

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

MAY 13, 1927.

### I.—GENERAL; PLANT; MACHINERY.

**Heat transfer alinement charts.** M. ROULLEUX (Chem. Met. Eng., 1927, 34, 148—151).—The practical solution of most problems relating to the transfer of heat between two fluids in circulation and separated by a solid wall involves the determination of the mean temperature difference. Curves have been derived mathematically by means of which this factor may be easily deduced. C. A. KING.

**Compressed gas handling technique.** F. A. ERNST (Chem. Met. Eng., 1927, 34, 151—153).—Attempts to adapt hydraulic and compressed air equipment to use with compressed gases has in most cases proved uneconomical. Several types of high-pressure valves and unions designed for this special use are described. Alloy steel tubing, although of higher unit price, is desirable by reason of the reduced weight of the fitting allowable. In a union fitting the gas pressure is held by a metal gasket, the threads serving merely to hold the tube ends against the gasket. For a closure for a reaction vessel, instead of an end plate being bolted to the flanged vessel with consequent distortion in use, the plate is fitted internally with a gasket, and the bolts are in compression. C. A. KING.

**Turbidimetry.** RENWICK.—See XXI.

#### PATENTS.

**Cooling and drying or mechanically treating hot material which must not come in contact with the air until cold.** N. V. CARBO-UNION IND. MAATSCHAPPIJ (G.P. 435,737, 4.12.24).—Activated charcoal, ultramarine, calcium cyanamide, and similar material is discharged direct into a chamber in which it is cooled by a current of a non-reacting gas which circulates through the chamber and through a heat-exchanger. Means are provided for collecting separately the coarse particles of the material and the fine particles carried along with the gas. L. A. COLES.

**Heating of liquids.** C. CALLEBAUT and J. DE BLICQUY (E.P. 266,075, 4.12.25).—When a bath of liquid is heated by a steam pipe dipping into the bath, the vertical part of the pipe is liable to cause ebullition and splashing before the bulk of the liquid is sufficiently heated. To prevent this, the vertical part of the steam pipe is covered with a casing or sleeve, which may be either closed at the top and open at the bottom, or closed at the bottom and open at the top above the level of the liquid with the open end covered by a hood, or closed at both ends except for a vent pipe at the top, bent down over the bath of liquor. B. M. VENABLES.

**Process of recovering volatile solvents [without explosions].** A. L. MOND. From METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 266,145, 9.3.26).—

In a process for recovering volatile solvents where a gaseous medium is continuously circulated through a drying or evaporating apparatus and a condenser or absorbing apparatus, explosions are prevented by using a mixture of oxygen and nitrogen containing less oxygen than air and/or by allowing the gases to take up solvent vapour until the upper explosion limit has been passed, and keeping above that point even in the condensing stage. To provide more internal heat for the evaporation a gas of high specific heat, *e.g.*, carbon dioxide, may be used in the mixture. B. M. VENABLES.

**Machines for emulsifying, mixing, or grinding materials.** R. SURTEES (E.P. 266,098, 5.1.26).—In a colloid mill of the type wherein two discs rotate closely to each other at a high relative speed, each disc is formed with a groove on the working face, and fixed curved guides and collecting channels are arranged so as gradually to deflect and reduce the speed of the issuing emulsion. B. M. VENABLES.

**Wet separation of constituents of mineral and other pulp.** T. G. MARTYN (E.P. 264,129, 30.8.26. Conv., 7.1.26).—Several forms of apparatus are described in which the pulp flows horizontally between confining surfaces which are a short distance apart, and so inclined that the pulp is continually meeting a surface after only flowing a short distance horizontally, and the solid matter when deposited in the nearly stationary film touching the surfaces slides along them to collecting means in the lower part of the apparatus. B. M. VENABLES.

**Apparatus for the distillation or drying of coal or other solid materials.** F. G. HOFFMAN (E.P. 261,740, 15.11.26. Conv., 17.11.25).—Through a cellular metallic structure comprising alternate rows of vertical and horizontal passages, the material passes downwards once only, and the heating gases zig-zag across. For continuous operation a number of cellular units may be arranged as the spokes of a wheel on a rotating carriage, the bottom being closed by an annular plate interrupted only at the discharging point. Products of distillation are collected by a surrounding casing, and the heating gases are supplied to and withdrawn from the central space. B. M. VENABLES.

**Process and apparatus for spraying or projecting viscous, liquid, or granular material.** K. KISSE (E.P. 262,066, 25.10.26. Conv., 30.11.25).—Material which may be of heterogeneous nature and liable to separate into its constituents, is delivered to the interior of a centrifugal bowl, up which it rises by centrifugal force, and is discharged over the top rim by a fixed scraper, which delivers it at a high speed in the required direction. The fixed scraper may be supported by the fixed central-feed hopper. B. M. VENABLES.



**Apparatus for mixing liquids with solid or semi-solid substances or with other liquids.** N. BENDIXEN, and MILKANIC, LTD. (E.P. 266,950, 25.5.26).—The device comprises a propeller and surrounding perforated screen rotating together in a vessel of the liquid to be emulsified. The rake of the propellers is such as to give a downward flow to the liquid, so that considerable pressure is created in the screen basket, and any entrained air will expand on emerging from the basket, thus aiding emulsification. A stationary guiding screen may be provided to circulate the liquid back to the basket.

B. M. VENABLES.

**Separation of the constituents of gaseous mixtures by liquefaction.** L'AIR LIQUIDE, SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (E.P. 263,732, 17.3.26. Conv., 24.12.25).—A mixture of gases containing a wanted low-boiling constituent, e.g., hydrogen, is compressed and cooled (1) by heat exchange with the returning unwanted liquefied gases, (2) by the hydrogen before expansion, and (3) by the hydrogen after expansion. The cooling stages (1) and (3) are separate, but (2) is effected simultaneously on the same portion of gas as (1) and (3). Stage (2) also effects reheating of the hydrogen prior to its doing work in the expanding machine.

B. M. VENABLES.

**Apparatus for separating constituents of gaseous mixtures.** C. C. VAN NUYS, ASSR. to AIR REDUCTION Co., INC. (U.S.P. 1,619,169, 1.3.27. Appl., 6.12.24; cf. U.S.P. 1,539,528; B., 1925, 575).—In the apparatus described, compressed cooled air is subjected successively to the cooling action of cold expanded gases and liquid products, and the liquid fraction resulting is rectified to produce a liquid as rich as possible in oxygen. This liquid is subsequently rectified, and the vapours produced are brought in contact with liquid nitrogen in quantity sufficient to ensure the separation of all the oxygen.

T. S. WHEELER.

**Apparatus for treating gases and vapours with silent electric discharges.** W. CARPMAEL. From I. G. FARBERIND. A.-G. (E.P. 266,983, 19.7.26).—Tubes of suitable size are placed loosely one inside the other, and are spaced at their respective upper ends by projections or pegs on the inner or outer tubes, and at the lower ends by constrictions on the outer tubes.

J. S. G. THOMAS.

**Absorption refrigerating apparatus.** P. M. LE ROY, ASSEE. of G. BEAUMONT (E.P. 255,053, 1.7.26. Conv., 8.7.25).—Ammonia vapour is conducted from the boiler absorber through a non-return valve to the ammonia liquefier, which is water-cooled and outside the space to be chilled. At the bottom of the liquefier are two valves, one leading to the evaporator or refrigerator proper, and the other for leading residual aqueous ammonia solution back to the boiler. The boiler absorber is also water-cooled during the absorption stage.

B. M. VENABLES.

**Refrigerating apparatus.** J. O. BOVING (E.P. 266,920, 26.3.26).—The condenser-evaporator and boiler-absorber of a refrigerator of the reversing absorption type are invertible, and are coupled together side by side as one unit by the transfer pipe, which is perforated within the boiler absorber and leads from one end of that

vessel to the remote end of the condenser evaporator. All the necessary operations to complete a cycle of refrigeration are effected by tilting the apparatus to various positions. During the boiling off stage the boiler is heated and the absorber placed in a vessel of water.

B. M. VENABLES.

**Refrigerating and heat interchanging apparatus.** D., M., S. R., and S. GUGGENHEIM, J. K. MACGOWAN, and E. A. C. SMITH, ASSEES. of C. L. BURDICK (E.P. 261,731, 5.11.26. Conv., 21.11.25).—A tubular heat interchanger is provided with devices for spreading the two liquids in a thin film on the interior and exterior of the tubes respectively. For the inner liquid, cones at the top, with their bases nearly touching the walls of the tubes, may be used, and for the exterior, one or more trays may be provided with holes slightly larger than the outside of the tubes.

B. M. VENABLES.

**Heat exchangers.** CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (E.P. 266,100, 6.1.26).—A tubular heat exchanger is constructed of silicon carbide tubes which are good conductors, and are intended to be heated by radiation from the hot gases which may be "live" gases of combustion, and metal tubes which are used where the heat is not so great. All are supported and interconnected by a double metal shell, which is screened where necessary from excessive heat by refractory linings.

B. M. VENABLES.

**Heat exchange apparatus.** BABCOCK & WILCOX, LTD., A. HALL-BROWN, and E. W. JONES (E.P. 266,206, 23.7.26).—A fan with alternate passages for gas and air is arranged at the cold end of a stationary, cellular heat interchanger. One fluid, say the gas, enters the eye of the fan and leaves the rotor tangentially, while the air enters through ports on the back plate and leaves in an axial direction, i.e., the flows of gas and air are substantially at right angles.

B. M. VENABLES.

**Furnaces.** RADIATION, LTD., S. N. BRAYSHAW, and E. R. BRAYSHAW (E.P. 266,487, 12.12.25).—In a regenerative gas furnace constructed as described in E.P. 119,553 (B., 1918, 678A) those parts of the gas and air inlet pipes where the hot gases first strike them are covered by a loose, renewable piece of metal, and a deflector is placed in the combustion chamber in the path of the gas jet.

B. M. VENABLES.

**Filtering apparatus.** R. POWLEY & SONS, LTD., and M. POWLEY (E.P. 266,615, 28.8.26).—A vacuum drum filter is rotated by the incoming unfiltered liquid, and the vanes or buckets for this purpose may be on the drum itself.

B. M. VENABLES.

**Asbestos filter for the dry recovery of solid constituents of blast-furnace gases and the like.** DEUTSCHE MASCHINENFABR. A.-G. (G.P. 435,653, 3.8.22).—The filter fabric has warp and weft constructed of tightly woven asbestos yarn, the warp and/or weft being wound round with loosely woven asbestos material.

L. A. COLES.

**Process and apparatus for obtaining powders of great fineness.** E. PODSZUS (U.S.P. 1,621,270, 15.3.27. Appl., 2.9.24. Conv., 12.7.21).—See E.P. 183,134; B., 1923, 385.

**Separation of constituents of gaseous mixtures containing hydrogen.** G. CLAUDE, ASSR. to LAZOTE



INC. (U.S.P. 1,620,192, 8.3.27. Appl., 12.3.23. Conv., 4.4.22).—See E.P. 195,950; B., 1924, 123.

**Method of distillation [of tar etc.].** J. S. MORGAN, ASS. TO THERMAL INDUSTRIAL AND CHEMICAL (T.I.C.) RESEARCH CO., LTD. (U.S.P. 1,622,964, 29.3.27. Appl., 30.1.23. Conv., 13.11.22).—See E.P. 207,366; B., 1924, 82.

**Roof for industrial ovens and furnaces.** O. MÜLLER-TANNECK (E.P. 266,211, 20.8.26).

**Roof for kilns or ovens.** J. LAURENT (E.P. 259,251, 4.10.26. Conv., 3.10.25. Addn. to 242,294).

**[Fluid-cooled wall for] furnaces.** T. E. ROBERTSON. FROM POWER SPECIALTY CO. (E.P. 266,166, 10.4.26).

**Checker work for regenerators of furnaces fired by gas producers.** W. LINDNER (E.P. 266,932, 21.4.26).

**Irradiation apparatus for treating fluids with ultra-violet rays.** QUARZLAMPEN-GES.M.B.H. (E.P. 247,231, 9.2.26. Conv., 9.2.25).

**Heat insulating process, devices, and materials.** E. SCHMIDT and E. DYCKERHOFF (E.P. 266,177, 14.5.26).

**Timing devices for centrifugal separators.** E. A. ALLIOTT and MANLOVE, ALLIOTT & CO., LTD. (E.P. 266,529, 10.2.26).

**Liquid seals.** A. H. STEVENS. FROM SHARPLES SPECIALTY CO. (E.P. 250,171, 19.11.25).

**Apparatus for automatically governing the discharge of one liquid into another in proportion to the flow.** R. AMES (E.P. 266,883, 4.2.26).

**Means for heating by oil circulation.** MANUF. DE MACHINES AUXILIAIRES POUR L'ELECTRICITÉ ET L'IND. (E.P. 262,753, 22.11.26. Conv., 10.12.25).

**Vapour generating systems.** SIEMENS-SCHUCKERT-WERKE G.M.B.H., and W. ABENDROTH (E.P. 267,012, 10.9.26).

**Drying cylinders.** W. P. EVANS (E.P. 266,481, 4.12.25).

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

**Laboratory methods of determining the inflammability of coal dusts.** A. L. GODBERT (Safety in Mines Res. Bd., Paper No. 31, 68 pp.).—Previous work on the inflammation of coal dust is fully surveyed. The inflammability of a coal dust cloud is dependent on the concentration of the dust, its fineness, the nature of the coal, the presence of incombustible material, and the nature of the atmosphere in which the dust is suspended. Determination of the ignition temperature is unsatisfactory as a measure of the inflammability, the order of values obtained with a series of dusts being dependent on the method of measurement used. Moreover a dust of high ignition temperature may give rise to a more violent explosion than one of lower ignition temperature. The determination of speed of flame propagation presents experimental difficulties, particularly on a small scale. The most satisfactory laboratory method depends on the determination of the amount of incombustible dust which must be added to prevent inflammation. The results also are directly applicable to the problem

of the prevention of explosions in coal mines. Details of the method which the Safety in Mines Research Board propose to adopt in their future experiments are briefly outlined.

A. B. MANNING.

**Methods of analysis of coal.** (Dept. Sci. Ind. Res., Fuel Res. Bd. Survey Paper No. 7, 35 pp.).—The methods of analysis recommended in the present report differ only slightly from those in the Interim Report (B., 1924, 206). The method of determining phosphorus in ash is slightly modified, and an appendix added dealing with possible errors arising from the presence of arsenic, silica, titanium, or vanadium. A somewhat more detailed account is given of the Gray-King carbonisation assay (B., 1921, 205 A).

