BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 22, 1927.

I.—GENERAL; PLANT; MACHINERY.

Recovery of volatile solvents by "chemical washing." H. WIESENTHAL (Chem.-Ztg., 1927, **51**, 373).—The "Cheminova" apparatus as used for the recovery of alcohol and ether vapours in the collodion artificial silk process is described. The air containing 20 g./cub. m. of vapour is passed through a dust filter to a series of towers, where it is treated with a suitable reagent. From this alcohol, ether, and water are recovered by distillation, and the distillate is fractionated. C. IRWIN.

Determination of pressure of liquefied gases within containers. R. NITZSCHMANN (Continental Met. Chem. Eng., 1927, **2**, 79).—Equations and charts defining the pressures developed between — 10 and + 45° in containers of liquid chlorine in relation to the volume and the filling temperature and pressure indicate that, under certain conditions, the containers are advantageously filled at relatively high temperatures. CHEMICAL ABSTRACTS.

Further applications of a mobilometer. H. A. GARDNER and A. W. VAN HEUCKEROTH (Ind. Eng. Chem., 1927, **19**, 724—726).—A mobilometer is described in which the time taken to push a metallic disc (perforated or otherwise), suitably loaded with a known weight, through a given column of material is measured. Applications in the paint and varnish and in other industries are described. The time of "flow" usually varies appreciably when diluents are added to the material being tested. S. K. TWEEDY.

PATENTS.

Ball mill. P. L. CROWE (U.S.P. 1,627,487—8, 3.5.27. Appl., [A, B], 30.9.25).—A cylinder rotating on a horizontal axis is divided by a vertical screen into a grinding chamber containing steel balls and a blowing chamber containing fan blades. These latter induce a current of hot air, which carries pulverised coal from the grinding chamber through the screen into the furnace. In a modification of the apparatus the grinding chamber surrounds the blowing chamber. T. S. WHEELER.

Grinding mill. J. R. TORRANCE (E.P. 270,465, 25.3.26).—In a roller mill for, *e.g.*, paint, the material is fed to one end of a pair of rolls and is not removed until it arrives at the other end, guiding and scraping blades being provided. The rolls are given a considerable differential speed, the roll with the higher speed being of larger diameter than the other. They may be either cylindrical or frusto-conical with equal and opposite tapers. B. M. VENABLES.

Rotary drying apparatus. H. A. MARSTON (E.P. 270,503, 3.6.26).—Hot gases from a furnace pass first over the outside of a rotary drying drum and then through the interior, counter-current to the material. The drum is provided with one or more enlarged zones containing baffles and helical blades, which delay and mix the material and give the hot gases a whirling motion. B. M. VENABLES.

Mixing and agitating machines and appliances. B. GOLDMAN (E.P. 267,480, 8.2.27. Conv., 9.3.26).—In a tank containing liquid, which is set in motion by any desired means, baffles are suspended by cords or chains in such a way that the drag of the liquid will cause them to move upwards towards the surface of the liquid. B. M. VENABLES.

Method and apparatus for leaching. A. B. KENNEDY (U.S.P. 1,628,787, 17.5.27. Appl., 21.2.22).—A tank for leaching tannins etc. from bark has the bottom formed of a series of troughs of semicircular cross-section placed edge-to-edge. A paddle wheel rotating in each trough passes the material under treatment into the adjacent trough against the flow of extracting liquid, and prevents the material from rising to the surface of the liquid. T. S. WHEELER.

Apparatus for dissolving and filtering minerals and other inorganic material. COMP. DE PROD. CHIM. ET ÉLECTROMÉTALLURGIQUES ALAIS, FROGES, ET CAMARGUE (F.P. 614,862, 21.4.26. Conv., 22.4.25).—A cylindrical vessel is divided into two sections by a filtering plate upon which the material is treated with the solvent. Means are provided for heating and agitating the material upon the plate. L. A. COLES.

Filtration. E. A. ALLIOTT, A. E. HATFIELD, and ACHILLE SERRE, LTD. (E.P. 270,461, 20.3.26).—To each batch of the soiled liquid flowing from a clothes-washing or cleaning machine a definite volume of filter-aid pulp is added at a constant rate. When the measuring vessel delivering to the pulp has emptied itself to a certain level, the vessel is refilled and the pulp diluted with fresh or soiled washing liquid, so that, the outflow remaining constant, the amount of filter aid added per sec. decreases in the later stages; this compensates for the fact that the washing liquid carries most dirt in the early stages. The dilution of the pulp may be effected several times. The apparatus comprises mainly a measuring vessel with internal float which governs the dilution, and another float chamber which prevents any vacuum in the pipe conveying the washing liquor to the filter from affecting the delivery of filter-aid. B. M. VENABLES.

Filter. J. E. SPALDING and R. L. ARCHER (U.S.P. 1,630,079, 24.5.27. Appl., 20.6.24).—An ascending outlet

conduit leads from the sand bed of the filter to a descending conduit connected to it at the normal level of the liquid within the filter. An adjustable vent admits air above the liquid in the first conduit to prevent draining of the filter through the outlet. H. HOLMES.

Purification, decoloration, and deodorisation of liquids and solutions. L'ULTRA-FILTRE D.M.S. (E.P. 262,131, 26.11.26. Conv., 26.11.25).—The liquid is passed through superposed porous membranes having adsorptive properties, such as nitrocellulose, cellulose acetate, or gelatin. The membranes are divided into groups which can be put into operation independently, the saturated membranes being regenerated by passing through them in a reverse direction the same liquid at a changed temperature, and to which may be added other products or a different liquid. W. G. CAREY.

Apparatus for treating air or gas with liquid. VISCO ENGINEERING CO., LTD., and F. C. SMITH (E.P. 269,790, 4.10.26).—In a wet scrubber of the type where a number of rotating perforated plates dip into a liquid bath, the perforations are provided with raised edges or lips on the side facing the oncoming gas, with the object of preventing dirty liquid being carried through the perforations. B. M. VENABLES.

Distributing apparatus. A. MÖDER (G.P. 428,929, 15.1.25).—Apparatus suitable for treating liquids with gases, for crystallising salt solutions, etc. comprises a number of superposed box-shaped structures of unequal height rotating about a common axis, and provided with helical grooves or channels. L. A. COLES.

Drying and separation from gases of soluble and/or condensable vapours and impurities. CARRIER ENGINEERING CO., LTD., and S. L. GROOM (E.P. 269,714, 18.3.26).—The gases (particularly coal gas) are scrubbed with refrigerated water; they preferably pass through two chambers in series, each being provided with water sprayed in opposite directions to the gases from separate refrigerating-washing circuits, and drop arrestors are placed between the two chambers and at the final exit. The refrigerators are preferably of the evaporative type, and a weir, trap, or water seal may be provided for drawing off condensed light liquids, such as benzol, also the excess water due to condensation. B. M. VENABLES.

Apparatus for the purification of gases. G. KÖNIG (G.P. 438,833, 7.2.25).—Dust particles are precipitated by passing the gas upwards through a honeycomb filter in which lateral and vertical movements are prevented by vertical or sloping partitions which are intersected by cross-partitions to facilitate the precipitation. A settling chamber may be attached to the filter. L. A. COLES.

Separation of gas mixtures. M. FRANKL (F.P. 613,755, 16.1.26. Conv., 16.1.25).—Liquid collectors used in separating gas mixtures by liquefaction are divided into two or more sections, or consist of separate apparatus provided with corrugated sheets of metal, the separate sections being used in rotation. L. A. COLES.

Furnace for supplying drying gases. GENERAL RUBBER CO., Assees. of D. E. MANSFIELD (E.P. 267,464, 10.9.26. Conv., 13.3.26).—A combustion chamber is surrounded by a jacket, and both are supplied at the same end with air from a common blower, the air being preferably delivered tangentially into the jacket. The united gases are exhausted either to atmosphere when lighting up and smoky, or to pass over the goods to be dried. The fuel is preferably liquid, and refractory material is used to promote complete combustion. B. M. VENABLES.

Kiln. CARBORUNDUM CO., LTD. From B. M. JOHNSON (E.P. 270,520, 6.7.26).—A number of heating chambers each provided with internal "radiating" combustion chambers are interconnected by underground flues, so that air before combustion may be passed over already burnt material, and products of combustion may be used to preheat new material. B. M. VENABLES.

Refrigerant. G. BARSKY, Assr. to AMERICAN CYANAMID CO. (U.S.P. 1,631,573, 7.6.27. Appl., 5.3.26). —A mixture of monomethylamine and ammonia is claimed. H. HOLMES.

Manufacture of an adsorbent. K. IKEDA, H. ISOBE, and T. OKAZAWA, Assrs. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,630,660, 31.5.27. Appl., 27.12.23).—See E.P. 206,190; B., 1924, 3.

Apparatus for the extraction and recovery of volatile liquids. E. A. IRONSIDE (U.S.P. 1,631,036 31.5.27. Appl., 6.11.25. Conv., 8.11.24).—See E.P. 246,930; B., 1926, 345.

Utilising the heat of materials discharged from furnaces and kilns. BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX CO. (E.P. 269,813, 11.12.26).

Refrigerating apparatus. G. ROOS (E.P. 249,873, 24.3.26. Conv., 28.3.25).

[Cover for] hydro-extractors for laundry and like purposes. G. E. EDEN (E.P. 270,515, 22.6.26).

Heat-retaining coverings or lagging. J. S. GRIMASON, and KEASBEY-MATTISON, LTD. (E.P. 270,589, 7.12.26).

Gas-purifying plant (G.P. 438,834 and F.P. 614,442). —See XI.

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

Autoxidation and anti-oxygenic action. XXII. Mode of action of anti-detonators. C. MOUREU, C. DUFRAISSE, and R. CHAUX (Chim. et Ind., 1927, 17, 531—535).—Comparison of the action of substances which suppress knocking with the "anti-oxygenic" action described in preceding papers (A., 1926, 581, 1031, 1215), of which the inhibition of the autoxidation of benzaldehyde by traces of quinol and the "poisoning" of certain catalysts may be taken as typical, leads to the conclusion that both actions result from the same cause. None of the various theories of knock suppression which have been proposed up to the present accounts fully for the facts. The theory proposed by the authors is summarised in the equations: $A + O_2 \rightarrow A[O_2]$; $A[O_2] + B \rightarrow A[O] + B[O]$; $A[O] + B[O] \rightarrow A + B + O_2$. A is the oxidisable material or

fuel, and $A[O_2]$ an unstable peroxide which, in the absence of the anti-knock compound B, would pass directly into stable oxidation products. This peroxide, however, in the presence of B interacts with it to form the two peroxides A[O] and B[O], which interact further, in the known manner of antagonistic peroxides, giving A, B, and O_2. Some of the consequences of the theory are briefly discussed. A. B. Manning.

Mechanism of "knock" suppression. H. S. Taylor (Nature, 1927, **119**, 746; cf. Egerton and Gates, A., 1927, 318; B, 1927, 402).—Not only do the lead atoms obtained on thermal decomposition of lead tetraethyl function as oxidation centres, but so also do the free organic radicals producing homogeneous combustion. The effect is supplementary to the inhibitory action of the metal alkyl on the oxidation of the aldehydes produced by partial oxidation of hydrocarbons. A. A. Eldridge.

Active charcoal industry and the oxidation of phosphorus. E. Urbain (Chim. et Ind., 1927, **17**, 536—540; cf. B., 1926, 1003).—The gases produced in the manufacture of active charcoal by the carbonisation of cellulosic material mixed with phosphoric acid contain carbon monoxide and phosphorus vapour. It is not economic to burn these directly; the constituents therefore are separated, the carbon monoxide being used for any suitable purpose, and the phosphorus being oxidised with steam to give phosphoric acid and hydrogen. The latter reaction proceeds conveniently at 400—500° in the presence of active charcoal, which absorbs the phosphoric acid produced; the speed of the reaction is increased by the addition of a trace of a halogen acid. This process is distinct from that described in the patents of Liljenroth (B., 1925, 242; 1926, 320). Since neither the oxidation of carbon monoxide by steam nor the deposition of carbon ($2CO \rightarrow CO_2 + C$) proceeds appreciably under the conditions of the reaction, the mixture of carbon monoxide and phosphorus vapour may be submitted directly to the oxidation process, the resulting gas then corresponding in composition to $CO + H_2$. The disadvantage of using a shaft furnace for the production of phosphorus, namely the low concentration of the latter in the exit gases, can be overcome by absorbing the phosphorus in active charcoal. The slag from this process can be used for cement manufacture. A. B. Manning.

Briquette binder containing residues from alcohol manufacture. W. C. Moore and H. A. Myers (Ind. Eng. Chem., 1927, **19**, 147—149).—Molasses residue from alcohol manufacture, evaporated down to *d* 1·3, will form emulsions with petroleum-pitch asphalt if dust from incinerator flue gases or, better, calcium chloride is used as an emulsifying agent. These emulsions may be employed as a binder in the manufacture of briquettes from anthracite culm. The addition of sulphur to the residue gives a briquette less liable to leaching of the binder, though it does not increase the strength of the briquette. A suitable binder has the formula, in pts. by wt.; concentrated molasses residue, 28; anhydrous sodium carbonate, 9; anhydrous calcium chloride, 10; sulphur, 9; asphalt (60°), 28; and water, 14. The solids are ground and mixed with the residue, added to the asphalt at a temperature somewhat above its m.p., and boiling water is added. The briquettes are generally baked at 315° for 30 min. H. Moore.

Sulphur in coal and coke. W. A. Selvig and A. C. Fieldner (Ind. Eng. Chem., 1927, **19**, 729—733).—From an examination of sixteen samples of coal and coke ranging in sulphur content from about 0·5 to 17% by the Eschka, bomb-washing, and sodium peroxide fusion methods, it is concluded that the last two methods yield results comparable with the standard Eschka method. The three methods are described in detail. The bomb-washing method requires slow and uniform release of the gases after combustion, and thorough washing of the bomb, including valve openings etc. C. O. Harvey.

Design and operation of horizontal retort settings [for producer-gas]. T. H. Goldsmith (Gas J., 1927, **178**, 375—380).—In producer construction, it is recommended that the front wall should slope towards the grate in order to reduce clinker formation above the top steps, whilst for efficient combustion the velocity of producer gas and secondary air entering the combustion chamber should be 9—10 ft./sec. A regenerator is described which allows a uniform speed of travel of the gases throughout its length, a condition necessary for efficient working. Aluminium-coated steel tubes are suggested for use in regenerators at temperatures below 900°. H. D. Greenwood.

Removal of tar fog from coal gas. N. A. Ross (Gas World, 1927, **86**, Coking Sect., 53—55).—The principles of the different methods of removing tar fog are briefly outlined, and a number of arrangements suitable for the recovery of tar fog on the laboratory scale are described. For small quantities, the most efficient combination is a glass-wool extractor, followed by a cotton-wool extractor. A glass-wool diaphragm of 6—8 mm. diam. dealt successfully with the gases (730 litres) from 5 lb. of coal carbonised in a silica retort, collecting 28—30 c.c. of tar. For larger quantities of tar fog, a laboratory modification of the Pelouze and Auduin extractor, followed, if necessary, by a plug of cotton-wool, is recommended. A. B. Manning.

Burning gases in nitrous oxide. H. B. Dixon and W. F. Higgins (Fuel, 1927, **6**, 232—235; cf. A., 1926, 689).—Flames of hydrogen, methane, ethylene, and propylene show a marked increase in size and luminosity, together with the appearance of characteristically coloured zones when the surrounding atmosphere of air is replaced by nitrous oxide. Carbon is deposited from the flame of propylene burning in nitrous oxide, and gradually forms a sheath round the luminous zone. The ignition temperatures of hydrogen, ethylene, and propylene in nitrous oxide have been determined at pressures from 150 to 1000 mm. and times of lag from 0·5 to 5 sec., by the concentric tube method; the values for the three gases at 760 mm. and a lag of 0·5 sec. were 597°, 592°, and 564° respectively. The curves showing the variation of the ignition temperature with pressure exhibit maxima, and are similar to those for ignition in oxygen, the temperatures at the corresponding pressures, however, being always lower in nitrous oxide. Nitrous

oxide alone undergoes no appreciable spontaneous decomposition at temperatures below 700°.

A. B. MANNING.

Occurrence of synthol in the process of petroleum synthesis under pressure, and the synthesis from water-gas of paraffin hydrocarbons of high molecular weight. F. FISCHER and H. TROPSCH (Brennstoff-Chem., 1927, **8**, 165—167).—When water-gas is passed over a catalyst comprising finely-divided iron and copper, in the ratio 4 : 1, at 280° and 10—15 atm., a mixture of hydrocarbons and oxygenated compounds is formed. As long as the catalyst is fresh, oily and aqueous products predominate, but after prolonged action a greater quantity of solid paraffins is produced. These adhere to the catalyst, and are extracted with benzene or xylene. After recrystallising, they are colourless and melt in the neighbourhood of 110°. Their mol. wt. is about 1000, and they are of the order of $C_{70}H_{144}$.

W. T. K. BRAUNHOLTZ.

Action of sulphur monochloride on petroleum hydrocarbons. E. LORAND (Ind. Eng. Chem., 1927, **19**, 733—734).—Sulphur monochloride reacts vigorously with unsaturated compounds. With normal paraffin hydrocarbons the reaction is slow even upon heating, but side-chain paraffin hydrocarbons react more readily. The reaction is accompanied by the formation of polymerisation products and the evolution of hydrogen chloride, and it is suggested that, in the case of paraffins, ethylenic linkings are formed, whilst ethylenic compounds yield two hydrogen atoms to form hydrogen chloride with the formation of a triple linking. The reaction might serve as a test for unsaturated compounds.

C. O. HARVEY.

Ethyl sulphate in the examination of hydrocarbon oils. J. N. TAYLOR (Ind. Eng. Chem., 1927, **19**, 76—77).—The non-poisonous ethyl sulphate is used to differentiate paraffins and naphthenes from aromatic and unsaturated hydrocarbons, the procedure, though less simple than that with methyl sulphate, being more reliable. 5 c.c. each of mixtures of white oil and benzene to be tested are placed with 5 c.c. of white oil in Egertz tubes, and 16 c.c. of ethyl sulphate are added, the amount of residue being observed at 25°. The results are comparable with the sulphonation residue, with a maximum error of 12%. Dips from low-temperature coal-tars and petroleum oils were tested with concordant results.

H. MOORE.

Changes in properties of four unblended mineral oils produced by prolonged treatment with ozone. M. V. DOVER and J. H. CROMWELL (Ind. Eng. Chem., 1927, **19**, 94—96).—Two oils of paraffin and two of asphaltic base were examined before and after treatment with ozone from a laboratory ozoniser at room temperature for 8 hrs., and at 27° for 16 hrs. Lowering of interfacial tension has no direct relation to lowering of the static coefficient of friction, and in some cases the viscosity was inversely proportional to the coefficient of static friction. A more unsaturated oil is generally a more efficient lubricant than a more saturated one.

H. MOORE.

Composition of gasoline as indicated by close fractionation. J. B. HILL, L. M. HENDERSON, and S. W. FERRIS (Ind. Eng. Chem., 1927, **19**, 128—130).—Four gasolines of various origins were fractionated, and curves prepared showing the relation between the sp. gr. and b.p. of the fractions. Fractionation was conducted in a 5-ft. Snyder column with 47 sections run adiabatically, with a controlled reflux of 5 : 1 at the top, fractions of 2·5% being taken, and also in a 16-ft. column packed with glass rings with electric insulation and controlled reflux, with cuts of 1·2° range. The curves showed regular peaks and troughs, the peaks (high sp. gr. and n values and low aniline point) showing a concentration of the more naphthenic hydrocarbons, and the troughs a higher concentration of the paraffins. All samples have their peaks and troughs in the same places. Re-fractionation caused the troughs to be lowered and the peaks raised.

H. MOORE.

Measurement of knock characteristics of gasoline in terms of a standard fuel. G. EDGAR (Ind. Eng. Chem., 1927, **19**, 145—146).—A standard fuel for the comparison of knocking tendencies of gasolines is made by mixing pure n-heptane, obtained by the distillation of Jeffrey pine oil with a new octane, which is β-*methyl*-β-iso*butylpropane*. A method for preparing this octane from *tert*.-butyl alcohol is given. It has b.p. 99·3°, d 0·6914, viscosity 0·00543 at 18·5°, and n_D^{20} 1·3921. It has knocking tendencies less than those of any commercial gasoline, whilst n-heptane has greater tendency to knock than commercial gasoline. By mixtures varying from 40 to 60% of the two substances respectively, any gasoline may be matched as regards knocking tendencies.

H. MOORE.

Bowie-Gavin process for shales. BOWIE.—See III.

Pure sulphur from gas sulphur. GLUUD, SCHÖNFELDER, and RIESE.—See VII.

"Spalling" in gas retorts. REES.—See VIII.

Flames of atomic hydrogen. LANGMUIR.—See XI.

ERRATUM.—B., 1927, 435, col. 1, line 33, *for* "G. VARGA" *read* "J. VARGA."

PATENTS.

Drying coal and other fuel, or other granular or powdered material. INTERNAT. COMBUSTION, LTD., and F. H. ROSENCRANTS (E.P. 265,374, 2.2.26. Addn. to E.P. 234,327; B., 1925, 577).—A central drum rotatably mounted on a vertical shaft carries on lugs on its outside a series of superimposed annular plates. These plates have a series of steam-heating pipes cast within them, the steam and drainage connexions being made by two common header pipes carried on the frame of the rotating drum. The material to be dried falls on to the uppermost plate, where it remains for almost one complete revolution of the drum, after which it is scraped on to the next lower plate, and so on to the bottom. Preheated air is admitted to one side of the casing which encloses the rotating member, and, after passage over the material, leaves the casing on the opposite side.

S. PEXTON.

Coking coal. H. G. C. FAIRWEATHER. From URBANA COKE CORP. (E.P. 269,994, 1.2.26).—A heated fluid is passed through the coal until its temperature is just below the temperature at which it becomes plastic.

The coal is then fused throughout its mass and finally raised to a coking temperature. It may be necessary to maintain the fused condition of the mass in order to permit the escape of volatile matter, the coking temperature being about 750°. The hydrocarbon vapours may be either assisted from the coal by the admission of steam to the carbonisation chamber before the temperature is raised to the coking temperature, or removed merely by the maintenance of a requisite temperature. During the initial heating stages the coal mass may be agitated or remain quiescent. R. A. A. Taylor.

Process and apparatus for coking coal. H. G. C. Fairweather. From Urbana Coke Corp. (E.P. 270,003, 2.1.26).—The coal is heated in two stages—(*a*) at a temperature just below the critical temperature, and (*b*) at a temperature sufficient to complete the coking process. During the first stage only, the fuel is agitated or tumbled, the second being completed rapidly by heating quickly to the temperature of evolution of the hydrocarbon vapours; or the fuel may be heated slowly to the temperature for evolving the vapours. The second stage may be carried out by pouring the coal into a retort previously raised to the requisite temperature. A reservoir may intervene between the first stage and the retort for the second stage, the rate of discharge from it being so regulated as to allow the coal to reach the desired temperature.

R. A. A. Taylor.

Manufacture of briquettes. S. F. Walton (U.S.P. 1,618,248—9, 22.2.27. Appl., 3.12.25).—(a) Wet-ground coal (9 pts.) of predetermined moisture content is mixed with an oil binder (1 pt.) and briquetted. (b) Coal is bonded with an uncarbonised water-soluble sulphide and waterproofed with a cellulosic agent.

