

Micro-determination of sulphur dioxide in wine and fruit syrups. K. WOIDICH (Mikrochem., 1930, 8, 147—150).—The liquid (5 c.c.) is acidified with 5 drops of dilute hydrochloric acid in a pear-shaped bulb which is heated with steam inside a specially designed Erlenmeyer flask. The distillate is collected in 5 c.c. of 0.01N-iodine solution and the excess iodine is titrated with thiosulphate. A second portion of the liquid (5 c.c.) is treated with dilute iodine until a blue colour is obtained with starch and the distillation is repeated after addition of 5 drops of acid. The first titration gives the total, and the second the combined, sulphur dioxide. A. R. POWELL.

Determination of aldehydes in rectified spirits. E. WOJCIECHOWSKA-STRUSZYŃSKA (Przemysl Chem., 1930, 14, 222—227).—The intensity of the coloration given by reagents of the Schiff type with aldehydes is the greater the greater the fuchsin content and the smaller the sulphur dioxide content of the reagent; for this reason such reagents become more sensitive on keeping. The sensitivity of the reagent also increases with increase in alcohol concentration, until, at very high concentrations of alcohol, the reaction is positive even in the absence of aldehyde. The following minimum aldehyde contents (%) in absolute alcohol can be detected by the following reagents: Girard's and Mohler's, about 0.0005; Schiff's, Schiff and Gayon's, and Villavecchia's, about 0.00005. The following method is proposed for the determination of aldehyde in absolute alcohol. One c.c. of 10% m-phenylenediamine hydrochloride solution, kept in a dark glass bottle over animal charcoal, is added to 10 c.c. of the alcohol, and the fluorescence produced in the solution by the light of a mercuryvapour lamp is compared after 4 hrs. with that of a sample of alcohol of known aldehyde content. This standard is best prepared by fractional vacuum distillation of alcohol over m-phenylenediamine hydrochloride, and then adding to the aldehyde-free distillate a known quantity of acetaldehyde. Aldehyde-free alcohol gives a violet coloration in ultra-violet light under the above conditions, whilst a blue fluorescence appears at concentrations of 0.00002% of aldehyde; as the aldehyde concentration increases the fluorescence become yellowish-green. Accurate results are given by this method for concentrations of 0.00002-0.001%.

R. TRUSZKOWSKI. Coils for stills. BUTCHER.—See I.

See also A., June, 731, Phthaleinoscope for acidimetry in coloured solutions (BRUÈRE).

## PATENTS.

Manufacture of malt liquids. C. A. MITCHEL, Assr. to H. E. FREES Co. (U.S.P. 1,747,785, 18.2.30. Appl., 13.2.28).—The volatile constituents are distilled from hops at a pressure less than 1 atm., and the residual hop material is boiled with malt wort. After separation from the hop residues the wort is concentrated and, after cooling, is incorporated with the volatile constituents of the hops. C. RANKEN.

Tinting of beer. H. FINLAY (B.P. 328,999, 3.1.29).— A range of tinted glasses are so coloured that the progressive numbers which they bear correspond to the number of units of colouring matter in a unit volume of caramel solution. The glasses are mounted on a rotatable disc, so that they may be viewed side by side with a glass-sided tank containing the solution to be matched. The colour number of the wort and that to which it is to be raised are determined, and the difference between the two numbers on the glasses gives the number of colour units to be added to unit volume of the wort. C. RANKEN.

Apparatus for determining alcohol in liquids. B. STEIN (U.S.P. 1,747,742, 18.2.30. Appl., 21.12.28).— Two conical receptacles, one of which contains water and the other the liquid to be tested, are placed side by side and heated by a single source of heat situated in a flue between the two vessels. Thermometers are mounted in both vessels to indicate the b.p. of the contained liquid, and the second receptacle is also fitted with a condenser for the alcoholic vapours.

C. RANKEN.

Treatment of distiller's wash, and other byproducts and agricultural or industrial residues. M. BERNIER, A. and F. DURIEZ, and H. SCHOTSMANS (U.S.P. 1,747,538, 18.2.30. Appl., 29.3.23. Fr., 19.1.23). —Distiller's wash and other fermentable by-products are sterilised and fermented in a closed vessel by bacteria of the type of *B. aminophilus*. Before and during fermentation the  $p_{\rm H}$  of the solution is adjusted to between 6 and 8. C. RANKEN.

Glues from yeast (B.P. 328,645).-See XV.

## XIX.-FOODS.

Wheat and flour studies. XVI. Effect of yeast fermentation on viscosity of flour-water suspensions. A. H. JOHNSON and J. GREEN (Cereal Chem., 1930, 7, 117—132).—Fermentation lowers the viscosity of flour-water suspensions, the rate of decrease depending on the hydrogen-ion concentration during the process, high concentrations increasing and low decreasing this rate. The presence of sodium chloride and phosphates prevents the decrease in viscosity during fermentation periods extending over 10 hrs. The viscosity cannot be restored by addition of salts after fermentation.

#### W. J. BOYD.

Certain effects of varying the  $p_{\rm H}$  of the tempering water on the wheat proteins. M. C. MARKLEY (Cereal Chem., 1930, 7, 154—161).—For tempering wheat 0.1Nsulphuric acid and 0.1N-sodium hydroxide, respectively, do not differ from water at 92 hrs., but at 20 hrs. they depress the amino-acid content and increase both the water-soluble nitrogen and the copper-non-precipitable nitrogen. Sodium sulphate (0.05N) depresses these three values at all times. Sodium hydroxide combines more freely with the proteins than does sulphuric acid, but dilute solutions of these as tempering agents have nearly identical effects on the nitrogen distribution.