A. B. MANNING.

**Determination of nitrogen in fuels.** G. LAMBRIS (Brennstoff-Chem., 1927, 8, 89–93; cf. B., 1927, 289).—Full details are given for carrying out the determination, and the accuracy and reliability of the method are exemplified by analyses of coals, coke, and various organic compounds. Two samples can be placed in the combustion tube at the same time, and their analysis carried out one after the other without any discontinuity. In this way two nitrogen determinations can be completed in 2–2½ hrs. W. T. K. BRAUNHOLTZ.

**Ten years of coal research.** F. FISCHER (Chim. et Ind., 1927, 17, 367–374).—A brief account of the work of the Kaiser-Wilhelm Institut für Kohlen-Forschung at Mülheim-Ruhr.

**Sulphur balance in coking practice.** A. WEINDEL (Brennstoff-Chem., 1927, 8, 85–89).—The quantity of sulphur present in raw coke-oven gas is calculated, and methods of recovering it, particularly the Burkheiser and Feld processes, are discussed. Whilst desulphurisation is essential for town's gas and gas for certain catalytic processes, the recovery of sulphur is nowadays less profitable than formerly, since gypsum is now largely used in the manufacture of ammonium sulphate and nitric acid is produced by oxidising ammonia.

W. T. K. BRAUNHOLTZ.

**Removal of hydrogen sulphide from coke-oven gases.** W. GLUUD and R. SCHÖNFELDER (Stahl u. Eisen, 1927, 47, 453–456).—A process is described, due to the Ges. für Kohlentechnik, in which the coke-oven gas immediately after the separation of the tar enters the bottom of a washing tower and meets a dilute suspension of ferric hydroxide in counter-current. The liquor containing ferrous sulphide is pumped into a tall tower called an "oxidiser." The sulphide liquor-air mixture rises into a settler where the sulphur is separated and the regenerated liquor passes again into the gas washer. The sulphur is floated out by the air into another vessel, where it is washed and dried. The air is led into an acid washer from which ammonium sulphate is drawn off from time to time. 85% of the sulphur in the gas is removed in the crude form, the balance in the solution being oxidised to sulphite and thiosulphate, the total recovery being as much as 99.5%. In order to prevent the solution getting too rich in salts, it is passed into the ammonia recovery apparatus, the iron lost being replaced by the addition of iron sulphate. Instead of iron sulphate or hydroxide, an alkaline soda solution may be used, which, although it obviates the



necessity of an acid washer for the air, is only recommended on special occasions. In conjunction with this installation a cyanogen washer was introduced, the cyanogen being recovered in the form of ammonium thiocyanate.

T. H. BURNHAM.

**Recovery of sulphur from gas.** K. N. CUNDALL (Chem. Met. Eng., 1927, 34, 142—147).—The removal of sulphur gases from fuel gas is based on a process devised by the Koppers Co., in which, however, the regeneration of the absorbing solution involved the emission of hydrogen sulphide into the atmosphere. Hydrogen sulphide is absorbed, according to the equation  $\text{Na}_2\text{CO}_3 + \text{H}_2\text{S} \rightleftharpoons \text{NaHCO}_3 + \text{NaHS}$ , by passing the gas through a tower filled with wooden hurdles over which a solution of sodium carbonate is sprayed. During experimental work on the interaction of sulphur dioxide and hydrogen sulphide in the presence of a catalyst, it was discovered that the oxidation of hydrogen sulphide to sulphur was completed in the presence of oxygen only, the most active catalyst being colloidal nickel sulphide formed from the addition of nickel sulphate to the foul solution. The process was therefore resolved into securing maximum contact between air and solution. Three types of aerators are in use. The "thioniser" type supplies air by means of canvas-covered perforated pipes placed at the bottom of a series of tanks through which the liquor flows. The Sirocco emulsifier is a superimposed set of four tanks each fitted with a turbine-driven, basket-type impeller. Very little power and no primary air are required for this installation. Large capacity is the feature of the Feld type emulsifier, a single 5 ft. 2½ in. cone in a 13 ft. tank activating up to 100% efficiency at a rate of 1500 gals. of liquor per min. In all the plants sulphur separates as a grey muddy foam, which may form a layer several feet thick, and may then be filtered and washed. The cake contains about 50% of water and 3% of ash, the recovery in the form of free sulphur averaging about 85%. The presence of cyanides reduces the efficiency of the catalyst. C. A. KING.

**Flame temperature of producer gas with air and gas preheating.** R. NITZSCHMANN (Chem.-Ztg., 1927, 51, 197—198).—Equations are constructed based upon the composition of the producer gas, the volume of oxygen available for combustion, and the temperatures of the oxygen or air and producer gas. These are reduced to three constants, expressed in terms of the volumes of nitrogen and oxygen present before combustion, of carbon dioxide and water vapour after combustion, and of the specific heats and calorific values of reacting gases. The combination of these constants gives the theoretical flame temperature. A diagram is included for determining the flame temperature of producer gas of given composition when there is no preheating. H. D. GREENWOOD.

**Gaseous explosions. IV. Rate of rise of pressure, velocity of flame travel, and the detonation wave. V. Probable mechanism causing "detonation" in the internal-combustion engine.** G. G. BROWN and G. B. WATKINS (Ind. Eng. Chem., 1927, 19, 363—369. Cf. B., 1927, 243).—When *isohexane*-oxygen mixtures are exploded under conditions of constant initial

temperature and pressure, the maximum rate of rise of pressure in the detonation wave occurs in mixtures containing slightly more oxygen than that just necessary to form carbon monoxide and hydrogen. A comparison of these results with those of Dixon on the explosion of ethylene-oxygen mixtures (Phil. Trans., 1893, 184A, 97) and a consideration of other previous work indicates that the velocity of flame travel and the rate of rise of pressure in exploding mixtures are closely related, and vary in the same way with changing initial conditions. The amount of nitrogen which had to be added to various fuel-oxygen mixtures in order to reduce the intensity of detonation to an arbitrary standard varied directly as the rate of rise of pressure in the exploding mixture, which may therefore be taken as a measure of the tendency of the fuel to initiate detonation. The relative values of the rise of pressure for paraffin and aromatic hydrocarbons are entirely different from their relative tendency to produce "knocking" in internal-combustion engines; the cause of this phenomenon must therefore involve other factors than the detonation wave. A review of the available experimental evidence indicates that "knocking" in internal-combustion engines is caused by a heterogeneous reaction occurring at the heated cylinder walls, and not by the setting up of the detonation wave. The tendency of a fuel to cause knocking varies as the ratio of the rate of rise of pressure in an explosive mixture of the fuel to its auto-ignition temperature, which suggests that knocking is due to auto-ignition of the unburned mixture adiabatically compressed against the hot walls of the cylinder. A. B. MANNING.

**Combustion in the gasoline engine. I. Determination of rate of burning by chemical analysis. II. The burning of hydrogen and carbon monoxide.** W. G. LOVELL and J. D. COLEMAN [with T. A. BOYD] (Ind. Eng. Chem., 1927, 19, 373—378).—The composition of the gases in an engine cylinder has been determined at various times after ignition by means of a specially designed quick-acting, water-cooled sampling valve. The mixture of gases in the cylinder at any moment appears to be almost homogeneous. The data obtained from an air-cooled Delco-Light engine running at 1200 r.p.m. on gasoline with 75% of the theoretical air requirement have been plotted, and permit an estimate being made of the rate at which combustion is progressing in the cylinder. When "knocking" is induced by the use of a gasoline-kerosene mixture, or by the addition of a small quantity of *isopropyl* nitrite, the rate of burning is more rapid than normal, the normal rate being restored by the addition of lead tetraethyl. The gas composition-time curves obtained when the engine is driven on a mixture of equal volumes of hydrogen and carbon monoxide indicate that the simultaneous burning of these gases takes place thus:— $2\text{H}_2 + \text{O}_2 = 2\text{H}_2\text{O}$ ;  $2\text{CO} + \text{O}_2 = 2\text{CO}_2$ , the ratio of the velocity constants  $k_{\text{H}_2} : k_{\text{CO}}$  being 2.3. Neither alteration of the compression ratio nor the presence of ethyl nitrite or of lead tetraethyl alters the mechanism or the relative rates of these reactions. The results do not accord with the conception of gaseous combustion in which the flame is regarded as a boundary surface between burned and unburned gases, the gases in the cylinder at any instant being apparently homogeneous



and the reactions following the laws of homogeneous gas reactions.

A. B. MANNING.

**Laboratory method of determining the starting properties of motor fuels.** W. G. LOVELL and J. D. COLEMAN [with T. A. BOYD] (Ind. Eng. Chem., 1927, 19, 389—394).—A measured amount of gasoline is charged into a bomb of 300 c.c. capacity, maintained at constant temperature, and the air-gasoline mixture vigorously agitated by means of a fan, the gasoline being contained in a small cup attached to the fan shaft. The bomb is provided with a spark gap, and after 2 min. agitation the fan is stopped and the ignition current turned on. The air-fuel ratio just necessary to produce an explosive mixture under these conditions is a measure of the ease of starting an engine when using the fuel, as is shown by a comparison with the results obtained directly in engine starting tests at the Bureau of Standards. Determinations have been made for a number of fuels at temperatures ranging from  $-15^{\circ}$  to  $20^{\circ}$ . No direct relationship exists between the values so determined and the dew-points of the fuels, nor does it appear possible to obtain a quantitative measure of the readiness of starting from the Engler distillation curves.

A. B. MANNING.

**Improving fractionation in petroleum refining.** D. B. KEYES (Chem. Met. Eng., 1927, 34, 164—165).—It is suggested that in the refining of petroleum by means of a bell-cap fractionating column, without reflux, connected to a pressure still by a vapour line and a liquid return, over-cracking may be prevented by the application of more modern systems of fractionation as used in other processes, *e.g.*, the distillation of alcohol. Owing to the formation of gummy substances during fractionation, the introduction of a number of storage plates in the fractionating column would be desirable in order to allow sufficient time for the deposition of gums.

C. A. KING.

**Solubility of naphthalene.** RHODES and EISENHAUER.—See III.

#### PATENTS.

**Coal washing jig.** U. S. JAMES (E.P. 266,635, 21.10.26).—A jig is constructed so that the pulsations of water are upwards, alternating with periods of quiescence, there being no downward flow through the material under treatment. The pulsations are produced by pistons and suitable valves.

B. M. VENABLES.

**Heating of jacketed rotary retorts for the carbonisation of fuels at definite low temperatures.** E. K. STACKMANN (G.P. 435,573, 1.1.21).—Retorts are heated by circulating hot gases through the surrounding jacket, and are maintained at the required temperature by the regulated addition of hot or cold gases to the circulating current.

A. B. MANNING.

**Recovery of constituents, other than sulphur dioxide, from gas mixtures.** M. SCHROEDER (G.P. 435,587, 18.9.24).—In the recovery, *e.g.*, of benzene from coke-oven gas, of ethylene from illuminating gas, or of carbon dioxide from combustion gases, by absorption in a liquid under pressure, the potential energy of the compressed residual gases is utilised to generate power in a suitable engine, which energy is increased by heating the gas before it passes into the engine.

L. A. COLES.

**Apparatus for treating [cracking] hydrocarbons.** G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,618,645, 22.2.27. Appl., 15.7.20. Renewed 14.8.26; cf. U.S.P. 1,524,818; B., 1925, 276).—An apparatus for cracking hydrocarbons comprises a still in the form of a horizontal cylinder, the greater portion of which rotates around the axis. Heating is effected by combustion within a tube lying in the axis. All vapour outlets and other pipes are connected to the stationary portion of the cylinder. Thorough agitation of the liquid and uniform heating are obtained.

T. S. WHEELER.

**Method and apparatus for treating petroleum hydrocarbons.** W. M. CROSS (U.S.P. 1,621,475, 15.3.27. Appl., 24.11.24).—In a continuous process for treating petroleum hydrocarbons the oil is intimately mixed with an acid for about  $\frac{1}{2}$  min. (to prevent polymerisation), and is separated mechanically.

C. O. HARVEY.

**Process of treating acid sludge to provide a fuel.** W. D. KINKADE and C. J. BAUGH (U.S.P. 1,621,071, 15.3.27. Appl., 8.1.27).—Acid sludge from petroleum refining is added to a fuel oil, the mixture is heated to reduce the viscosity, and is agitated with amounts of soda and lime far less than those required for complete neutralisation of the sludge.

C. O. HARVEY.

**Apparatus for heating stills.** G. D. WHITE, Assr. to TEXAS Co. (U.S.P. 1,621,298, 15.3.27. Appl., 5.4.23).—An apparatus for cracking hydrocarbon oils comprising a battery of spaced vertical stills surrounded by a furnace chamber, even distribution of heat being attained by (a) spacing the stills from the furnace walls, (b) avoiding direct contact between the flames and the still walls by a special distribution of the gas burners, which extend through opposite walls of the furnace, and (c) a series of flue passages.

C. O. HARVEY.

**Combined condenser and oil separator.** F. C. BELL (U.S.P. 1,620,713, 15.3.27. Appl., 5.2.24).—The oil and gas are admitted to a cylindrical chamber containing an interior cylindrical casing carrying a spiral cooling coil. Condensation and separation occur, and the oil is drawn off from the base of the chamber by means of a pipe extending upwards into the oil strata.

C. O. HARVEY.

**Oil and gas separator.** D. G. LORRAINE (U.S.P. 1,620,771, 15.3.27. Appl., 26.5.24).—A device comprising a settling chamber and a gas chamber, both fitted with valvular discharge controls operated automatically by means of a float.

C. O. HARVEY.

**Making grease.** J. MCKEE, Assr. to SUN OIL Co. (U.S.P. 1,619,352, 1.3.27. Appl., 27.2.25).—Calcium hydroxide (100 pts.), mixed with a mineral lubricating oil (300 pts.), is heated at  $130^{\circ}$  to expel water, cooled to  $38^{\circ}$ , treated with oleic acid (600 pts.), mineral lubricating oil (4000 pts.), sodium hydroxide solution,  $d_{15} 1.21$  (35 pts.), and a small quantity (1%) of water, and is finally heated with agitation at about  $110^{\circ}$ .

T. S. WHEELER.

**Manufacture of hydrocarbons and cyanides.** J. C. CLANCY (E.P. 266,809, 2.12.25).—See U.S.P. 1,567,241; B., 1926, 486.

**Apparatus for coal distillation** (E.P. 261,740).—See I.

**Asbestos filter for gases** (G.P. 435,653).—See I.



Organic lead compounds (U.S.P. 1,622,228).—See XX.

Production of alcohols and hydrocarbons (F.P. 593,648—593,650, 594,121, and 598,447).—See XX.

### III.—TAR AND TAR PRODUCTS.

**Solubility of naphthalene in certain aromatic hydrocarbons.** F. H. RHODES and F. S. EISENHAEUER (Ind. Eng. Chem., 1927, 19, 414—416).—The solubility of naphthalene in toluene, xylene, refined heavy solvent naphtha, and refined coal-tar creosote oil has been determined. For temperatures over a range of 0° to 50° the solubility curves for toluene, xylene, and solvent naphtha are roughly parallel, and the solubility at any given temperature decreases as the mol. wt. of the solvent increases. The addition of cresol has no specific effect in increasing the solubility of naphthalene in solvent naphtha or creosote oil. E. H. SHARPLES.