S. Pexton.

Manufacture of activated carbon. R. Threlfall (E.P. 270,505, 4.6.26).—Charcoal is heated with sulphur or is exposed at a high temperature to sulphur vapour; the charcoal is subsequently treated to remove the sulphur from it. The charcoal product is washed with an acid, then with water, and finally dried by heat alone or at a high temperature in an atmosphere of hydrogen.

R. A. A. Taylor.

Treatment of wet peat with porous materials. E. von Niessen (G.P. 432,712, 5.10.19).—To remove water from peat the latter is subdivided and mixed with its own weight or more of an incombustible, absorbent material, *e.g.*, pumice, porous pot, etc., in a powdered or small-grained form, and, after the distribution of the water between the peat and the added material has reached an equilibrium, the water is removed by evaporation, the added material being finally separated from the peat by shaking or sieving. A very considerable drying of the peat is thus attained without the use of pressure or other mechanical means.

A. B. Manning.

Production of water-gas and coal-gas from bituminous fuel with simultaneous recovery of ammonia and tar by-products. Power-Gas Corp., Ltd., and N. E. Rambush (E.P. 270,009, 3.2.26).—In a process whereby bituminous fuel is gasified in a shaft generator under conditions which permit of the recovery of by-products, the mixture of coal-gas and water-gas is enriched by carburation by means which do not interfere with the recovery of the by-products. The down-run is effected only through the generator zone, and the gas so made is carburetted; the up-run is effected both through the generator and distillation zones, and the gaseous products treated for recovery of by-products. Two or more generators may work in conjunction with one common carburettor, which may be heated by waste blow-gases successively from each generator. The blow-gases may also be used to raise steam for aiding the distillation of the fuel.

R. A. A. Taylor.

Manufacture of water-gas. N. J. Bowater (E.P. 270,948, 28.9., 1.10., and 9.11.26).—Pulverised fuel is gasified by steam so highly superheated that the formation of carbon dioxide is practically precluded. The heat of combustion during the blow period is returned to the process in the sensible heat of the superheated steam; the sensible heat of the water-gas and surplus steam preheats air for the blow period. The steam is raised in waste-heat boilers by the sensible heat of the gases produced in both blow and make periods. The temperature in the generator is not, during the make period, allowed to fall below that requisite for the immediate combustion of the fuel in the run period. Further heat may be introduced from the generator lining to counter the endothermicity of the water-gas reaction. Completeness of the reaction may be assured by turbulent and counter-flow of the reacting phases, the time and intimacy of contact being so regulated as to produce a product equivalent to the equilibrium product of a lower temperature. R. A. A. Taylor.

Manufacture of carburetted water-gas. A. C. Klein, Assr. to Stone & Webster, Inc. (U.S.P. 1,630,300—1, 31.5.27. Appl., [a], 26.2.21, [b], 18.11.21).—The plant comprises a generator, a carburettor, and a superheater. (a) The fuel bed is heated by an air-blast; the blast gases are excluded from the carburettor, which is simultaneously treated with air to burn out any carbon deposit from the previous gas-making cycle and to heat the carburettor. Steam is introduced through the fuel bed, and the water-gas is then heated and enriched with oil in the carburettor. (b) Air-blasting of fuel occurs in the generator, and the hot blast-gases are passed through the carburettor; steam is then passed into the heated fuel in the generator to form water-gas, which is passed into and enriched in the carburettor; and, finally, the carbon deposit in the carburettor is burned with air, the products of combustion from the generator being excluded. The oil used for carburation is such as will leave a substantial carbon residue. R. A. A. Taylor.

Eliminating carbon monoxide in the exhaust of internal-combustion engines. M. von Wertaur and S. Caplan (E.P. 270,541, 26.8.26. Conv., 19.6.26).—Attached to the exhaust pipe is a shell containing absorbent baffles placed transversely to the path of the gases. The baffles are holders filled with gas-absorbing material, together with a reagent capable of oxidising the carbon monoxide. R. A. A. Taylor.

Separation of tar and ammonia from fuel gases. W. C. Holmes & Co., Ltd., J. Parker, and D. M.

Henshaw (E.P. 270,852, 5.3.26).—The condensing chamber is situated above and in communication with the vessel into which the condensate is received, both vessels being provided with cooling means. The receiver has outlets at different levels in order that the aqueous and tarry extracts may be drawn off separately. Alternatively, the height of the means for drawing off tar may be adjustable, so that the level of the tar may be varied in the space in which the liquor collects and the liquor drawn off through an outlet provided.

R. A. A. Taylor.

Extraction of [aromatic] hydrocarbons from [coal] gas. A. P. Bjerregaard, Assr. to Doherty Research Co. (U.S.P. 1,624,206, 12.4.27. Appl., 17.1.22).—Coal-gas, freed from tar, ammonia, and pyridine, is washed with mineral oil at about 90° to separate phenolic compounds, and then with mineral oil at ordinary temperature to separate benzene and toluene. Due to the absence of phenolic compounds, no frothing occurs in the subsequent steam-distillation of the mineral oil containing aromatic hydrocarbons. T. S. Wheeler.

Gas-purifying apparatus. Deutsche Erdöl-A.-G. and K. Bomcke (E.P. 271,018, 8.3.27).—The dry removal of dust from gas is effected by means of a dust-separating chamber, leading to and from which are several restricted passages through which the gas passes in a horizontal direction at a uniform velocity. These equalising devices consist of small objects, *e.g.*, glass or aluminium beads, shingle, etc., arranged between wire grids, which are so arranged as to offer a minimum of resistance, and are suitably spaced from each other. Each equalising device has a filling port at the top and an emptying port at the bottom, and from one device to the other plates may stretch, the gas passing between them, and the dust collecting upon ribs, waves, etc., which are arranged in staggered relation with each other.

R. A. A. Taylor.

Process and apparatus for distilling small samples of coal. R. Geipert (E.P. 267,082, 22.9.26. Conv., 4.3.26).—The experimental retort is already heated at the temperature of the test. A sample of coal is introduced in the form of a moulded block or of a number of blocks of regular shape and size, which can be strung together on a wire. The blocks thus occupy the same space in the retort, and accurate comparative tests can be made by successive distillations with similar blocks. The retort is enclosed in an externally-heated heat accumulator, or the retort itself forms a heat accumulator. A gas-tight, refractory tube having one end within the retort and the other outside may be used to introduce the coal and to lead off the gaseous products of distillation. The length of the tube inside the retort should be at least twice the internal diameter of the tube. The tube should be vertical, and have at its outer end a stopper through which passes the wire holding the blocks, the wire being held by a clip so that it can be released for dropping the blocks down the tube. R. A. A. Taylor.

Preparation of synthetic petroleum hydrocarbons. E. Legé (F.P. 602,007, 17.11.24).—Coal, alkali or alkaline-earth salts, alkali or alkaline-earth carbonates, and water are allowed to interact under pressure and at the necessary temperature. After being brought into contact with alkali or alkaline-earth chlorides in a further working chamber, the gas mixture produced is allowed to interact at 400—700° in the presence of iron and other metals or metallic oxides as catalysts, then passed over iron or copper at 300—400°, and the products condensed in the solid or liquid form. The residual gases are used for heating purposes. A. B. Manning.

Fuel for internal-combustion engines. E. G. E. Meyer (E.P. 270,772, 12.11.25).—Hydrocarbon distillates having initial b.p. of 70—80° and final b.p. of 175—225°, and of which not more than 5% distils below 80°, whilst only a relatively small proportion boils above 160°, are rendered suitable for use in internal-combustion engines by the addition of up to 5% of commercial ether, together with a small proportion of a substance adapted to retard the spontaneous evaporation of the ether (*e.g.*, phenol, benzoic acid, ammonia). C. O. Harvey.

Regeneration of lubricating oils. H. Bensmann (E.P. 268,284, 16.10.26. Conv., 25.3.26).—In the recovery of used motor oils by removal of colloidally dispersed particles of soot and graphite, and of decomposition (cracking) products from the oil, it is treated at 40—70° with substances which exert a polymerising action on the decomposition products (*e.g.*, stannic chloride, aluminium chloride, sulphuric or phosphoric acid, etc.), whereby asphaltic substances are precipitated and carry down with them the colloidal carbon. The oil passes from a settling tank to a filter containing hydrated aluminium magnesium silicate, which adsorbs organic acids and tarry products. C. O. Harvey.

Manufacture of a lubricant. A. E. Becker, Assr. to Standard Development Co. (U.S.P. 1,628,646, 17.5.27. Appl., 21.11.24).—Soap is added to a hydrocarbon oil having lubricating properties, the mixture being then heated at between 176° and the b.p. of the oil. H. Royal-Dawson.

Lubricant. H. H. Willock, S. J. Caplan, and J. E. Babb, Assrs. to Waverly Oil Works Co. (U.S.P. 1,625,969, 26.4.27. Appl., 30.8.21. Renewed 3.9.26).—A lubricant for use at high temperatures comprises petroleum oil, aluminium oleate, oleic acid, and a small proportion of a calcium soap. T. S. Wheeler.

Treating hydrocarbon oils. W. L. Coultas, jun. (U.S.P. 1,628,532, 10.5.27. Appl., 12.7.20).—The temperature of the oil is gradually raised by passage through heating coils contained in a series of furnaces, and free hydrogen at a temperature above 760° is introduced into each coil at the point where the oil has reached the vaporisation state. The vapours are fractionated and the high-boiling condensate from each column is re-treated in a second furnace. C. O. Harvey.

Distilling and cracking hydrocarbon oils. S. L. Tingley (U.S.P. 1,627,937—8, 10.5.27. Appl., [A], 12.10.23, [B], 28.12.23).—(A) The oil is heated in a water-jacketed coil contained in a furnace, the water in the jacket flowing in the same direction as the oil, and protecting the latter from direct heat from the furnace. The oil is thus progressively heated and vaporised, and the vapour superheated at the same progressively increasing temperatures as those of the water and the superheated

steam formed. (B) The coil is divided into horizontal sections, and means are provided for the automatic control of the oil level. Prior to heat-treatment, the oil is subjected to radioactive emanations. C. O. Harvey.

Absorption and distillation of hydrocarbons. D. L. Newton (U.S.P. 1,628,055, 10.5.27. Appl., 9.12.25).—Each of a series of superimposed chambers carries a partition holding an absorbing liquid medium, and the gases pass upwards from one chamber to another, the rate of flow of gas into each chamber being controlled independently of the rate of flow into other chambers. C. O. Harvey.

[Cracking] treatment of hydrocarbon oils. C. P. Dubbs, Assr. to Universal Oil Products Co. (U.S.P. 1,628,127, 10.5.27. Appl., 17.12.21).—Oil under pressure passes through a heating zone, and thence to a distilling zone, whence the vapours are withdrawn. The oil supply to the heating zone is controlled by the liquid oil level in the distilling zone, and the oil in the former is kept in circulation. C. O. Harvey.

Treatment of petroleum residue. J. C. Morrell, Assr. to Universal Oil Products Co. (U.S.P. 1,618,669, 22.2.27. Appl., 28.6.26).—The liquid medium is separated from cracked petroleum residue containing suspended coke-like particles, and the residue is rendered homogeneous, so that the suspended particles are stabilised in the mixture with the residual oil, and the product assumes a creamy consistency. S. Pexton.

Extraction and decomposition of bitumen. Deutsche Erdöl A.-G. (G.P. 437,210, 12.11.25).—Bituminous materials, in particular brown coals, are extracted with liquid sulphur dioxide at moderately high temperatures. At 70—80° the whole of the montan wax can be brought into solution, whilst at lower temperatures only the resinous materials can be separated. A. B. Manning.

Recovery of volatile solvents [from gas]. R. Oertel, Assr. to Metallbank u. Metallurgische Ges. A.-G. (U.S.P. 1,631,052, 31.5.27. Appl., 8.6.26. Conv., 7.4.24).—See E.P. 266,145; B., 1927, 319.

Purifying gases [from iron carbonyl]. J. Jannek, Assr. to I. G. Farbenind. A.-G. (U.S.P. 1,631,823, 7.6.27. Appl., 8.6.25. Conv., 16.6.24).—See E.P. 247,050; B., 1926, 351.

Delivery of pulverised fuel to furnaces, kilns, etc. H. A. Procter (E.P. 265,434, 12.5.26).

Apparatus for cooling coke and the like. G. M. Gill (E.P. 265,337, 10.12.25, and 28.5.26).

Apparatus for receiving coke discharged from retorts used in gas manufacture. F. J. Harrisson and J. W. Drake (E.P. 271,260, 16.6.26).

Controlling gas burners for furnaces. J. Keith & Blackman Co., Ltd., and G. Keith (E.P. 270,786, 5.2. and 19.11.26).

Utilisation of ultra-violet rays for supporting combustion in heat engines and the like. H. A. Gill. From Studienges. für Wirtschaft & Ind.m.b.H. (E.P. 269,808, 9.11.26).

Ammonium sulphate and bisulphate, and sulphur (E.P. 250,990).—See VII.

Resins from hydrocarbons (U.S.P. 1,627,054).—See XIII.

III.—TAR AND TAR PRODUCTS.

The Bowie-Gavin process and its application to the cracking of tars and heavy oils, and to the recovery of oil from sands or shales. C. P. Bowie (U.S. Bur. Mines, Tech. Paper 370, 1926. 42 pp.).—The possibility of cracking tars, heavy oils, etc. without the formation of excessive carbon deposits in the apparatus is claimed. The oil is mixed with varying proportions of inert material such as shale, sawdust, etc. which retains the greater part of the carbon formed. Laboratory experiments showed that on heating heavy oils, tar, etc. with finely-ground shale in a small retort at about 400°, a yield of 75—85% of oil of *d* 0·934 was obtained, the loss consisting of fixed gas and carbon. Preliminary work indicated that on a commercial scale it would be necessary to feed the oil-inert mixture and to extract the inert residue continuously, to maintain the mixture in thin layers to prevent over-heating, and to remove the vapour and any condensate immediately from the retort. A continuous semi-commercial apparatus is described, the throughput being 12 barrels of oil per day. The base of the retort consists of a plate composed of a number of cast-iron hollow segments with tongued and grooved joints, arranged in three concentric rings, the whole being 8 ft. in diameter. Supported on this plate is a wrought-iron cone fitted with a feed pipe and a vapour outlet pipe. Any oil refluxing in the cone is trapped and drained to the vapour outlet. The oil and inert material are mixed externally and, after preheating, fed on to the plate, where the mixture is kept in motion by rabble arms rotated by a central shaft passing through the apex of the cone. The spent material is forced to the periphery of the plate by the rotation of the arms, and falls into a pocket cast in one of the plates, from which it is removed by a spiral conveyor. The retort is heated by hot gases passing through the hollow segments of the plate from the periphery to a central flue below the apparatus. The following results are typical of those obtained from cracking Casmalia crude oil (*d* 0·9935). Oil used, 65 gals.; shale added, 590 lb.; oil recovered, 58 gals. (*d* 0·934) = 81% recovery; gas produced, 2130 cub. ft., calorific value 741—1203 B.Th.U./cub. ft. On distillation, the cracked oil gives gasoline (0—220°) 17%, kerosene (220—275°) 15·5%. The viscosity of the residue above this temperature is such as to render it suitable for fuel purposes. H. D. Greenwood.

Viscosity curve of coal tar and the question of its mathematical validity. H. Mallison and F. Soltau (Brennstoff-Chem., 1927, **8**, 169—173; cf. Spiers, J.S.C.I., 1926, **45**, 396 T).—Results obtained with two gasworks tars indicate the impossibility of determining the viscosity at 25° from observations at only two other temperatures, although Spiers' formula affords information if a greater number of different observations are made. In practice, however, a few

viscosity determinations at higher and lower temperatures are sufficient, without the introduction of mathematical formulæ, which cannot be expected to be generally valid for a mixture of the complexity of coal tar. W. T. K. Braunholtz.

Tar fog from gas. Ross.—See II.

Patents.

Tar preparations. Prodorite S.A. (Swiss P. 113,922, 25.2.25).—Tar is treated with a solvent, *e.g.*, "gasöl," heated, with stirring, at 300°, allowed to settle, the solution of heavy tar oils drawn off, and any solvent still remaining in the residue removed by distillation. The residue can be used as cement in the construction of concrete vessels resistant to hot water. A. B. Manning.

Manufacture of fluorescent oil. L. Lilienfeld (U.S.P. 1,625,415, 19.4.27. Appl., 23.7.24. Renewed 30.12.26).—See E.P. 163,271; B., 1922, 50 A.

Tar from gas (E.P. 270,009 and 270, 852).—See II.

Fractional distillation (Austr.P. 104,137).—See II.

Wood preservative (G.P. 435,146).—See IX.

IV.—DYESTUFFS AND INTERMEDIATES.

Patents.

Manufacture of anthraquinone derivatives. O. Y. Imray. From I. G. Farbenind. A.-G. (E.P. 270,840, 25.2.26).—When anthraquinone or one of its homologues is heated below 180° with an arylamine salt having a free *p*-position, diaminodiarylanthrones of the type, $CO<^{C_6H_4}_{C_6H_4}>C(C_6H_4\cdot NH_2)_2$, are obtained. At higher temperatures *meso-pp'*-diaminodiarylanthracenes are formed. 9 : 9-*Bis*-p-*aminophenylanthrone*, m.p. 298°, and 9 : 9-*bis*-(4'-*amino*-3'-*methylphenyl*)*anthrone*, m.p. 255°, are obtained from aniline and *o*-toluidine hydrochlorides, respectively, by heating with anthraquinone at 175° (oil-bath temperature). At 185—190° the products are 9 : 10-*bis*-p-*aminophenylanthracene*, m.p. 300°, and 9 : 10-*bis*-(4'-*amino*-3'-*methylphenyl*)*anthracene*, m.p. above 300°. C. Hollins.

Manufacture of anthraquinone derivatives. British Dyestuffs Corp., A. Shepherdson, W. W. Tatum, and H. M. Bunbury (E.P. 270,778, 11.1.26).—Acylated 1 : 4-diaminoanthraquinones (*e.g.*, mono- and di-benzoyl derivatives and the dioxamic acid) are obtained in a single operation by treating leuco-1 : 4-diaminoanthraquinone with acylating agents in nitrobenzene or nitrotoluene, the solvent acting also as an oxidant (cf. E.P. 271,023; following abstract). C. Hollins.

Manufacture of anthraquinone intermediates. British Dyestuffs Corp., A. Shepherdson, W. W. Tatum, and H. M. Bunbury (E.P. 271,023, 11.1.26).—Halogenated 1 : 4-diaminoanthraquinones (*e.g.*, 2 : 3-dichloro-1 : 4-diaminoanthraquinone) are obtained in a single operation by treating leuco-1 : 4-diaminoanthraquinone with halogenating agents in an inert or oxidising solvent (*e.g.*, dichlorobenzene or nitrobenzene). C. Hollins.

Manufacture of anthraquinone intermediates. British Dyestuffs Corp., and W. W. Tatum (E.P. 270,779, 11.1.26).—Leuco-1 : 4-diaminoanthraquinones (*e.g.*, leuco-derivatives of 1 : 4-diamino-, 1 : 4-dimethylamino-, 8-amino-1 : 4-dimethylamino-5-hydroxy-, 1 : 4-dimethylamino-5-hydroxy-, and 1 : 4-dimethylamino-5 : 6-dihydroxy-anthraquinones) are oxidised by heating a salt (*e.g.*, the hydrochloride) of the leuco-base in an oxidising solvent (*e.g.*, nitrobenzene) or in an inert solvent with an added oxidant. C. Hollins.

Manufacture and application of new dyes. Soc. Chem. Ind. in Basle (Ges. für Chem. Ind. in Basel) (E.P. 258,854, 8.9.26. Conv., 24.9.25).—Dyes, especially suitable for chroming on wool, are obtained by the action of formaldehyde and sodium bisulphite on the azo dyes formed by coupling diazotised 1-amino-2-naphthol-4-sulphonic acid with α- or β-naphthol. Wool is dyed from a formic or acetic acid bath, and on chroming gives deep black shades. C. Hollins.

Manufacture of alkylnaphthalenes chlorinated in the nucleus. I. G. Farbenind. A.-G. (E.P. 263,844, 29.12.26. Conv., 29.12.25).—Alkylnaphthalenes are chlorinated in the presence or absence of a solvent such as ether or carbon tetrachloride by treatment with sulphuryl chloride. 1-*Chloro*-4-*methylnaphthalene*, b.p. 278—283°, 1-*chloro*-2-*methylnaphthalene*, b.p. 273—275°, a *monochloro*-2 : 6-*dimethylnaphthalene*, m.p. 39°, b.p. 293—297°, and a *dichloro*-2 : 6-*dimethylnaphthalene*, m.p. 135°, b.p. 328—333°, are described. C. Hollins.

New azo dyes and process of dyeing cellulose acetate. British Dyestuffs Corp., J. Baddiley, and J. Hill (E.P. 270,428, 12.2.26).—Brown shades fast to light and washing are obtained on cellulose acetate materials by the use of insoluble disazo dyes of the type: nitroarylamine → amine → amine of the benzene series. Suitable first components are *m*- and *p*-nitroanilines, 2 : 4-dinitroaniline, picramic acid, 4 : 5-dinitro-α-naphthylamine, etc. The middle component may be aniline, *m*-toluidine, cresidine, α-naphthylamine, 1-amino-2-naphthyl ethyl ether, etc. Suitable end components are *m*-phenylenediamine, *m*-toluidine, cresidine, and the like. Sulphonic groups must be absent. The dyeing may be done from an aqueous suspension in the presence of a dispersing agent. C. Hollins.

Manufacture of new azo dyes and process of dyeing. British Dyestuffs Corp., J. Baddiley, P. Chorley, and R. Brightman (E.P. 270,446, 24.2, and 8.4.26).—Azo dyes obtained by coupling diazo compounds, excepting diazotised aminonaphthols and their derivatives, with 8 : 8'-dihydroxy-2 : 2'-dinaphthylamine-6 : 6'-disulphonic or -3 : 3' : 6 : 6'-tetrasulphonic acid ("di-γ-acid" or "di-2R-acid") give level dyeings on viscose and other regenerated cellulose silks. In suitable cases the dye may be diazotised on the fibre and developed, or may be coupled on the fibre with diazo components. Thirteen examples are given, the shades being brown to violet, blue, and black. C. Hollins.

Separation of aromatic amines (E.P. 270,930).—See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Effect of partial hydrolysis on the alkali solubility of wood. L. F. Hawley and W. G. Campbell (Ind. Eng. Chem., 1927, **19**, 742—744).—Sitka spruce

sawdust was treated with varying concentrations of hydrochloric acid (0·5—15·0%) for 6 hrs. at 100°, and the product analysed both before and after treatment with 1% sodium hydroxide solution at 100° for 1 hr. Whilst the original wood could be completely accounted for by determinations of cellulose, lignin, "pentosans not in cellulose" (*i.e.*, total pentosans — pentosans in crude cellulose) and water-soluble (total 102%), this was not so in the case of the partially hydrolysed products (total 93—98%). The latter probably contain a cellulose degradation product which does not appear in the above determinations, and is not present in the original wood. The solubility of cellulose in 1% sodium hydroxide at 100° was considerably increased by acid hydrolysis, whilst that of lignin was only slightly affected. Very mild hydrolysis, which had little influence on cellulose, considerably increased the alkali-solubility of the pentosans present. The total alkali-solubility of partly decayed wood was much greater than that of wood which had been hydrolysed to the same extent, *i.e.*, had suffered equal loss in weight. W. J. POWELL.