## W. J. BOYD.

Effect of severe weathering on the protein and ash contents of wheat and flour. W. O. WHITCOMB and A. H. JOHNSON (Cereal Chem., 1930, 7, 162—168).— No change was observed in the protein and ash contents of wheat subjected to severe weathering throughout the winter, although marked changes occurred in physical properties (e.g., in weight per bushel) and in germination.

#### W. J. BOYD.

Water imbibition of frosted wheat. C. H. BAILEY and E. G. BAYFIELD (Cereal Chem., 1930, 7, 108—116).— Unripe wheat kernels frozen when their moisture content is above 40% subsequently show higher water imbibition and higher amino-nitrogen content than normal kernels. Freezing in the unripe stage appears to arrest protein formation from the amino-acids. On rewetting, such kernels tend to revert to the moisture content at the time of freezing. W. J. BOYD.

Sundry means of hastening the determination of protein in wheat. H. L. WILKINS (Cereal Chem., 1930, 7, 168—188).—Various devices are described for handling reagents and samples in the routine determination of the protein in a large number of wheat samples. A reliable modification of the method recommended by the American Association of Cereal Chemists is described, whereby the protein in a sample may be determined in 25 min. In this, 0.5 g. of sample is digested in a 500-c.c. Kjeldahl flask for 12 min. on a 600-watt pre-heated Gilmer heater. Half portions of concentrated sulphuric acid, alkali thiosulphate solution, and mercuric oxide-potassium sulphate mixture are used, with the other reagents similarly reduced in amount, and 75 c.c. of liquid are distilled over. W. J. BOYD.

Adaptability of the quinhydrone electrode to cereal work. L. V. Sorg (Cereal Chem., 1930, 7, 143-153).—The quinhydrone electrode gives  $p_{\rm H}$  values in close agreement with those obtained with the hydrogen electrode, and by its use a constant potential is more readily reached (about 1 min.). The temperature coefficient is -0.00074 volt per degree. Under ordinary laboratory conditions variations of pressure have no measurable effect. Duplicate determinations agree within 0.000802 volt. For flour-water extracts bare gold wire is the best inert electrode, but bare platinum wire may be used for most solutions free from suspended matter. There is no error due to the water-soluble proteins in determining the  $p_{\rm H}$  of flour-water extracts by the quinhydrone electrode. W. J. BOYD.

Fermentation of bread. J. SPOUSTA and A. KUBÁSEK (Chem. Listy, 1930, 24, 121—129, 145—150).—Dough made from wheat and rye flours, and from mixtures of the two, with the addition of different quantities and strengths of yeast, is allowed to ferment for different periods of time before baking. For each case a certain optimum time is found, above or below which the porosity of the bread obtained is smaller. R. TRUSZKOWSKI.

Speed of germicidal action of chlorine compounds on bacteria commonly occurring in milk. C. K. JOHNS (Sci. Agric., 1930, 10, 553-563).—The effects of certain chlorine preparations used as sterilising rinses on *Esch. coli*, *Aer. aerogenes*, *S. lactis*, and *B. subtilis* are recorded. Chloramine-T preparations were uniformly too slow in action, whereas liquid hypochlorite preparations were very effective against all but sporing organisms. None of the materials examined affected spores of *B. subtilis*. Losses of available chlorine during storage were least in chloramine-T solutions. Homemade (prepared from bleaching powder) were more stable than proprietary preparations. A. G. POLLARD.

Determination of sugar content of condensed milk. J. DE GRAAFF (Chem. Weekblad, 1930, 27, 339— 340).—The error introduced in the polarimetric determination by the mutarotation of the lactose is discussed, and a modified procedure for preparing the solution is described. S. I. LEVY.

Cholesterol as a measure of egg yolk in milk products. L. M. LAMPERT (Ind. Eng. Chem. [Anal.], 1930, 2, 159—162).—A colorimetric method based on the Liebermann-Burchard procedure is described for the determination of cholesterol in dairy products, *e.g.*, icecream, by which the amount of egg used in the preparation may be estimated. A determination of the total cholesterol content of the ice-cream is made and a correction is applied for that introduced by the other constituents. It is found that whole, fresh egg-yolk contains 1.36% of cholesterol. H. INGLESON.

Method of Uglow and Schapiro for determination of caffeine in tea. F. C. VAN EEKHOUT (Chem. Weekblad, 1930, 27, 189—191).—Numerous methods for the determination of the caffeine content of tea are surveyed. The method of Uglow and Schapiro (B., 1928, 544) is considered inferior to that of Romburgh, Nanninga, and Deuss, and is criticised on account of the employment of sodium carbonate solution for the extraction : the caffeine obtained has usually a lower m.p. than that obtained by the other method. H. F. GILLBE.