#### PATENTS.

**Preparation of pure *p*-xylene.** E. MERCK, Assr. to A. DÜTZMANN (G.P. 434,988, 27.9.24).—*p*-Methylbenzyl chloride, b.p. 80°/8 mm., prepared, for example, by condensing toluene with trioxymethylene and hydrogen chloride at 60° in the presence of zinc chloride, is reduced to pure *p*-xylene. A quantitative yield is obtained with palladium and hydrogen, rather less with zinc dust or aluminium amalgam. C. HOLLINS.

**Distillation of tar** (U.S.P. 1,622,964).—See I.

### IV.—DYESTUFFS AND INTERMEDIATES.

**Naphthalenesulphonic acids. VII. Hydrolysis of naphthalene-1:6-disulphonic acid.** J. A. AMBLER and J. T. SCANLAN (Ind. Eng. Chem., 1927, 19, 417—420).—Quantitative examinations have been made of the degree of hydrolysis occurring when pure naphthalene-1:6-disulphonic acid is heated in sealed tubes with concentrations of sulphuric acid ranging from 1% to 85% and at temperatures varying from 100° to 230°. The temperatures of the commencement of hydrolysis for each concentration of sulphuric acid and the nature of the reaction products have been determined. The following general rule has been deduced. Naphthalene-1:6-disulphonic acid, when heated with sulphuric acid of a concentration not high enough to sulphonate naphthalene at the temperature employed, and at a temperature sufficiently high to effect hydrolysis, is converted directly into naphthalene. If the temperature is high enough to hydrolyse the 1:6-acid and the sulphuric acid strong enough to sulphonate naphthalene at the temperature used, isomeric disulphonic acids, *i.e.*, those which would be produced by the interaction of naphthalene and sulphuric acid under the same conditions of temperature and concentration, are formed. During hydrolysis both sulphonic acid groups of the 1:6-acid are removed simultaneously, and there is no intermediate formation of the  $\beta$ -acid. On heating the 1:6-acid at temperatures at which hydrolysis begins in an open system, the reaction will go to completion if provision is made for the removal of the naphthalene, and thus, in the vapour phase sulphonation of naphthalene, which is carried out in

an open system at 220—245° in 80—95% sulphuric acid, the 1:6-acid will not be present in the product. Also the 1:6-acid would not be affected in the steam treatment for the removal of tar, which is conducted at 100° in 10% sulphuric acid. (Cf. B., 1925, 66, 310.)

E. H. SHARPLES.

**Behaviour of oxides of lead towards dyestuffs. I.** A. N. ADAMSON and J. K. WOOD (J. Soc. Dyers and Col., 1927, 43, 47—51).—The absorption of the acid dyes Disulphine Green I, Patent Blue, Methyl Orange, Bordeaux B, Orange II, and Orange IV (the basic dyes Methylene Blue, Toluidine Blue, Safranin T, and Methyl Violet 10 B were not absorbed) by various samples of impure lead dioxide containing 92.6—98.5% PbO<sub>2</sub>, 1.0—5.8% PbO, and 0.4—1.2% of moisture appears to be a case of adsorption. Less dye (Orange II or Methyl Orange) is sorbed in the presence of sodium hydroxide, the reduction in the degree of sorption being independent of the amount of alkali present, whereas the presence of an acid increases the sorption. Dyes adsorbed by the samples were not completely removed by washing with water. It is probable that lead dioxide free from lead monoxide would have but little power of adsorption. The complete inability of lead dioxide to adsorb basic dyes is in striking contrast to the behaviour of the oxides of titanium and tin (Morley and Wood; B., 1923, 544 A, 545 A), and is probably to be associated with the definite basic properties of lead dioxide and its low degree of hydration. A. J. HALL.

#### PATENTS.

**Triarylmethane dyes.** I. G. FARBENIND. A.-G., Assees. of A.-G. F. ANILIN-FABR. (E.P. 249,160, 13.3.26. Conv., 14.3.25; and E.P. 250,576, 1.4.26. Conv., 9.4.25. Addn. to E.P. 249,160).—Basic violet dyes of the Victoria Blue type, which may be converted into acid dyes by sulphonation, are obtained by condensing either 1 or 2 mols. of a tetra-alkyl-4:4'-diaminobenzophenone with 1 mol. of an *NN'*-diarylated ethylenediamine in the presence of phosphoryl chloride. Michler's ketone is thus condensed with *NN'*-diphenyl-*NN'*-dimethyl- (or diethyl- or dibenzyl-) ethylenediamine, *NN'*-di-*o*-tolylethylenediamine, and *NN'*-di- $\alpha$ -naphthylethylenediamine. C. HOLLINS.

**Manufacture of sulphuric acid esters of oxyalkyl compounds of the aromatic series [sulphatoalkyl ethers of phenols].** W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 266,940, 4.5.26).—Intermediates, suitable for manufacture of soluble dyes particularly for acetate silk, are prepared by the action of sulphating agents (sulphuric acid, chlorosulphonic acid, etc.) on hydroxyalkyl ethers of phenols, naphthols, anthrols, etc. 3-Nitro-*p*-tolyl  $\beta$ -hydroxyethyl ether, m.p. 40°, obtained from 3-nitro-*p*-cresol and ethylene chlorohydrin, treated with concentrated sulphuric acid, yields a sulphate isolable as the potassium salt, NO<sub>2</sub>·C<sub>6</sub>H<sub>3</sub>Me·O·CH<sub>2</sub>·CH<sub>2</sub>·O·SO<sub>3</sub>K, which may be reduced, diazotised, and coupled with  $\beta$ -naphthol for a soluble bluish-red dye, or the reduced nitro-compound may be coupled with *p*-nitrodiazobenzene for a soluble yellowish-red dye suitable for acetate silk. Ethylene glycol mono- $\alpha$ -anthracyl ether, m.p. 117—118°, and 2- $\beta$ -hydroxyethoxynaphthalene also form sulphates.



From 1- $\beta$ -y-dihydroxypropoxynaphthalene the mono-sulphate,  $C_{10}H_7 \cdot O \cdot CH_2 \cdot CH(OH) \cdot CH_2 \cdot O \cdot SO_3Na$ , is obtained.

C. HOLLINS.

**Manufacture of coeruleinsulphonic acids.** I. G. FARBENIND. A.-G., Assees. of A.-G. F. ANILIN-FABR. (E.P. 251,968, 26.4.26. Conv., 7.5.25).—Coeruleins, *e.g.*, from pyrogallolphthalein or from hydroxyquinolphthalein, are converted into soluble sulphonic acids, suitable for mordant dyeing, by treatment with fuming sulphuric acid and boric acid at 130–140°.

C. HOLLINS.

**Manufacture of [perylene] dyestuffs.** A. ZINKE (E.P. 242,306, 30.10.25. Conv., 3.11.24).—Halogenated diaroylperylene, in particular 3:9-dibromo-4:10-dibenzoylperylene (*cf.* A., 1925, i, 383), are treated at high temperatures with alkalis or alkaline-earths in the presence or absence of organic solvents, in particular aniline. The products dye cotton from a blue vat in violet shades fast to chlorine, acids, alkalis, and washing.

A. DAVIDSON.

**Copper [compounds of] azo dyestuffs.** T. H. LEAMING, Assr. to NATIONAL ANILINE & CHEMICAL CO., INC. (U.S.P. 1,618,762, 22.2.27. Appl., 22.6.25).—A solution of copper sulphate or other soluble copper salt is added to a neutral or slightly alkaline aqueous solution of an azo dyestuff obtained by coupling, in presence of sodium carbonate, 1 or 2 mols. of a diazotised anthranilic acid, or 1 mol. of the latter and 1 mol. of another diazo-compound, with 1 mol. of 5:5'-dihydroxy-2:2'-dinaphthylamine-7:7'-disulphonic acid. The mixture is made alkaline, and the copper derivative of the dyestuff formed is salted out. These products dye cotton and silk bright reddish-violet shades.

T. S. WHEELER.

**Manufacture of [azo] dyestuffs.** F. STRAUB and H. SCHNEIDER, Assrs. to SOC. OF CHEM. IND. IN BASLE (U.S.P. 1,617,872, 15.2.27. Appl., 11.3.26. Conv., 28.3.25; *cf.* E.P. 249,884).—Azo dyes, derived from *o*-phenolazo-2- $\alpha$ -naphthol-8-sulphonic acid in which positions 3, 4, and 6 of the naphthalene nucleus are substituted at least once, and at most twice, by non-adjacent sulfo-groups, of which one is in the 3- or 4-position, are treated with a metallic, *e.g.*, copper or chromium, salt to yield dyes, which give on wool in an acid bath fast, pure, bordeaux to violet, blue, and black shades. *E.g.*, diazotised *p*-chloro-*o*-aminophenol is coupled with  $\alpha$ -naphthol-3:8-disulphonic acid, and an aqueous solution of the product is treated with copper sulphate and sodium acetate to give a substance which dyes wool red-violet shades.

T. S. WHEELER.

**[Mordant] azo dyes.** BRITISH DYESTUFFS CORP., LTD., K. H. SAUNDERS, and H. GOODWIN (E.P. 266,561, 17.4.26. Addn. to E.P. 242,061; B., 1926, 7).—1-Amino-2-hydroxy-6-sulpho-3-naphthoic acid is diazotised and coupled with a sulphonated naphthol or dihydroxynaphthalene, to give reddish- to greenish-blue chrome printing colours for cotton or mordant dyes for wool. Examples of end-components are  $\alpha$ -naphthol-4:8-disulphonic acid, 1:8-dihydroxynaphthalene-3:6-disulphonic acid, and 1:8-dihydroxynaphthalene-4-sulphonic acid.

C. HOLLINS.

**Disazo dye of diphenylcarbamide and chromotrope acid.** R. J. FLETCHER, Assr. to AMALGAMATED

DYESTUFF AND CHEMICAL WORKS, INC. (U.S.P. 1,617,244, 8.2.27. Appl., 19.4.26).—Diazotised *p*-nitroaniline is coupled with 1:8-dihydroxynaphthalene-3:6-disulphonic acid, and the product is treated with sodium sulphide to reduce the nitro-group and then with phosgene to give *s*-di-(1:8-dihydroxy-3:6-disulphonaphthalene-2-azo-*p*-phenyl)carbamide, which gives direct brilliant violet shades, fast to light, but soluble in water. The compound can also be prepared by coupling diazotised *p*-aminoacetanilide with 1:8-dihydroxynaphthalene-3:6-disulphonic acid, hydrolysing the acetamido-group, and treating the product with phosgene, or by coupling tetrazotised *pp'*-diaminodiphenylcarbamide with 1:8-dihydroxynaphthalene-3:6-disulphonic acid.

T. S. WHEELER.

**Manufacture of azo dyes.** W. CARPMAEL. From FARBENFABR. VORM. F. BAYER & Co. (E.P. 266,771, 5.10.25).—Arylsulphamic acids coupling in the *para* position are used as middle components in the manufacture of secondary disazo pigment dyes or ice-colours. Examples are: *o*-anisidine  $\rightarrow$   $\alpha$ -naphthylsulphamic acid  $\rightarrow$  2:3-hydroxynaphthoic anilide or  $\beta$ -naphthylamide (black on the fibre) or 1-phenyl-3-methylpyrazolone (scarlet on the fibre); aniline  $\rightarrow$  bisacetacetotolidine (orange on the fibre). [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 238,683 (B., 1926, 840).]

C. HOLLINS.

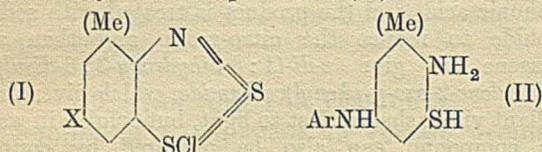
**Manufacture of monoazo dyes.** W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 265,767, 30.1.26).—Toluidine- $\omega$ -sulphonic acids are diazotised and coupled with non-sulphonated, non-carboxylated arylamines, and nitrotoluidinesulphonic acids are diazotised and coupled with  $\beta$ -naphthylamine derivatives, including aminonaphthols and carboxylic and sulphonic acids. The products dye acetate silk in shades fast to washing, and unmordanted wool in level shades fast to light. 23 examples are given, including the coupling of diazotised *p*-toluidine- $\omega$ -sulphonic acid with diphenylamine (yellow) and methyl- $\beta$ -naphthylamine (orange); 5-nitro-*o*-toluidine- $\omega$ -sulphonic acid with benzylethylaniline (red), methyldiphenylamine (red), 2- $\beta$ -hydroxyethylnaphthylamine (red-violet), 2-methylaminonaphthalene-7-sulphonic acid (red-violet),  $\gamma$ -acid (violet), and methyl- $\gamma$ -acid (blue-violet); 3:5-dinitro-*o*-toluidine- $\omega$ -sulphonic acid (G.P. 141,783) with ethyl- $\beta$ -naphthylamine (blue-violet), methyl- $\gamma$ -acid (blue), 2:3-aminonaphthoic acid (red-violet), 2:8-aminonaphthol (blue-black), Brönner's acid (bordeaux), phenyl-Brönner's acid (violet); and 3:5-dinitro-*p*-toluidine- $\omega$ -sulphonic acid (G.P. 134,988) with  $\beta$ -naphthylamine (blue-red), ethyl- $\beta$ -naphthylamine (bordeaux), and 2-methylamino- or 2-ethylaminonaphthalene-7-sulphonic acid (bordeaux). Monoazo mordant dyes (*cf.* E.P. 18,139 of 1902) are disclaimed.

C. HOLLINS.

**Manufacture of new aromatic compounds and dyes therefrom.** A. J. RANSFORD. From L. CASSELLA & Co., G.M.B.H. (E.P. 265,641, 7.8.25).—Compounds of the type (I), prepared from arylamines and sulphur chloride (E.P. 17,417/14), where X is a labile substituent such as halogen or alkoxyl, react with arylamines to form



arylamino-derivatives, which are converted by alkalis, especially in the presence of a reducing agent, into 2-amino-5-arylaminothiophenols (II). The latter



compounds condense with benzo- or naphtha-quinones to give violet to blue to black quinone vat dyes for wool, which probably contain the thiazine nucleus. Examples are given of the preparation of 5-anilino-3-mercapto-*o*-toluidine (II, Ar = Ph) and its condensation with chloranil for a violet-blue dye; of 1-amino-4-anilino-2-thionaphthol and its condensation with 2-hydroxy-1:4-naphthaquinone or 2:3-dichloro-1:4-naphthaquinone for grey-blue to black dyes; of 5-anilino-3-mercapto-*o*-anisidine and its condensation with chloranil for an indigo-blue dye; of 5-(*o*- and *m*-carboxyanilino)-3-mercapto-*o*-toluidines and their condensation with chloroanil etc. for violet to greenish-blue dyes. Alternative routes to the dyes and intermediates are also covered.