Constants of cellulose pulp. Change with time of cooking. J. CHINTSCHIN (Zellstoff u. Papier, 1927, 7, 235).—Prolonged boiling of pulp results in decrease of its lignin content, suppression of the bromine number or chlorine factor, decrease in the quantity of active chlorine absorbed in bleaching, and increase in the amount of the fibre stained by Congo Red. The copper number (Schandrock and Häen-Low) increases, and correspondingly the resistance to baryta (which refers to increase in the quantity of cellulose decomposition products) decreases. Decrease of the total sulphur dioxide content of the cooking liquor apparently produces an increase in the pentosan content. Fluctuations in the α-cellulose content show no definite relation to the duration of boiling, concentration of the liquor being apparently a more important factor, but definite conclusions cannot be drawn owing to the insignificant differences in the amounts of α-cellulose. The duration of boiling exerts a proportionally smaller action on the decrease of the resin content, whilst the amount of ash is also decreased by prolonged cooking. B. P. RIDGE.

Pulping of pine wood by the sulphite process. C. G. SCHWALBE and K. BERNDT (Cellulosechem., 1927, 8, 66—68).—A criticism of Hägglund's conclusions (B., 1927, 294). The latter's own results show that pine wood after extraction with benzene or ether alone cannot be successfully pulped by the sulphite process, even under the conditions used in his sealed-tube experiments, which differed widely from those obtaining on the large scale in regard to such factors as time of boiling, means of agitation, acid concentration, maximum pressure, and size of chips. His conclusion that the age of the wood has no effect on the pulping properties is not valid for the same reason. W. J. POWELL.

Recovery of solvents. WIESENTHAL.—See I.

Nitrocellulose for lacquers. VON MÜHLENDAHL and SCHULZ.—See XIII.

Sulphite extract as tanning material. WALLACE and BOWKER.—See XV.

PATENTS.

Manufacture of artificial silk. F. C. NIEDERHAUSER and A. E. SUNDERLAND, Assrs. to VISCOSE CO. (U.S.P. 1,625,562, 19.4.27. Appl., 9.11.20).—In the manufacture of viscose silk, the addition of glue (2%) to the sulphuric acid setting bath restricts the deleterious action of the acid on the filament and inhibits the crystallisation of any salt left in the filament after removal from the bath. T. S. WHEELER.

Preparation of hollow artificial textile fibres from viscose. BRITISH ENKA ARTIFICAL SILK CO., LTD. From N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (E.P. 253,477, 5.3.26).—Solutions of low viscosity, such that when tested by the steel ball method the time of descent is 3 sec. or less (steel ball ⅛ in. diameter, length of tube 8 in.) are spun, without degasification, into a bath which precipitates the viscose quickly. The presence of a zinc salt (*e.g.*, 2% of zinc sulphate) in this bath is an advantage, and a finely-powdered solid (*e.g.*, pumice) may be mixed with the viscose before spinning in order to promote the formation of gas bubbles in the fibres. B. P. RIDGE.

Manufacture of plastics. L. LILIENFELD (U.S.P. 1,625,416, 19.4.27. Appl., 9.1.26. Conv., 15.5.20).—The products described in E.P. 163,271 (B., 1922, 50 A) are of value as plasticising agents for cellulose ethers. T. S. WHEELER.

Composition for use in making sulphate or kraft pulp. H. B. KIPPER (U.S.P. 1,629,393, 17.5.27. Appl., 26.11.24).—Sodium hydrogen sulphate mixed with sufficient sodium carbonate to give sodium sulphate, is added to black liquor from the sulphate-cellulose process before evaporation, to make up losses in sodium sulphide during the process. T. S. WHEELER.

Insulating material for electric condensers and the like. J. LAHOUSSE (E.P. 251,970, 27.4.26. Conv., 8.5.25).—A dielectric for condensers consists of paper etc. impregnated with compounds of the amide series solid at ordinary temperatures, but fusible at conveniently low temperatures, particularly urethanes, anilides, carbamides, their sulpho- and chloro-derivatives. By adding to the amide a small quantity of a second substance, the two being reciprocally soluble, a sufficient amount of eutectic (m.p. 30—70°) is formed at the end of cooling, acting as a binding agent, and thus avoiding the detrimental effects of a break in the dielectric mass on solidification. S. S. WOOLF.

Production of insulating paper containing phenol resins. C. A. HAANEN, Assr. to FELTON & GUILLEAUME CARLSWERK A.-G. (U.S.P. 1,630,424, 31.5.27. Appl., 27.11.26. Conv., 29.4.26).—Phenol resin solutions are added to fibrous pulp in which the water has been replaced by solvents for phenol resins, and the resin is precipitated in and on the fibres by the addition of water. The mass is worked up to form paper and pressed articles. S. S. WOOLF.

Treatment of fibres for spinning. L. UBBELOHDE (U.S.P. 1,629,241, 17.5.27. Appl., 26.6.25. Conv., 23.2.25).—See E.P. 254,357; B., 1926, 782.

Manufacture of artificial silk or the like. J. O. ZDANOWICH (U.S.P. 1,630,285, 31.5.27. Appl., 12.8.26. Conv., 6.5.25).—See E.P. 260,642; B., 1927, 103.

Machine for grinding paper pulp. K. G. V. Sörbom (E.P. 262,070, 27.10.26. Conv., 27.11.25).

Filtration (E.P. 270,461).—See I.

Dyeing cellulose acetate (E.P. 270,428).—See IV.

Dyeing process (E.P. 270,446).—See IV.

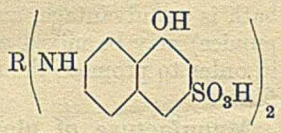
Ligno-tanning materials (U.S.P. 1,629,448).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Chlorine in bleach liquors. Hausner.—See VII.

Patents.

Dyeing artificial silk. British Dyestuffs Corp., J. Baddiley, P. Chorley, and R. Brightman (E.P. 270,883, 16.4. and 11.9.26).—Regenerated cellulose (*e.g.*, viscose) silks are dyed in level shades by azo dyes made by coupling suitable diazo components (1 or 2 mols.) with compounds of the type:



in which R represents a carbonyl or thiocarbonyl group or a straight chain made up of two or more methylene and/or carbonyl groups. Suitable diazo components are aniline, its homologues and derivatives (including carboxylic and sulphonic acids), naphthylamines and their sulphonic acids. 23 examples (reds, violets, and browns) are given, the coupling components being 8 : 8′-dihydroxy-2 : 2′-dinaphthylcarbamide-6 : 6′-disulphonic acid, the corresponding thiocarbamide, *NN′*-8 : 8′-dihydroxy-6 : 6′-disulpho-2 : 2′-dinaphthylethylenediamine (R = ·CH : CH·), *NN′*-8 : 8′-dihydroxy-6 : 6′-disulpho-2 : 2′-dinaphthylglycinamide (R = ·CO·CH_2·), 8 : 8′-dihydroxy-2 : 2′-dinaphthyloxamide-6 : 6′-disulphonic acid (R = ·CO·CO·), and *NN′*-8 : 8′-dihydroxy-6 : 6′-disulpho-2 : 2′-dinaphthyl-$\alpha\gamma$-diaminoacetone (R = ·CH_2·CO·CH_2·). In suitable cases the dyes may be developed or coupled on the fibre. C. Hollins.

Dyeing mixed textiles. I. G. Farbenind. A.-G., Assees. of Farbw. vorm. Meister, Lucius, & Brüning (E.P. 247,224, 8.2.26. Conv., 7.2.25).—Wool and silk in mixtures are equally dyed in level yellow to orange shades by pyrazolone dyes containing one or more carboxylic ester groups in the diazo or the coupling components or in both. Only such sulphonic or free carboxylic groups as are necessary to give the requisite solubility should be present. Examples are: *o*-chloroaniline → ethyl 1-*p*-sulphophenyl-5-pyrazolone-3-carboxylate; ethyl anthranilate → 1-*p*-sulphophenyl-3-methyl-5-pyrazolone; ethyl *m*-aminobenzoate → ethyl 1-*p*-sulphophenyl-5-pyrazolone-3-carboxylate. The preparation of ethyl 1-(6′-sulpho-2′ : 4′-dimethylphenyl)-5-pyrazolone-3-carboxylate from *m*-4-xylidine-5-sulphonic acid is described. C. Hollins.

Finishing, mercerising, and ornamenting textiles. E. Hausheer (E.P. 266,466, 24.8.25).—The material is treated wholly or locally with a solution containing 8—18% of sodium hydroxide as basis, with or without the addition of 0·1% or more of natural silk, other protein, or agar-agar, and/or up to 5% of dissolved cellulosic material which may also contain pigments or powdered metal. It is then subjected to the action of ammonia, carbamide or its derivatives, washed with acid and water, and dried. Alternatively, 2—10% of ammonia is added to the sodium hydroxide, the material treated therewith, mangled down to about twice its original weight, subjected in the tensioned state for several minutes to a temperature of − 7° to − 12°, and then treated with a 5% sulphuric acid solution, washed, and dried; or the material impregnated with sodium hydroxide is exposed to an ammonia atmosphere at a temperature of − 7° to − 15°. Before acid washing, the excess of the impregnating agent is removed by brushing, mangling, or scraping, treated yarns being passed through circular holes, corresponding to the thread diameter, in divided, hinged bars. Tensioning of the material may be regulated to produce various effects. B. P. Ridge.

Application of dyes (E.P. 258,854).—See IV.

Algin compounds (U.S.P. 1,625,301).—See XX.

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

Preparation of sulphuric acid from gypsum. H. Molitor (Chem.-Ztg., 1927, **51**, 329—332, 370—373).—Details are given of the important deposits of gypsum and anhydrite on the Moselle followed by an account of the various methods which have been proposed to utilise them as a source of sulphuric acid. The Bambach process effects the decomposition of gypsum at 1400° by means of the surface combustion of producer gas with air on the gypsum itself, which is crushed to small pieces, but not powdered. Alternatively, the air may be shut off at intervals so as to allow the alternate formation and decomposition of calcium sulphide. The principal difficulty is the tendency to slagging, due to the closeness of the decomposition temperature to the m.p. of calcium sulphate, and the risk of formation of basic sulphate or sulphite of lower m.p. Anhydrite is more difficult to work than gypsum. The quicklime produced is "dead-burnt." With care, however, the process can be carried out successfully. Basset heats gypsum in a rotary furnace with clay and coal, steam being blown in, producing cement as residue. The Bayer process, by which 2800 tons of sulphur trioxide are produced monthly at Leverkusen, uses powdered anhydrite and waste calcium sulphate. This is mixed with powdered shale and coke, and fired with powdered coal in rotary cement kilns, in a slightly oxidising atmosphere. The gas contains 6—7% SO_2. The B.A.S.F. have given large-scale trials to a process in which gypsum is allowed to interact with magnesia and carbon dioxide, whereby it is partly converted into magnesium sulphate. The latter is decomposed by heating with powdered coke at 450—500°. The calcium carbonate produced as a by-product is difficult to dispose of. This difficulty also arises with the process operated by the same firm on a large scale in which the magnesia is replaced by ammonia. In the Bambach process the carbon dioxide is replaced by sulphur dioxide. In this case the by-product is calcium sulphite, which is either sold as such, or converted into sulphate by interaction with sodium hydrogen sulphate, sulphur dioxide being also liberated. The main reaction may be carried out in towers or in a series of closed iron cylinders,

and is best performed at the b.p. of the solution. The second step is chiefly of interest as a means of utilising the acid content of nitre-cake and converting the residue into saleable saltcake. The Badische process for recovering elementary sulphur from gypsum has been abandoned owing to the poor yield. It has also been proposed to recover elementary sulphur by reducing gypsum with methane, or by reducing to calcium sulphide, decomposing with magnesium chloride solution, and proceeding as in the Claus process. C. Irwin.

Production of pure sulphur from gas sulphur. W. Gluud, R. Schönfelder, and W. Riese (Brennstoff-Chem., 1927, **8**, 168—169).—Crude sulphur, recovered from coal-gas by the method of the Ges. für Kohlentechnik (cf. B., 1927, 321) may be purified by distillation, although difficulties are met with when it contains much tarry matter or when large quantities have to be dealt with. An alternative method is to burn part of the sulphur, absorbing the sulphur dioxide in concentrated ammonia liquor, and to dissolve a further part of the crude sulphur in the ammonium sulphite solution so obtained. From the resulting ammonium thiosulphate solution pure sulphur is precipitated by adding sulphuric acid, and the liquor, after centrifuging, is worked up for ammonium sulphate. W. T. K. Braunholtz.

Determination of available chlorine in bleach-liquor. J. Hausner (Chem.-Ztg., 1927, **51**, 373—374).—A method intended for unskilled control. A stoppered measuring cylinder is filled with the sample to be tested up to a zero mark. A solution containing 0·85 g. of crystalline sodium thiosulphate and 0·14 g. of sodium indigodisulphonate per litre is run in until the colour changes to greenish-blue. The percentage of available chlorine is then read direct from a graduation. The standard solution, in concentrated form, remains unchanged for upwards of five months, and the method is reasonably accurate. C. Irwin.

Manganese interference in the *o*-tolidine test for available chlorine. E. S. Hopkins (Ind. Eng. Chem., 1927, **19**, 744—746).—In the presence of easily reducible salts of manganese and other metals the results of the *o*-tolidine test for free chlorine are unreliable, since a yellow colour similar to that due to chlorine is produced; *e.g.*, 0·01 pt. of potassium permanganate per million produces a colour intensity equal to that of 0·03 pt. of chlorine per million. Stable salts of manganese such as the sulphate or chloride at a concentration as high as 100 pts. per million do not produce a colour, but manganous hydroxide in the presence of air readily yields a positive result. The test is unsuitable for a determination of residual chlorine in water supplies containing manganese. W. J. Powell.

Charcoal and phosphorus. Urbain.—See II.

Barytes in rubber industry. Dawson and Hartshorne.—See XIV.

Patents.

Manufacture of sulphuric acid. H. Petersen (E.P. 244,764, 270,988—9, [a], 11.12.25, [b, c], 16.12.26. Conv., [a], 16.12.24, [b], 12.10.26).—(a) Gases containing sulphur dioxide are treated in reaction enclosures filled with packing material and irrigated with solutions of nitrosylsulphuric acid in high-grade sulphuric acid containing such an amount of nitrogen oxides that the sulphur dioxide is in excess. A fine jet of water is introduced in small quantity, so that the resulting nitrous sulphuric acid has the same nitrous content as the acid fed to the same reaction enclosure. The acid is denitrated in a tower in front of the system. (b) Excess of water in sulphurous gases is removed by treatment with acid (*d* 1·71) withdrawn from the circulating acid in a tower in front of the system, the resulting weak acid being returned to the plant instead of water for acid formation. (c) Sulphur dioxide gases containing excess of moisture are divided into two portions, one of which effects the denitration of the commercial acid by intimate contact of acid and gas in a tower packed with inert material in grains of not more than 40 mm. diameter, the other portion being introduced into the plant at points where water is necessary. W. G. Carey.

Sulphuric acid chambers. Packards & J. Fison (Thetford), Ltd., and R. T. Maudsley (E.P. 270,826, 17.2.26).—Externally water-cooled chambers of the Mills-Packard type are flexibly supported by a series of links pivoted at one end to a vertical lead lug burnt on to the chamber from top to bottom, and at the other on to brackets secured to the framework of the chamber, the brackets having adjustable eyebolts. W. G. Carey.

Distillation of hydrochloric acid. L. C. Drefahl, Assr. to Grasselli Chemical Co. (U.S.P. 1,628,829, 17.5.27. Appl., 5.4.23).—A continuous stream of crude hydrochloric acid of greater strength than the constant-boiling acid flows over a heated surface in a thin layer, the vapours being condensed and withdrawn; the rate of flow is regulated according to the rate of vaporisation. H. Royal-Dawson.

Manufacture of acetic acid. H. Dreyfus (E.P. 268,845, 7.11.25).—Methyl alcohol and carbon monoxide at atmospheric pressure are heated below 400—500°, and preferably at 200—350°, with catalysts capable of forming acetates which split off acetic acid at temperatures below 400—450°, and preferably at 200—300° (cf. E.P. 264,558; B., 1927, 268). Such catalysts are copper oxide or acetate, zinc oxide, acetate, or methoxide, and finely-divided zinc, aluminium, or copper, or mixtures of any of these with potassium acetate or sodium acetate. W. G. Carey.

Production of phosphoric acid. W. H. Waggaman and H. W. Easterwood, Assrs. to Victor Chemical Works (U.S.P. 1,630,283, 31.5.27. Appl., 23.1.24).—A mixture of phosphate rock, silica, and coke is heated with solid fuel, and oxidising gases are introduced at one end of the mass to burn the fuel, while additional oxidising gases are introduced at various points to oxidise the phosphorus evolved. W. G. Carey.

Synthetic production of ammonia. F. Uhde (E.P. 247,225 and 259,230, [a] 8.2.26, [b] 30.9.26. Conv., [a] 9.2.25, [b] 2.10.25).—(a) Catalysts entirely free from water for the production of ammonia from its elements at about 425° are prepared from ferro- or ferri-cyanides and metallic salts of the iron group by solution or suspension in water-free polyhydric alcohols, *e.g.*, glycol. (b) Superheating of the catalyst is obviated

by conveying the gases through double pipes arranged in the contact chamber in a double counter-current, so that the fresh gases enter through the inner pipes in the same direction in which they are conducted through the catalyst, and are then led through the outer pipes in counter-current to the entering and to the reacting gas current, and thence to the contact mass for reaction. The diameters of the pipes are calculated so that the heat transference assures a practically uniform temperature throughout the whole contact chamber. W. G. Carey.

Simultaneous manufacture of ammonium sulphate and bisulphate and of sulphur. C. Harnist (E.P. 250,990, 20.4.26).—Solutions of ammonium salts of one or several oxygenated acids of sulphur are heated under pressure. If bisulphite or normal sulphite is used risk of explosion is prevented by the direct introduction of steam, which starts the reaction and agitates the liquid. Solutions to be so treated are obtained from ammonia and hydrogen sulphide gases from coal distillation by absorption, while hot and before tar removal in water, in saturators or scrubbers and treatment with sulphur dioxide. The decomposition of hydrogen sulphide by sulphur dioxide is accelerated by the action of a strong electric field. W. G. Carey.

Manufacture of ferric oxide. D. G. Zalocostas (U.S.P. 1,630,881, 31.5.27. Appl., 28.3.25. Conv., 16.4.24).—Ferrous sulphate crystals are passed through a dehydrating chamber having gas burners which project flames over the surface of the crystals and are so regulated that the water of crystallisation is vaporised as it is liberated, a temperature of 64° being maintained in the chamber. The dehydrated product is ground, oxidised, and roasted. W. G. Carey.

Manufacture of chromic chloride. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 269,028, 26.7.26).—Anhydrous chromic chloride is made by treating ferrochromium with chlorine, with or without a small quantity of a reducing agent, *e.g.*, carbon monoxide, in a heat-insulated revolving furnace. Heat is necessary only to start the reaction; thereafter the temperature is controlled by varying the amount of chlorine so that the iron chloride formed is sublimed. W. G. Carey.

Manufacture of hydrated chromic chloride. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 271,016, 26.7.26).—Anhydrous chromic chloride is converted into the hydrated salt without the aid of reducing agents by exposing an aqueous suspension to the action of a cathodically polarised conductor, *e.g.*, in a lead-lined iron vessel having a chromium anode and using a very low current density. The heat generated in the process enables a highly concentrated solution to be produced, which is filtered while hot and solidifies on cooling. W. G. Carey.

Production of high-acid phosphates. E. C. R. Marks. From Parker Rust Proof Co. (E.P. 270,820, 16.2.26).—The dihydrogen orthophosphate of manganese, zinc, cadmium, or iron is made by dissolving the metal in a 60—70% solution of orthophosphoric acid at 100—115°, filtering, cooling, and crystallising, finally drying under non-oxidising conditions below 60°. Ferromanganese is used if the manganese and iron salts are required together. W. G. Carey.

Manufacture of barium salts, especially the carbonate, free from sulphur. F. Falco (E.P. 270,559, 5.10.26).—Barium carbonate, precipitated from barium sulphide by carbon dioxide, is mixed into a paste with a reducing agent which decomposes and volatilises on heating, such as a 2% solution of formic or oxalic acid, and is heated in a rotary drum to drive off the hydrogen sulphide and sulphur dioxide and to decompose the reducing substance. Barium salts may be treated in solution by the same reducing agents, acidifying with the appropriate acid to liberate the sulphur compounds. W. G. Carey.

Continuous purification of crude carbon disulphide. E. Legeler and P. Esselmann (E.P. 254,676, 1.12.25. Conv., 6.7.25. Addn. to E.P. 238,489; B., 1925, 803).—Crude carbon disulphide is run through the inner part of a double concentric rectifying column filled with Raschig rings and surrounded with a heating jacket at the bottom. From the inner part of the column hydrogen sulphide is carried off, and from the outer part carbon disulphide free from hydrogen sulphide is distilled from the upper end, while the concentrated solution of sulphur in carbon disulphide remaining is continually withdrawn for further separating treatment. W. G. Carey.

Method of chlorinating solutions. J. H. MacMahon, Assr. to Mathieson Alkali Works, Inc. (U.S.P. 1,616,134, 1.2.27. Appl., 9.7.25).—The liquid is circulated by a pump and the pressure is increased on the discharge side of the pump by restricting the flow, chlorine being introduced in the region of increased pressure. W. G. Carey.

Manufacture of phosphorus acids and hydrogen. I. G. Farbenind. A.-G. (E.P. 262,447, 2.12.26. Conv., 3.12.25).—Heat produced by the interaction of phosphorus and steam is recovered by separating the phosphorus acids formed from the reaction mixture by a water-operated condenser maintained at 100° or over, the condenser generating steam at atmospheric or higher pressure. The excess of steam and hydrogen from the reaction passes through a second condenser and generates steam below atmospheric pressure, which is compressed and superheated for use in the process. W. G. Carey.

Purification of burner gases and manufacture of chemically-pure sulphuric acid. M. Krafft (U.S.P. 1,627,977, 10.5.27. Appl., 25.6.23. Conv., 30.6.22).—See E.P. 220,413; B., 1924, 868.

Production of hydrocyanic acid. R. Koepp & Co., Assees. of G. Bredig and E. Elöd (U.S.P. 1,627,144, 3.5.27. Appl., 21.11.24. Conv., 7.12.22).—See E.P. 229,774 and 229,973; B., 1925, 315.

Ammonia from gas (E.P. 270,009 and 270,852).—See II.

Depilatory (G.P. 436,149).—See XV.

VIII.—GLASS; CERAMICS.