Vitamin-C in fresh and canned tomatoes. B. CLOW and A. L. MARLATT (J. Agric. Res., 1930, 40, 767-775).—Various methods of canning cause only a slight loss, or none at all, in the vitamin-C content of field-ripened tomatoes. The amount of vitamin-C in field- or greenhouse-matured green tomatoes allowed to ripen by exposure to the air or ethylene is equal to that of tomatoes ripened naturally on the plant. This result is in agreement with that of House, Nelson, and Haber (A., 1929, 610). The vitamin-C content increases with maturation of the tomato. F. O. HOWITT.

Freezing of bacon. C. R. BARNICOAT (New Zealand J. Sci. Tech., 1930, 11, 409-410).—Bacon stored at --10° freezes slowly, giving large ice crystals which disrupt the flesh, but rapid freezing at lower temperatures, if anything, improves the quality. C. W. GIBBY.

Oxidation-reduction indicators as a means of determining overheating in walnuts during dehydration. A. R. C. HAAS (Bot. Gaz., 1930, 89, 200–204).—Overheating during the dehydration of walnuts is marked by the increased reducing power of the products. The latter is determined by the extent of the colour change in suitable oxidation-reduction indicators (e.g., an  $\alpha$ -naphthol-2-sulphonate-indophenol, methylene-blue, or the mono-, di-, or tetra-sulphonates of indigo) when added to an infusion of mashed walnut in a neutral buffer solution.

A. G. POLLARD. Detection of hardened fats. KAUFMANN AND KELLER.—See XII. Casein plastics. SIMMONS.—See XV. Xylose. EMLEY.—See XVII.

See also A., June, 825, Evaluation of plant products (NIETHAMMER). 827, Composition of odoriferous compounds of roasted chicory (REICHSTEIN and BEITTER).

## PATENTS.

**Rendering victuals radioactive.** H. STRATMANN and F. G. WERNER (STRATMANN & WERNER) (B.P. 329,292, 11.2.29).—Victuals coated with an electrically conducting film of water vapour are exposed at a high negative potential in the open air, until atmospheric radium emanation is transferred to them.

J. S. G. THOMAS. **Preparation of tapioca and apparatus therefor.** R. L. M. MORIN (B.P. 316,291, 16.5.29. Fr., 28.7.28).— Cassava fecula after treating with water until the moisture content is 50—55% above normal is spread out in layers 1 cm. thick on non-ferrous metal, then heated by steam for 2—3 min., and the product dried in a kiln. E. B. HUGHES.

Preparing an infusion of coffee from which the poisonous constituents have been removed. J. LOCKHOVEN (B.P. 327,662, 20.8.29).—Before infusion the coffee is mixed with granulated active carbon, e.g., in the proportion of 4:1. A small quantity of boiling water is added to the mixture, and a further quantity after 5—10 min. F. G. CLARKE.

Pretreatment of materials used in the preparation of pectin. PECTINERIE DU KERVOR, S.A.R.L. (B.P. 302,734, 21.12.28. Fr., 21.12.27).—In the preliminary lixiviation of the pulp used for the extraction of pectin, hot dilute acid is used instead of organic acid. It is claimed that this treatment gives a larger yield and purer pectin. E. B. HUGHES.

**Preservation of meat and fish refuse and the like.** W. CONNEMANN (B.P. 328,547, 21.12.28. Ger., 23.11.28).—The material, *e.g.*, raw shrimps, is mixed with sufficient quick lime, plaster of Paris, etc. to remove chemically the greater part of the water, and most of the hydrated lime etc. is removed from the product by screening. The product may be dried further by treatment with hot gases which, when lime has been used and the product is required for the manufacture of fodder, may contain carbon dioxide to convert residual calcium hydroxide into the carbonate. L. A. COLES.

**Treatment of [edible] animal matter.** D. Тномзом (U.S.P. 1,758,987, 20.5.30. Appl., 30.11.25. Renewed 11.10.29).—See B.P. 252,754; B., 1926, 689.

Apparatus for production of fish meal or the like. C. DOWNS and R. A. BELLWOOD (B.P. 329,723, 4.3.29).

Freezing cream etc. (B.P. 328,434).—See I. Bleaching materials (B.P. 328,544).—See VI. Irradiation of substances (B.P. 328,544).—See VI. Margarine (B.P. 327,519).—See XII. Increasing the vitamin content of foodstuffs (B.P. 328,942).—See XX.

## XX.-MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Pharmaceutical specialities and secret remedies. VII. Identification of pharmaceutical and technical solvents. C. A. ROJAHN, M. GREISS, and A. LINK (Pharm. Zentr., 1930, 71, 337—346).—Tables are given for the separation and identification of 61 of the more common organic solvents. The fractions of the pro duct soluble, and insoluble, in water are distilled through a column and the constituents identified by the special tests described previously (cf. B., 1927, 617).

H. E. F. NOTTON. Employment of the Seitz filter for pharmaceutical solutions. A. JACOBSEN (Dansk Tidsskr. Farm., 1930, 4, 146-154).-Experiments on the filtration of solutions of (a) arsenious oxide buffered with sodium phosphate, (b) morphine hydrochloride, (c) caffeine sodium benzoate, using the above filters, both of the pressure and suction type, showed that only 2-3% of the dissolved constituent was adsorbed by the filter; in the case of a 1: 10,000 solution of methyleneblue, 59% of the latter was retained. The material of the filter was found to yield magnesia to solutions passing through it, hence if solutions of alkaloids are being filtered these must either contain free acid (0.001N) or be buffered  $(p_{\rm H} 5.5)$ , otherwise a separation of the free base occurs in the filtrate.