C. HOLLINS.

**Preparation of 2:7-dinitroanthraquinone.** GRASSELLI DYESTUFF CORP., Assees. of B. STEIN (U.S.P. 1,622,168, 22.3.27. Appl., 28.6.26. Conv., 25.6.25).—Tetranitrodianthrone is treated with pyridine to form pyridine salts of tetranitrodianthranol, which is converted into 2:7-dinitroanthraquinone by removal of pyridine with acid followed by oxidation with concentrated nitric acid.

C. HOLLINS.

**Manufacture of alanines of the anthraquinone series and derivatives thereof.** I. G. FARBENIND. A.-G., Assees. of FARBENFABR. VORM. F. BAYER & CO. (E.P. 246,840, 26.1.26. Conv., 31.1.25).— $\beta$ -Chloropropionic acid is condensed with aminoanthraquinones, or  $\beta$ -alanine with halogenated anthraquinones, giving  $\beta$ -alanines of the anthraquinone series. Examples are the condensation of  $\beta$ -chloropropionic acid with 2-amino-, 1:4-diamino-, and 1:4:5:8-tetra-amino-anthraquinones. The products dye cellulose acetate and animal fibres.

C. HOLLINS.

**Manufacture of vat colouring matters of the anthraquinone series [acylated aminoanthraquinones].** J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 264,561, 10.10.25).—Aminoanthraquinones are acylated in at least one  $\alpha$ -amino-group by benzoic acid derivatives substituted in the *m*-position by a difficultly attackable group such as alkyl, alkoxy, etc. The acylation is accomplished by boiling the amine in a suitable solvent with the *m*-substituted benzoyl chloride or benzoic anhydride, or by heating a mixture of the amine and the *m*-substituted benzoic acid in the presence of a condensing agent such as thionyl chloride, phosphorus pentachloride, etc. The examples given include, as acylating agents, *m*-methoxy- and -ethoxy-benzoic, *m*-toluic, isophthalic, *m*-benzamidobenzoic, 3-methylthiolbenzoic, diphenyl-*m*:*m'*-dicarboxylic, trimesic, and 5-methoxyisophthalic acids, whilst the amines used include 1-aminoanthraquinone, 1:4- and 1:5-diaminoanthraquinones and their monobenzoyl derivatives,

1:5-diamino-4-hydroxy- and -4-methoxy-anthraquinones, and 4:8-diaminoanthrarufin. 1-Amino-5-methoxyanthraquinone, m.p. 225°, obtained from 5-chloro-1-aminoanthraquinone and sodium methoxide, is also used. The products are vat dyes of good affinity and fastness to light.

A. DAVIDSON.

**Manufacture of vat dyestuffs [dialkoxyisodibenzanthrones].** J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 264,631, 19.11.25).—Alkyl ethers of hydroxybenzanthrones substituted by halogen in the 3-position, but with a free 4-position, are treated with alkaline condensing agents. *E.g.*, 3-chloro-2-methoxybenzanthrone (obtained, *e.g.*, by treating 2-methoxybenzanthrone with sulphuryl chloride in nitrobenzene solution or by methylating 3-chloro-2-hydroxybenzanthrone) is heated with potassium ethoxide for 1 hr. at 145–150°, diluted with water, boiled, and aerated, when the dye is precipitated. It gives a blue vat, and dyes cotton greenish-blue. Excluded from the patent are those halogenoalkoxybenzanthrones obtained by nitrating 3-chlorobenzanthrone, reducing the nitro-compound, substituting a hydroxyl for the amino-group, and alkylating the product.

A. DAVIDSON.

**Manufacture of condensation products of the anthracene series [Bz-methylbenzanthrones].** O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS, UND BRÜNING (E.P. 244,120, 4.12.25).—Anthrones or anthranols are condensed with crotonaldehyde in the presence of pyridine and piperidine and subsequently cyclised to Bz-methylbenzanthrones by means of aluminium chloride. By using an acid condensing agent (hydrogen chloride, sulphuric-acetic acid) in place of the base the reaction proceeds to completion in one stage. An oxidant (arsenic acid) may be added. 1-(or 3-)Methylbenzanthrone, m.p. 113–114°, from anthranols and crotonaldehyde in sulphuric-acetic acid at 115–120°, distils in steam superheated at 300°. By the two-stage process anthrone and crotonaldehyde give a Bz-methylbenzanthrone, m.p. 168°. From  $\alpha$ -hydroxyanthranol a hydroxy-Bz-methylbenzanthrone is obtained.

C. HOLLINS.

Colour lakes (E.P. 265,032).—See XIII.

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

**Sizing of artificial silk.** W. BRUCKHAUS (Kunstseide, 1926, 8, 319–320).—Pastes containing glue or gelatin are much more suitable than those containing ordinary starch for sizing artificial silk yarn so that it retains its lustre and acquires suitable elasticity for purposes of weaving. Suitable sizes are prepared by (1) boiling 1 kg. of bone-gelatin (previously steeped in cold water for 12–24 hrs.), 1 kg. of gelatin, and 24 litres of water, then adding 200 g. of olive oil, 50 g. of anhydrous sodium carbonate, and 100 g. of glycerin, or (2) boiling 2 kg. of glue (previously steeped in cold water for 12 hrs.) and 25 litres of water, then adding 500 g. of Marseilles soap and 150 g. of Brilliant Monopol Oil G; both sizing preparations are used at 50°.

A. J. HALL.

**Viscose solutions. II. Turbidity measurements of the changes of state.** T. MUKOYAMA (Kolloid-Z., 1927, 41, 158–163).—The effects on the



turbidity of solutions of viscose due to changes of concentration, age, temperature, and alkali content were studied. The turbidity of a freshly prepared sol increases linearly with concentration, but after a few days the increase of turbidity with concentration becomes much greater, reaches a maximum, and then falls off, the departure from the original curve increasing with the age of the sol. The maximum values for middle concentrations correspond with the maximum viscosity, and in old sols the phenomena of gelatinisation and syneresis are observed. In preparations containing a high percentage of alkali the maximum is followed by a minimum, but this is due to removal of some of the substance by precipitation. With rise of temperature the turbidity diminishes. With increasing content of alkali the turbidity also diminishes, the effect being greater the older the sol.

E. S. HEDGES.

**Viscosity of viscose solutions.** P. WAENTIG (Kolloid-Z., 1927, 41, 152–157).—Experiments are given which show that when cellulose is very finely powdered before conversion into viscose the product has a much smaller viscosity than ordinary viscose. Such finely-divided cellulose is also much more readily attacked by solutions of sodium hydroxide. The decrease in viscosity of solutions of viscose (cf. Heuser and Schuster; B., 1926, 399) with age is discussed in the light of these experiments. Such an ageing effect takes place only when the excess of caustic soda has been removed.

E. S. HEDGES.

**Solubility of cellulose derivatives.** W. VON NEUENSTEIN (Kolloid-Z., 1927, 41, 183–185).—Ostwald's peptisation rule (A., 1927, 310) is found to hold for the dissolution of cellulose acetate in chloroform and in ethyl acetate, and for cellulose nitrate in a mixture of alcohol, benzene, and acetone, the amount dissolved increasing with the amount of colloid initially present. Cellulose nitrate after extraction gave a curve showing that the amount dissolved by the liquid mixture is, above a certain concentration, independent of the initial quantity present.

E. S. HEDGES.

**Autoxidation of lignin.** H. DIRTZ and R. MAY (J. pr. Chem., 1927, [ii], 115, 201–215).—Fischer and Schrader (A., 1922, i, 637; 1923, i, 185) found that, in presence of sodium hydroxide solution, lignin absorbs oxygen more readily than does pine-wood sawdust, and suggested that the lignin in the latter has a protective covering of cellulose. Consideration of the lignin content of the two specimens indicates, however, that that of the pine-wood is actually the more readily oxidised. This is attributed to the presence of catalysts. Since manganese is an important inorganic constituent of pine-wood, its effect on the autoxidation of purified, manganese-free lignin suspended in 2*N*-sodium hydroxide has been examined. Addition of 0.05% of bivalent manganese produces an initial increase in the rate of oxidation, which disappears after the first 24 hrs. With 0.5% of manganese the increase is not so marked. This effect is probably not purely catalytic, the oxidation of the lignin being induced by the simultaneous autoxidation of the manganese to the quadrivalent state (cf. Meyer and Nerlich; A., 1921, ii, 509).

H. E. F. NOTTON.

**Pulping flax straw.** Hydrolysis with sodium

sulphite. M. W. BRAY and C. E. PETERSON (Ind. Eng. Chem., 1927, 19, 371–372).—The digestion of flax straw at 155° with 20% and 40% of sodium sulphite at a concentration of 40 g./litre and with 40% of sodium sulphite at concentrations of 40 and 80 g./litre has been studied. Whilst an increase in the percentage of sodium sulphite greatly accelerates the rate of cooking, an increase in the concentration of the cooking liquor has but little effect. During the first hour the incrustants are removed at a rapid rate, and it is during this period that the loss of cellulose by hydrolysis is most pronounced. Beyond this point the rate of change of yield and chemical properties with time is comparatively slow. Pentosans in the cellulose as obtained by Cross and Bevan's method remain practically constant during the whole cooking period. Details are given of the chemical properties of the pulps obtained in these experiments.

D. J. NORMAN.

**Pulping flax straw. II. Chemical studies with chlorine as a pulping agent.** E. R. SCHAFER, M. W. BRAY, and C. E. PETERSON (Pulp and Paper Mag., 1927, 25, 209–212).—The pulping of flax straw by digestion with soda followed by chlorination has been studied in order to determine the relationship between the proportion of soda used in the preliminary digestion and the quantity of chlorine required to complete the pulping. For soda pulps prepared by digestion for 2–12 hrs. at a maximum temperature of 155° and showing soda consumption of 2–18.7% (on the weight of straw), there is a linear relationship between the soda consumption and the chlorine requirements, the latter ranging from 23.9 to 2.7% on the weight of straw. The final pulps showed practically identical properties irrespective of the relative amounts of soda and chlorine used in their preparation.

D. J. NORMAN.

**Soda pulp investigation. II. Yield and quality of pulp obtained from the birches and maples.** D. E. CABLE, R. H. MCKEE, and R. H. SIMMONS (Pulp and Paper Mag., 1927, 25, 243–247; cf. B., 1926, 1007).—The data of a series of experimental cooks on white birch (*Betula papyrifera*), yellow birch (*Betula latea*), silver maple (*Acer saccharinum*, L.), hard maple (*Acer saccharum*, Marsh), and red maple (*Acer rubrum*, L.) are given. The samples were digested with 25% of soda on the weight of dry chips at 170° for 4–7 hrs., 4–5 hrs. being sufficient for white birch, yellow birch, and silver maple. Variations in the causticity of the fresh cooking liquor from 60 to 98% had no apparent effect on the yield and character of the resulting soda pulps, provided that the other cooking conditions were unchanged. The yields of pulp varied from 43.3% for hard maple and red maple to 44–48.6% for the other woods; calculated on a cord basis, these figures are equivalent to 1620–1980 lb. of dry pulp per cord of wood. The bleach requirements of these pulps were 10.7–14.7% (on the weight of air-dry pulp). Pitting of digester covers, which is attributed to the presence of traces of volatile organic acids in the digester vapours, should not be more serious when cooking birch and maple than is at present the case with aspen.

D. J. NORMAN.

**Recovery and utilisation of waste liquors in the pulp industry.** U. POMILIO (Ind. Eng. Chem., 1927,



19, 344—346).—In view of the fact that one of the main difficulties attending the economic treatment of the waste liquors from the cellulose industry is their low concentration, attention is drawn to the chlorination process for manufacturing pulp, in which, by continued re-use of the liquors, it is possible to obtain a final alkali waste liquor containing about 150 g./litre of organic matter. Hägglund's process (cf. E.P. 258,035; B., 1927, 165), which involves heating the liquor under pressure, is suggested as being the most promising method of treating these liquors and also those obtained in the soda process.

D. J. NORMAN.

**Waste sulphite liquor as an agricultural spray.** C. S. FLEMING and J. H. REEDY (Chem. Met. Eng., 1927, 34, 159).—For use as an agricultural spray, it is suggested that sulphite waste liquor be saturated with hydrogen sulphide, thereby reducing calcium hydrogen sulphite with the separation of free sulphur. The liquor contains 7—9% of a sugar-like substance which aids the suspension of the sulphur particles, the permanence of which is improved by the addition of 0.5% of gelatin or maize sugar. If the sulphur is flocculated and separated, the solution contains fermentable material which might be utilised for the production of alcohol.

C. A. KING.

**Degree of sizing and finish [of paper].** H. SCHWALBE (Papier-Fabr., 1927, 25, 173—176).—A review of various methods for determining the degree of sizing of paper is given, that due to Albrecht being particularly described (Finska Pappers- och Trävarutidskr. för Finland, 1926, Nos. 10 and 11). With this apparatus reliable results may be obtained, and the method also enables the disposition of the size to be observed. The humidity of the air and temperature of the ink both influence the size testing, 65% R.H. and 24° being recommended as standard conditions. Curves for samples examined show that for a rise in temperature from 10° to 30° the time of penetration of a water-glass-sized wood-pulp paper decreased only slowly, but for rosin- and animal-sized rag papers a rapid decrease was shown. Concordant results are obtained for samples lying adjacent to one another in the same sheet. Variation in the experimental conditions (e.g., increasing the depth of the ink column in penetration tests) affects the results. Differences due to the effects of moist- and dry-glazing actions of the paper machines and also of calendering are indicated for samples tested before and after these operations.

B. P. RIDGE.

**Spanish moss.** SCHORGER.—See XX.

## PATENTS.

**Manufacture of artificial silk.** BRITISH ENKA ARTIFICIAL SILK CO. LTD., Assees. of N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (E.P. 259,528, 18.11.25. Conv., 12.10.25).—Threads of artificial silk of different denier are kept apart during manufacture by dyeing the silk with different colours according to the denier, either during or after spinning, such colours being subsequently removed or decolorised. B. P. RIDGE.

**Manufacture of artificial silk.** C. C. JESSEN, Assr. to ATLAS POWDER CO. (U.S.P. 1,621,590, 22.3.27. Appl., 20.3.25).—The thread is wound first into a cake

in a centrifuge and then from the outside of the cake on to a cylinder, the latter being of such size with respect to the diameter of the cake that, as the thread is wound upon it, those portions which were under the greatest tension in the centrifuge are subjected to the least tension during winding, and *vice versa*.