Crystallisation of glass. A. Lecrenier (Bull. Soc. chim. Belg., 1927, **36**, 137—148).—A rare example of crystallisation is furnished by a specimen of glass in the collection of the Institut Chimique at Nancy. Analysis

of the glass yielded the following numbers: SiO_2, 75·54%; PbO, 0·37%; $Fe_2O_3 + Al_2O_3$, 0·35%; Mn_3O_4, 0·25%; CaO, 7·51%; MgO, 0·18%; Na_2O, 14·51%; SO_3, 0·43%; Sb_2O_3, traces; total, 99·16%. Spherolites of varying size, the majority, however, being about 4—5 mm. in diameter, are formed within the material, and their number increases from year to year. The spherolites have the same composition as that of the surrounding glass. The crystalline phase appears to be cristobalite. On allowing a fused barium crown leger glass (SiO_2, 48·08%; As_2O_3, 0·57%; BaO, 29·83%; Al_2O_3, 1·65%; ZnO, 8·09%; K_2O, 6·87%; Na_2O, 1·36%; B_2O_3, 3·55%) to cool, a large number of spherolites of devitrification, about 5—7 mm. in diameter, were formed within the material. As in the preceding case, the spherolites have the same composition as the surrounding glass. Devitrification was not accentuated by heating at 600°, but was completed on raising the temperature to 700°. The crystalline phase appears to be the silicate, $BaSi_2O_5$. No well-defined crystal which could possibly have acted as a centre of crystallisation was found in any of the spherolites. Crystal soda glass (Na_2O, PbO, SiO_2) decomposes on cooling with separation of cristobalite. Glasses containing cuprous oxide crystallise to form a vitreous, opaque mass having a characteristic reddish-brown colour. The crystalline phases are cristobalite and tridymite. Wollastonite has been found in numerous specimens of plate glass.

J. S. Carter.

Ionometric measurement of the acidity and alkalinity of glasses by means of the Luers quinhydrone potentiometer. A. Mauri (Giorn. Chim. Ind. Appl., 1927, **9**, 168—169).—Tests of the alkalinity, similar to those already carried out by Clark's colorimetric method and by the potentiometric method with hydrogen, have been made on Muranese (Tenax), Neapolitan (Crist. Naz.), and Jena (Fiolax) glasses. The p_H values of the solutions obtained by treating these glasses with water of p_H 5·43 in the cold (at 134° for 1 hr.) are, in the above order, 5·65 (5·45), 4·74 (5·62), and 5·75 (6·33). These results differ in absolute magnitude from, but are parallel to, those previously obtained.

T. H. Pope.

"Spalling" in silica gas retorts. W. J. Rees (Trans. Ceram. Soc., 1926, **25**, 321—325).—Normally there is little or no penetration or deposition of carbon within silica retort material, but in the two cases investigated several small patches of carbon were visible on the internal face of the "spalled" pieces. Faint cracks were detected round these small black patches. Analyses showed that the black patches contained 12·5% C and 5·18% Fe_2O_3, whereas the normal material contained no carbon and only 0·92% Fe_2O_3. It is suggested that small segregations of iron oxide in the silica retort material have acted as nuclei for the deposition of carbon. This has caused a weakening of the silica material round the deposited carbon, with the gradual development of cracks. In a laboratory test, "carbonised" iron oxide spots were obtained by subjecting silica brick impregnated with iron oxide to the action of a current of carbon monoxide at 700—900°.

A. T. Green.

Discoloration of clays during firing. J. Konarzewski and A. E. J. Vickers (Trans. Ceram. Soc., 1927, **26**, 1—20).—The influence of water vapour and sulphur dioxide upon the colour of fired ware was investigated. Six different clays were tested; they were fired in an electric tube furnace through which air, water vapour, and sulphur dioxide, or a mixture of these, could be passed at will. Two series of experiments were made, the firing period in the first being 7½ hrs. and in the second 10 hrs. The colour of the fired clays was measured by means of the Lovibond tintometer. It was observed that the presence of water vapour during the dehydration and oxidation periods of firing, and the presence of sulphur dioxide during the whole time of firing, produced a reddish coloration of white and yellow burning clays. The presence of water vapour during the last stage of firing produced a lighter colour than was obtained under normal conditions. The action of the gases on clays is strongest between 500° and 600°. The discoloration may be removed more or less by prolonged firing in an oxidising atmosphere. The sulphur dioxide seemed to be adsorbed by the clays, and the presence of water vapour retarded the process of dehydration, which then took place more suddenly and at a higher temperature, causing more complete breakdown of the iron compounds.

F. Salt.

Patents.

Manufacture of glass. Soc. Anon. des Manuf. des Glaces et Prod. Chim. de St. Gobain, Chauny & Cirey (E.P. 264,495, 11.1.27. Conv., 12.1.26).—In glasses used for absorbing *X*-rays and rays of similar short wavelength, and containing more than 50% of lead oxide or 20—50% of lead oxide with 40—10% of barium oxide, zirconium oxide, up to 10%, is employed to replace some of the silica. The rate of setting of the glass on cooling is thereby increased, whilst the glass also possesses increased opacity to the rays, and is less sensitive to exterior agents. Batch materials used are zirconia or zircon.

A. Cousen.

Manufacture of vitreous silica. L. B. Miller, Assr. to General Electric Co. (U.S.P. 1,628,468, 10.5.27. Appl., 29.12.26).—Vitreous silica is prepared from crystal quartz by heating at a temperature but little below 1700°, then cooling to below 270°, and finally fusing *in vacuo*.

A. Cousen.

Glass composition. W. F. Bleecker (U.S.P. 1,629,648, 24.5.27. Appl., 22.7.22).—A mixture consisting of 75 pts. of sand, 80 pts. of soda ash, 70 pts. of litharge, 20 pts. of borax, 30 pts. of nitre, and 4—24 pts. of uranium oxide is used, and gives a transparent henna-coloured lead glass.

H. Royal-Dawson.

Glass. F. M. Locke (U.S.P. 1,626,042, 26.4.27. Appl., 28.8.16. Renewed 29.10.26).—A borosilicate glass with a low coefficient of expansion contains 70—79% of silica, not above 28% of boron trioxide, not above 5% of alumina, and not above 9% of alkali oxides, which latter are added to the batch as nitrates prior to fusion.

T. S. Wheeler.

Manufacture of synthetic aquamarine stones. J. F. Riera (E.P. 271,316, 4.10.26).—A mixture of silica and hydrated or anhydrous beryllia is fused with alumina

and colouring matter containing cobalt nitrate, together with a flux, *e.g.*, lithium carbonate or borax. The fused product is broken into fragments, softened by heat, and moulded under pressure to the desired shape before polishing. B. W. Clarke.

Manufacture of a ceramic product. T. C. and W. O. Prouty, Assrs. to American Encaustic Tiling Co., Ltd. (U.S.P. 1,628,910, 17.5.27. Appl., 7.3.25).—A bisque of talc, clay, and calcium sulphate, containing 27% of magnesium oxide, 6% of alumina, 61% of silica, and 4% of calcium, together with water and other impurities, is formed, and fired at 1200—1230°. T. S. Wheeler.

Production of cemented acid- and alkali-resisting vessels. B. Ehrig & Co. (G.P. 438,879, 26.10.24).—The inner walls of the vessels are coated with an acid- and alkali-resisting surface formed by heating refractory material to which a suitable flux or glazing powder has been added. L. A. Coles.

Kilns for burning clay products, glazed ware, and pottery. H. Webster (E.P. 269,344, 17.10.25).—A battery of kilns of the continuous type is provided with central longitudinal flues connecting adjacent kilns, a parallel flue passing alongside all the kilns of each series, and a pair of transverse flues between adjacent kilns in operative communication by means of dampers with the longitudinal and parallel flues and with a chimney. B. W. Clarke.

Apparatus and process for burning ceramics and the like. H. Koppers, Assr. to Koppers Development Corp. (U.S.P. 1,631,536, 7.6.27. Appl., 31.8.21. Conv., 21.8.19).—See G.P. 349,951; B., 1922, 711 a.

IX.—BUILDING MATERIALS.

Re-use of plaster of Paris moulds. M. Farnsworth (Ind. Eng. Chem., 1927, **19**, 714—717).—Plaster of Paris moulds used for die-casting metals become pitted and dehydrated by the heat and unfit for further use. The anhydrite formed can be rehydrated to gypsum and recalcined (cf. B., 1925, 804), but the regenerated moulds show a large decrease in tensile strength. Changes in the character of plaster during five recalcinations were studied by determinations of water of crystallisation, water-carrying capacity, setting time, and tensile strength, but the cause of loss of tensile strength does not appear to be a chemical one. Examination of the regenerated samples of calcined plaster and gypsum by an *X*-ray photographic method showed no changes in the particle size of the former, but the crystals in the gypsum became continuously larger with each successive calcination, due, it is believed, to incomplete calcination, the particles of uncalcined gypsum acting as centres of crystallisation. This effect may to some extent be obviated by adding to the ground plaster, before recalcination, a small amount of aluminium oxide, the optimum proportion being about $\frac{1}{4}$%, but this or further additions of the oxide do not prevent further loss of tensile strength upon second recalcination. C. O. Harvey.

Concrete deterioration, more especially with reference to its use for conduits and in lining iron pipes. J. R. Baylis (Amer. Soc. for Municipal Improvements, 1926, 217—234).—The extent of the carbonation of the calcium compounds in set cement or concrete exposed to the air or to water is an important feature in determining the resistance of the concrete to deterioration. Not only is the calcium carbonate formed less resistant to the attack of saline waters than the original high calcium silicates, but its formation results in setting free the sulphates in the concrete with the result that needles of calcium sulphoaluminate are formed which rapidly disintegrate the content. The original SO_3 content of the cement or water is not a sufficient indication of the durability of the concrete, since the distribution of the pores may result in local concentrations of sulphates with consequent disintegration. In view of the possibilities of deterioration it is pointed out that for conduits especial care must be taken to make good dense concrete; for the concrete lining of cast-iron water-pipes, the usual thickness of about $\frac{1}{16}$ in. is not sufficient, except for non-corrosive waters, and a thickness of $\frac{1}{2}$—1 in. concrete would be a more suitable lining. B. W. Clarke.

Patents.

Production of cements. I. G. Farbenind. A.-G. (E.P. 263,124, 6.12.26. Conv., 15.12.25).—The slag obtained by fusing a crude phosphate with sand and carbon is tapped off and mixed with calcareous or aluminous materials in proportions suitable to form a cement clinker. B. W. Clarke.

Production of porous concrete. E. I. Lindman (E.P. 262,394, 11.10.26. Conv., 5.12.25).—Clay or clayey material is calcined at above the sintering temperature and the operation interrupted before the expanded mass begins to shrink; on cooling, the clinker produced is ground and used as an admixture with cement, effervescing means, and water in the production of porous concrete. Preferably, the mixture is calcined at such temperature that the mass is melted, a further supply of gases being developed which remain occluded in the material. The light product obtained on cooling (*d* 0·5) has 100% increased heat-insulating properties compared with porous concrete produced in the usual way. B. W. Clarke.

Production of a hydraulic aluminous binding material and an iron alloy. L. de Lambert, Assr. to P. Zucco (U.S.P. 1,628,872, 17.5.27. Appl., 17.4.24).—Bauxite (47 pts.), limestone (45 pts.), phosphorite (8 pts.), and carbon (10—15 pts.), heated at 800°, yield impure iron and a hydraulic cement. T. S. Wheeler.

Process and apparatus for tempering plaster. L. E. Chassevent (E.P. 266,335, 11.2.27. Conv., 20.2.26).—Plaster is mixed with hot water at 60—100°. The plaster can be kept for several hours in this condition without affecting its setting properties, and when used it will harden rapidly when the temperature falls to 38—43°. B. W. Clarke.

Continuous [brick] kiln and drier. C. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. (U.S.P. 1,627,841, 10.5.27. Appl., 21.2.20).—Bricks are burnt in a fire-heated tunnel kiln, the products of combustion being utilised in drying the bricks before

burning. The bricks and transporting device are air-cooled, the heated air being injected into the drying chamber at intervals along its length in order to supply heat thereto and to serve as a vehicle for carrying away moisture from the bricks. B. W. Clarke.

Preservation of stone, brick, and like work. W. Anderson (E.P. 271,203, 17.3.26).—A mixture of silicates and silicofluorides with or without the addition of fluorides is claimed. B. W. Clarke.

Wood preservative. L. P. Curtin, Assr. to Western Union Telegraph Co. (U.S.P. 1,624,930, 19.4.27. Appl., 28.12.25).—Barium hydroxide solution emulsified with petroleum is employed to impregnate wood. T. S. Wheeler.

Preservative for telegraph poles etc. J. Himmelsbach (G.P. 435,146, 28.9.24).—An addition of 10% of natural bitumen is made to the mixture of coal-tar pitch and petroleum pitch used for the preservation of the wood of telegraph poles etc., the petroleum pitch being preferably one of very high m.p. A. B. Manning.

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

Production of iron sponge. M. Wiberg (Amer. Electrochem. Soc., April, 1927, **51**. Advance copy. 26 pp.).—The reduction of magnetite by carbon monoxide may be regarded as occurring in two stages (1) $Fe_3O_4 \rightarrow FeO$; (2) $FeO \rightarrow Fe$. With ore fed into the top of the furnace shaft and carbon monoxide introduced at the bottom, reaction (2) will occur in the lower part of the shaft and at 1000°, if equilibrium were attained, the gas leaving this zone would contain 29% CO_2. If this gas then produced the corresponding quantity of ferrous oxide by reaction (1) in the upper zone of the furnace, its carbon dioxide content would only reach 38%, whereas the equilibrium concentration for this reaction at 1000° is 82%. In the Wiberg process (cf. U.S.P. 1,401,222, B., 1922, 108 A) utilisation of the reducing capacity of carbon monoxide is attained by removing a large proportion of the gas leaving the lower zone, enriching it in carbon monoxide by passing through hot coke or charcoal (water may also be added to produce some hydrogen), and then returning it to the bottom of the shaft. Low-grade fuels may be used for this purpose, any sulphur derived from the coke being removed by passing the gases through a layer of lime. By removing 78% of the gas in this way, the remainder, supposing it to contain 29% CO_2, would, if used completely in reducing the corresponding amount of magnetite, leave the furnace containing 72% CO_2. Actually gases containing about 20% CO_2 were withdrawn and the carbon dioxide content of the remainder rose to about 50% in the upper zone. The carbon monoxide not utilised in reduction is burnt by introducing an air blast above the upper reduction zone, whereby the incoming ore is preheated to an extent which is regulated by the composition of the gas. Semi-commercial trials were carried out at Sandviken, Sweden. The regenerator was electrically heated and reduced the carbon dioxide content of the gas to 1% or less, its temperature being maintained above 1000°. The iron sponge is obtained as lumps of about the same size as those of the ore, and may contain over 1% C. On smelting, the carbon monoxide produced by the oxidation of this carbon protects the iron against oxidation. If the carbon content of the sponge is high enough, good quality steel may be produced by smelting in the crucible or electric furnace without addition of pig iron. Steel produced from iron sponge is of a higher quality than that obtained from ordinary raw materials, probably because the sponge has never been in a molten condition during its production, and therefore has not been able to form slag inclusions. Foreign material in the ore remains as relatively big grains in the sponge, and easily rises to the surface on smelting. The sponge obtained from low-grade ores (30—50% Fe) may be treated by magnetic separation to remove the gangue (which contains most of the sulphur and phosphorus). The powdered iron sponge is readily compressed into solid briquettes without addition of a binder. For the production of 1000 kg. of iron sponge from ore containing 95·3% of magnetite using coke in the electric regenerator, 222 kg. of coke was used and 1280 kw.-hrs. of electrical energy, the electrode consumption being only 2 kg. In places where electrical energy is expensive the electric regenerator could be replaced by two "alternating" producer-gas generators. H. J. T. Ellingham.

[Production of steel by] carbonising scrap iron. R. Hennecke (Stahl u. Eisen, 1927, **47**, 777—780).—Good quality steel may be produced by melting scrap iron and steel without addition of cast iron scrap or raw pig iron. The scrap is briquetted together with 1—2% of carbonaceous material, as free as possible from sulphur, the briquettes being melted with 3% of limestone in an acid-lined open-hearth furnace. When the charge has melted down and the evolution of gas has almost ceased 1% of fluorspar and a further 3% of limestone are added in two portions to assist in the removal of sulphur. Finally the steel is deoxidised in the ladle by the addition of 5—7 kg. of 80% ferromanganese per ton of steel. The output of steel per hour is about 5·8—5·9 tons per 20 m.^{2} of hearth. Cost data compare favourably with those of the usual process. A. R. Powell.

Action of pure carbon monoxide on iron at elevated temperatures. W. P. Fishel and J. F. Wooddell (Trans. Amer. Soc. Steel Treat., 1927, **11**, 730—740).—On heating samples of Armco iron in carbon monoxide at temperatures from 800° to 1100° the amount of carbon dioxide formed was greater than that given for the equilibrium values for the system CO–CO_2–C. Iron carbide was formed during the reaction, which was reversible. High temperature gave a low-carbon case with deep penetration, low temperature produced a high-carbon case with low penetration. The rate of penetration of carbon monoxide and dioxide into an Armco cylinder with $\frac{3}{32}$ in. walls was measured at 950—1100°. The rate for both gases increased with rising temperature, but more quickly for the former. It would not, however, account for the depth of case produced by carbon monoxide. T. H. Burnham.

Properties of materials at high temperatures. I. Mechanical properties of Armco iron, 0·17% carbon steel, and 0·24% carbon steel, with special

reference to creep. H. J. Tapsell and W. J. Clenshaw (Dept. Sci. Ind. Res., Eng. Res., Spec. Rep. No. 1, 1927, 60 pp.).—Tensile, torsion, impact, hardness, and fatigue tests have been carried out on Armco iron, and on 0·17% and 0·24% carbon steels at temperatures up to 700°, and the phenomenon of creep has been specially investigated. In the tensile tests at normal rates of loading the ultimate strength reached a maximum between 200° and 250°. No yield point, as determined by the drop of the testing-machine beam, was evident above 250° for Armco iron, or above 200° for the carbon steels. Under a steady load the extension–time curves show different forms according to the stress and temperature. With a given load above a certain temperature the velocity of creep first decreases and then increases, the extension–time curves passing through a point of inflexion, but below a definite temperature the creep gradually ceases with time. In the same way at a given temperature a limiting stress can be found below which the creep ceases after a time, although this same stress produces a certain amount of initial creep. These limiting stresses are very sensitive to temperature; thus a change of 20° may cause a change of 20% in the limiting stress for Armco iron. There is considerable similarity between the torsion test and tensile test results, but the maximum value of the modulus of rupture occurs at a higher temperature than the corresponding maximum ultimate tensile strength. The effect of different types of notches in the impact test specimens was investigated. The fatigue tests, in a Haigh alternating-stress testing machine at 2400 cycles/min., showed that the fatigue ranges for zero mean stress, based on 10^7 reversals, are considerably higher at 300—400° than at air temperature, although the limiting creep stresses are falling rapidly at these temperatures. This is probably due to the rapid rate of reversal.

W. Hume-Rothery.

Graphite in grey cast iron. P. Bardenheuer (Stahl u. Eisen, 1927, **47**, 857—867).—In grey cast iron of normal composition the volume of the precipitated graphite is relatively so great that its form and degree of fineness have a far more important effect on the mechanical properties than has the composition or structure of the ground mass. The tensile strength, bending strength, and resistance to shock are greater the finer and more evenly distributed are the graphite particles; coarse and irregular graphite inclusions not only impair the physical properties, but also reduce the resistance of the metal to atmospheric corrosion. The formation of coarse graphite lamellæ is hindered by the presence of the maximum amount of solid solution at the temperature at which the eutectic freezes, owing to the action of the solid solution in accelerating the rate of solidification of the eutectic. The absence of undissolved graphite nuclei is important if a fine structure is to be obtained; this is assured by keeping the carbon content relatively low or by heating the metal at a sufficient temperature to dissolve the carbon completely. Addition of manganese above 1%, chromium, and more especially sulphur, increases the stability of the carbide; aluminium, nickel, and silicon, on the other hand, decrease it. The simplest method of obtaining a high-grade cast iron, however, is to add sufficient steel scrap to the melt to reduce the carbon content to 1% below that of the eutectic composition.

A. R. Powell.

Determination of silicon in steel and pig iron. A. Stadeler (Stahl u. Eisen, 1927, **47**, 966—969).—Comparative tests of six methods used in ironworks for the determination of silicon showed that there is little to choose between the methods as regards accuracy provided that, in all cases, the filtrate from the first silica residue is evaporated again to recover traces that escape the first filtration. The most rapid and economical method is that involving evaporation with hydrochloric acid followed by evaporation of the weighed residue with hydrofluoric acid; in the case of pig iron, addition of bromine to the acid in which the metal is dissolved is recommended. Fusion of the silicious residue from the acid evaporation is unnecessary in any case, and serves only to increase possible sources of error.

A. R. Powell.

Determination of molybdenum in iron and steel. E. Färber (Chem.-Ztg., 1927, **51**, 171).—The sample (1—2 g.) of the metal chips is dissolved in hydrochloric acid (*d* 1·19), with subsequent addition of 2 g. of ammonium persulphate. The filtrate is boiled, saturated for 20 min. with hydrogen sulphide, and the molybdenum sulphide collected, washed with hot dilute hydrochloric acid and with hot water, dried, ignited, and weighed. The impure oxide so obtained is extracted for 1 hr. with 15% potassium hydroxide solution, and the insoluble oxides of copper and iron are collected, dried, ignited, and weighed, this weight being deducted from that of the impure oxides to give the weight of molybdenum trioxide.

F. R. Ennos.

Formation of ferrite and diminution of impact resistance in tempered nickel-chromium steels. A. Schleicher (Rev. Mét., 1927, **24**, 293—295).—After forging at 1100°, followed by annealing at 875°, quenching in oil, and tempering for 4 hrs. at 650°, the impact resistance of steels containing 0·45—0·51% C, 0·2—0·4% Cr, and 1·4—1·5% Ni, decreased from 8—10 kg./cm.^{2} to 0·9—1·2 kg./cm.^{2} This is ascribed to the formation of aggregates of acicular ferrite owing to the prolonged heating at a temperature near the Ac1 point. Subsequent annealing at 840° (A3 point) and cooling in air resulted in the formation of a Widmannstätten structure without improvement in the resistance to shock. A steel containing 0·26% C, 0·72% Cr, and 3·77% Ni, after a complicated forging and heat-treatment, followed by an annealing operation at 660° for 3 hrs., showed large columnar masses of ferrite in the microstructure and a correspondingly low impact resistance. Hence steels of the above composition should not be tempered, after forging, at a temperature too near the A1 point, and the period of heating must be as short as possible.

A. R. Powell.

Effect of tin on the mechanical properties of copper. W. Stahl (Chem.-Ztg., 1927, **51**, 427).—Addition of a small quantity of tin, *e.g.*, 1%, to tough-pitch copper removes any adsorbed hydrogen and carbon monoxide and increases the sp. gr. from 8·625 to 8·913; at the same time the tensile strength is improved and the

ductility only slightly reduced. Overheating during annealing of this alloy, however, induces brittleness and internal fractures due to segregation of a constituent rich in tin. A. R. Powell.

Determination and separation of the oxidisable constituents of aluminium alloys. G. Jander and F. Baur (Z. angew. Chem., 1927, **40**, 488—490. Cf. Jander and Wendehorst; A., 1922, ii, 529).—When hydrogen chloride free from oxygen is passed over aluminium alloys containing magnesium at 200°, iron, copper, manganese, and part of the magnesium remain behind as metals or chlorides. Aluminium and silicon chlorides volatilise with some magnesium chloride which is carried over as a fine dust. By application of an external electric field to a dust-settling chamber through which the mixture passes, practically the whole of the magnesium chloride dust is deposited therein. The analysis of the various fractions is carried out by usual methods. When pure hydrogen chloride is used in this way to decompose commercial aluminium, the whole of the silicon is found as chloride, no silica remaining in the reaction boat. L. M. Clark.