H. F. HARWOOD.

Industrial extraction of santonin. F. CHEM-NITIUS (Chem.-Ztg., 1930, 54, 335).—An account of a small plant for the production of 40 kg. per month of the pure substance is given. S. I. LEVY.

South African pepper-tree oil. G. W. B. VAN DER LINGEN (Perf. Ess. Oil Rec., 1930, 21, 154).—Steamdistillation of fresh pepper-tree leaves with green berries gave 0.42% of a practically colourless oil having: d 0.8486,  $\alpha_{\rm D} + 58^{\circ} 24'$ ,  $n_{\rm D}^{\circ} 1.4732$ , acid value below 0.1, ester value (phenol-free oil) 46.70, ester value after acetyation (phenol-free) 115.80. On addition of absolute alcohol the solution immediately becomes turbid. Thymol could not be detected in the oil, but a phenol (carbanil ester, m.p. 140°), probably carvacrol, is present and also 26.3% of phellandrene. E. H. SHARPLES.

Leaf oil from Dacrydium Franklini, Hooker. A. R. PENFOLD and J. L. SIMONSEN (J. Proc. Roy. Soc. New South Wales, 1930, 63, 95–101).—The air-dried leaves of D. Franklini gives 0.3-0.76% of essential oil having  $d_{15}^{15}$  0.8752-0.8911,  $\alpha_{20}^{20}$   $+8.4^{\circ}$  to  $+18.6^{\circ}$ ,  $n_{20}^{20}$ 1.4805-1.4871, ester value 2.7-4.5, ester value after acetylation 16.6-36.9, insoluble in 10 volumes of 80%alcohol. l- $\alpha$ -Pinene,  $\beta$ -pinene, and d-limonene are present in the oil as also is phyllocladene, m.p. 95°. The hydrocarbon "dacrydene" stated to be present in this oil (Hooker, Baker, and Smith, "Pines of Australia," p. 397) is probably identical with d- $\Delta$ 4-carene.

E. H. SHARPLES.

Australian "tea trees " of economic value. I. A. R. PENFOLD and F. R. MORRISON (Bull. Tech. Mus., Sydney, 1929, No. 14, 15 pp.).-The following essential oils were obtained by steam-distillation of the leaves and terminal branchlets. Melaleuca linariifolia gave 1.5-2.0% of oil having d 0.8927-0.8992,  $\alpha$  +3.3° to  $+6.8^{\circ}$ , n 1.4752-1.4780, ester value 1.3-2.7, ester value after acetylation 58-82, and consisting principally of  $\alpha$ - and  $\gamma$ -terpinene, cymene, cineol (16-20%),  $\Delta^{1}$ -terpineol-4, and sesquiterpenes. M. alternifolia yielded about 1.8% of oil having d 0.8958-0.8961,  $\alpha + 6 \cdot 8^{\circ}$  to  $+7 \cdot 4^{\circ}$ ,  $n \cdot 1 \cdot 4782 - 1 \cdot 4790$ , ester value 4-7, ester value after acetylation 80-84, and containing d- $\alpha$ -pinene,  $\alpha$ - and  $\gamma$ -terpinene, cymene, cineol (8%)  $\Delta^1$ -terpineol-4, and sesquiterpenes. Both the above oils have pleasant odours, are non-poisonous, and are 11-13 times as powerful in destroying typhoid germs as is carbolic acid. From Leptospermum citratum is obtained 1-1.5% of oil having d 0.8792-0.8856,  $\alpha$  +3.5°, n 1.4688-1.4757, solubility in 70% alcohol 1 in 1—1.2 vols., and containing about 50% of citral and 35% of citronellal. E. H. SHARPLES.

Essential oils of *Melaleuca decora* (Salisbury) Druce, and *M. nodosa*, var. *tenuifolia* (de Candolle), from the Port Jackson district. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. New South Wales, 1930, 63, 102—110).—Steam-distillation of the leaves and terminal branchlets of *M. decora* yielded 0.12-0.31%of bright yellow essential oils having  $d_{15}^{15}$  0.8895—  $0.9069, \alpha_{25}^{20} + 12 \cdot 2^{\circ}$  to  $+13 \cdot 25^{\circ}, n_{25}^{20} 1 \cdot 4785 - 1 \cdot 4872$ , ester value  $8 \cdot 1 - 11 \cdot 9$ , ester value after acetylation  $30 \cdot 4$ —  $52 \cdot 6$ , solubility in 80% alcohol insoluble in 10 vols., and consisting chiefly of d- $\alpha$ -pinene (50-60\%), sesquiterpenes (25-30\%), with small quantities of  $\alpha$ -terpineol (?), dipentene, and a sesquiterpene alcohol. Leaves and terminal branchlets of M. nodosa, on steam-distillation, yielded 0.9-1.0% of lemon-yellow coloured oils having  $d_{15}^{15} 0.9073 - 0.9125$ ,  $\alpha_{20}^{20} + 5.15^{\circ}$  to  $+7.75^{\circ}$ ,  $n_{20}^{20} 1.4641 - 1.4649$ , ester value 3.5-5.8, ester value after acetylation 40.7, solubility in 70% alcohol 1 in 1.3-5.5 vols., and containing cineol 40-55%,  $\alpha$ -pinene, dipentene,  $\alpha$ -terpineol, and sesquiterpenes. E. H. SHARPLES.