B. P. RIDGE.

**Manufacture of viscose solutions.** S. NEUMANN (E.P. 265,685, 10.11.25).—Suitable liquids are emulsified with completely dissolved viscose in high-speed mixers running at speeds greater than 1000 r.p.m. This treatment homogenises the viscose solution, provides a means of introducing liquid paraffin and other hydrocarbons, and accelerates the ripening process to such an extent that by adjusting the duration of the mechanical treatment and the temperature, which, however, should not exceed 50—60°, it is possible to complete the ripening process in a few hours. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 234,039, 189,114, 184,533, 143,253, and 1020/98 (cf. B., 1925, 914; 1923, 179 A; 1922, 806 A; 1921, 689 A).]

D. J. NORMAN.

**Production of cellulosic material.** A. H. LYMN and C. LEYST (E.P. 265,697, 14.11.25).—A process is described for preparing crude bamboo pulp, which, when formed into blocks and dried, coheres sufficiently well to be transported. Bamboo stems are cut into pieces 1—5 mm. in thickness and boiled for 10 hrs. at 95—100° with about 35% of lime on the weight of dry bamboo. The product is washed, ground with water for 1.5—2 hrs., pressed into cakes containing approximately 50% of water, and dried. This crude pulp may be subsequently converted into paper pulp by digestion with 6—9% of caustic soda (calculated on dry material) for about 4 hrs. at a pressure of 2 atm. followed by treatment with 25—50% of bleaching powder.

D. J. NORMAN.

**Treatment of wood and like porous material.** M. SMITH and P. PHIPPS (E.P. 265,643, 17.8.25).—Woods and like porous material, particularly those varieties of wood which are not naturally suitable for the manufacture of articles such as lead pencils, are seasoned, softened, and uniformly tinted throughout by impregnating, *in vacuo* if desired, the material, previously dried at 105—127°, with a softening medium, e.g., an 18% emulsion of Turkey red oil, and subjecting it to a comparatively high temperature so that the softening agent volatilises and completely penetrates the interstices of the wood. Suitable apparatus is described.

D. J. NORMAN.

**Production of moulded pieces from dry mixtures of cellulose derivatives and albuminous substances.** F. SCHMIDT (E.P. 248,781, 8.3.26. Conv., 6.3.25).—Albuminous substances are kneaded with cellulose derivatives in the presence of suitable swelling agents, e.g., water in admixture with volatile or non-volatile solvents. The resulting homogeneous mixture is dried, powdered, and subjected to heat and pressure in moulds of the desired shape. *Example*: An intimate mixture of glue (30 pts.), water, and ethylene chlorohydrin is incorporated with a homogeneous cellulose acetate composition containing 50 pts. of cellulose acetate, 20 pts. of water, and 20 pts. of methyl phthalate.



Fillers and hardening agents, *e.g.*, formaldehyde, may be added. D. J. NORMAN.

**Manufacture of felt.** C. and E. PICHARD (U.S.P. 1,622,883, 29.3.27. Appl., 12.2.26. Conv., 28.2.25).—See E.P. 248,343; B., 1926, 532.

**Effecting agglomeration in paper pulp and other suspensions and mixtures.** K. SVEEN (U.S.P. 1,622,474, 29.3.27. Appl., 17.7.25. Conv., 21.7.24).—See E.P. 237,292; B., 1925, 843.

**Pumps for artificial silk spinning machines.** P. HILLEBRAND and A. OLSON (E.P. 267,265, 25.1.26).

**Artificial silk spinning or like machines.** J. L. RUSHTON and H. HILL (E.P. 266,821, 5.12.25).

**Artificial silk spinning machines.** J. L. RUSHTON and J. LEVER (E.P. 266,835, 8.12.25).

**Apparatus for drying fibrous materials such as fabric, leather, yarns and the like.** BARROW, HEPBURN, & GALE, LTD., and A. HAWKLYARD (E.P. 266,464, 27.11.25).

**Softening, spinning, and twisting artificial silk.** R. C. BOGER (E.P. 266,438, 23.11.25).

**Paper-making machines.** INDIA RUBBER, GUTTA PERCHA & TELEGRAPH WORKS CO., LTD., and B. WALKLEY (E.P. 266,465, 27.11.25).

**Machine for drying or chemically treating continuous lengths of fabric or paper.** A. LAMBRETTE (E.P. 266,649, 13.12.26. Addn. to 255,297; B., 1926, 740).

**Drying cylinders** (E.P. 266,481).—See I.

**Purification of caustic soda solutions from viscose treatment** (E.P. 265,126).—See VII.

**Alloys** (G.P. 435,170).—See X.

**Solvent for cellulose ester** (E.P. 252,203).—See XIII.

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

### PATENTS.

**Producing fast coloured resists under Aniline Black.** I. G. FARBERIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, UND BRÜNING (E.P. 247,211, 5.2.26. Conv., 5.2.25).—An enol ester ("indigo-sol") of a leuco-vat dye, to which is added ammonium oxalate or other substance producing an acid reaction when steamed, is printed on material padded with aniline. The material is then steamed, passed through a weak dichromate bath, washed, and soaped. Under these conditions the leuco-ester is oxidised in preference to the aniline, and so produces coloured resists under Aniline Black. C. HOLLINS.

**Dyeing leather with acid azo dyes.** I. G. FARBERIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, UND BRÜNING (E.P. 247,187, 1.2.26. Conv., 3.2.25).—Full brown shades are obtained on leather by using polyazo dyes of the type: amine (I)  $\rightarrow$  mono- or di-sulphonated naphthylamine or aminonaphthol (II)  $\rightarrow$  non-sulphonated dihydric phenol (III) such as resorcinol  $\leftarrow$  amine (IV), the amines (I and IV) being any diazotisable arylamines, including aminophenols, amino-

azo compounds, and their carboxylic or sulphonic acids. The dyeing of chrome leather with the dye, picramic acid  $\rightarrow$  H-acid  $\rightarrow$  resorcinol  $\leftarrow$  *p*-nitroaniline, is described. Other examples are *o*-toluidine-5-sulphonic acid or sulphanilic acid or naphthionic acid with H-acid, resorcinol, and *p*-nitroaniline; sulphanilic acid  $\rightarrow$  Cleve's acids  $\rightarrow$  resorcinol  $\leftarrow$  *p*-nitro-*o*-aminophenol; *p*-nitro-*o*-aminophenol  $\rightarrow$  Cleve's acids  $\rightarrow$  resorcinol  $\leftarrow$  naphthionic acid; and picramic acid  $\rightarrow$  H-acid  $\rightarrow$  resorcinol  $\leftarrow$  picramic acid. C. HOLLINS.

**Machine for washing, dyeing, or treating material.** E. C. R. MARKS. From MAX AMS CHEMICAL ENGINEERING CORP. (E.P. 266,551, 25.3.26).

[Vat for] dyeing knitted, woven, or the like articles. C. CALLEBAUT and J. DE BLICQUY (E.P. 266,227, 30.11.25).

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

**Determination of water content in concentrated sulphuric acid by thermometric titration.** T. SOMIYA (J. Soc. Chem. Ind. Japan, 1927, 30, 106—112).—When fuming sulphuric acid is added to concentrated sulphuric acid containing a small percentage of water, the temperature of the solution rises, but when the water content is exhausted no further heat is evolved by the continued addition of the fuming acid. By tracing curves correlating temperature elevation and amount of fuming acid, two curves are obtained and their point of intersection gives the end point of the reaction, by which the amount of water in the concentrated acid is determined. Fuming acid is gradually added from a burette into concentrated acid contained in a test tube inserted into a Dumas flask, and the temperature elevation of the mixture is measured. The method is best applied when the concentration of the sample is 90% or more. The error is 0.2—0.5%, according to the amount of the material taken. Commercial fuming acid of any concentration can be used as the standard solution, but that of 10—25% is most suitable, due to its low m.p. The standardisation should be carried out thermometrically with 80% sulphuric acid. K. KASHIMA.

**Sulphur from gas.** CUNDALL.—See II.

**Magnesium carbonate for rubber.** TANAKA.—See XIV.

### PATENTS.

**Concentration of nitric acid.** M. KALTENBACH (Addn. No. 30,931, 11.9.25, to F.P. 594,865; B., 1926, 236).—The sulphuric acid used in the prior process is completely denitrated before entry into the third element of the concentrator, by passing a small quantity of steam through it in the second element.

L. A. COLES.

**Apparatus for the purification of impure solutions of caustic soda or the like on osmotic principles.** L. CERINI (E.P. 265,126, 16.6.26. Conv., 29.1.26).—Caustic soda containing colloidal impurities from viscose treatment etc. is purified in an iron reservoir containing vertical partitions dividing it into intercommunicating parallel compartments through which the caustic soda circulates. In each compartment are



disposed semi-permeable diaphragms fastened at their edges to form a tubular bag or sack and in communication with each other to form a conduit for water, which may traverse the apparatus in counter-current to the impure solution. The diaphragms are of cotton, linen, or hemp either in the natural condition or parchmented by acids, alkalis, or salts. W. G. CAREY.

**Process and apparatus for drying salt.** P. H. MÜLLER (G.P. 435,837, 1.9.23).—Salt is dried in a current of superheated steam in a chamber shut off from the air, in which the salt drops over a series of hot tubes rounded on the lower side and of acute-angled cross-section on the upper side. L. A. COLES.

**Heat economy in the production of salt.** METALLBANK AND METALLURGISCHE GES. A.-G., and W. GENSECKE (G.P. 435,589, 15.3.24).—The waste heat of the steam generated in a closed pan for evaporating brine is used for heating the next pan of the series, and the steam generated in this pan is mixed with air which has preferably been preheated. L. A. COLES.

**Electrolysis of alkali chlorides.** F. GERLACH (Addn. No. 30,954, 22.9.25. Conv. 22.12.24, to F.P. 596,918; B., 1926, 284).—Mercury or other fluid metal is transported in the presence of liquids, *e.g.*, water, alkali lyes, salt solutions, etc., by a device comprising a vertical or inclined screw provided above with a curved plate which participates to some extent in the rotation. J. S. G. THOMAS.

**Production of alkali iodides from absorption charcoal containing iodine.** N. V. BOMMAATSCHAPPIJ ARINA (Dutch P. 15,378, 16.4.25).—The charcoal is treated with approximately the theoretical quantities of an alkali bisulphite solution and an alkali carbonate, and extracted with water. L. A. COLES.

**Production of azoimide solution from its alkali salts.** DEUTSCHE GASGLÜHLICHT-AUER-GES.M.B.H., Assees. of J. EINIG (G.P. 435,654, 8.9.23).—Alcohol and oxalic acid in sufficient quantity to precipitate sodium dioxalate or potassium dioxalate or tetroxalate are added to a solution containing an alkali azide, and the precipitate is removed. L. A. COLES.

**Production of nitrates and a dry mixture of chlorine and nitrosyl chloride.** W. DOMINIK (F.P. 611,652, 24.2.26. Conv., 11.3.25).—Metal chlorides, *e.g.*, potassium chloride, are treated with an excess of hot nitric acid containing more than 440 g. HNO<sub>3</sub> per litre, under such conditions that there is a difference of not less than 7 g.-equivalents per litre between the concentration of the nitric acid and that of the chlorine ions. L. A. COLES.

**Production of alkali bisulphites or metabisulphites from alkali sulphates and alkaline-earth bisulphites.** CHEM. FABR. KALK G.M.B.H., H. OEHME, and E. HERRMUTH (G.P. 435,528, 5.11.25).—Alkali sulphates are added in small quantities to a solution or suspension of an alkaline-earth bisulphite. L. A. COLES.

**Preparation of a mixed salt of calcium phosphate and carbonate for nutritive purposes.** C. MÜNSTER and K. THORMANN (G.P. 398,093, 30.11.21).—After the acid phosphate solution has been treated with sufficient

milk of lime to transform the free phosphoric acid into monocalcium phosphate, it is thoroughly mixed with the calculated amount of calcium carbonate required to yield a mixed salt with a definite phosphoric acid content. F. R. ENNOS.

**Manufacture of active colloids.** I. G. FARBENIND. A.-G. (E.P. 255,863, 16.7.26. Conv., 22.7.25).—Active silica is made from a silica jelly containing at least 50 g./litre of silica and no free alkali, by drying the jelly rapidly above 120° with hot combustion gases in a channel or revolving furnace, and heating the mass to incandescence. The process is applicable to all colloids the gels of which can be retransformed into hydrosols. W. G. CAREY.

**Drying and grinding Glauber's salt.** G. POLYSIUS EISENGIESSEREI U. MASCHINENFABR. (G.P. 435,725, 27.9.23. Addn. to G.P. 398,048; B., 1925, 10).—The salt leaving the drying drum passes direct into an attached grinding mill. L. A. COLES.

**Extraction of beryllia from minerals.** L. PETIT-DEVAUGELLE (F.P. 611,095, 20.5.25).—The powdered mineral is treated with sufficient alkali hydroxide solution to yield a solution containing an aluminium beryllium alkali trisilicate, and the solution is filtered, treated with carbon dioxide to precipitate silica and alumina, which are removed, and then boiled to precipitate beryllia. L. A. COLES.

**Production of beryllium oxide.** O. LÉONARD (F.P. 611,457, 9.6.25).—Minerals containing beryllium and hydrofluoric acid, fluorides, or difluorides, are treated successively with sulphuric acid and with excess ammonium carbonate, and the solution obtained is boiled. L. A. COLES.

**Treatment and digestion of natural aluminium hydroxides.** F. KLIENMANN, and BÜTTNER-WERKE A.-G. (E.P. 266,225, 25.8.25).—Unroasted bauxite is ground with caustic alkali solution in a wet-grinding mill, the resulting fine sludge is treated with chlorine or hypochlorite to free it from organic impurities, and the aluminate-hydrate solution is filtered. The whole operation may be conducted in a ball mill with a long tube so that the alkali and oxidising agents are admitted during the operation, the filtration taking place through the outlet end of the tube, which is pierced and fitted with a filter. The caustic alkali and chlorine may be supplied by an electrolysed solution of sodium chloride. W. G. CAREY.

**Preparation of hydrogen cyanide.** I. G. FARBENIND. A.-G. (Swiss P. 115,702, 6.4.25. Conv., 25.8.24).—Mixtures of methyl formate and ammonia are led at 200° over a catalyst, *e.g.*, activated alumina, giving much hydrogen cyanide together with formamide. C. HOLLINS.

**Preparation of sulphur monochloride.** E. TERLINCK (F.P. 611,141, 27.5.25).—Chlorine is passed into a hot or boiling solution of sulphur in sulphur dichloride. J. S. G. THOMAS.

**Separation of a mixture of hafnium and zirconium.** W. J. TENNANT. From N. V. PHILIPS' GLOELAMPENFABR. (E.P. 266,800, 28.11.25).—A mixture of hafnium and zirconium phosphate is introduced into



a medium containing hydrofluoric acid or a difluoride, and is fractionally decomposed by adding a soluble zirconium salt which withdraws the fluorine from the mixture, the hafnium and zirconium phosphates being precipitated, giving at each fractionation a mixture richer in hafnium.