Aluminium cans for foods. Serger.—See XIX.

Patents.

Manufacture of pure iron. J. Y. Johnson. From I. G. Farbenind. A.-G. (E.P. 269,677, 23.1.26).—Iron carbonyl in a liquid or vapour state is introduced into a vessel heated preferably at 250—300° in such manner that decomposition is effected mostly in the hot free space and not by contact with the walls of the vessel. Temperatures above 400° are to be avoided because of the interaction between carbon monoxide and iron at such temperatures. Gases or other substances which act catalytically may be introduced to assist the decomposition. C. A. King.

Production of dense iron and iron alloys directly out of oxide ores. H. G. E. Cornelius (E.P. 252,677, 26.3.26. Conv., 26.5.25).—Briquettes formed from a mixture of finely-divided iron ore, carbonaceous material, and vanadium or titanium oxide are reduced and melted while floating on a slag bath in an electric furnace. The source of vanadium may be slag produced in a previous heat. C. A. King.

Gaseous reduction of iron ores. I. G. Farbenind. A.-G. (F.P. 613,677, 30.3.26. Conv., 11.4. and 1.7.25). —The reducing gases used in the process are obtained by treating low-grade solid fuels in powder form or vapours, or gases containing hydrocarbons, with a deficiency of oxygen or gases rich in oxygen. The reduced iron may be melted by means of the same gases, and the issuing gases from the melting operation used for reducing further quantities of ore, if necessary, after the addition of steam or other gas. The final spent gases may be utilised in the synthesis of ammonia or of methyl alcohol. A. R. Powell.

Reduction of metal oxides, especially iron ores. A./S. Norsk Staal (Elektrisk-Gas-Reduktion) (G.P. 438,558, 13.10.25).—Iron ore is reduced by means of water-gas in a closed circuit from which a portion of the used gases is periodically removed and treated with steam at a high temperature, whereby the carbon monoxide present is oxidised to carbon dioxide with the liberation of hydrogen. After removal of the carbon dioxide, the remainder of the gas is passed back into the circuit with the other gases, which have meanwhile been regenerated in the usual way. The whole of the oxygen of the ore is thus removed as carbon dioxide and water. A. R. Powell.

Alloy. V. Fener (U.S.P. 1,626,925, 3.5.27. Appl., 14.1.26).—To an alloy of 100 pts. of copper and 3—25 pts. of tin, while in the molten state, are added 1 pt. of ammonium chloride and 0·02—0·10 pt. of potassium ferrocyanide. F. G. Crosse.

Wrought metal article. W. H. Bassett, jun., Assr. to American Brass Co. (U.S.P. 1,630,999, 31.5.27. Appl., 28.1.26).—An alloy is used consisting of 95·5% Cu, 2% Sn, 2·5% Al, approximately, the aluminium being in excess of the tin. F. G. Crosse.

Soldering aluminium. S. Smith (E.P. 270,453, 10.12.26).—A solder for aluminium consists of a mixture of tin and zinc, the zinc being in greater proportion than tin, but not exceeding 70%. Vaseline or petroleum jelly is used as a flux. C. A. King.

Aluminium alloys. W. Guertler and W. Sander, Assrs. to T. Goldschmidt A.-G. (U.S.P. 1,629,699, 24.5.27. Appl., 6.5.26. Conv., 22.11.23).—Aluminium is alloyed with 4—26% by wt. of a magnesium-zinc compound, $MgZn_2$, and subjected to annealing, quenching, and ageing. H. Royal-Dawson.

Light aluminium alloy. H. G. C. Fairweather. From Établ. Métallurgiques de la Gironde (E.P. 270,824, 17.2.26).—A light alloy of high tensile strength and high coefficient of expansion contains 1·5—5 (2)% Cu, 0·3—3 (0·37)% Ni, 0·2—2 (0·27)% W, 0·2—2 (0·45)% Mg, and 97·8—88 (96·91)% Al (commercial). C. A. King.

Alloys for use as platinum substitutes. H. Limbourg (E.P. 270,974, 27.11.26).—Alloys suitable for use in place of platinum comprise 75—85% Au, 10—15% Pd, and 2·5—10% Pt. The m.p. varies from 1300—1400° according to the composition, and the alloys resist the action of any single mineral acid and of fused alkali carbonates and nitrates, but are slightly attacked by fused alkali hydroxides. A. R. Powell.

Treatment of ores. J. T. Terry, Assr. to T. H. Sheridan (U.S.P. 1,627,582, 10.5.27. Appl., 7.9.22).—Ground ore containing silver is treated with an ammoniacal solution of copper sulphate and sodium thiosulphate, the extracted silver being recovered by treatment of the filtrate with sodium sulphide. T. S. Wheeler.

Method of metallising ores. H. N. Tracy (U.S.P. 1,628,012, 10.5.27. Appl., 29.5.23).—Ground ore is heated in a reducing atmosphere, and is introduced by a centrifugal thrower into the bottom of a bath of molten lead. The metal present alloys with the lead, and the gangue which rises to the surface is removed in a gas current. Means are provided for maintaining a reducing atmosphere in contact with the surface of the bath. T. S. Wheeler.

Manufacture of malleable nickel. N.V. Hybinette (U.S.P. 1,628,149, 10.5.27. Appl., 17.1.21).—In the production of malleable nickel by electrodeposition,

use is made of an anode of nickel containing 1% C, 1% Si, and 1% S, and of an aqueous electrolyte containing nickel chloride (40—60 g./litre), and boric acid (10—20 g./litre). A current density of 5—200 amp./sq. ft. is employed at a temperature of 40—50°. A portion of the electrolyte is continuously removed, neutralised by boiling in presence of nickel carbonate, filtered, and returned to the process. The deposited metal is rendered homogeneous by hot-working.

T. S. WHEELER.

Manufacture of nickel catalyst. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 255,884, 22.7.26. Conv., 22.7.25).—A highly active catalyst is formed by precipitating nickel, or a mixture of nickel and a metal of the iron or silver group, in the form of a crystalline hydroxide or carbonate in the presence of a carrier, *e.g.*, silica gel. The temperature for precipitation is preferred at about 70°. The product is reduced in hydrogen at about 400°.

C. A. KING.

Production of molybdenum and tungsten trioxides. P. SCHWARZKOPF (U.S.P. 1,629,004, 17.5.27. Appl., 23.7.26. Conv., 26.4.26).—Ore containing molybdenum or tungsten compounds is heated in air to form the respective trioxide, which, when oxidation is complete, is sublimed into a condensing chamber in a current of air.

T. S. WHEELER.

Recovering antimony from alloys. L. and M. MEYER (HÜTTENWERKE TEMPELHOF A. MEYER) (E.P. 264,139, 9.12.26. Conv., 8.1.26).—A quantity of sulphur equivalent to all the metals except antimony is added to the alloy when in a molten condition, molten antimony being separated from the other metal sulphides.

C. A. KING.

Reclaiming [easily oxidisable] metals. T. D. STAY and C. O. TESSIER, Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,630,361, 31.5.27. Appl., 28.10.21).—Light, readily oxidisable metals are melted in a furnace provided with mechanical agitation. A continuous feed of scrap metal is maintained to the furnace, and means for concentrating the slag on the surface of the molten metal are provided.

C. A. KING.

Protection of molten baths of easily oxidisable metals. G. MICHEL (E.P. 257,221, 10.2.26. Conv., 18.8.25).—Molten baths of easily oxidisable metals, *e.g.*, magnesium, are protected by a covering of a suitable salt, *e.g.*, magnesium fluoride, which is sprinkled with sulphur to form a local non-oxidising atmosphere.

C. A. KING.

Tough annealing of metal alloys. J. L. LA COUR and F. O. M. LINDH (E.P. 243,006, 13.11.25. Conv., 13.11.24).—Non-ferrous alloys containing copper, zinc, tin, lead, and aluminium are heated at a suitable annealing temperature in a neutral atmosphere and then slowly cooled to a temperature below 75% of the annealing temperature, before the alloy is exposed to the atmosphere.

C. A. KING.

Treatment of zinc-bearing ores for the recovery of zinc by electrolytic deposition. ELECTROLYTIC ZINC CO. OF AUSTRALASIA, LTD. (E.P. 258,575, 8.9.26. Conv., 17.9.25).—Prior to the recovery of zinc by electrodeposition an impure solution of zinc is treated with zinc dust while passing through an enclosed vessel in which the solution is agitated, sufficient time being allowed for the precipitation of copper, cadmium, etc. without the production of basic zinc sulphate. Excess of zinc dust is separated by gravity, and the solution containing suspended impurities is filtered.

C. A. KING.

Electric bright-annealing furnace. SIEMENS-ELEKTROWARME-GES.M.B.H. (E.P. 262,468, 4.12.26. Conv., 5.12.25. Addn. to E.P. 152,405; B., 1927, 115).—In a furnace constructed as in the prior patent, the resistance heating winding is arranged within the protective bell.

J. S. G. THOMAS.

Production of rustless iron and steel. D. W. BERLIN, Assr. to AKTIEBOLAGET FERROLEGERINGAR (U.S.P. 1,631,512, 7.6.27. Appl., 20.7.23. Conv., 28.8.22).—See E.P. 202,952; B., 1924, 717.

Aluminium[-copper-silicon] alloy. V. FUSS, Assr. to VER. ALUMINIUMWERKE A.-G. (U.S.P. 1,631,930, 7.6.27. Appl., 31.5.24. Conv., 16.8.23).—See E.P. 220,602; B., 1925, 886.

Electro-deposition of chromium and its alloys. OLAUSSON & CO. AKTIEBOLAG (E.P. 264,442, 17.3.26. Conv., 14.1.26).—See F.P. 612,880; B., 1927.

[Carriers for heating elements of] electric resistance annealing furnaces. A.-G. BROWN, BOVERI, & CIE. (E.P. 271,003, 13.1.27. Conv., 19.5.26).

Apparatus for sherardising metal articles. RUSTPROOF PROCESSES, LTD., and M. TROUTON (E.P. 269,680, 25.1.26).

Iron alloy (U.S.P. 1,628,872).—See IX.

XI.—ELECTROTECHNICS.

Action of iron as an impurity in the lead accumulator. I. Capacity loss due to self-discharge. II. Permanent capacity loss. Adsorption and desorption of the iron by the positive plate. F. M. LEA and J. T. CRENNELL (Trans. Faraday Soc., May, 1927. Advance proof).—I. The presence of iron in the electrolyte of a lead accumulator causes (*a*) a loss of capacity as measured by discharging immediately after charging (permanent loss), and (*b*) a further decrease in capacity when allowed to remain for some time before discharging (self-discharge loss). No other effect of iron on the life of performance of the cells has been found. The self-discharge loss increases notably with temperature, and at given temperature is practically proportional to the iron concentration, but at the highest temperature used (48·9°) the effect of iron is masked by losses due to other causes. For a cell with pasted plates and a capacity about 17 amp.-hrs. at 23·9°, an iron concentration of 0·84 g./litre caused an average self-discharge loss during 9 days of 0·22 amp.-hrs./day, the rate of self-discharge decreasing slightly during this period. There is evidence that self-discharge occurs to the same extent at both positive and negative plates. There is a definite tendency for iron to accumulate in the lower layers of the electrolyte, this being attributed to the action of concentration cells caused by the higher acid concentration at the bottom of a recently-charged cell. Assuming that

self-discharge is due to alternate oxidation and reduction of iron at the respective plates (Dolezalek, "The theory of the lead accumulator," 1904), an approximate relation is deducted between the rate of self-discharge and the cell dimensions, iron concentration, and the diffusion coefficients of ferrous and ferric ions; this gives self-discharge rates of the order observed. The view that iron also acts by accelerating the reaction $Pb + H_2SO_4 = PbSO_4 + H_2$ at the negative plate is not supported. II. For a cell of 17 amp.-hr. capacity at 23·9°, the permanent loss caused by 0·52 g. of iron per litre was 5% of the total cell capacity when discharging at a 5 hr. rate. This permanent loss is practically proportional to the iron concentration, and increases with increase of temperature or decrease of rate of discharge. It is connected with the adsorption of iron by the positive plate. Iron is readily adsorbed by lead peroxide, especially if freshly precipitated, and the adsorption increases with temperature, but decreases with increasing acidity of the solution. The adsorption is largely irreversible, but desorption occurs during the electrical working of cells in which the iron in the electrolyte and the paste are not in equilibrium. Removal of iron from the system by successive changes of electrolyte removes the permanent loss. The probable mechanism of the production of the permanent loss is discussed. The titanous chloride method of determining iron has been adapted to the determination of small amounts of iron in sulphuric acid solutions.

H. J. T. Ellingham.

Electric furnace for gas reactions. O. Scarpa (Giorn. Chim. Ind. Appl., 1927, **9**, 167—168).—In the usual types of electric furnaces for gas reactions, replacement of the metallic electrodes by carbons renders possible the lowering of the voltage of the arc, but the necessity of either injecting violent gas currents to dilate the arc sufficiently or employing complicated and costly devices remains. This necessity may be avoided by arranging the one carbon electrode perpendicular to the plane of three or four electrodes of the opposite sign, so that three or four arcs are formed. The voltage used is of the order 80, the intense electrodynamic actions induced by the arrangement causing great elongation of the arcs in the direction of the single electrode.

T. H. Pope.

Platinum-wound resistance furnace. E. Orton, jun., and J. F. Krehbiel (J. Amer. Ceram. Soc., 1927, **10**, 373—387).—A description is given of an endeavour to construct a furnace with an effective hot chamber 4—5 in. long and 3 in. in diameter, in which the temperature would be uniform throughout, the control accurate, the atmosphere clean and oxidising, and able to melt cone 20. The furnace has not been working long enough to furnish knowledge of its practical value.

A. T. Green.

Flames of atomic hydrogen. I. Langmuir (Ind. Eng. Chem., 1927, **19**, 667—674; cf. B., 1926, 549, 550).—The maximum temperature of the atomic hydrogen flame is calculated to be 4030° Abs., the degree of dissociation at this temperature being 0·642. When acetylene burns with an equal volume of oxygen the flame may attain 3270° Abs., the flame gas containing (by vol.) 60·9% CO, 21·8% H (molecular), and 17·3% H (atomic). The rate at which heat is delivered to a tungsten or copper surface by the atomic hydrogen flame produced by a 60 amp. arc (1330 watts/cm.^{2}) is 26 times as great as that from a Bunsen burner flame, and about double that from the oxy-acetylene flame. The behaviour of refractory substances in the flame is described. Except in the case of difficultly volatile metals (*e.g.*, molybdenum and tungsten), cooling by evaporation limits the temperature to which molten metals can be heated by allowing the atomic-hydrogen flame to play on the metal surface; the following maximum temperatures were observed (° Abs.): nickel, 2870° (b.p. 3650°); copper, 2840° (b.p. 3110°); silver, 2660° (b.p. 2740°).

S. K. Tweedy.

Patents.

Preparation of an electron-emitting cathode. F. Holborn, Assr. to Hazeltine Corp. (U.S.P. 1,625,776, 19.4.27. Appl., 14.1.26).—Natural guaiac is mixed with barium or strontium hydroxide or carbonate and water, and the mixture is applied to a thermionic valve filament, which is then heated first at 200° and then at 1100°.

T. S. Wheeler.

Metal-coated [arc] electrode. E. L. Swift, Assr. to V. Swift (U.S.P. 1,626,104, 26.4.27. Appl., 21.2.22).—Carbon mixed with an organic binder, a mineral acid, and a halogen salt is formed into an arc electrode, and is plated first with copper and then with a thin layer of brass, nickel, silver, or the like, to protect the copper from oxidation.

T. S. Wheeler.

***X*-Ray filter.** A. St. John, Assr. to Union Carbide & Carbon Research Laboratories, Inc. (U.S.P. 1,624,443, 12.4.27. Appl., 20.10.21).—An *X*-ray filter for use with a molybdenum target comprises a screen coated with calcium zirconium fluoride, which has been rendered fluorescent by strong ignition.

T. S. Wheeler.

Neon tube. R. R. Machlett, Assr. to Rainbow Light, Inc. (U.S.P. 1,628,256, 10.5.27. Appl., 16.10.26).—The life of a neon tube is increased by forming the electrodes with cavities containing magnesium carbonate, so that during operation there is a slow evolution of carbon monoxide.

T. S. Wheeler.

Metal filaments. T. E. Foulke, Assr. to General Electric Co. (U.S.P. 1,628,456, 10.5.27. Appl., 29.7.22).—Tungsten wire is passed through a furnace at 950°, so that a film of oxide is formed on it, and is then coated with a getter.

T. S. Wheeler.

Activation [of thermionic valve filaments] by means of hydrogen-free, carbon-bearing gas. J. W. Marden, T. P. Thomas, and J. E. Conley, Assrs. to Westinghouse Lamp Co. (U.S.P. 1,626,685, 3.5.27. Appl., 10.4.23).—Thoriated tungsten wire for thermionic valve filaments is activated by passage at a dull-red heat through carbon monoxide or cyanogen.

T. S. Wheeler.

[Filling for] gas-filled electric lamps. Siemens & English Electric Lamp Co., Ltd., and P. D. Oakley (E.P. 270,900, 10.5.26).—The harmful action of traces of water vapour in gas-filled lamps in which a tungsten filament is raised to incandescence in an atmosphere of nitrogen or argon or a mixture of these gases, is overcome by admitting into the lamp a small proportion,

e.g., 0·5—1%, of an oxyhalide of carbon, *e.g.*, carbonyl chloride, which may be introduced directly into the lamp or produced synthetically therein by the interaction of carbon monoxide and chlorine. J. S. G. Thomas.

Depolariser for primary batteries. G. W. Armstrong, Assr. to National Carbon Co., Inc. (U.S.P. 1,624,460, 12.4.27. Appl., 28.2.25).—Copper oxide is treated at below 100° with hydrogen sulphide until a sulphur content of 0·5% is reached. The product is of value as a depolariser for primary cells with an alkaline electrolyte. T. S. Wheeler.

Electrolytic cell. A. K. Croad. From Jessup & Moore Paper Co. (E.P. 270,104, 2.6.26).—An electrolytic cell, designed more especially for the production of caustic soda, comprises a casing supporting outer and inner cathode members having the form of a perforated iron basket. The inside of the outer member and the outside of the inner member are lined with asbestos or other material serving as a diaphragm which, when assembled, forms an asbestos-lined endless channel into which anode slabs of graphite are inserted, these being spaced so as to allow thorough circulation of electrolyte. A gas chamber or head adapted to clamp the cathode and diaphragm gas-tight between itself and the cell casing is provided. An additional outlet is provided for gaseous products liberated between the diaphragm and the outer casing, and another for liquid products at the bottom of the casing. Means comprising a float container automatically feed electrolyte, thus maintaining a constant level of electrolyte in the cell. J. S. G. Thomas.

Electrolytic rectifier. L. J. Keeler, Assr. to Le R. P. Bensing and G. P. Koelliker (U.S.P. 1,628,785, 17.5.27. Appl., 18.1.26).—A permanent electrolytic rectifier comprises electrodes of nickel and magnesium, and an aqueous electrolyte containing 30% of potassium fluoride and 10% of sodium borate. T. S. Wheeler.

Apparatus for the electrical purification of gases. L. Drault and C. Raulot-Lapointe (F.P. 614,442, 12.4.26).—The apparatus comprises a moving precipitation electrode kept clean by scrapers, a high-tension electrode directed towards the precipitation electrode, and means for cleaning the insulators. L. A. Coles.

Electrical gas-purifying plant, containing alternate discharge and precipitation electrodes placed transversely to the gas stream. Siemens-Schuckertwerke G.m.b.H., Assees. of C. Hahn (G.P. 438,834, 28.7.22).—The precipitation electrodes, all of which have the same mesh, are arranged in pairs, and between each pair is a sieve or set of sieves, the mesh of which decreases in the direction of the gas stream. L. A. Coles.

Mounting for electrodes of closed electric furnaces. I. G. Farbenind. A.-G. (E.P. 263,756, 5.11.26. Conv., 28.12.25).

Insulating paper (U.S.P. 1,600,424 and E.P. 251,970).—See V.

Annealing furnace (E.P. 262,468).—See X.

Rubber [in cables] (E.P. 269,124).—See XIV.

Rubber deposits (E.P. 257,885).—See XIV.

Oxidation of compounds (E.P. 265,672).—See XX.

Silver from photographic solutions (U.S.P. 1,629,212).—See XXI.

XII.—FATS; OILS; WAXES.

Utilisation of waste in the fat-hardening industry. K. Butkovski (J. Oil and Fat Ind., Moscow, 1926, 11—12; Chem. Zentr., 1927, I, 821—822).—After removal of the nickel from the deposit of catalyst and adhering fat obtained in fat-hardening, the hitherto wasted residue, which amounts to 4% by wt. of the hardened fat and contains considerable amounts of kieselguhr from which the fat cannot be recovered by melting, is treated with hot lye (*d* 1·05), 70% of the fat being recovered as soap. S. S. Woolf.

Change in sp. gr. of curd soap during the drying-out process. W. Kristen (Seifensieder-Ztg., 1926, **53**, 669—670, 689; Chem. Zentr., 1926, II, 3123).—The sp. gr. of soap curd (62—63%) rose, with shrinkage on drying out, from an initial value of 0·905 to 1·0482 in 39 days, and to 1·075 in 68 weeks. The outer layers were less dense than the mass in general. S. S. Woolf.

Oxidation of linseed oil. II. G. E. Holden (J. Soc. Dyers and Col., 1927, **43**, 157—158. Cf. B., 1918, 429 A).—Linoxyn films prepared by exposing linseed oil to atmospheric oxidation on roughened glass plates (*a*) at 18° for 12 days, and (*b*) at 100° for 8 hrs., were washed with ether, alcohol, and water, dried over concentrated sulphuric acid *in vacuo* to constant weight, and analysed. The figures for linoxyn obtained at 18° agree with the results of earlier observers, whilst that produced at 100° corresponds to a higher state of oxidation, and is more serviceable in industrial practice. Linoxyn has analytical figures agreeing closely with hexahydroxylinoleic triglyceride. S. S. Woolf.

Gossypol in cottonseeds. Gallup.—See XIX.

Patents.

Edible fat composition. H. E. Dubin, Assr. to H. A. Metz Laboratories, Inc. (U.S.P. 1,624,164, 12.4.27. Appl., 19.2.25).—The product, with m.p. below 37°, free from objectionable odour and taste, and of therapeutic value, comprises glyceryl margarate mixed with at least an equal weight of ethyl margarate. T. S. Wheeler.

Machine for preparation of artificial edible fats. G. Scheffler (G.P. 437,670, 4.9.23).—Melting kettle, emulsifier, and cooling and kneading rollers are combined in one machine, in which, *e.g.*, the complete manufacture of margarine can be carried out. S. S. Woolf.

Recovery of fats by extraction with volatile solvents, from emulsions stabilised with solid material. A. Marx (F.P. 609,806, 30.4.25).—The emulsion and the solvent circulate through apparatus comprising a suitable number of mixers and centrifugal separators for separating the fat from the solvent. L. A. Coles.

Apparatus for treating fish meal, guano, or the like products by means of a solvent for the recovery of residual oils or fats. G. W. Bradshaw (E.P. 270,869, 30.3.26).—Material to be extracted is placed in a perforated cylinder, which is disposed in an outer cylinder so that an annulus is left between them. Solvent, under increased pressure or temperature if desired, is led into the material by means of a perforated central

tube placed axially in the inner cylinder, and percolates radially through the mass, the extract being discharged from the annulus. A filtering medium may be wrapped round the inner cylinder. The reverse process (of inward radial percolation) is also claimed.