Occurrence of a number of varieties of Eucalyptus dives as determined by chemical analysis of the essential oils. III. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. New South Wales, 1930, 63, 79-84; cf. B., 1929, 537).-Essential oils from E. dives grown in Victoria are described. Most of the trees examined were Type with an admixture of var. "A," and this was reflected in the varying piperitone contents of the oils (2-54%). Previous results have been confirmed, and all forms of E. dives are well established as separate varieties according to the chemical composition of the respective oils. Where the piperitone content fluctuates between 26% and 36% it is due almost invariably to admixture of var. "A" with Type. Four representative E. dives, Type oils from Victoria had  $\begin{array}{l} d_{15}^{15} \ 0.8866 - 0.9062, \ \alpha_{\rm D}^{20} \ -57 \cdot 3^{\circ} \ \ {\rm to} \ \ -68 \cdot 7^{\circ}, \ n_{\rm D}^{20} \ 1 \cdot 4783 \\ -1 \cdot 4810, \ {\rm solubility} \ {\rm in} \ \ 70\% \ \ {\rm alcohol} \ 1 \ {\rm in} \ 1 \cdot 4 - 10 \ {\rm vols.}, \\ {\rm piperitone} \ \ 36 - 53\%. \ \ {\rm The} \ {\rm yields} \ {\rm were} \ \ 2 \cdot 9 - 4 \cdot 0\%. \end{array}$ E. H. SHARPLES.

Determination of citronellal and citral in Australian essential oils. A. R. PENFOLD and W. G. ARNE-MAN (J. Proc. Sydney Tech. Coll. Chem. Soc., 1927-1929, 4, 53-60).—The method of Holtappel (B., 1928, 587) for the determination of citronellal gives low values in the examination of oils rich in this aldehyde, and the following modification is described. About 2 g. of the oil are cooled to  $-10^{\circ}$ , 20 c.c. of reagent [5 g. of hydroxylamine hydrochloride in 9 c.c. of water, 80 c.c. of neutral rectified spirit, 2 c.c. of bromphenol-blue indicator (0.1 g. in 3 c.c. of 0.05N-sodium hydroxide and 22 c.c. of water), made up to 100 c.c. with rectified spirit] and 30 c.c. of 0.5N-potassium hydroxide are mixed and also cooled to  $-10^{\circ}$ , and the two solutions are mixed, kept at  $-10^{\circ}$  for 15 min., and then allowed to attain room temperature. The excess alkali is then titrated with 0.5N-alcoholic hydrochloric acid. A good end-point is obtained and results, using pure citronellal and essential oils rich in this compound, are more accurate than those given by the original method, by Kleber's phenylhydrazine method, or by Dupont and Labaune's oximation method. Oils from Eucalyptus citriodora gave aldehyde contents of 70-85%, Leptospermum citratum 70-85%, and Backhousia citriodora 94%. Oils containing citral and citronellal soon change in aldehyde E. H. SHARPLES. content on keeping.

Hydrogen peroxide. SCHWYZER. Antiformin. GJALDBÆK. Bismuth salts. MAUERSBERGER.—See VII. Testing of ampoules. PRITCHARD.—See VIII. Tobacco pest. AYYAR.—See XVI. Xylose. EMLEY.— See XVII. Determination of caffeine in tea. VAN EEKHOUT.—See XIX.

See also A., June, 741, Bactericidal dialkylacetic acids (ARMENDT and ADAMS). Bactericidal olefinic acids (BROWNING and others). 765, Mono- and polyalkoxy- or halogeno-substituted derivatives of benzhydroxylamine and  $\alpha\beta$ -diphenylethylamine (VALETTE). 772, Ethyl esters of chaulmoogric acid (LORA Y TAMAYO). 786, Substituted piperidinoalkyl benzoates (BAILEY and MCELVAIN). Catalytic reduction of 1-phenyl- and 1-benzyl-4-piperidones (BOLYARD). 788, 5:5-Substituted barbituric acids (VOLWILER and TABERN). Microscopical identification of barbituric acid (DENIGÈS). 793, Synthesis of thiazoles containing phenol and catechol groups (SUTER and JOHNSON). 794, The alkaloid coclaurine (Kondo and Kondo). 795, Decomposition of berberine in aqueous solution (DIETZEL and SOLLNER). 796, Microchemical reactions of veratrine (WAGENAAR). Fritzsche's reagent for alkaloids (DE LISSNER). 803, Preparation of lecithin (MALTANER). 825, Evaluation of plant products (NIETHAMMER).

#### PATENTS.

Vehicle for acetylsalicylic acid tablets. W. C. BAUSCH (U.S.P. 1,746,984, 11.2.30. Appl., 31.10.27).— The tablets contain 3—4 pts. of acetylsalicylic acid and 1 pt. of stearic acid, which latter inhibits hydrolysis in the stomach, but not in the intestine. L. A. COLES.

[Medical] preparation of solid compounds, capable of being spread, containing iodine and potassium iodide. R. GELLER (B.P. 310,869, 8.1.29. Ger., 2.5.28).—A solution of sodium hydroxide or ethoxide in alcohol is added to a hot alcoholic solution of benzoic acid, iodine, and potassium iodide are added to the neutral mixture, and the whole is poured into moulds and cooled. The benzoic acid may be replaced by salicylic, phthalic, palmitic, or stearic acid, or by mixtures of these. H. ROYAL-DAWSON.