W. G. CAREY.

**Decomposition of hydrogen sulphide and its removal from industrial gases.** W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 267,018, 17.9.26).—The hydrogen sulphide is oxidised by bringing pulverised active carbon into suspension in the gases in a disintegrator mill, the gases then being freed from carbon and sulphur by passing them through a direct-current, high-tension field. The carbon charged with sulphur is regenerated by contact with heated surfaces arranged in cascade, or by sprinkling it while upon a travelling band with a solvent for sulphur.

W. G. CAREY.

**Manufacture of sulphuric acid.** J. V. SKOGLUND (E.P. 266,397, 23.9.25).—See U.S.P. 1,559,292; B., 1926, 12.

**Production of phosphoric acid.** H. A. BRASSERT, W. H. WAGGAMAN, and H. W. EASTERWOOD (U.S.P. 1,622,082, 22.3.27. Appl., 23.1.24).—See E.P. 228,185; B., 1925, 846.

**Manufacture of hydrocyanic acid.** W. GLUUD (U.S.P. 1,622,372, 29.3.27. Appl., 18.4.24. Conv., 23.4.23).—See G.P. 410,418; B., 1925, 757.

**Production of phosphorus pentoxide.** G. PISTOR, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,622,206, 22.3.27. Appl., 8.12.25. Conv., 30.12.24).—See G.P. 426,388; B., 1926, 584.

**Treating leucite and other sodium and potassium silicates with lime for extracting potassium or sodium aluminates.** F. JOURDAN (U.S.P. 1,620,212, 8.3.27. Appl., 27.12.24. Conv., 28.12.23).—See E.P. 226,819; B., 1925, 630.

**Apparatus for making anhydrous metallic chlorides.** F. T. WOHLERS (E.P. 266,437, 23.11.25).—See U.S.P. 1,564,302; B., 1926, 126.

**Process for dissolving a mixture of hafnium and zirconium phosphates and for separating hafnium and zirconium.** A. E. VAN ARKEL and J. H. DE BOER, Assrs. to N. V. PHILIPS' GLOELAMPEN-FABR. (U.S.P. 1,618,494, 22.2.27. Appl., 4.1.26. Conv., 6.6.24).—See E.P. 235,217; B., 1925, 713.

**Manufacture of cyanides** (E.P. 266,809).—See II.

**Hydrocyanic acid as an insecticide** (G.P. 435,714).—See XVI.

**Mercury vapour for inhalation purposes** (G.P. 435,689).—See XX.

### VIII.—GLASS; CERAMICS.

**Surface devitrification of glass.** III. Devitrification of alkali silicates. IV. Devitrification of alkali lead silicates. K. TABATA (J. Soc. Chem. Ind. Japan, 1927, 30, 37—42, 43—47. Cf. *ibid.*, 29, 473).—The surface devitrification of alkali silicates having the composition of  $R_2O \cdot xSiO_2$  is examined. Mixtures of a silicate (97%  $SiO_2$  and 2.75%  $Al_2O_3$ ) with potassium and sodium carbonates are melted in a furnace. When

the temperature of the mixture is above  $1000^\circ$ , the melt is taken out and quickly cooled in the air. Two pieces of glass from the same sample are heated in a muffle furnace at  $625$ — $750^\circ$  for 2 hrs., one of them being lightly scratched with quartz before treatment. After heating, it is quickly cooled and examined under a microscope. According to the mode of crystal production on the glass surface, the devitrification is divided into five classes: (a) No crystals; (b) a few minute crystals at specified places, such as at the sharpest edges of cicatrices; (c) many crystals along sharp edges, or at the cicatrised portion; (d) crystals on all the surfaces having neither sharp edges nor cicatrices; and (e) crystals entirely cover the surfaces. Curves are plotted, showing the relation between the number of molecules of silica dissolved and the percentage  $K_2O$  in the alkalis. The curve connecting the points of zero degree is a straight line called the "boundary line of devitrification" or "saturation line of silica dissolution." The compounds present in the melts are assumed to be always metasilicates. The dissolving power of each mol. of  $Na_2O \cdot 2SiO_2$  and  $K_2O \cdot 2SiO_2$  for silica is about 0.5 and 4.5 mols. respectively, at the temperature of the experiment. The dissolving power of mixed alkali silicates for silica is entirely additive. The remarkable differences in the behaviour of both alkali silicates towards silica are considered to be due to the different atomic volumes of the atoms of sodium and potassium. A series of glasses with the composition  $R_2O \cdot PbO \cdot xSiO_2$  were tested by the foregoing method, and similar curves have been plotted. Points of the same degree of devitrification give straight lines respectively. The zero degree line runs parallel to the corresponding line for alkali silicates. The composition is suggested for the alkali-lead silicate glasses  $x(R_2O \cdot 2SiO_2) + y(PbO \cdot 2SiO_2) + 2SiO_2$ .

K. KASHIMA.

**Importance of silica conversions in the burning process and in the behaviour of refractory materials in industry.** E. STEINHOFF (Gas- u. Wasserfach, 1927, 70, 237—240, 264—268).—The thermal conversions of silica are reviewed and classified into two groups:—(a) Non-reversible conversions occurring after prolonged heating or by the aid of catalysts and accompanied by considerable volume change and alterations in crystal structure, e.g., quartz  $\rightarrow$  cristobalite,  $1300$ — $1350^\circ$ , expansion 17.4%. (b) Reversible conversions occurring practically instantaneously, but with little volume change or alteration in crystal structure, e.g.,  $\alpha$ -quartz  $\rightleftharpoons$   $\beta$ -quartz,  $575^\circ$ , expansion 2.4%. Reference is made to the anomalous behaviour of  $\beta$ -quartz in passing directly into cristobalite, and to the variable conversion temperature of  $\alpha \rightarrow \beta$ -quartz ( $220$ — $260^\circ$ ), which is attributed to the presence of non-volatile impurities. The velocity of the different conversions is largely determined by whether the silica is present in the free state or in solid solution, and also varies inversely as the size of the particles. In the manufacture of silica refractories the greater part of the quartz is converted to cristobalite, but complete conversion is only effected by prolonged heating at  $1400$ — $1450^\circ$ . The reversible changes which introduce expansion and contraction mainly take place below  $600^\circ$ , the reversible expansion being greatest with cristobalite and least with tridymite.



The use of a mixture of converted and semi-converted silica frequently results in the formation of cracks and fissures, owing to the expansion of the non-converted material, and these allow of the penetration of slag and dust. Kieselguhr is converted into cristobalite by heating at 1100–1150°, and, after conversion, its resistance to load between 1000° and 1700° is almost equal to that of 95% silica material.

H. D. GREENWOOD.

**Terra cotta.** H. SPURRIER (J. Amer. Ceram. Soc., 1926, 9, 773–778).—The structure and physical characteristics of different varieties of terra cotta showed wide variations. No agreement appears to exist as to what constitutes the best terra cotta. A microscopical study showed great differences in the number of voids in a given area; their existence was associated with coarse grog particles. The heterogeneity of the grog was frequently the cause of a number of defects. In the preparation of thin sections for the microscope, the tendency of the sections to curl was taken as evidence of internal strain in the body. Osmosis is not an important factor in the breakdown of terra cotta over large areas. On the other hand, freezing and thawing of wet terra cotta caused marked internal deterioration of the body; this was confirmed by porosity tests.

F. SALT.

**Colouring of stoneware with cobalt sulphate.**

A. F. ALZNER (Keram. Rundsch., 1926, 34, 650; Chem. Zentr., 1926, II, 2995).—The calculated amount of cobalt must be added to mask the colour of the iron, together with a suitable excess to act as a colour. The iron content of the clay must be uniform.

B. W. CLARKE.

**Characteristics of pyrometric cones.** C. O. FAIRCHILD and M. F. PETERS (J. Amer. Ceram. Soc., 1926, 9, 701–743).—The characteristics of Orton cones were determined under standardised conditions of heating resembling those occurring in ceramic practice. An electric tube furnace was used for the experiments with cones fusing below 1200°, and a graphite spiral furnace was designed for the more refractory cones. Means were provided for varying the nature of the atmosphere within the furnaces. Thus the experiments were carried out under definite and reproducible conditions. The temperatures of the "end-points" and of the bending intervals of a complete set of cones were measured in clean air and in various kiln gases. With few exceptions, the end-points were higher when the rate of heating was 150°/hr. than when it was 20°/hr. Under slow heating in gases free from sulphur dioxide, the end-points of cones 015–01 were higher than in air; kiln gases free from sulphur dioxide had little effect upon cones 022–016 and 1–42. The accuracy of the method developed in this investigation was within 5° up to 1400° and within 10° from 1400° to 2000°. The end-point of cones is variable, depending upon time, temperature, and atmosphere. Given a constant firing schedule and kiln atmosphere, a cone will always come down at the same temperature. Hence cones measure the temperature at which ware will mature under given rate of heating and character of kiln gases. The combined use of cones and pyrometers is recommended for kiln control, but cones are inherently unsuited for the

determination of the refractoriness of clays in laboratory tests.

F. SALT.

**Comparison of the softening points of some foreign and American pyrometric cones.** R. F. GELLER and E. E. PRESSLER (J. Amer. Ceram. Soc., 1926, 9, 744–757).—The results of a comparative study of the softening points of English, French, German, and American cones are presented graphically, and by means of drawings and photographs. The softening points of cones 022–20 were determined in a gas-fired, recuperative, down-draught kiln, whilst a graphite resistance furnace was used for cones 26 to 35.

F. SALT.

**Silicon carbide refractories for water-gas generators.** M. L. HARTMANN and J. A. KING (J. Amer. Ceram. Soc., 1926, 9, 758–765).—The rapid deterioration of ordinary firebrick linings in water-gas generators is due mainly to clinkering trouble. Clinker adhesion and slag erosion have been largely eliminated by the use of Bernitz blocks made of "Carbofrax" (bonded silicon carbide). These are hollow, square, or rectangular shapes pierced with a number of holes which taper inward from the face nearest the fire. Air or steam can be passed through these holes at various points into the fire. The success of these blocks when used for lining water-gas generators is due to their great mechanical strength at high temperatures, lack of chemical activity in contact with viscous slags, and high thermal conductivity.

F. SALT.

**Harper electric kiln.** F. A. J. FITZGERALD (J. Amer. Ceram. Soc., 1926, 9, 766–772).—The construction of an electric tunnel kiln, 68 ft. long, designed for firing biscuit ware, is described. A temperature of 1500° has been attained in the kiln when firing porcelain.

F. SALT.

#### PATENTS.

**Process and apparatus for the production of silica articles.** H. L. WATSON, Assr. to GENERAL ELECTRIC Co. (U.S.P. 1,621,446, 15.3.27. Appl., 1.4.26).—Silica is shaped by heating to fusion a part only of the mass, so that the unfused silica serves as a support for the fused silica. The fused silica is then withdrawn, leaving behind the unfused mass.

W. G. CAREY.

**Production of white opacifying media for glasses and enamels.** I. KREIDL (E.P. 245,757, 29.12.25. Conv., 10.1.25).—Uncoloured oxides and compounds of metals other than tin and zirconium are converted into the colloidal gel state in a colloid mill, and are maintained in a highly polymerisable form, even at the annealing temperature of the enamel, by the addition of tin or zirconium compounds as protective substances.

W. G. CAREY.

**Manufacture of ceramic materials, building elements, and the like, from mud.** A. LOESSIN (E.P. 259,236, 1.10.26. Conv., 1.10.25).—Mud deprived of about one half of its water by settlement for some 4 months and decanting, or by centrifugal action, is kneaded in a kneading machine with Glauber's salt, with or without sand, and after preliminary burning at 400–500° is cooled and given a final burn, forming ceramic products. Clinker is made by giving a two-stage preliminary burning with intermediate cooling and a final burning. Paving is formed by a final burning at



1300°, whilst a thin flowing casting material is produced at 1350—1400°. W. G. CAREY.

**Quartz working.** E. R. BERRY, Assr. to GENERAL ELECTRIC Co. (U.S.P. 1,620,511, 8.3.27. Appl., 18.4.23).—See E.P. 214,630; B., 1924, 713.

**Treatment of earthy minerals.** W. FELDENHEIMER (U.S.P. 1,622,099, 22.3.27. Appl., 30.10.25).—See E.P. 242,358; B., 1926, 55.

**Methods and apparatus for forming sheet glass.** L. MELLERSH-JACKSON. From HARTFORD-EMPIRE Co. (E.P. 266,182, 31.5.26).

**Manufacture of double-walled glass flasks.** F. SIEGHEIM (E.P. 267,005, 1.9.26).

## IX.—BUILDING MATERIALS.

### PATENTS.

**Burning cement.** H. KÜHL (Austr. P. 104,148, 31.3.25. Conv., 20.1.25).—An oxygen carrier is added to a finely pulverised mixture of raw materials for cement and fuel, in order to facilitate the combustion of the fuel. Thus, *e.g.*, ferric oxide is added to the raw materials to make the proportion one twelfth that of the lime content. B. W. CLARKE.

**Burning cement and the like.** M. BÜTNER (G.P. 435,077, 29.8.24).—A rotary kiln is provided with one or more constrictions, which form a sintering zone or "condensing" chamber, into which penetrates the fuel inlet pipe, having a large surface area. The speed of rotation of the kiln is arranged so that the product of the square of the number of rotations per min. and the greatest internal diameter in metres of the lining of the calcining zone is 500 or more. The process results in a considerable lowering of the coal consumption and of the waste-gas temperature. B. W. CLARKE.

**Rotary tubular kilns for treating cement and the like.** O. BOUZIN (E.P. 266,939, 4.5.26).—A metal heat exchanger, arranged in the preheating zone of the kiln, is provided with a series of radial plates at the periphery which spread the material to be treated in thin sheets over a central tube or bundle of tubes through which the hot gases pass. The inner wall of the calcining zone has ring-shaped projections which spread the material along a succession of horizontal planes, thereby impeding its progress and increasing the capacity of the kiln. The tubular clinker cooler is provided with a similar heat exchanger, and the amount of air admitted is limited to that strictly necessary to maintain combustion in the kiln. B. W. CLARKE.

**Apparatus for preheating slurry, especially cement slurry.** G. POLYSIUS, EISENGIESSEREI U. MASCHINENFABR. (G.P. 435,792, 24.2.24).—The slurry inlet pipe passes through the smoke chamber, where it is heated by the waste kiln gases, and is connected to the kiln by a pipe provided with a jacket through which the waste gases are conducted. This prevents the heat taken up by the material in the smoke chamber from being lost during its passage to the kiln. B. W. CLARKE.