S. S. Woolf.

Cooling apparatus for fatty emulsions and the like. W. G. Schröder (E.P. 269,416, 15.10.26).—Fatty emulsions are air-cooled by a reverse current of cold air in the jacket of a revolving drum which is nearly filled by an inserted hollow body, the fluid circulating through the annular space thus left, in a tortuous path induced by a series of staggered circumferential ribs on the inside walls of the drum. The external cooling means is improved by the provision of a heat exchanger, which is built up with the main cooler, and through which the cooling air is circulated. The receiver for the charge is fitted with a fluted roller which acts as stirring gear, producing a constant axial flow of emulsion. Solidified material is removed from the drum by means of a knife to which is given a reciprocating movement parallel to the axis of the drum, thus causing complete removal of the deposit and at the same time keeping the knife edge sharp. The apparatus may be used in the manufacture of margarine. S. S. Woolf.

Preparation of fats of a plastic and pliable consistency. K. Erslev (E.P. 269,384, 17.8.26).—Small proportions of gummy plasticisers, *e.g.*, unvulcanised rubber, rubber latex, chicle, etc., either alone or dissolved in suitable solvents which may be subsequently recovered, are incorporated into naturally occurring hard and brittle fats, *e.g.*, cacao butter, palm-kernel fat, rendering them pliable without greatly lowering their m.p. The process may be accelerated by leading a heated and preferably inert gas through the mass. S. S. Woolf.

Condensation apparatus for preparation of pale fatty acids. Continentale A.-G. für Chemie, Assees. of R. Tern (G.P. 436,890, 2.3.26).—A system of three connected rows of vertical air-coolers, those in the central row having greater cross-section than the others. By means of an air pump the volatile distillate is led to the top of the middle coolers, and the condensate is drawn off from the bottom of the outer ones.

S. S. Woolf.

Polymerisation of oils. S. Cabot (E.P. 250,538, 10.2.26. Conv., 10.4.25).—A readily polymerisable oil is heated with a non-volatile and more slowly polymerising oil in which it is soluble, the polymerised product of the first oil remaining colloidally dispersed in the second without gelatinising the mass. A typical example is the heating of 1 pt. by wt. of tung oil with 2 pts. by wt. of linseed oil for 1 hr. at 300°. S. S. Woolf.

Vitamins from cod-liver oil (U.S.P. 1,629,074).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Settling of oil paints. A. V. Blom (Farben-Ztg., 1927, **32**, 2082—2083).—Paste red lead was shaken up with linseed oil, alone and thinned with light petroleum, turpentine, and ether, the volume ratio pigment : medium being kept constant. As anticipated, the rate of settling varied inversely as the viscosity of the medium, whilst the final volume of sediment lay between the volume occupied by the packed dry pigment and the actual volume of solid particles. On pouring off the supernatant liquor, replacing it by various solvents, and re-shaking, the rate of settling and final volume of sediment were anomalous. The probable causes for these anomalies are discussed, the wetting, solvating, and flocculating action of the medium being considered.

S. S. Woolf.

Linseed oil stand oil. H. Wolff (Farben-Ztg., 1927, **32**, 2019—2020).—From consideration of the analytical constants of five samples of stand oil, the empirical relationships $2\cdot7\,R - D - 0\cdot143\,I =$ (approx.) 3045, and $I_o = (R - 1458\cdot5 + 0\cdot11\,I)/0\cdot224$ (where $R = 1000\,n_D^{20}$, $D = 1000\,d^{20}$, and I = iodine value of the stand oil, whilst I_o = iodine value of the original oil) are developed from earlier relationships of similar character due to Lund, saponif. value, which appeared in the latter's expressions, being incorporated into the constant term. By the use of these relationships, information may be obtained as to the purity of linseed oil stand oils in the absence, which must be established, of tung oil. S. S. Woolf.

Rubber pigments. Ditmar.—See XIV.

Patents.

Manufacture of a substitute for turpentine. L. L. Odom, Assr. to M-O-R Products Co. (U.S.P. 1,625,622, 19.4.27. Appl., 18.3.24).—A mixture of rosin (100 pts.) and calcium oxide (10 pts.) is heated at 190° for 2 hrs., petroleum distillate, d 1·53, is added at 80°, and the product distilled. T. S. Wheeler.

Quenching lithopone. A. S. Krebs, Assr. to Krebs Pigment and Chemical Co. (U.S.P. 1,630,267, 31.5.27. Appl., 29.6.23).—A continuous fine stream of hot lithopone is fed into a running stream of water which conveys the quenched pigment to a settling tank.

S. S. Woolf.

Drying and calcining lithopone. W. G. Graves, Assr. to Grasselli Chemical Co. (U.S.P. 1,630,613, 31.5.27. Appl., 23.1.26).—The entire drying and calcination of lithopone is carried out as a continuous operation in one vessel. S. S. Woolf.

Manufacture of a zirconium oxide-silica composite. C. J. Kinzie, Assr. to Titanium Alloy Manuf. Co. (U.S.P. 1,618,288, 22.2.27. Appl., 6.11.25).—A zirconium ore or mineral, *e.g.*, zirconium silicate, is heated with an equal weight of sodium carbonate at 900°, and the product is ground, washed with water, and treated with sodium chloride and a 50% solution of sulphuric acid. On being heated and stirred the mixture rapidly solidifies. It is calcined at 1000° and the product is ground, washed and dried. It consists of an intimate mixture of zirconium dioxide and silica, and is of value in the manufacture of pigments. T. S. Wheeler.

Writing fluids. Marconi's Wireless Telegraph Co., Ltd., Assees. of R. H. Ranger (E.P. 257,954, 4.9.26. Conv., 5.9.25).—A freely flowing, non-clogging, quick-drying writing fluid, particularly designed for use in radio-transmission of pictures, consists of a base of waxy material, *e.g.*, paraffin wax, to which is added colouring matter, *e.g.*, aniline dyes. The mixtures are usually

solid at ordinary temperatures, and must be heated above their m.p. before use. S. S. Woolf.

Production and utilisation of dichroic inks. C. Bittinger (U.S.P. 1,629,250, 17.5.27. Appl., 3.1.25).—A pigment, *e.g.*, Paris Green, is ground in a vehicle to form a coloured ink, with which an organic dye of a different colour, *e.g.*, Methyl Violet, is then mixed. The pigment colour is predominant until the ink is subjected to mechanical pressure, when the dye colour is obtained. By varying the pressure in printing with this ink it is possible to produce one or other colour. T. S. Wheeler.

Purification of condensation products produced from phenols and aldehydes. Bakelite G.m.b.H. (E.P. 248,726, 17.2.26. Conv., 6.3.25. Addn. to E.P. 247,956; cf. G.P. 431,514; B., 1926, 889).—Phenol-aldehyde condensation products are treated, in the presence of water, with metallic oxides or hydroxides, other than the alkali hydroxides or compounds capable of furnishing such substances, in amount sufficient to neutralise free phenols without combining with the artificial resins. The condensation products are precipitated by water or by aqueous solutions of substances of hydrotropic action. Solvents for the condensation products may be added prior to the treatment with oxides etc., and filling substances may be present. The formation of resin salts, to which the process described in the prior patent was liable, is avoided, and greater economy is attained. S. S. Woolf.

Manufacture of synthetic resins. Commercial Solvents Corp., Assees. of E. R. Littmann, B. K. Brown, and W. J. Bannister (E.P. 250,265, 1.4.26. Conv., 1.4.25).—The multivalent metal salts of alkyl hydrogen phthalates, formed from an alkali alkyl phthalate and a soluble salt of the metal, are friable insoluble resins. The following salts of *n*-butyl hydrogen phthalate are described: *Zinc*, m.p. 150°; *lead*, pale amber; *ferric*, ruby-red; *cupric*, bluish-green. C. Hollins.

Manufacture of homogeneous glass-like condensation products of carbamide and formaldehyde. K. Ripper, Assr. to F. Pollak (U.S.P. 1,625,283, 19.4.27. Appl., 9.9.22. Conv., 31.5.21).—See E.P. 181,014; B., 1923, 988 A.

Condensation product. H. C. P. Weber, Assr. to Westinghouse Electric & Manuf. Co. (U.S.P. 1,630,365, 31.5.27. Appl., 24.9.20).—See E.P. 169,451; B., 1922, 978.

Grinding mill (E.P. 270,465).—See I.

Photographic varnish (E.P. 270,387).—See XXI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Nature of matured rubber. II. G. Bruni and T. G. Levi (Giorn. Chim. Ind. Appl., 1927, **9**, 161—164).—The aqueous fraction, obtained when the acetone extract of matured slab rubber is shaken with ether and water, contains (1) 10% of a mixture of an α-aminovaleric acid and two α-aminohexoic acids, one giving a readily soluble and the other a sparingly soluble copper salt; these acids, which have no influence on the vulcanisation, are obtained also by similar treatment of smoked sheet. (2) About 20% of tetra- and pentamethylenediamines, which greatly accelerate vulcanisation. (3) More than 30% of potassium phenylacetate, which, like the potassium salts of other fatty and fatty-aromatic acids, is an energetic accelerator of vulcanisation; the corresponding sodium salts are less active in this respect. Potassium salts of aromatic acids are weak accelerators, and the sodium salts are almost inactive. The ethereal fraction of the acetone extract contains phytosterol, which does not affect vulcanisation. T. H. Pope.

Röntgen-spectrography of rubber and of similar extensible substances. Amorphous rings and their alterations with extension. J. R. Katz (Chem.-Ztg., 1927, **51**, 381—385).—A review of the subject dealing with the significance of the "amorphous spectra" of liquids, the behaviour of polymerides and substances with several similar side-chains, the broad amorphous rings shown by various forms of rubber, the dividing of these rings when the rubber is stretched, and the view of the structural condition of rubber and similar extensible substances as intermediate between that of the amorphous and crystalline states. D. F. Twiss.

***Hevea* latex. VI. Proteins in serum from frozen latex.** R. O. Bishop (Malayan Agric. J., 1927, **15**, 27—34).—Fresh latex was frozen for several days until on thawing it gave a clear serum; this showed the same p_H as the original latex, viz., 6·5. By successive acidification to p_H 4·5, saturation with sodium chloride, and heating at 100° three protein substances were obtained designated A, B, and C. After purification by dialysis, protein A retained a compound containing phosphorus and nitrogen which was extractible by alcohol. No indication of a lipin complex was obtained. The serum had d^{30} 1·015 (average), and the total yield of proteins was 0·6% by wt. of the serum. D. F. Twiss.

Determination of sulphur in rubbers. E. Kahane (Caoutchouc et Gutta-Percha, 1927, **24**, 13,549—13,550).—1 g. of the sample is added to a mixture of 10 c.c. of nitric acid and 5 c.c. of perchloric acid. In the vigorous reaction, induced by gentle heating if necessary, the rubber disappears. The mixture is then heated strongly when it first darkens and then clears. After these operations, which occupy from 7 to 8 min., the precipitation of barium sulphate is effected in the customary manner. The use of a mixture of 5 c.c. of perchloric acid with 5 c.c. of sulphuric acid for the oxidation of 1 g. of rubber also enables a Kjeldahl determination of nitrogen to be completed in 10 min. D. F. Twiss.

Jelutong. B. J. Eaton, C. D. V. Georgi, and G. L. Teik (Malayan Agric. J., 1927, **15**, 65—77).—Jelutong latex has p_H 7·0 (approx.) when fresh, but after 24 hrs. the value may attain 5·5. An average sample showed total solids 19·87%, ash 0·03%, coagulum (by alcohol) 15·54%, nitrogen 0·025%. The latex contains a large proportion of gummy material and sugars. Satisfactory coagulation of fresh latex can be effected by acidifying to p_H 5·0 with acetic acid (0·1 g. of acetic acid per 100 c.c. of latex), and boiling for a few minutes. Satisfactory coagulation is also produced by formic acid (0·05 g.), sulphuric acid (0·02 g.), and potash alum

(0·05 g.). The liability of jelutong to deterioration can be reduced by immersion in boiling water for 10 min. and then pressing. The removal of soluble organic matter thus effected reduces the tendency to fungoid development. D. F. Twiss.

Effect of the colour of inorganic and organic rubber pigments on organic accelerators of vulcanisation. R. Ditmar (Chem.-Ztg., 1927, **51**, 332—333).—The behaviour of a number of rubber mixtures, selected to reveal the influence of various organic accelerators on such pigments as vermilion, antimony sulphide, and a number of "Vulkan" organic colours, previous to or during vulcanisation between glass plates at 135°, indicates that this follows no simple general rule. It is necessary to make a practical test of the influence of each accelerator on each pigment under varying conditions. D. F. Twiss.

Barytes and its employment in the rubber industry. T. R. Dawson and N. H. Hartshorne (India-rubber J., 1927, **73**, 885—889, 926—930, 961—966).—Twenty samples of commercial barytes were submitted to a comprehensive chemical and physical examination. Particle size was investigated by elutriation and sedimentation methods, a simplified form of apparatus being described for the latter. Comparative vulcanisation experiments were made with two samples, the vulcanisates being examined as to tensile strength, elongation, stress–strain curve, permanent set, sp. gr., hardness, and colour. The results indicate that, for use in rubber, particle size is easily the most important feature, the practice of grading barytes according to colour bearing no rational relation to the properties desirable for rubber compounding. D. F. Twiss.

Patents.

Production of homogeneous rubber deposits from rubber latex. Anode Rubber Co., Ltd. (E.P. 257,885, 23.2.26. Conv., 4.9.25. Cf. E.P. 223,189; B., 1925, 46).—In order to avoid the anodic liberation of gases during the electro-deposition of rubber from rubber latex on a metallic anode such as zinc, cadmium, iron, or lead, the ammonia concentration of the latex should be at most 0·1*N*. Reduction of the proportion of ammonia in preserved latex can be effected by dialysis, aeration, partial evaporation, addition of formaldehyde (with formation of hexamethylenetetramine) or other carbonyl compounds, or even neutralisation. Alternatively, latex may be used which has been preserved with a less ionised substance, either alkaline or germicidal. Mere dilution of ammonia-preserved latex is not satisfactory as it adversely affects the deposition of the rubber. A further alternative is to effect deposition under such conditions that the anode potential is insufficient for electrolysis with gas formation; this, however, involves slow deposition. The condition of the rubber deposit formed can be modified by previously coating the anode with a liquid-absorbing substance, *e.g.*, gelatin or collodion; the anode surface also may be provided with any desired pattern, and individual parts may be differently constituted so as to give a porous or smooth rubber deposit. D. F. Twiss.

Manufacture of rubber goods. Anode Rubber Co., Assees. of P. Klein and A. Szegvári (E.P. 253,069, 12.1.26. Conv., 5.6.25).—In order to avoid the possibility of difficulty arising from undesirable features in certain ingredients for latex mixtures used in the manufacture of goods by electrophoretic methods, these ingredients are combined with a conditioning substance or substances such that in the particles of the resulting combination the undesirable characteristics are diminished. For example, oils or litharge may be blended with kieselguhr; sulphur may be used with kieselguhr, lamp-black, clay, or even with vulcanised rape oil; zinc oxide may be used as a dispersion of the product obtained by heating with sulphur and rape oil. [Reference is directed, in pursuance of Sect. 8, Sub-sect. 2, of the Patents and Designs Acts, 1907 and 1919, to E.P. 254,765.] D. F. Twiss.

Production of rubber goods directly from [rubber] latex. Anode Rubber Co., Ltd. (E.P. 252,673, 23.2.26. Conv., 30.5.25).—When a porous mould is immersed in rubber latex, preferably concentrated and, if desired, compounded, a layer of agglomerated rubber forms on the surface. The agglomeration may be increased by applying suitable agents such as acetic acid, calcium chloride, or zinc chloride to the side of the mould which is not in contact with the latex; it may also be aided by accelerating the migration of the serum through the mould, *e.g.*, by using a hollow mould and applying a slightly reduced pressure or promoting evaporation in the interior. Water-absorbing substances also may be introduced inside the porous mould. D. F. Twiss.

Rubber compounds and articles. Simplex Wire & Cable Co., Assees. of C. R. Boggs and J. T. Blake (E.P. 269,124, 24.7.26. Conv., 6.4.26).—Composite rubber products resistant to the action of ozone are made with an elastic portion imparting the desired physical characteristics covered with a protective plastic portion free from internal stress. A suitable resistant plastic gum comprises balata (35 pts.), reclaimed rubber (5 pts.), and oil substitute (60 pts.); the elastic inner material may be of any of the customary rubber "compounds." The invention is of especial value in connexion with insulated electrical cables. D. F. Twiss.

Manufacture of vulcanite and of composite materials, including vulcanite. H. P. Stevens (E.P. 269,693, 18.1.26).—Vulcanite is produced by heating a mixture of rubber, sulphur, and a powerful accelerator, *e.g.*, zinc diethyldithiocarbamate, at a low temperature, *e.g.*, below 100°, for a prolonged period, such as 10 days. The mixture may contain other additional ingredients, and may be converted into solution for coating articles before vulcanisation. D. F. Twiss.

[Precipitation] treatment of rubber. W. G. O'Brien, Assr. to Goodyear Tire & Rubber Co. (U.S.P. 1,628,326, 10.5.27. Appl., 22.3.23).—For the manufacture of a rubberised fibrous composition, rubber solution, after treatment with hydrogen sulphide and sulphur dioxide, is diluted before gelation, and is then mixed with loose fibrous material and with agents for the

precipitation of the rubber on the fibres. The fibres are then removed from the liquid and allowed to gel.
D. F. Twiss.

Combining halogen-containing materials with rubber etc. C. E. Bradley and W. A. Gibbons, Assrs. to Naugatuck Chemical Co. (U.S.P. 1,627,725, 10.5.27. Appl., 20.6.21).—"Chlorinated rubber hydrochloride" is obtained by treating a mixture of rubber and solvent with hydrogen chloride and chlorinating the reaction product. D. F. Twiss.

Reclaiming rubber. M. J. Fessler (U.S.P. 1,626,800, 3.5.27. Appl., 29.1.27).—Old or used rubber stock is comminuted and then treated with tetrahydronaphthalene. D. F. Twiss.

Manufacture of a new jelutong product. S. S. Yates (E.P. 271,329, 30.10.26. Conv., 19.6.26; cf. E.P. 245,405; B., 1926, 555).—Jelutong or pontianac is prepared by coagulation of the latex, *e.g.*, with acetic acid or alum. Drying is then effected until a very low proportion of water remains, *e.g.*, 5% or less, instead of the customary 40% or more. The product is obtained in the form of sheet or compact material which can be transported without objectionable deterioration and needs a minimum of refining. D. F. Twiss.

Manufacture of an age-resisting rubber compound. E. R. Bridgwater and D. H. Powers, Assrs. to E. I. du Pont de Nemours & Co. (U.S.P. 1,628,528, 10.5.27. Appl., 3.7.26).—Substances, *e.g.*, *p*-aminophenol, which inhibit the ageing of rubber, can be readily dispersed through it prior to vulcanising if they are first intimately mixed with an equal weight of a finely-divided inorganic substance, *e.g.*, barium sulphate, calcium carbonate, or zinc oxide. T. S. Wheeler.

Preparation of a rubber composition. H. W. Elley, Assr. to E. I. du Pont de Nemours & Co. (U.S.P. 1,624,724, 12.4.27. Appl., 5.5.26).—Ageing of rubber is inhibited by mixing a nitroaminophenol, *e.g.*, *p*-nitro-*o*-aminophenol, with it. T. S. Wheeler.

Vulcanisation of rubber. N. A. Shepard and S. Krall, Assrs. to Firestone Tire & Rubber Co. (Reissue 16,648, 7.6.27, of U.S.P. 1,576,072, 20.6.23).—See B., 1926, 453.

XV.—LEATHER; GLUE.

Mechanism of tanning. K. H. Gustavson (J. Amer. Leather Chem. Assoc., 1927, **22**, 236—243).—Portions of ordinary and deaminised hide powder, respectively, were tanned for 48 hrs. with different basic chromium chloride and sulphate solutions respectively, and also at p_H 5·0—the isoelectric point. The p_H value of each liquor was increased to 5·0 by adding the same amount of sodium formate. The p_H values of the exhausted liquor and the chromium content of the tanned powders were determined. The chromium content was lower in the case of tannage at the isoelectric point for a 50% acid chromium chloride solution and for the 41% and 63% acid chromium sulphate solutions, but higher for the 61·5% acid chromium chloride solution. In the deaminised series, the 41% acid chromium sulphate at ordinary p_H value showed a much greater percentage drop in chromium content than the other chromium salts. The results favour the view that the inhibition of cationic chromium fixation by hide protein possessing a less number of reactive basic groups than ordinary hide protein is connected with the diminished acid combining capacity of the structurally altered hide protein. There are disturbing factors, *e.g.*, the formato-complexes, the effect of which is not known. Experiments with ordinary hide powder, powder treated with a buffer solution of p_H 8·0, and powder tanned with formaldehyde at p_H 8·0, using a 61·5% acid chromium chloride solution, showed greater increase in the rate of chromium fixation by hide powders pretreated at p_H 8·0 whether formaldehyde-tanned or only treated with a buffer solution. Chemical changes involving the structure of the protein and its degree of chemical activation, besides colloidal alterations of the micells, are probably the cause of this increase.
D. Woodroffe.

Standardisation of hide powder. III. H. G. Bennett (J. Soc. Leather Trades' Chem., 1927, **11**, 87—93; cf. B., 1927, 52).—The alkalinity of hide powders due to organic matter is usually greater in influence than that due to inorganic sources. Determinations of the soluble matter for any one type of manufacture of hide powder yield results which vary concomitantly with the results both of acidity and of alkalinity determinations. Determinations of mineral ash and its alkalinity may or may not vary in the same sense. The absorptive properties of a powder are best indicated by determinations of the soluble matter and its mean variation. The "alkalinity" or "acidity" of the soluble matter is a minor factor. The percentage of ash and its alkalinity are indications of adsorptive power only as they influence the soluble matters present. The alkalinity of the powder should be determined by observing the change in p_H caused by the infusion of 6·5 g. of hide powder in 100 c.c. of saturated boric acid solution, and by titrating the alkalis extracted from hide powder by three successive infusions in saturated boric acid. The results must be related both to the weight of the powder and to the weight of the soluble matter determined. Experiments with two hide powders from the same source showed that high non-tans were obtained with the powder containing a higher proportion of soluble matter and having a higher acidity and alkalinity. The latter are due to amphoteric, soluble organic constituents. D. Woodroffe.

Use of sulphite-cellulose extract as a tanning material. E. L. Wallace and R. C. Bowker (U.S. Bur. Standards, 1927, Tech. Paper No. 339, 309—321).—Experiments have shown that sulphite-cellulose extracts contain a satisfactory amount of material absorbable and fixed by hide powder. They compare favourably with vegetable tanning extracts in these respects. They may be blended with ordinary vegetable tanning extracts without loss of tannin content, and give a satisfactory colour on leather used either alone or in mixtures. They brighten the colour of most tanning materials on leather, moderate the red colour of quebracho and mangrove, and show a marked brightening effect on chestnut extract. They solubilise some of the quebracho insoluble matter. Pieces of hide pre-tanned in sulphite-cellulose liquors of ½%, 1%, and 1½% tannin content

and finished in ordinary mixed tanning extracts required 28 days only as compared with 40 days for hides not pre-tanned. Blends of equal parts of sulphite-cellulose extract, chestnut, and mangrove gave as good a tannage in 35 days as required 40 days with oak bark extract. The chemical, physical, and ageing properties of the leather tanned with sulphite-cellulose extracts compared favourably with those of leathers tanned without their use. D. Woodroffe.