Production of extracts for direct application as medicaments and for increasing the vitamin content of foodstuffs, medicaments, and the like. G. M. CLARK. From MATRO GES.M.B.H. (B.P. 328,942, 4.2.29).—Rootlets of germinated grain or maize from the drying kilns in maltings contain up to about 0.15% of sterol which is antirachitically active even in the unirradiated condition. The rootlets, either alone or mixed with substances such as caustic soda, lime, etc., are extracted with alcohol, the extract is freed from the solvent *in vacuo*, and the residue extracted with a solvent for fat. The solvent-free extracts so obtained are used for increasing the vitamin content of foods etc. E. H. SHARPLES.

Manufacture of solutions of cholesterol or its esters or mixtures of these substances. F. PASSEK (B.P. 328,922, 4.2.29).—Solutions of cholesterol or its esters etc. are prepared by dissolving these substances in a small amount of chloroform, ethylene dichloride, or similar type of chlorinated hydrocarbon and adding sufficient aqueous alcohol to ensure the production of a permanently clear solution. E. H. SHARPLES.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Effect of grain size in photographic emulsions on failure of reciprocity law and a theory of its origin. A. P. H. TRIVELLI and R. P. LOVELAND (J. Franklin Inst., 1930, 209, 639—648).—The results of Jones and Hall for the effect of difference in grain size on the intensity-time function of exposure of a photographic emulsion (B., 1926, 1030) have been confirmed and supplemented. If the larger grains contain most of the larger sensitivity specks, the small grains will require more light energy than the larger grains to convert their specks into centres large enough to be developable, which accounts for their, being more sensitive to variations in intensity. If photographic sensitivity depends on the photo-conductivity of the silver halide lattice, the observed rate of increase of the photo-electric current with illumination adequately explains the failure of the reciprocity law at both low and high intensities. R. CUTHILL.

Reactions of photographic materials to light. S. E. SHEPPARD (Ind. Eng. Chem., 1930, 22, 555-563).— A lecture. The applications of photochemistry to the problems of photography are discussed.

H. INGLESON.

Properties of [photographic] fixing baths. J. I. CRABTREE and H. A. HARTT (Brit. J. Phot., 1929, 76, 743-745, 758-761, 777-778; 1930, 77, 4-6, 19-22). -Various properties of fixing baths have been investigated and special tests are outlined for the determination of the velocity of fixation, the acidity, the sulphurisation life, i.e., the time which elapses before deposition of sulphur, the hardening action on the gelatin film, and finally the developer capacity, i.e., the quantity of standard developer which may be added before precipitation of aluminium sulphite and undue loss in hardening action occur. For a given emulsion a maximum velocity of fixation is reached with 30-40% sodium thiosulphate solution, and this velocity increases with temperature and with the degree of agitation of the film, being most markedly accelerated when the film surface is brushed. The hardening action is dependent on the balance between the potash alum, acetic acid, and sodium sulphite contents of the bath, and is unaffected by its age or thiosulphate content. With constant quantities of sulphite and alum the hardening decreases with addition of acid, whilst with increasing quantities of sulphite in presence of constant alum and acid concentrations the hardening rises to a maximum and then falls away, i.e., for maximum hardening there is a critical sulphite concentration for each acid concentration. The maximum hardening with any combination is increased by addition of alum. Extension of time of fixation increases the hardening action, and over the temperature range 5-27° a slight increase is shown at the higher temperatures. For equal concentrations of alum and acid the sulphurisation life is rapidly increased by addition of sulphite, whilst the addition of acid to baths containing equal quantities of alum and sulphite shortens the life. Alum itself also slightly shortens the life. The application of these results to the compounding of fixing baths of definite properties is discussed; formulæ for satisfactory non-hardening and acid-hardening baths are also given, together with methods by which such baths can be revivified during continuous use. The causes and means for avoiding various defects in the fixing process are described.

J. W. GLASSETT

Photographic chemistry. I. Dispersion of silver halides through the aqueous medium. T. SUZUKI (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1930, 12, 285-293).-Ability to cause ready dispersion of freshly precipitated silver bromide in water appears to be the property of aqueous or alcoholic solutions of various dyes and the common alkaloids. Amongst the specific dyes are included the usual sensitisers, and desensitisers for gelatin-silver bromide plates. The dispersed silver halide is not stable, but after flocculation redisperses easily on agitation. Silver chloride and iodide, mercurous chloride, and thallous chloride can also be dispersed in the same manner. Substances which disperse silver bromide do not necessarily alter the sensitivity of the silver bromide plate, but plates made from dispersed silver bromide are suitable for J. O. CUTTER. photographic use.

See also A., June, 716, Photomicrography of silver halide grains (TRIVELLI and LOVELAND). Photochemical properties of silver bromide in aqueous gelatin or gum arabic (REYCHLER). Blackening of photographic plates by electron rays (NACKEN). 717, Photothermometry (HENCKY and NEUBERT).

## PATENTS.

Kinematograph films. P. REHLÄNDER (B.P. 303,794, 8.1.29. Ger., 9.1.28).—The deleterious effect of scratches is minimised by the use of a support for the positive film comprising a mixture of celluloid with material of high albedo (magnesia, white lead, lithopone), the support having a transparency of about 55-60% and a diffusion value of 0.11-0.16. L. A. COLES.