**Manufacture of acid-proof cementing compositions.** I. G. FARBEININD. A.-G., Assees. of FARBERWERKE VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 256,258 and

258,616, [A], 30.7.26, [B], 18.9.26. Conv., [A], 30.7.25, [B], 18.9.25).—(A) A powdered modification of silicic acid, such as opal, chalcedony, etc., which is capable of reacting strongly with alkali and will show a loss of weight of not less than 40% of its original weight when boiled for 2 hrs. with 25 times its weight of 15% sodium hydroxide solution, is added to a water-glass solution forming an acid-proof cementing composition. Powdered fireclay or other similar fillers may be added to increase the binding power. (B) The addition of 3—4% of powdered silicon to the above or other water-glass cement produces an acid-proof composition with a thermal conductivity comparable with that of metals, thereby increasing the durability of the cement when used with metal vessels. B. W. CLARKE.

**Production of cementitious material.** A. E. HILLS (E.P. 266,775, 20.10.25).—A suitable proportion of the hydrated oxide of iron or aluminium is added to hydraulic cements, especially Portland cement, to combine with the lime set free on setting, thereby accelerating the maturing of the cement and preventing efflorescence. The mixture is suitable for use in manufacturing cement-asbestos sheets, tiles, etc. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 24,718/1908 (*cf.* B., 1909, 984).] B. W. CLARKE.

**Insulating and building materials.** Soc. ANON. CIMENTI (Swiss P. 115,884, 19.3.25).—Vegetable fibres, *e.g.*, straw, maize, wood fibre, etc., rendered fireproof with solutions of magnesium salts or water-glass, are mixed with materials, such as Portland cement or plaster, which will react with the moisture in the fibrous material, the mixture is poured into moulds, and removed to complete the hardening process after a sufficient preliminary setting. B. W. CLARKE.

**Porous compounds or compositions formed from gypsum.** G. O. CASE (E.P. 266,524, 3.2.26).—Calcium carbonate is added to partly dehydrated gypsum, or natural gypsum rock containing 3—13% of calcium carbonate is calcined until about 6.5% of water is left, and either product is treated with hydrochloric or other acid of *d* 1.02 to the extent of 40—60% by wt. of the dehydrated gypsum. The mixture is cast in moulds having porous tops. The resulting blocks may be made waterproof by a layer of cement or concrete, or by the addition of ferric or magnesium sulphate to the mixture. W. G. CAREY.

**Manufacture of priming material for coating porous surfaces.** E. W. FRENKEL and A. J. H. BRUST (E.P. 266,401, 26.9.25).—A stable suspension of finely-divided light magnesia, magnesium carbonate, levigated alumina, etc. in linseed oil, the suspensoid condition of which is maintained by adding an altered fatty oil such as coagulated wood oil or oxidised linseed oil, is used for priming absorbent material. The operation may be performed by altering linseed oil while in admixture with the pore filler in a mill by oxidation or by sulphur chloride, and stopping the reaction when desired by adding more linseed oil together with turpentine substitute as thinning agent and siccatives if desired. W. G. CAREY.



**Manufacture of tarred macadam and the like.** J. F. WAKE (E.P. 266,420, 30.10.25).—The agglomeration in cold weather of tarred macadam aggregate is prevented by the addition of a coating of petroleum, linseed oil, or other film-producing substances, the lubricant being introduced by spraying or sprinkling the aggregate. W. G. CAREY.

**Kiln for burning cement.** C. NASKE (U.S.P. 1,622,337, 29.3.27. Appl., 7.2.25. Conv., 8.1.24).—See E.P. 227,444; B., 1926, 409.

**Production of artificial stones.** J. JAKOB (E.P. 244,724 and 266,789. Appl., [A], 3.11.25, [B], 23.11.25. Conv., [A], 22.12.24).—See G.P. 417,360; B., 1926, 130.

**Compositions for laying or preventing dust and their application to road surfaces.** P. G. EKSTRÖM (E.P. 252,378, 19.5.26. Conv., 22.5.25).—See U.S.P. 1,606,928; B., 1927, 110.

**Manufacture of cement concrete and apparatus for use therewith.** A. C. KNIPE (E.P. 266,814, 3.12.25).

**Building materials from mud** (E.P. 259,236).—See VIII.

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

**Chromium-cobalt steels.** F. M. OSTROGA (Rev. Mét., 1927, 24, 135—145; cf. B., 1927, 77).—Two steels containing 1.21% C, 13.16% Cr, 4.88% Co, 0.64% Mo, 0.59% Ni and 1.78% C, 13.06% Cr, 2.40% Co, 1.14% Mo, 0.11% Ni, used for exhaust valves for internal-combustion engines have been studied by dilatometric and microscopical methods and their Brinell hardnesses determined. The steels are not readily oxidisable at high temperatures, the transformation temperature on heating is 850°, and the hardness is 60 at 800°. In the malleable condition they are easily machineable. That the carbon content is less towards the edge than at the centre of the rod is regarded as a favourable factor in the application of the steels to exhaust valves, and in spite of their fragility, which may be accentuated by the presence of impurities or eutectic formed by heating above 1200°, chromium-cobalt steels have under certain conditions given satisfactory results. M. COOK.

**Silicon construction steel from the Siemens-Martin furnace.** J. MEISER (Stahl u. Eisen, 1927, 47, 446—448).—Particulars are given of eight heats of silicon steel made in an 80-ton tipping open-hearth furnace at the Dortmund Union Works for bridge construction. The analyses had the following limits:—0.13—0.19% C, 0.80—1.37% Si, 0.84—1.14% Mn. Also at a Rhenish works, 6 heats were made in a smaller (17-ton) furnace of a steel for railway wagon construction, with analysis range: 0.15—0.19% C, 0.85—1.10% Si, 0.90—1.10% Mn. The results of a large number of acceptance tests show that such constructional steels can be satisfactorily produced in ordinary open-hearth furnaces. T. H. BURNHAM.

**Advantages of smelting fine-grained ores in the blast furnace.** K. HOFMANN (Trans. Amer. Electrochem. Soc., 1927, 51, 91—102).—In the reduction of ferric and ferrosferic oxides by hydrogen, the

degree of reduction is a minimum at 750° owing to surface welding of the particles already reduced; this action commences at about 600° and prevents the diffusion of the hydrogen into the interior. At temperatures below 600° ferric oxide is much more readily reduced than ferrosferic oxide of the same grain size, an indication that the latter is not an intermediate product of the reduction of ferric oxide to metallic iron. In the case of iron ores, similar results are obtained, but the retardation of the action above 600° is intensified by the presence of silica, which forms ferrous silicate at 700° with the ferrous oxide produced in the reduction, and this forms a glassy, impervious coating on the oxide particles. If fine iron ores, therefore, could be used in the blast furnace in such a way that they were completely reduced at a relatively low temperature, the height of the furnace could be reduced and the boshes and hearth could be cylindrically shaped and of the same diameter. A. R. POWELL.

**Gaseous cementations. Cementation by cyanogen. II.** M. PÉROT (Rev. Mét., 1927, 24, 79—92; cf. B., 1927, 45).—Examination of test pieces of a steel containing 0.2% C, which have been heated for varying periods in cyanogen at 1000°, shows that rapid cementation takes place during an initial time of 45 min., and that the rate then quickly diminishes. A layer of non-assimilable carbon is deposited on the specimens, and carbon slowly diffuses towards the interior from the hypereutectoidal zone already rich in carbon. Evidence is obtained to show that renewal of the cyanogen atmosphere increases the rate of cementation. Cementation is little affected by variations in cyanogen pressure from 767 to 60 mm. of mercury. Values are given for the depth of cementation by cyanamide with varying times and temperatures of heating. Sodium cyanide is not useful as a cementation agent, since its fusion at a comparatively low temperature appears to stop the reaction. L. M. LARK.

**Existence of limiting states in alloy studies. Equilibrium diagram involving the existence of limiting states.** L. GRENET (Rev. Mét., 1927, 24, 93—107).—A theoretical investigation of limiting states in alloys. The anomalous behaviour of alloys (such as is observed in the iron-nickel series) is examined from the point of view of the existence of critical points in phase equilibria and transformations. L. M. CLARK.

**Recrystallisation [of iron, steel, and copper].** H. HANEMANN (Stahl u. Eisen, 1927, 47, 481—489).—The rate of recrystallisation of iron, which has been deformed by hammering at a high temperature and immediately annealed at the same temperature, is a logarithmic function of that temperature, the equation to the recrystallisation curve being  $R = 1.016^{(T - 500)}$ , when the degree of deformation is constant. During recrystallisation, the average number of new grains per unit area decreases hyperbolically with the time of annealing, and is smaller, after any given time, the higher the temperature of annealing. As is the case with copper, there is a certain minimum degree of deformation to which iron and steel must be subjected before recrystallisation will ensue on annealing; this minimum value decreases hyperbolically with rise of temperature up to the transformation point. There is also a minimum



temperature of annealing below which no recrystallisation will take place, however severe the deformation; in the case of soft iron this is  $450^{\circ}$  in the  $\alpha$  range and  $910^{\circ}$  in the  $\gamma$  range. Parameter curves for the recrystallisation of copper, iron, and steel together with the corresponding equations are given, also curves showing the grain size of iron after cold deformation and recrystallisation, and of soft iron forged at temperatures between  $900^{\circ}$  and  $1100^{\circ}$ .

A. R. POWELL.

**Working of steel containing copper.** W. HERWIG (Stahl u. Eisen, 1927, 47, 491—493).—The presence of copper in steel sheets renders them unsatisfactory for use in the manufacture of deeply recessed articles, but such steel may be used in making heavy parts of machinery without fear of fracture. Steel sheets containing much copper acquire a rough, hackly surface during rolling, and it is difficult to remove the scale from their surfaces. Cupriferous steel is much more easily corroded than steel free from copper, and, in the case of open-hearth steel, the rate of corrosion is roughly proportional to the copper content.

A. R. POWELL.

**Influence of the [heat and mechanical] treatment of transformer iron on the wattage losses.** G. EICHENBERG and W. OERTEL (Stahl u. Eisen, 1927, 47, 262—269).—The effect of various methods of rolling and annealing transformer iron with 4% Si on the wattage losses has confirmed the observations of Yensen (Bull. Univ. Illinois, No. 83, 1915) that the losses are smaller with sheets consisting of large, evenly distributed crystallites than with those containing unevenly oriented and irregular crystal grains. The effect of grain size, however, is often masked by other influences such as the so-called "gas content." Prolonged annealing results in the interaction of oxide inclusions with the carbon content, and the subsequent elimination of the carbon monoxide so formed results in an appreciable decrease in the wattage loss. This beneficial action is obtained by annealing at  $750^{\circ}$  for the final rolling operation.

A. R. POWELL.

**Sulphur in iron and steel.** J. CIOCHINA (Chim. et Ind., 1927, 17, 383—384; cf. B., 1927, 167).—Free sulphur in iron and steel is determined by passing carbon dioxide through a Corleiss flask, in which the sample (10—20 g.) is being dissolved in concentrated hydrochloric acid, for 2 hrs., during which time the flask is gently heated. The reaction having ceased, the current of gas is continued for  $\frac{1}{2}$  hr. to remove any remaining hydrogen sulphide. 25 c.c. of hydrofluoric acid are added and carbon dioxide is again passed for  $\frac{1}{2}$  hr. After cooling, excess of potassium hydroxide solution is added, the whole boiled, then cooled, and 200—300 c.c. of concentrated hydrochloric acid are added, the mixture being again heated. Before the acid addition, carbon dioxide is passed into the flask, and, after heating for 1 hr., the contents are titrated with iodine and thio-sulphate solution. Results obtained by this method have shown that in iron containing 0.15—0.2% S, 0.005—0.009% exists as free sulphur, and in slag containing 1.84% S, 0.012% is present as free sulphur. If a sample of iron is ground with mercury before analysis, mercury sulphide is formed, and only traces of free sulphur can be found. The determination of sulphur by com-

bustion gives higher results than the hydrogen sulphide method, for with the latter only the result for sulphur occurring as sulphide is obtained, whereas the former yields the total sulphur. A sample of iron rapidly cooled and another sample, from the same ladle, slowly cooled were found to contain 0.148 and 0.106% S respectively. The difference between the two is regarded as sulphur in the gaseous form which, when the iron is quickly cooled, does not escape but combines to form iron sulphide and 3—4% of it remains as free sulphur.

M. COOK.

**Determination of small amounts of titanium in alloy steels.** K. ROESCH and W. WERZ (Chem.-Ztg., 1927, 51, 149—150).—The sample (5—10 g.) of turnings is dissolved in 1:1 hydrochloric acid. If tungsten is a constituent of the steel the tungstic acid which is precipitated contains part of the titanium, and is fused with sodium carbonate. On treating with hot water the sodium titanate is not dissolved, but is treated with hydrochloric acid which may be added to the original solution. Iron is separated by the Rothe ether process. Chromium if present is brought into solution as chromate by treatment with sodium peroxide or carbonate, vanadium with sodium hydroxide, and molybdenum by either of these means. Nickel and cobalt, on treatment with excess of ammonia, pass into solution as hydroxides. The titanium in hydrochloric acid solution is reduced by zinc to the trichloride with exclusion of air, a freshly prepared 50% solution of potassium or ammonium thiocyanate is added, and titrated with 0.3N-ferrie chloride solution until a red-brown colour persists. The experimental error of the process is as follows:— $\text{Ti} = 0.01—0.10\%$ ,  $\pm 0.003$ ;  $\text{Ti} = 0.10—1.00\%$ ,  $\pm 0.005$ .

T. H. BURNHAM.

**Crystallite orientation of copper in relation to the degree of rolling.** G. TAMMANN and H. H. MEYER (Z. Metallk., 1927, 19, 82—84).—On rolling copper the crystallites slip along an octahedral plane and, until a reduction of 30—40% in thickness has been effected, the proportion of octahedral planes in the plane of rolling increases. Further work results in the breaking up of the elongated crystals in the direction perpendicular to the direction of rolling, and finally these lamellæ split up into long, narrow fibres, which are apparently twinned. On etching, these fibres develop a series of parallel lines arranged at an angle of  $35^{\circ} \pm 5^{\circ}$  to the direction of rolling, the lines running towards that direction and away from it in alternate fibres. Up to about 40% reduction the proportion of octahedral planes on the surface increases, but with 50% reduction they have practically disappeared, giving place to dodecahedral planes. After annealing at  $400—500^{\circ}$ , the surface crystallites are practically all so oriented that an octahedral plane lies with its apex in the direction of rolling. Severely cold-rolled sheets, however, pass through a transition state in which there is a large proportion of unevenly oriented dodecahedral planes in the surface.