Unhairing action of stannous salts. H. B. Merrill (J. Amer. Leather Chem. Assoc., 1927, **22**, 230—236).—Three samples of hair were treated with saturated lime water, 1% stannous chloride for 24 hrs. followed by lime water for 24 hrs., and 1% stannous chloride solution for 48 hrs. respectively. The lime water alone and stannous chloride solution alone had little action on the hair, but the combined treatment of stannous chloride solution followed by lime water completely disintegrated the hair. Stannic chloride had no such effect. The stannous chloride can be used instead of sodium sulphide and in similar quantity for sharpening milk of lime for loosening the hair on raw skin. The effect of the stannous chloride is attributed to its reducing properties. It acts on hair only in alkaline solution. Its action confirms the theory of Atkin and Thompson (Leather Trades' Year Book, 1926, 56) that sulphides reduce cystine to cysteine, which latter acts as an oxygen carrier, thus oxidising the proteins of the hair and epidermis.

D. Woodroffe.

Patents.

Manufacture of ligno-tanning material from waste sulphite liquor. V. Drewsen, Assr. to West Virginia Pulp & Paper Co. (U.S.P. 1,629,448, 17.5.27. Appl., 14.2.19. Cf. U.S.P. 1,303,176—7; B., 1919, 507 A).—Waste sulphite-cellulose liquor is heated at 130° under pressure with 2% of calcium oxide for about 1 hr. The liquor is then treated with carbon dioxide, filtered, and concentrated, and sufficient sulphuric acid to precipitate the calcium present as calcium sulphate is added. The filtrate is of value as a tanning agent.

T. S. Wheeler.

Solution for depilating hides. H. Benfey (G.P. 436,149, 5.12.24. Cf. E.P. 246,114; B., 1926, 505).—Sodium hydrosulphide solution, *d* 1·22, containing 12·6% of active sulphur, is obtained by scrubbing waste gases from the manufacture of carbon disulphide with a mixture of sodium sulphide solution, *d* 1·24, and milk of lime, *d* 1·16, and subsequently allowing the calcium hydroxide sludge to settle. L. A. Coles.

Leaching apparatus (U.S.P. 1,628,787).—See I.

XVI.—AGRICULTURE.

Effect of soil micro-organisms on paraffin used as a coating to decrease the injurious action of lead arsenate on plant roots. W. E. Fleming (J. Agric. Res., 1927, **34**, 335—338).—Soil micro-organisms, especially various species of fungi, are able to decompose paraffin in the soil and convert it into organic acids. The growth of many soil fungi was markedly stimulated in the presence of paraffin, whether added alone or as a coating on particles of lead arsenate. This would explain the results of Leach (cf. B., 1926, 840), who found that the injury to plants caused by lead arsenate, applied to the soil as an insecticide, was not reduced when the particles were coated with paraffin.

C. T. Gimingham.

Determination of organic matter in soils by means of hydrogen peroxide. W. O. Robinson (J. Agric. Res., 1927, **34**, 339—356).—A method is described for the determination of organic matter in soils by the loss in weight on digestion with hydrogen peroxide. About 1 g. of soil is digested on the water-bath with 10 c.c. of water and 10 c.c. of 30% hydrogen peroxide until bubbles cease to be evolved. The liquid is then filtered through asbestos in a Gooch crucible, and the filtrate and washings are evaporated to dryness, ignited, and weighed. The insoluble matter on the pad is dried for 18 hrs. at 110°. The difference in weight between the water-free soil and the dried residue plus the ash from the filtrate, is taken as organic matter. In some soils an appreciable amount of organic matter is unattacked by hydrogen peroxide; in certain cases this consists mainly of charcoal. Graphite is not attacked; charcoal and coal are partially decomposed. The method tends to give low results, but it is on the whole about as accurate as the combustion method, and, in special cases, much more accurate. It is not applicable to soils high in calcium carbonate, manganese dioxide, or chromium sesquioxide. Various forms of organic matter, including cellulose, humus, and lignite are almost completely destroyed by hydrogen peroxide in the presence of soil, and the method does not determine any clearly defined kind of soil organic matter. The author does not therefore agree that hydrogen peroxide can be used to differentiate between humified and non-humified material, as proposed by G. W. Robinson and Jones (cf. B, 1925, 140).

C. T. Gimingham.

Alkaline and neutral permanganate methods [for nitrogen determination]: comparison of results on raw materials and fertiliser mixtures. H. C. Moore and R. White (J. Assoc. Off. Agric. Chem., 1927, **10**, 202—216).—The results of over 2000 determinations of the "activity" of fertilisers by the alkaline and neutral permanganate methods are summarised, and attention is drawn to the discrepancies. Both methods are empirical, and certain points in the official regulations need to be made more explicit to ensure greater uniformity. The following possible sources of variation are indicated. Samples should be ground just fine enough to pass a 1-mm. sieve. The exact amount of material should be taken in determining water-soluble nitrogen, so that 50 mg. of water-insoluble nitrogen are left in the residue. In the alkaline method the distillation should be carried out in a definite time, *e.g.*, 1 hr., and exactly 95 c.c. of distillate should be collected. The addition of paraffin to prevent frothing leads to a reduction of some permanganate; the use of glass beads is preferable. In the neutral method, complete preliminary extraction with water is essential, as some of the water-soluble nitrogen may be rendered insoluble by the subsequent permanganate digestion. The complete methods embodying these modifications are described. H. J. Dowden.

Modification of the salicylic-thiosulphate method suitable for determination of total nitrogen in plants, plant solutions, and soil extracts. E. R. Ranker (J. Assoc. Off. Agric. Chem., 1927, **10**, 230—251).—The salicylic-thiosulphate method of determining total nitrogen in fertilisers, which is based on the reduction of nitrates in acid medium, was found to give low results when applied to plants and plant materials, recoveries of 27—68% being secured. By testing the vapours for nitrates, nitrites, and ammonia at each individual stage of the determination, the operations in which losses of nitrogen may occur have been detected, and modifications of the procedure to counteract these losses are suggested. The sample must first be rendered neutral or faintly alkaline, by addition of a predetermined quantity of acid or alkali and not by colorimetric adjustment in the presence of an indicator, since the latter method leads to loss of nitrogen during the subsequent evaporation. If water is present, the sample must be evaporated just to dryness under reduced pressure, on a waterbath. Evaporation to ash dryness may in some cases cause loss of nitrogen during the drying, whilst incomplete evaporation will involve losses during the addition of the acid and during the subsequent digestion. The method as thus modified showed small limits of error when compared with the Devarda alloy method. H. J. Dowden.

Determination of carbon and nitrogen on the same soil sample. B. E. Brown (Ind. Eng. Chem., 1927, **19**, 629—630).—The determination is made with fair accuracy by the wet-combustion method. After the carbon has been converted into carbon dioxide by the action of the chromic-sulphuric acid mixture, the nitrogen is present in the residue as ammonium sulphate, and is determined as ammonia in the usual way. Chromic acid gives more satisfactory results than mercuric oxide or manganese dioxide. W. J. Powell.

Inaccuracies of the Devarda method when applied to plant materials. E. R. Ranker (J. Assoc. Off. Agric. Chem., 1927, **10**, 252—256).—The Devarda method for determining nitrate-nitrogen, although extremely accurate for soils when applied to plant extracts and biological materials, often gives rise to erroneous results. Samples were selected which had been proved free from nitrates, nitrites, and ammonia by tests with diphenylamine and with Nessler's solution. Out of 24 samples, 15, including materials such as sugar cane water-extract, germinated pea seed, carbamide, and glycine, gave a positive nitrate nitrogen determination, although none was present. To avoid incorrect deductions from these results, it is recommended that nitrogen determined by this method should be described as "Devarda nitrogen" rather than "nitrate-nitrogen." H. J. Dowden.

Patents.

Manufacture of materials serving both as means for destroying plant pests and as fertilisers. M. Lange (G.P. 438,006, 10.12.20).—The materials consist of solutions of nitrated hydrocarbons or phenols in sulphuric acid to which phosphates are added. *E.g.*, sulphuric acid, containing 10% of its weight of nitrated hydrocarbons, or phenols or their homologues, is mixed with phosphorite in the proportions required to render the phosphate soluble. A distributable powder is obtained, which, in use, does not harm plant roots. C. T. Gimingham.

Fertiliser. E. Cerasoli (E.P. 270,957, 16.10.26).—Residues from the distillation of beetroot molasses are mixed with kieselguhr and phosphorite or calcium sulphate at ordinary temperature. C. T. Gimingham.

Insecticide composition. O. F. Hedenburg and D. S. Pratt, Assrs. to Toledo Rex Spray Co. (U.S.P. 1,627,389, 3.5.27. Appl., 1.7.19).—An insecticide comprises lead arsenate and an emulsoid colloid, such as gum arabic, mixed with lime-sulphur. C. T. Gimingham.

Manufacture of calcium arsenate [insecticide] products. W. J. Liipfert, Assr. to General Chemical Co. (U.S.P. 1,626,942, 3.5.27. Appl., 6.2.24).—An aqueous suspension of calcium hydroxide is treated under agitation at below 38° with arsenic acid solution, and the paste obtained is heated at the b.p. and dried. T. S. Wheeler.

Manufacture of an insecticide. W. C. Piver (U.S.P. 1,626,872, 3.5.27. Appl., 30.3.23).—Arsenic acid is treated with sufficient excess of calcium oxide to yield a dry product, and with 0·5—5% of aluminium sulphate. T. S. Wheeler.

Solutions for destroying animal and plant pests. F. Hoffmann-La Roche & Co. A.-G. (Swiss P. 114,438, 14.11.24).—The solutions contain nicotine soaps of the higher fatty acids, resin acids, or naphthenic acids. L. A. Coles.

Material for destroying animal and plant pests. Farbenfabr. vorm. F. Bayer & Co. (F.P. 595,974, 31.3.25. Conv., 10.4.24).—Copper compounds of aromatic bases, such as aniline and its derivatives and homologues, prepared by treating the bases with copper sulphate solution, are used for the purpose. L. A. Coles.

XVII.—SUGARS; STARCHES; GUMS.

Refining qualities of raw [cane] sugars. T. B. Wayne (Int. Sugar J., 1927, **29**, 94—100).—It is fallacious to grade raw cane sugars for their refining value strictly on polarisation, as this basis of purchase does not differentiate between the superior refining qualities of some sugars and the inferior nature of others. Characteristics of a good raw sugar are that the crystals shall be uniform, hard, and sharp, having an average diameter of about 0·8 mm., with few needle-shaped crystals. Viewed by projection of their images on a screen, the crystals should be regular and of good shape; the colour value by spectrophotometric analysis at a wave-length of 560 μ expressed as — log (sp. transmissivity), should not exceed 0·7500. With a total colour value of less than 0·7500, the higher the absorption trend at 440 μ, the more suitable the sugar is for refining purposes, since this indicates that the colouring matter present is due to caramelisation of an incipient nature, the absence of greyish colouring matter indicating improper clarification of ferric iron-polyphenol compounds. Polarisation to be 96° or more, ash at a maximum of 750° F. not exceeding 0·45%, and moisture by drying *in vacuo* at 80° to constant weight to allow a safety factor of 0·25. J. P. Ogilvie.

Evaporating and heating systems in cane [sugar] factories. G. W. CONNON (Int. Sugar J., 1927, **29**, 100—103).—Following beet factory practice, cane houses, especially in Cuba, are now following the "extra steam" system for heating the raw juice, this method reducing the total fuel consumption about 4½%. A development of this method is the use of the "dead-end" double effect, the first cell of the evaporator consisting actually of two parts, the first operated with exhaust steam, and the second sending all its vapour to the juice-heaters. There are no connexions on the steam or vapour side between these evaporators and the quadruple effect, but the partly concentrated juice from the dead-end evaporator is sent to the multiple effect to be made into syrup. In this way 8½% less steam is used, compared with the ordinary quadruple effect, the total heating surface being no greater. A number of the larger and more efficient factories in Cuba employ the pre-evaporator or Pauly cell, using live steam at 40 lb. pressure, the evaporation being discharged into the exhaust steam main. A very high rate of evaporation can be obtained, and this system provides the cheapest way to increase the capacity and economy of the evaporation department of a cane factory. One of the factories in Cuba now operates an evaporator employing thermo-compression, with a good economy in its bagasse consumption. J. P. OGILVIE.

Sugar-house incrustations. J. W. SCHLEGEL and J. P. MANLEY (Ind. Eng. Chem., 1927, **19**, 219—221).—A heavy sticky sludge forming on the upper surfaces of the char filters was found to consist mainly of silica, which had probably originated from the use of diatomaceous earth in clarification. Scales found in the outlet pipes from the low-grade char filters were found to have a similar composition. A deposit inside the flue of a char kiln was found to consist essentially of ammonium and calcium sulphates. Scale deposited on the corrugations and faces of the plates of ordinary plate-and-frame presses used for filtering sugar liquors were found to consist mainly of CaO, 21·33; Fe_2O_3 and Al_2O_3 12·41; SiO_2, 14·89; P_2O_5, 7·25; MgO, 6·48; CO_3 6·50; and SO_3, 0·91%. Examination of yellowish-brown stalactitic deposits around the caps of water-tube boilers, caused by leakage from within the boilers, showed that traces of waste-waters from the char filters had leaked into the boilers where it had interacted with the soda ash present. Analyses were also made of the scales found in the Hoppes water purifier, and of sludge taken from a return steam-trap. J. P. OGILVIE.

Beet dehydration process. B. J. OWEN, L. F. MANÉS, and J. L. DOUGAN (J. Min. Agric., 1927, **33**, 986—996).—Cossettes are dehydrated in a single operation (without the preliminary drying advocated by de Vecchis) by a drying agent, *e.g.*, heated air, continuously and uniformly applied until their temperature is raised to about 93°, and the water content has been reduced to about 5% of the weight of the product. This drying may be effected either in mass or in layers, and plant is described for doing so on the large scale in field or factory. An extraction apparatus for exhausting the dried cossettes is employed by means of which the sugar may be obtained in the form of a dense syrup in continuous and expeditious manner, this being in essence a vertical vessel to which the material is fed continuously and impelled upwards, being exhausted by means of a counter-current of water caused to gravitate through the ascending mass. In purifying this dense syrup, it is treated with phosphoric acid, heated at 80°, and limed to neutrality; or it may be heated, centrifuged, and treated with a solution of phosphoric or sulphurous acid, followed by a suitable quantity of dry tricalcium sucrate or dry calcium hydrate, the alkalinity later being adjusted by means of phosphoric acid. This method ensures that the syrup can be filtered under comparatively low pressure. J. P. OGILVIE.

Determination of phosphates in sugar-cane juice. H. B. SPRINGER and J. G. DAVIES (J.S.C.I., 1927, **46**, 143—144 T).—In determining the phosphate content of cane juice as a guide to the manurial requirements of the soil (cf. Walker; B., 1923, 322 A), a modification of the Pemberton–Neumann molybdate method is used, which is stated to give rapid and reasonably accurate results, even in the presence of the usual amounts of silicate occurring in cane juice. The uranium acetate procedure followed by Walker gives results lower than the truth. J. P. OGILVIE.

Determination of sugar in the beet and the use of the Krüger method. A. LE DOCTE (Int. Sugar J., 1927, **29**, 214—218).—The Krüger method is considered unreliable because the volume of liquid added to 26 g. of beet pulp is too small to obtain a perfect admixture and a complete diffusion of the sugar within a reasonable time, and is also too small to obtain rapidly a sufficient quantity of filtrate for use with the Pellet continuous polarimetric tube. Compared with the Sachs and Le Docte procedure, the method is open to criticism on the grounds that it is less rapid and more liable to error in factory routine operation. J. P. OGILVIE.

Hydrogen-ion concentration and the defecation of cane juice. H. S. PAINE and R. T. BALCH (La. Planter, 1927, **73**, 127—132, 148—150).—Provided that sufficient filtration capacity be available, the procedure to ensure a maximum clarification and to prevent inversion losses is to lime to p_H 8—9, measured either before or after heating, but the hydrogen-ion concentration for the maximum defecation varies with different juices, and in any case the possibilities of colour formation and of scaling of the tubes of evaporators and heaters are to be considered. J. P. OGILVIE.

Use of hyposulphites in the [beet] sugar factory. M. MESTRE (Bull. Assoc. Chim. Sucr., 1926, **44**, 72—79).—Treatment of the juices and syrups with hyposulphites prevents the oxidation of insoluble gelatinous compounds and of soluble metapectic compounds into substances that considerably add to the viscosity, and retard boiling and crystallisation. J. P. OGILVIE.

Use of chlorine for [beet] juice purification. O. SPENGLER and R. WEIDENHAGEN (Z. Ver. deut. Zuckerind., 1927, 119—121).—A comparison of the results obtained by Ochi's process (B., 1926, 559) with the usual method of clarification showed that on treatment of the juice with chlorine followed by 2% of "Norit," the purity was 90·8°; whereas with 2·5% of lime in ordinary carbonation it was 94·2°. In order to reach a purity

of 95°, it was necessary to use 5% of the decolorising carbon. In applying the process to the purification of beet molasses, it was not possible to effect a sufficient incorporation of chlorine with the dense liquid.

J. P. Ogilvie.

Error caused by the presence of invert sugar in the determination of sucrose in the beet by the aqueous digestion method. V. Staněk and J. Vondrák (Z. Zuckerind. Czechoslov., 1927, **51**, 220—224).—Under the influence of the basic lead acetate in the hot aqueous digestion method, the invert sugar which may be present suffers a loss of its lævo-rotatory power, and may even become dextro-rotatory, its cupric reducing power only slightly diminishing. If the invert sugar content is considerable, it is not exact to correct the polarisation by increasing it by one third of the invert sugar present. It is then necessary to determine the sucrose by double polarisation, or else to ascertain the change of polarisation of the invert sugar present caused by sodium hydroxide at the temperature of operation.

J. P. Ogilvie.

Patents.

Extraction of juice from sugar cane [by milling]. W. H. Morgan, sen. (E.P. 267,234, 14.12.25).—Each cane stalk is divided into short sections which are disintegrated longitudinally during their movement between relatively movable blades to produce a long-fibred, finely-shredded absorbent mass, the juice being finally removed from it by pressure or by diffusion.

J. P. Ogilvie.

Preparation of sugar cane stalks prior to the extraction of juice therefrom. W. H. Morgan (E.P. 267,235, 14.12.25).—Cane stalks are prepared for juice extraction (by milling or otherwise) by removing extraneous matter by washing, cutting the stalks into short-length sections, shredding, and reducing them to a clean fibrous hay-like mass containing the juice. The removal of the extraneous matter is effected during the movement of the stalks in a liquid bath in which they are subjected to attrition.

J. P. Ogilvie.

Apparatus for concentrating and evaporating liquids, such as syrups. Baker Perkins, Ltd., G. R. Baker, W. E. Prescott, and Soc. Anon. Anc. Etabl. A. Savy Jeanjeon & Cie. (E.P. 268,437, 1.1.26).—The liquid passes through an element containing a spiral passage of gradually increasing size formed between two concentric tubes, the outer of which is heated and the inner serving for the passage upwards of the evolved steam. Heating may be effected by gas, oil-firing, electricity, or other direct means.

J. P. Ogilvie.

Recovering sugar content in cane juices. C. G. Petree, Assr. to Petree & Dorr Engineers, Inc. (U.S.P. 1,625,680, 19.4.27. Appl., 20.4.25. Conv., 18.8.24).—Treatment of the rich and weaker juices coming from different stages of a cane-milling circuit consists in decanting the rich juice, adding the mud produced to the weaker juice, decanting the latter, and separately recovering the solids and juice from the residual mud by means of a filter press.

J. P. Ogilvie.

Extraction of the sugar present in [beet] molasses and vinasses. H. Friedrich and W. Rajtora (G.P. 435,515, 23.4.24. Conv., 17.5.23).—Thickened molasses or vinasse at about 40° is mixed with glacial acetic acid, and the precipitated sugar separated and dried with hot air, the acetic acid being recovered from the residues by distillation.

J. P. Ogilvie.

Decolorisation of [beet] sugar juices. L. J. Heuclin (F.P. 608,097, 19.12.25).—Ozone is applied for decolorising at two stages, viz., at the end of the last saturation, and before, during, or after concentration.

J. P. Ogilvie.

Treatment of [soft] sugar. H. C. Welles (U.S.P. 1,624,296, 12.4.27. Appl., 1.10.23).—The colour of soft (brown) sugar is improved by treatment in the centrifuge with a small quantity (0·1%) of 20% phosphoric acid solution.

T. S. Wheeler.

Fertiliser (E.P. 270,957).—See XVI.

XVIII.—FERMENTATION INDUSTRIES.

Briquette binder. Moore and Myers.—See II.

Patents.

Treatment and preparation of yeast and product. F. M. Hildebrandt and C. N. Frey, Assrs. to Fleischmann Co. (U.S.P. 1,625,121, 19.4.27. Appl., 4.6.21).—Yeast is submitted to a conditioning fermentation in a nutrient sugar solution, and is then mixed with about half its weight of maize meal, and of a saturated solution of calcium sulphate, and the mixture is slowly dried in a current of air at 17° until the moisture content is reduced to about 13%.

T. S. Wheeler.

Stabilisation of yeast. F. Sauer (G.P. 436,878, 21.1.25).—Pure yeast cultures are treated with weakly acid salt solutions, the strength and composition of which correspond with the salt content of the yeast plasma, and stabilised by the addition of substances such as alcohol or glycerol which do not hinder the growth of the yeast cells.

A. R. Powell.

Production of potassium bitartrate from grape residues. C. Spedicato (F.P. 602,243, 7.8.25).—The residues are stirred with sodium carbonate and treated with live steam for 20 min. in a vessel provided with a perforated false bottom, the liquid collecting below this being subsequently treated with concentrated sulphuric acid to precipitate potassium bitartrate.

L. A. Coles.

Fermentation processes for the production of butyl alcohol and acetone. Commercial Solvents Corp., Assees. of D. A. Legg (E.P. 249,833, 25.2.26. Conv., 30.3.25).—See U.S.P. 1,582,408; B., 1926, 563.

XIX.—FOODS.

Preparation and analysis of the various proteins of wheat flour with special reference to the globulin, albumin, and proteose fractions. W. F. Hoffman and R. A. Gortner (Cereal Chem., 1927, **4**, 221—229).—Details are given of the methods of isolation from a patent wheat flour of gliadin, glutenin, leucosin, and small amounts of a possible globulin, together with the results of the protein analysis by the Van Slyke method (cf. B., 1911, 1135). No proteose could be detected, but a gum was obtained which, in a state of purity, did not colour blue with iodine or reduce Fehling's solution before hydrolysis. Extraction of wheat flour with 5% potassium sulphate solution and with 10% sodium

chloride solution gave very different results, both as regards the nature and amount of the proteins which could be separated from the solutions. F. R. Ennos.