[Fireproofing of] cellulose [kinematograph] films. FILM OZAPHANE (B.P. 305,653, 10.1.29. Fr., 10.2.28. Addn. to B.P. 301,878; B., 1930, 457).— Ammonium bromide is used alone without other admixture. H. ROYAL-DAWSON.

Marking material by electrodeposition (B.P. 329,258).—See V. Transfer of wood grain (B.P. 303,804).—See IX.

## XXII.—EXPLOSIVES; MATCHES.

Duration of the explosion flame of quarry and coal-mine explosives. P. NAOÚM and A. BERTHMANN (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 193-197).-For these experiments the authors made certain modifications in the Mettegang apparatus, and used 100-g. cartridges of 30 mm. diam., which were fired in a steel mortar, whereas Kast and Selle used 25-g. cartridges and fired them in a lead mortar with consequent loss of efficiency. The explosives examined comprised Wetter-Detonit A and B, Wetter-Siegrit A, Wetter-Nobelit A and B, Gelatit 1, Ammonit 1, 2, and 5, Ammon-Gelatine 1, Chloratit 3, Dynamit 1, and Kieselguhr Dynamit. The duration and size of flame, explosion temperature, and specific pressure are tabulated, and photographic reproductions of the flames are given. The lengths of the flames varied, but not proportionately, with their duration. No relation could be found between explosion temperature and duration of flame. Contrary to the experience of Kast and Selle, it was found that the flame duration of coal mine explosives differed appreciably from that of quarry explosives, and even amongst the former there were obvious divergences.

W. J. WRIGHT.

Direct determination of nitric nitrogen in colloidal [explosive] powders. Acetone method. H. MURAOUR (Bull. Soc. chim., 1929, [iv], 45, 1189-1192).-The powder (about 0.8 g. for a nitric acid content of 12.5%) is agitated for 2 hrs. with 40 c.c. of acetone in a pyrex glass. 50 C.c. of 12-vol. hydrogen peroxide and 4 c.c. of 5N-sodium hydroxide are added, and, after agitating until dissolution is complete, 4 g. of sodium perborate. The mixture is left for 12 hrs., heated for 20 min. at 60°, and after adding 50 c.c. of water, at 50° till the perborate is destroyed. After cooling, 30 c.c. of 10N-sodium hydroxide, 80 c.c. of water, and 4 g. of Devarda alloy are added. The process is completed as in the ordinary Devarda method, about half the distillate being collected in 25 c.c. of N/3-sulphuric acid. The mixture gives concordant results with mixtures of nitrocellulose and nitroglycerin and the results are in agreement with those obtained by the Dumas method on guncotton etc. which is free from centralite and similar nitrogen compounds. Low results obtained by the ordinary Devarda method are due to the reducing action of organic matter in presence of soda on the nitrate in solution. R. BRIGHTMAN.

Xylose. EMLEY.-See XVII.

See also A., June, 708, Explosion regions in gas mixtures (POSTHUMUS). 725, Analysis of nitrating acid. (MÜLLER and KOGERT). 750, X-Ray investigation of nitrocellulose (TROGUS and others).

#### PATENTS.

Delay powder. F. OLSEN (U.S.P. 1,748,455, 25.2.30. Appl., 30.10.23).—Such powders contain a deterrent, such as triphenyl phosphate, to regulate the rate of burning. A suitable mixture contains charcoal  $15 \cdot 3\%$ , sulphur  $8 \cdot 4\%$ , potassium nitrate  $74 \cdot 1\%$ , and triphenyl phosphate  $2 \cdot 2\%$ . W. J. WRIGHT.

Manufacture of tracer compositions. W. S. HEITMANN (U.S.P. 1,748,288, 25.2.30. Appl., 8.9.25).— To obtain intimate admixture of the components of tracer composition, the combustible ingredients are first mixed together, and the binder (e.g., a metallic resinate) is dissolved in a solvent and incorporated with the mixture. The solvent is then driven off by evaporation, and the resultant material is ground, graded, and pressed. W. J. WRIGHT.

Preventing the formation of copper azide in copper lead azide detonators. W. ESCHBACH (B.P. 304,144, 14.1.29. Ger., 14.1.28).—Air is excluded from electric detonators with copper shells by fixing the fusehead in its cardboard case by means of sulphur or non-combustible materials such as chlorinated hydrocarbons, with or without other non-inflammable substances. After the whole has been inserted in the detonator, the free space above is sealed by a cast fusible material. W. J. WRIGHT.

Blasting cartridges. D. Hodge (B.P. 329,636, 20.2.29).

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## XXIII.—SANITATION; WATER PURIFICATION.

Bacterial population during [sewage-]sludge digestion. C. C. RUCHHOFT, J. G. KALLAS, and G. P. EDWARDS (J. Bact., 1930, 19, 269—294).—Changes occurring during the digestion of activated sludge, fresh sewage, and mixtures of these are examined. Reduction of the total organic solids was least in activated sludge and greatest in mixtures containing 20% of activated sludge. Changes in  $p_{\rm H}$  of sludges during digestion were small, the extremes for all tests being  $6\cdot4$ — $7\cdot4$ . The total bacterial population increased for the first 10—15 days of digestion, after which there was a steady decline throughout the process.