Concentration of glutenin and other proteins in various types of wheat flour. E. Grewe and C. H. Bailey (Cereal Chem., 1927, **4**, 230—247).—Determinations of glutenin by the method of Sharp and Gortner (B., 1924, 762) and by that of Blish and Sandstedt, for a number of flour samples milled from wheat of varying types, gave results in satisfactory agreement, but the relative baking qualities as judged by the volume of the loaves obtained in three different baking tests were not consistent. The ratio of glutenin to crude protein and of glutenin to the sum of glutenin and gliadin showed very little variation over the whole range of flours examined. The highest coefficient of correlation with loaf volume was shown by the crude protein content and the extensibility, and a lower one by the diastatic activity of the flour. No correlation was shown by the quality constant (Sharp and Gortner, B., 1924, 29), the size of the starch granules, or the heat of imbibition of the dried starch preparations.

F. R. Ennos.

Determination of casein in milk by approximately isoelectric precipitation. H. C. Waterman (J. Assoc. Off. Agric. Chem., 1927, **10**, 259—263).—For the determination, a buffer solution of p_H 4·7 is used. The optimum point is p_H 4·74 as shown by Rona and Michaelis (cf. A., 1910, i, 905). The buffer precipitant is prepared from 250 c.c. of *N*-acetic acid to which are added 125 c.c. of *N*-sodium hydroxide solution, the mixture being diluted to 1 litre. To avoid the tedious washing of the casein precipitate, with its inherent losses, casein nitrogen is determined as the difference between the total nitrogen in the milk and the nitrogen content of the filtrate after precipitation. To 20 c.c. of the milk sample add 50 c.c. of the buffer solution, dilute to 100 c.c., and, after shaking, warm at 50—60° for 15 min. Cool, filter (returning the filtrate twice), and determine both nitrogen (A) in 50 c.c. of the filtrate and total nitrogen (B) in 10 c.c. of the original milk. Then $6·38 \times (B - A)$ = grams of casein in 10 c.c. of the milk.

H. J. Dowden.

Morres and Schützler's method for the determination of fat in milk. O. Baumann (Pharm. Zentr., 1927, **68**, 289—290).—Comparative experiments by the new method (Morsin method) and by the Gottlieb-Röse and Gerber methods show that it is entirely reliable, even in presence of formaldehyde, for milks of which the acidity is below 20 on the Soxhlet-Henkel scale. With higher acidities, or after boiling, the separation of the fat is incomplete, but pasteurised milks give accurate results. The method is very rapid and convenient. S. I. Levy.

Determination of nitrate- and nitrite-nitrogen in meat. O. Noetzel (Pharm. Zentr., 1927, **68**, 321—323).—10 g. of the finely-divided meat and 150 c.c. of water containing a few drops of 25% sodium hydroxide are frequently shaken over a period of about 1½ hrs. After bulking to 200 c.c., 150 c.c. of the filtrate are treated drop by drop with 30 c.c. of dialysed iron solution, diluted to 200 c.c., shaken vigorously, and filtered. One portion (40—80 c.c.) of the filtrate, which should be quite clear, is used for the determination of total nitrogen by adding a slight excess of 0·1*N*-potassium permanganate solution, acidifying with sulphuric acid, destroying the excess of permanganate with alcohol, and boiling for 2 min. after the addition of 1 g. of powdered iron. 9 c.c. of 1 : 2 sodium hydroxide solution are added to the cooled solution and the ammonia steam-distilled into acid, the excess of which is titrated using Congo-red indicator. In the other portion of the filtrate nitrite is determined colorimetrically by means of *m*-phenylenediamine hydrochloride. The method is very satisfactory for the determination of nitrite in the presence of relatively large quantities of nitrate, but not *vice versa*.

F. R. Ennos.

Determination of benzoic acid in minced meat. E. Waltzinger (Chem.-Ztg., 1927, **51**, 170).—To 20 g. of the minced meat shaken with water are added 20 c.c. of Fehling's copper sulphate solution, and 6 c.c. of *N*-sodium hydroxide solution, and the process continued as in the determination of benzoic acid in egg yolk (B., 1927, 122). The extract obtained from meat with ether and light petroleum may contain other substances, such as fatty acids, especially if putrefaction has set in. The extract is weighed, heated at 100° to sublime the benzoic acid, and weighed again. With perfectly fresh meat titration of the extract with 0·1*N*-sodium hydroxide solution gives satisfactory results.

F. R. Ennos.

Heat and moisture as factors in the destruction of gossypol in cottonseed products. W. D. Gallup (Ind. Eng. Chem., 1927, **19**, 726—728).—Gossypol, the toxic principle of cotton seeds (cf. Carruth, A., 1918, i, 266), is readily changed on heating to the less toxic *d*-gossypol (cf. Sherwood; B., 1926, 564), which is slowly destroyed, but the time of heating necessary to obtain a non-toxic product is much longer than that employed in the oil mill. Heating the seeds with water in an autoclave at 20 lb. pressure destroys both the gossypol and *d*-gossypol in relatively short periods of time (1—4 hrs.), and feeding tests show that the product thus obtained yields a more suitable cattle food than the untreated seeds. Germination of the seeds does not decrease the toxicity. W. J. Powell.

Aluminium cans for preserving foods. H. Serger (Chem.-Ztg., 1927, **51**, 391—393, 411—413).—Tests with a large number of fruits and vegetables show that they may be advantageously preserved in aluminium cans without suffering any deterioration as regards taste, colour, odour, or edibility. The aluminium is much less attacked by the fruit acids than is the tin of the usual tinned-iron cans, and any minute amount of aluminium that dissolves has no toxic effect.

A. R. Powell.

Oils in emulsions. Baldwin. See II.

Patents.

Extraction of albumino-caseins of vegetable origin, and separation of these albumins from amylaceous matters. H. Beaufour (E.P. 260,242, 12.10.26. Conv., 23.10.25).—Vegetable substances such

as cereals or oleaginous seeds are treated below 50° with a 3—10% solution of a chloride of an alkali or alkaline earth, the albuminous solution being separated from the insoluble material. After coagulation by suitable means, the albumino-casein is collected by decantation, filtering, or centrifuging and is washed, dried, and bleached. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 147,513, 121,141, 17,116 of 1906, 9457 of 1905, 30,842 of 1897, and 25,786 of 1896.]

F. R. Ennos.

Preparation of concentrated vitamin-*B*. P. Caccia (U.S.P. 1,617,702, 15.2.27. Appl., 25.9.25).—A mixture of yeast and water is kept for several days and filtered. The residue is extracted with boiling dilute hydrochloric acid, and the extract mixed with the filtrate and evaporated to a paste, which is extracted with absolute alcohol. The alcoholic extract is treated with ethylene chloride to precipitate vitamin-*B*, which is recrystallised from sulphuric acid. T. S. Wheeler.

Deodorising of fluids (E.P. 270,509).—See I.

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

Cheap ethylene dichloride. D. H. Killeffer (Ind. Eng. Chem., 1927, **19**, 636—639).—The commercial possibilities of ethylene dichloride are examined. This substance is now cheaper than carbon tetrachloride on a volumetric basis of comparison, and is suitable for use as a non-inflammable solvent since it has a relatively low sp. heat, forms explosive mixtures with air only with difficulty, and will not support a flame. It is superior to carbon tetrachloride in stability towards water and steam, hence is less liable to cause corrosion of plant. The sharp reduction in price of ethylene dichloride has also made possible its utilisation as a raw material for synthetic products, since under suitable conditions both chlorine atoms are replaceable by other groups. With benzene it yields dibenzyl, which is readily oxidised by atmospheric air to benzaldehyde or benzoic acid, and with ammonia yields ethylenediamine. Glycol diacetate, a solvent for cellulose acetate and nitrate, can be made by heating ethylene dichloride with a small excess of anhydrous sodium acetate under 125—150 lb. pressure. The addition of about 5% of glycol diacetate to the original mixture furnishes a common solvent and hence accelerates the action, whilst excess of sodium acetate is necessary to prevent the formation of by-products. Succinic acid can be prepared without difficulty by the condensation of ethylene dichloride with sodium cyanide, the succinonitrile formed being hydrolysed by means of hydrochloric acid. W. J. Powell.

Application of the Stahre reaction to the accurate determination of citric acid. B. G. Hartmann and F. Hillig (J. Assoc. Off. Agric. Chem., 1927, **10**, 264—272).—The determination of citric acid by oxidation to acetone and precipitation as pentabromoacetone (cf. Wöhlk; B., 1902, 564) is quantitative if certain precautions are taken. The solubility of the pentabromo-compound is the chief source of error, and must be corrected for by allowing 1·7 mg. for every 100 c.c. of reaction mixture. The tendency to form a difficultly crystallisable oil is inhibited by adding asbestos and cooling in a refrigerator before addition of the ferrous sulphate solution. The precipitate is most conveniently dried by aspirating air. To 100 c.c. of citric acid solution add exactly 10 c.c. of dilute sulphuric acid (1 : 1) and 10 c.c. of saturated bromine water, and filter after 10 min., if necessary. To 100 c.c. of the filtrate add 5 c.c. of potassium bromide solution (15 g. in 40 c.c. of water) and a little asbestos, heat at 48—50° for 5 min., and add sufficient 5% potassium permanganate solution to render the supernatant liquid a brown colour, about 15 c.c. being required. Cool in ice-water, and, after adding sufficient ice-cold ferrous sulphate solution (20 g. in 100 c.c. of water containing 1 c.c. of sulphuric acid) to dissolve all the manganese dioxide, about 40 c.c. being required, shake for 5 min., and place in a refrigerator overnight. Filter by decantation, wash with cold very dilute sulphuric acid and with water, dry by aspirating air through the filter, and weigh. Remove the pentabromoacetone from the filter by washing with alcohol and then with ether, and re-weigh. After correcting for the solubility of the pentabromo-compound, the conversion factor 0·424 is used.

H. J. Dowden.

Determination of terpin hydrate in terpin hydrate elixir. A. G. Murray (J. Assoc. Off. Agric. Chem., 1927, **10**, 257—259).—Owing to the sparing solubility of terpin hydrate in ether or chloroform, the extraction of this substance from aqueous solution by these solvents is long and tedious, but may be facilitated by the addition of salt and a little alcohol. Since the hydrate is efflorescent, it must not be dried in a desiccator, nor must heat be used in evaporation of the solvent. The method suggested consists in adding to a measured volume of the elixir sufficient salt solution (20 g. of sodium chloride in 100 c.c. of water) to reduce the alcohol content to 10—15% by vol. Extract 4 times with chloroform containing 5—7% of alcohol by vol., washing each extract through 5 c.c. of the salt solution. Filter the united extract through cotton wool into a weighed dish, and evaporate the solvent by an air-blast without heating. Expose the residue to the atmosphere for 15 min. before weighing.

H. J. Dowden.

Determination of benzoic acid. Waltzinger.—See XIX.

Patents.

Catalyst for synthetic methyl alcohol production. J. C. Woodruff and G. Bloomfield, Assrs. to Commercial Solvents Corp. (U.S.P. 1,625,924—9, 26.4.27. Appl., [a], 26.5.26; [b], 21.6.26; [c], 25.9.26; [d, e], 14.10.26; [f], 25.10.26).—(a) A catalyst for use in the synthesis of methyl alcohol from hydrogen and carbon monoxide comprises one or more difficultly reducible metal oxides, *e.g.*, zinc oxide, magnesium oxide, cadmium oxide, one or more easily reducible metal oxides, *e.g.*, (c) nickel oxide, (d) cobalt oxide, (e) copper oxide, and a metallic halide, *e.g.*, zinc chloride. (b) The catalyst comprises one or more difficultly reducible group II metal oxides, *e.g.*, zinc oxide, one or more difficultly reducible metal oxides of groups III—VII, *e.g.*, chromium oxide, and a metallic halide. (f) A suitable catalyst can be obtained by evaporating to dryness a suspension of zinc oxide in

an aqueous solution of chromium nitrate, ferric nitrate, and zinc chloride, and heating the product to transform the nitrates to oxides. The mixed gases are passed over it at 420° and 250 atm. T. S. WHEELER.

Dehydration of ethyl alcohol. W., K., L., W., and F. MERCK (E. MERCK) (E.P. 270,612, 9.2.27. Conv., 30.10.26).—The dehydration of alcohol by lime is accelerated by heating in an autoclave at a pressure greater than atmospheric. A. DAVIDSON.

Concentration of dilute acetic acid. H. SUIDA (U.S.P. 1,624,812, 12.4.27. Appl., 9.2.26. Conv., 21.2.25. Cf. E.P. 218,271—2; B., 1925, 899).—In the application of the processes described in the patents cited, the cresol removed from the extracting apparatus by water vapour is recovered by keeping the aqueous condensate until most of the cresol separates, and then extracting the aqueous layer with oleic acid, from which the cresol is recovered by distillation *in vacuo*. T. S. WHEELER.

Purification of acetic anhydride. BRITISH CELANESE, LTD., and W. P. SKERTCHLY (E.P. 270,809, 13.2.26).—Acetic anhydride containing sulphur or sulphur derivatives is purified by treatment with an anhydrous powdered metallic acetate, then with chlorine in quantity sufficient to react with the sulphur etc., and subsequent distillation. B. FULLMAN.

Manufacture of acetaldehyde. CONSORTIUM FÜR ELEKTROCHEM. IND. G.M.B.H. (E.P. 260,305, 26.10.26. Conv., 26.10.25).—The manufacture of acetaldehyde by the action of acetylene on water in presence of a mercury compound is improved by the presence in the reaction mixture of an organic solvent for acetylene, containing a hydroxyl or carbonyl group, but not a carboxyl group. Mono- and poly-hydric alcohols, phenols, and ketones are suitable, as also are substances capable of yielding them such as acetals and esters. To promote rapid removal of acetaldehyde from the reaction liquid, thus avoiding resinification, a rapid current of acetylene is passed through, whereby also the liquid is suitably agitated and the temperature regulated. By fractional condensation of the vapours, water and solvent are returned to the reaction vessel, while acetaldehyde and excess of acetylene pass through washers which absorb the aldehyde, the acetylene being returned to the reaction vessel. Spent catalyst is removed continuously or at intervals and, after regeneration, *e.g.*, by dissolving the separated mercury in nitric acid and converting into sulphate, returned to the system. The reaction may be carried out in presence or absence of acid and at pressures higher than atmospheric. A. DAVIDSON.

Manufacture of acetals. CONSORTIUM FÜR ELEKTROCHEM. IND. G.M.B.H. (E.P. 264,791, 12.10.26. Conv., 25.1.26. Addn. to E.P. 257,622; B., 1927, 379).—The process described in the prior patent may be carried out with alcohols containing water, the water being converted into acetaldehyde. The acetaldehyde is separated as far as possible by washing the vapours or fractionally condensing them. The proportion of acetal in the product is increased by suitable partial condensation of the vapours, the condensate being returned to the reaction mixture, or by passing the products of reaction, after removing the aldehyde, repeatedly through the reaction apparatus. A. DAVIDSON.

Electrolytic oxidation of organic compounds. C. H. FIELD and G. OWEN (E.P. 265,672, 7.11.25).—The electrolytic oxidation of organic compounds, especially of anthracene, is effected in the presence of a catalyst prepared from a cerite ore (composition given) from Riddarhytton, Sweden, containing chiefly: SiO_2, 20%; Ce_2O_3, 22·38%; Di_2O_3, 33·1%; and La_2O_3, 10·64%. The ore is heated at 200° for 8 hrs. with sulphuric acid, and the product when cool treated with cold water, the resultant solution being precipitated with oxalic acid (the precipitate being heated to expel carbon dioxide) or an alkali. The red precipitate as produced is added to the electrolyte in quantities of about 5% by wt. of the solution. The cell may be a lead-lined vessel with lead electrodes, the cathode being covered with a diaphragm of asbestos cloth or earthenware. *E.g.*, 3½ cwt. of anthracene (40% upwards) which has been treated with sodium hydroxide is electrolysed in 2—3½ tons of 50—70% sulphuric acid containing 5% of catalyst, at 100°, 4—7 volts, 300 amp. (*D.C.*), for 2 hrs. A high yield of anthraquinone is obtained. Toluene (to benzaldehyde), *m*-xylene, or naphthalene may also be oxidised. The action of the catalyst is attributed to the presence in the ore of an unisolated element associated with the didymium. B. FULLMAN.

Catalytic dehydrogenation. I. G. FARBENIND. A.-G. (E.P. 262,120, 25.11.26. Conv., 30.11.25).—Zinc sulphide and other sulphides of the heavy metals, either alone or mixed mutually or with other materials, often on a carrier, are used for catalytic dehydrogenation. *E.g.*, *iso*butyl alcohol gives *iso*butaldehyde in 92% yield when passed over zinc sulphide on pumice stone at 460°. B. FULLMAN.

Potassium bitartrate (F.P. 602,243).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Calibration of photographic plates. F. S. HOGG and C. H. PAYNE (Nature, 1927, **119**, 925).—Density curves for plates with two different emulsions show no greater differences than those for plates with the same emulsion; also, the two kinds of developer employed give closely similar results. The Harvard individual plate calibration curves allow for changes in sensitivity due to the temperature and humidity during exposure in the telescope, to the ageing of the plates, to delayed development, and to sky fog. A. A. ELDRIDGE.

Chemical sensitisation of silver iodide-gelatin plates. H. FRIESER (Phot. Ind., 1927, 520—521).—The preparation of a silver iodide emulsion is described, and results are given of tests of the latent image and print-out effects, using a large number of organic and inorganic sensitisers. W. CLARK.

PATENTS.

Photographic media. H. WADE. From WADSWORTH WATCH CASE CO. (E.P. 270,387, 4.2.26).—A stable light-sensitive varnish consists of a hydrophobic colloid containing metal salts capable of combining with free halogen. The salts are present to a point

approaching saturation, and the free halogen is introduced in such amount as to combine with most of the salts and to avoid the presence of free halogen after the metal halides have been formed. As an additional sensitiser, a halogen-substituted hydrocarbon, such as iodoform, may be added. The varnish consists in the main of a light-sensitive oil such as tung oil, and a smaller amount of a resin, such as coumarone, and of gutta-percha may be added. A solution of iodine may be used as the halogen, and lead resinate, for example, as the metal salt. W. Clark.

Photographic process [using asphalt]. H. Wade. From Wadsworth Watch Case Co. (E.P. 270,386, 4.2.26).—A sensitive medium for use in the preparation of screenless grained litho-plates, line engravings, half-tone blocks, intaglio blocks, etc. consists of asphalt containing a sensitiser which will liberate a halogen under the influence of light. A suitable sensitiser comprises both a metallic halide and a halogen-substituted hydrocarbon, such as iodoform. One medium is prepared by mixing together approximately 10% of asphalt, 40% of solvent naphtha, 25% of benzine, and about 25% of a solution containing a halogen-liberating compound, *e.g.*, 34% of iodoform and methyl ethyl ketone mixture in the ratio of 12 : 22, and 66% of a solvent such as solvent naphtha. W. Clark.

Recovery of metals [silver] from solutions. G. J. Levy, Assr. to Hollywood Chemical Co. (U.S.P. 1,624,172, 12.4.27. Appl., 23.12.25).—Waste sodium thiosulphate solution containing silver is mixed with zinc hyposulphite solution, when all the silver present is precipitated in the metallic form. T. S. Wheeler.

Recovery of silver from photographic spent thiosulphate solution. E. K. Giffen (U.S.P. 1,629,212, 17.5.27. Appl., 19.9.25).—Spent sodium thiosulphate solution containing dissolved silver is employed as electrolyte in a cell containing a copper positive pole and a zinc negative pole, connected through an external resistance. Silver is deposited on the copper pole which is fixed at the bottom of the solution, while the zinc pole, immersed in a porous bag, is maintained at the top, so that the silver deposited cannot be contaminated by hydrogen sulphide evolved at the zinc pole. T. S. Wheeler.

XXII.—EXPLOSIVES; MATCHES.

Patents.

Manufacture of smokeless [flashless] explosive powder. F. I. and E. du Pont, Assrs. to U.S.F. Powder Co. (U.S.P. 1,627,691—4, 10.5.27. Appl., [a, b], 3.5.22; [c], 8.8.21; [d], 23.2.21).—(a) A flashless propellant comprises nitrocellulose, gelatinised in presence of finely-divided ammonium nitrate, with or without addition of glyceryl nitrate. The addition of acetone to the ethyl ether–alcohol gelatinising mixture is of advantage when glyceryl nitrate is present. The product is formed into grains perforated sufficiently to bring the loading density 15—50% below that of normal cellulose nitrate powders. (b) Nitrocellulose is ground in presence of water or petrol with 4—25% of a mixture of barium nitrate (2 pts.) and potassium nitrate (1 pt.). The product is dried, treated with a gelatinising mixture of ethyl ether and alcohol, and formed into grains. (c) The mixture of barium and potassium nitrates employed in (b) is replaced by black powder or (d) by a mixture of potassium dichromate and charcoal. T. S. Wheeler.

Manufacture of a smokeless [flashless] explosive powder. Flashless cannon powder. F. I. and E. du Pont, Assrs. to U.S.F. Powder Co. (U.S.P. 1,627,638—9, 10.5.27. Appl., [a], 10.11.25; [b], 13.11.26).—(a) The process described in U.S.P. 1,627,692—4 (preceding abstract) is modified, in that the nitrocellulose is ground with the added substance in presence of alcohol, to which ethyl ether is then added to form a gelatinising mixture. (b) In the application of the process described in (a) a mixture of potassium nitrate and charcoal is preferable to black powder if the propellant contains glyceryl nitrate. T. S. Wheeler.

Sodium azide (U.S.P. 1,628,380).—See VII.

XXIII.—SANITATION; WATER PURIFICATION.

Detection and determination of very small quantities of iron in drinking and industrial waters. F. Kröhnke (Gas- u. Wasserfach, 1927, 70, 510—511).—A solution of *iso*nitrosoacetophenone in chloroform (1·5 g. in 200 c.c.) is a more sensitive reagent for ferrous iron than alkali thiocyanates for ferric iron, and can be used to determine quantities down to 0·01 mg. per litre. To 50 c.c. of the water to be tested 1 c.c. of a 2% solution of hydrazine sulphate is added, and the solution boiled until reduced in bulk to 10—20 c.c. (10—15 min.). The solution is rapidly cooled and shaken with 1 c.c. of the reagent, a 1—2% solution of ammonia being gradually added until a blue colour appears in the chloroform. The colour is compared with standards prepared from a range of solutions of known iron content (0·01—0·1 mg. per litre, or higher if necessary). The presence of manganese does not interfere with the test, but copper, nickel, and cobalt must be absent. A. B. Manning.

o-Tolidine test for chlorine. Hopkins.—See VII.

Patents.

Manufacture of disinfecting, deodorising, or sanitising substances. W. & F. Walker, Ltd., and J. T. Freestone (E.P. 270,440, 20.2.26).—*p*-Dichlorobenzene, made by chlorinating benzol, separating the monochlorobenzene by distillation and the *o*-dichlorobenzene by crystallisation at low temperatures, is used in the form of compressed blocks. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 19,688 of 1912.] W. G. Carey.

Insecticide. A.-G. für Anilin-Fabr. (E.P. 253,065, 14.10.25. Conv., 2.6.25).—An insecticide for washing animals consists of a soluble inorganic parasite poison and an insoluble inorganic parasite poison in colloidal form, together with a protective colloid and, if necessary, a wetting agent. The colloidal poison may be replaced by a soluble derivative decomposable by acids or carbon dioxide to liberate the insoluble poison in colloidal form; *e.g.*, sodium sulphoarsenite gradually forms colloidal arsenic sulphides on the animal by the action of atmospheric carbon dioxide. W. G. Carey.

Filter (U.S.P. 1,630,079).—See I.