## A. G. POLLARD.

Germicidal efficiency of chlorine and the Nchloro-derivatives of ammonia, methylamine, and glycine against anthrax spores. F. W. TILLEY and R. M. CHAPIN (J. Bact., 1930, 19, 295–302).—Nitrogen trichloride, chlorine in neutral solution, and chlorine in solution in 0.02N-hydrochloric acid were effective against spores of *B. anthracis* in 15 min., with available chlorine concentrations of 10 p.p.m. or less. Corresponding values for chlorine in 0.02N-sodium hydroxide were 100 p.p.m., 2 hrs.; for monochloroamine 80 p.p.m., 45 min.; dichloroamine 80 p.p.m., 30 min.; dichloromethylamine 50 p.p.m., 30 min.; and dichloroglycine 50 p.p.m., 45 min. Monochloro-methylamine and -glycine were not effective in 2 hrs. with 200 and 240 p.p.m., respectively, of available chlorine.

#### A. G. POLLARD.

Toleration of fresh-water protozoa to increased salinity. H. E. FINLEY (Ecology, 1930, 11, 336-347).--Several among many species of protozoa examined survived transference from fresh to sea-water and others withstood lower concentrations of sodium chloride.

A. G. POLLARD.

Correlation of sulphite reducing and colon bacteria in water. I. M. LEWIS, T. C. GREEN, and V. HAMILTON (J. Amer. Water Works' Assoc., 1930, 22, 667-672).—Numerous samples of filtered river water and raw spring water were examined for the presence of *B. coli* of fæcal and non-fæcal origin and for organisms capable of reducing sulphites. The results obtained failed to show satisfactory correlation. Thus 20% of the samples returned as *coli* free, and therefore "good," contained over 11 sulphite reducers per 100 c.c., which would class them as "bad"; again, sulphite reducers were absent in 15% of the total, which samples, according to the *coli* test, would be "questionable" or "bad." It is considered that the sulphite-reduction test is of little or no value as a supplement to the standard test for *B. coli*.

## C. JEPSON.

Influence of chlorine treatment on acidity of water. A. MASSINK (Chem. Weekblad, 1930, 27, 304–307).—The publications of Haase (cf. B., 1928, 466; 1929, 341) are criticised, and it is shown both experimentally and on theoretical grounds that with normal waters no variation in  $p_{\rm H}$  sufficient seriously to affect the properties of the treated water is to be expected.

S. I. LEVY.

Determination of nitrates [in water]. G. W. BURKE, M. LEVINE, and G. H. NELSON (J. Amer. Water Works' Assoc., 1930, 22, 679-684).—Concentration of the alkaline sample prior to reduction, as recommended in "Standard Methods of Water Analysis," does not cause any loss of nitrite. Nitrites are destroyed if ammonium chloride (up to over 3500 p.p.m.) be added and the mixture evaporated to dryness before making alkaline. If the nitrites be destroyed in this way and the ammonia then driven off by concentration of the solution in presence of sodium hydrate, the nitrates may be determined directly by reduction in the usual way. C. JEPSON.

Prevention of nuisance from fumes and dust. TRAVERS.—See I. Determination of phenol in effluent waters. PIETERS.—See II.

See also A., June, 741, Bactericidal dialkylacetic acids (ARMENDT and ADAMS). Bactericidal olefinic acids (BROWNING and others).

## PATENTS.

Purification of polluted liquids. J. T. TRAVERS, Assr. to Ohio Sanitary Eng. Corp. (U.S.P. 1,747,803, 18.2.30. Appl., 19.8.27).-The putrescible matter, mostly present in colloidal form, is converted into a coagulated suspensoid by agitating the liquid with a slowly dissolving reagent containing as a main constituent (a) an electrolyte-producing material, together with (b) an alkali, and (c) a coagulant. On adding (a), the colloidal putrescible matter acquires a negative charge and is then enabled to adsorb the positive ions offered by (a). Accompanying this adsorption nascent oxygen is generated which assists in destroying bacteria and renders re-aeration of the liquid unnecessary. By way of example, (a) may be calcium or magnesium sulphate, or certain waste products from cement manufacture or alkali plants; (b) lime; and (c) ferrous or aluminium sulphate or calcium monophosphate. Apparatus for carrying out the process is described. S. K. TWEEDY.

Apparatus for treating impure water with chemical solutions. O. WALTER (G.P. 456,611, 25.3.26).—The water is admitted through a float-controlled valve, and an adjustable apparatus to compensate for the hydrostatic head of the chemical solution is provided. W. G. CAREY.

Purification and softening of water. F. SCHMIDT (F.P. 631,530, 28.3.27).—A mixture of caustic soda or potash, sodium or potassium carbonate, and water is finely ground and to it are added silicates, aluminates, or aluminosilicates. The mixed solutions are boiled before use. W. G. CAREY.

**Removal of oxygen from water.** H. MANZ (G.P. 456,459, 21.3.26).—The water is passed over the cathode of a low-tension electrolytic cell using an easily oxidisable anode, with a slight cathodic polarisation.

W. G. CAREY.

Gas mask and like appliances generating their own oxygen. W. W. TRIGGS. From INHABAD-GES. M.B.H. (B.P. 304,248, 17.1.29).

Ferric alumina ((U.S.P. 1,747,177). Oxygen preparations (U.S.P. 1,745,465).—See VII.