A. R. POWELL.

**Hardness and potential of zinc-copper alloys.** O. BAUER and O. VOLLENBRUCK (Z. Metallk., 1927, 19, 86—89).—The hardness of zinc-copper alloys containing up to 35% Zn is very little greater than that of pure copper. Between 35% and 39% Zn the hardness



risers to almost double its previous value, then remains stationary again throughout the  $\alpha + \beta'$  range. In the range of pure  $\beta'$  (47–49%) an abrupt increase in hardness takes place, and this continues to a maximum of 310 at 61% Zn, corresponding with the formation of  $\text{Cu}_2\text{Zn}_3$ . After this, further addition of zinc results in a rapid fall in hardness to a third constant value in the  $\epsilon + \eta$  range somewhat less than in the  $\alpha + \beta$  range; finally, there is a small but abrupt fall to the hardness of pure zinc (48). In a 1% sodium chloride solution at 18° the potential of pure copper measured against the normal calomel electrode is 0.19 volt. Almost the same potential is shown by copper–zinc alloys containing up to 39% Zn, but, as soon as the  $\beta'$  constituent appears, the potential rises to 0.31 volt; then increases steadily to 0.40 volt with pure  $\beta'$  (47% Zn), and falls again to 0.36 volt with 50% Zn, corresponding with the appearance of  $\gamma$ . Within the  $\beta + \gamma$ , and the  $\gamma$  ranges, a slight but steady increase of potential occurs with rise in the zinc content, followed by a sudden rapid increase to about 1 volt with the appearance of the  $\gamma + \epsilon$  constituent (70% Zn). The value then oscillates about this figure for alloys containing 70–100% Zn.

A. R. POWELL.

**Gases contained in brasses, aluminium, and its alloys.** L. GUILLET and A. ROUX (Compt. rend., 1927, 184, 724–727; cf. B., 1927, 15).—Gases are evolved from brasses containing copper (60–67%) and zinc, between 530° and 1040°, the maximum rates occurring at 720–790°, and principally at 930–980°. The volume of the gas was half that of the metal, and had the composition  $\text{CO}_2$  19, CO 9.5, H 35.9,  $\text{CH}_4$  22.2, and N 13.4%. Aluminium containing silicon and iron (0.6%) evolved 0.14 of its volume of gas in 3 hrs. between 400° and 550°, and this had the composition  $\text{CO}_2$  20, CO 12, H 68.0%. The physical properties of the metal were the same whether annealing was carried out in air or *in vacuo*. When the metal was heated to 820° at the rate of 7° per minute, the volume of gas was 1.6 times that of the metal, and its composition  $\text{CO}_2$  31.7, CO 2.45, H 26.0,  $\text{CH}_4$  26.0, and N 3.25%. Duralumin yielded in  $\frac{2}{3}$  hrs. at 500°, 0.23 of its volume of a gas containing CO 9, H 91%.

J. GRANT.

**Aluminium as constructional material in the inorganic chemical industry.** BUSCHLINGER (Z. Metallk., 1927, 19, 101–106; cf. B., 1927, 143).—The action of a large number of inorganic compounds on metallic aluminium at various temperature has been examined. Solutions of most salts of the mineral acids, with the exception of alkali nitrates, have a more or less corrosive action on the metal, halides being the most destructive. Dilute ammonia solutions and alkali carbonates have very little action either hot or cold. Sulphur may be melted without danger in aluminium pans, which are also suitable for use with hot ammonium sulphide liquors.

A. R. POWELL.

**Structure and tensile properties of very pure aluminium.** VON GÖLER and G. SACHS (Z. Metallk., 1927, 19, 90–93).—The course of recrystallisation of very pure aluminium (99.9% Al) compared with that of technical grades has been examined by means of X-ray interference figures. As is the case with technical grades, pure aluminium commences to recrystallise just

above 200°, but the crystals grow at a much more rapid rate, so that at 375–400° the structure of the pure metal consists of very large crystals, whereas a similar structure is produced in the technical grades only after prolonged heating above 600°. The curves showing the variation of tensile strength, elongation, and reduction of area, with the annealing temperature run practically parallel for the 99.9, 99.0, and 98.7% grades of aluminium, minimum tensile strength and maximum ductility being obtained in every case at 300°. These results show that even the small quantities of impurities in the 99.9% grade are not completely in solid solution at the ordinary temperature.

A. R. POWELL.

**Leaching of molybdenite ores.** C. SVENSSON (Trans. Amer. Electrochem. Soc., 1927, 51, 51–58).—Norwegian molybdenite ore (1.9%  $\text{MoS}_2$ ) occurs as very fine flakes dispersed throughout a silicious dolomitic rock, and is not amenable to concentration by any of the usual methods. Roasting in a revolving tube furnace at 750–800° effected complete conversion of the molybdenum to trioxide, and of the sulphur to calcium sulphate, and subsequent leaching with sodium carbonate solution at 80–90° extracted over 90% of the molybdenum as sodium molybdate, together with most of the sulphate. From the leach liquor, after acidification with sulphuric acid, the molybdenum could be precipitated as sulphide, or, alternatively, it could be recovered as calcium molybdate after removal of the sulphate from the acidified solution with barium chloride and subsequent neutralisation with milk of lime and ammonia. Cost data based on the laboratory results are given.

A. R. POWELL.

**Metal calorimeter for determination of the specific heats of metals, oxides, and slags.** W. GROSSE and W. DINKLER (Stahl u. Eisen, 1927, 47, 448–454).—An apparatus is described based on the Nernst metal calorimeter suitable for calorimetric measurements over a temperature range of 0 to 1600°. The specimen is pear-shaped, and is heated in a tubular furnace wound with nickel–chromium wire for use up to 1250°, and above this temperature with molybdenum wire or a carbon spiral *in vacuo*. It falls into a copper vessel the temperature of which is indicated by 20 copper–constantan thermo-couples arranged in series. For molten metals a quartz container is used or a drop is allowed to fall from a conical rod. Calibration is carried out under the same conditions as the sp. heat determinations, water in a thin brass vessel being used as calibrating medium. It is heated by steam, which is also used for determining heat contents up to 100°. The weight of the specimen is such that the same amount of heat is always introduced into the calorimeter. After each experiment the small unavoidable losses of heat are determined. By use of a Kurnakov apparatus the heat transmission from the specimen to the calorimeter is made self-registering.

T. H. BURNHAM.

**Damping properties of some metals [iron, copper, and aluminium] in torsional vibration.** O. FEUSSNER and E. RAMB (Z. Metallk., 1927, 19, 115–116).

PATENTS.

**Sponge iron.** COPPER SEPARATION, LTD., Assees. of P. W. NEVILL (Austral. P. 20,811, 1.12.24).—A mixture of finely-divided iron ore and coal is heated at



900—1000° in a shaft furnace to which a finely-divided air supply is admitted over a large surface so that at the most only a partial fusion of the mass takes place. The hot reduced product is quenched in water, the unchanged carbon washed away, and the iron recovered from the residue by crushing followed by magnetic separation.

A. R. POWELL.

**Tool steel without brittleness.** RÖCHLING'SCHE EISEN- U. STAHLWERKE G.M.B.H., and J. KUBASTA (Austr. P. 104,009, 18.11.21).—The formation of a metasilicate in the metal is avoided by removing the slag from its surface and allowing the silicon present to separate and the metal to take up oxygen. Subsequently the excess of ferrous oxide is removed by deoxidation in the usual manner.

A. R. POWELL.

**Heat treatment of steel.** E. F. KENNEY (U.S.P. 1,619,025, 1.3.27. Appl., 23.4.26).—Steel rails or the like are cooled from a temperature (about 950°) above the critical range, to a temperature (380°) below that range, but above blue-heat, heated at an intermediate temperature (540°) for 1 hr., and allowed to cool in air. The physical properties are greatly improved.

T. S. WHEELER.

**[Alloy] steel.** C. K. EVERITT, and E. ALLEN & Co. (E.P. 267,024, 28.9.26).—An alloy steel of high resistance to corrosion contains less than 2% and, preferably, not more than 1.5% Cu, 7—20% Cr, 4—20% Ni (preferably 6%), 0.05—0.5% C, refractory metals of the carbon group being absent.

C. A. KING.

**Alloy steel.** W. H. KEEN (U.S.P. 1,621,886, 22.3.27. Appl., 10.5.24).—An air-hardening steel capable of being forged contains, approximately, 3% V, at least 0.85% C, 18% W, and 4% Cr.

C. A. KING.

**Titanium alloy.** A. W. CLEMENT, Assr. to LUDLUM STEEL Co. (U.S.P. 1,621,523, 22.3.27. Appl., 18.12.17).—An alloy containing 10—30% Cr, 5—12% Ti, up to 1% Si, up to 10% Al, together with carbon sufficiently low for working purposes, the remainder being iron.

C. A. KING.

**Manufacture of alloy steel and iron.** B. D. SAKLATWALLA (U.S.P. 1,619,462, 1.3.27. Appl., 16.7.24).—A bath of molten steel or iron covered with slag is treated with successive small portions of chrome ore, or other unreduced compound of an alloying metal, and a reducing agent, *e.g.*, silica in the form of ferro-silicon.

T. S. WHEELER.

**Composition of matter. [Iron alloy.]** R. P. DRUMMOND, Assr. to PACIFIC CAST IRON PIPE & FOUNDRY Co. (U.S.P. 1,620,877, 15.3.27. Appl., 7.8.25).—The alloy consists of iron in combination with 1.75—2% C, 6.25—8% Mn, and 1.50—2% Si.

F. G. CROSSE.

**Production of iron and other carbon binding metals and alloys thereof with a certain percentage of carbon directly out of oxide ore.** H. G. FLÖDIN and E. G. T. GUSTAFSSON (E.P. 243,353, 18.11.25. Conv., 19.11.24).—A charge of a mixture of ore and carbonaceous material containing only sufficient carbon for the reduction process is, during or after reduction, mixed with carbon alone or with a high-carbon ore mixture to increase the carbon content of the metal to the desired amount. When the reduction is effected in an

electric furnace, the high-carbon mixture, which may be in the form of briquettes, is introduced on to the surface of the slag around the electrodes.

C. A. KING.

**Chemically resistant alloys [of iron, nickel, and copper].** R. WALTER (G.P. 435,170, 13.5.24).—Alloys suitable for use with sulphite-cellulose liquors comprise varying proportions of iron, copper, and nickel with 10% Si.

A. R. POWELL.

**Direct reduction process for producing carbon-binding metal or metal alloy.** H. G. FLÖDIN and E. G. T. GUSTAFSSON (E.P. 243,743, 26.11.25. Conv., 27.11.24).—A mixture of oxide ore and carbon together with necessary fluxes is reduced in an electric furnace having electrodes which can be raised or lowered, the heating being effected chiefly near the surface of the slag bath. The charge may be introduced into the furnace continuously, being interrupted for a sufficient time before tapping to allow the metal to be raised to a suitable temperature, during which period the electrodes are immersed in the slag bath.

C. A. KING.

**Conversion of sulphide ores into sulphates.** COMPLEX ORES RECOVERIES Co. (G.P. 434,948, 2.2.22).—Pyritic ores containing copper, lead, or zinc are roasted at 600—1000° to convert the sulphides into oxides, sulphates, and ferrite. The roasted material is then raked in the direction of flow of the sulphurous gases into a cooler part of the furnace so as to favour the formation of copper, zinc, and lead sulphates and to convert the iron into a basic ferric sulphate.

A. R. POWELL.

**Treating sulphide ores of lead and zinc.** N. C. CHRISTENSEN (U.S.P. 1,620,873, 15.3.27. Appl., 26.12.23).—Ores containing galena are agitated with cold acid brine until the liberation of hydrogen sulphide is completed, and lead is then recovered from the solution.

C. A. KING.

**Obtaining blende free from chlorides by melting lead-zinc ores with a mixture of potassium and zinc chlorides.** E. LANGGUTH (G.P. 435,704, 16.5.25).—Lead-zinc sulphide ores are melted with a mixture of potassium and zinc chlorides to convert the lead into chloride, which is then reduced to metal by the addition of zinc. The slag is extracted with the minimum of water, and hydrochloric acid is added in quantity just sufficient to dissolve the precipitated zinc oxychloride. The residue after filtration consists of relatively pure blende.

A. R. POWELL.

**Process and apparatus for the condensation of zinc vapours.** C. J. G. AARTS (E.P. 249,123, 10.3.26. Conv., 10.3.25).—Zinc vapours are condensed in a vertical duct provided with a number of water-cooled coils which may be regulated individually so as to produce a given temperature in any particular section of the duct. In practice, a number of such ducts may be used in parallel, forming at their lower parts a chamber for collecting liquid zinc, and being connected at the upper ends with a dust collector which discharges the collected dust to a charging device at the base of the condensing ducts.

C. A. KING.

**Shaft furnace for roasting ores and calcining fine-grained materials.** G. A. STRECKER (G.P. 435,703, 22.2.24).—The flues for the air supply of a



shaft furnace for roasting ores and the like are so arranged that the air passing through the cooling shaft, and there preheated, passes directly into the roasting zone and thence into the hottest zone. Regulating dampers are provided at intervals in the air flues to allow of the admission of fresh air to any desired zone of the furnace. A. R. POWELL.

**Smelting ores in the absence of air.** A. ROITZHEIM and W. REMY (G.P. 435,109, 26.6.25).—To avoid slagging of the walls of the reduction chamber the ore is mixed with an aqueous suspension of finely-divided carbon or aliphatic hydrocarbon and an inorganic colloid, *e.g.*, clay, and the mixture is charged into the furnace wet or after drying, briquetting, and carbonising. A. R. POWELL.

**Cadmium plating.** UDYLITE PROCESS CO., ASSEES. of A. W. YOUNG and M. E. LOUTH (E.P. 266,985, 28.7.26. Conv., 31.3.26).—In a process of cadmium plating from a cyanide bath, a mixture of an extract of a cereal, *e.g.*, bran, wheat, maize, etc. with sugar is used as an additive agent. An impure sugar, *e.g.*, molasses or the caramelised form, is more suitable than pure cane sugar. C. A. KING.

**Lead alloys.** H. YOSHIKAWA (E.P. 253,920, 17.6.26).—Binary alloys of lead and bismuth forming a one-phase solid solution contain 4%, 0.25%, and 0.25–0.025% Bi, respectively. The alloys are harder, and have a small coefficient of friction as compared with lead, and are also more resistant to chemical and electro-chemical attack than other lead alloys. C. A. KING.

**Extraction of gold.** J. L. WARNER (U.S.P. 1,621,697, 22.3.27. Appl., 19.12.23).—Gold-bearing material consisting of clay and sand is screened dry, the finer portion being crushed and agitated in water to separate the sand from the clay. Washings from the coarser material are added to the sludge from the smaller grained material, and sand is removed from the clay particles. C. A. KING.

**Making bi-metallic or compound metallic strips, plates, or the like, such as for use in thermostats.** F. W. MILLER (E.P. 266,645, 26.11.26).—A metal plate is covered with a non-metallic fusible material, *e.g.*, powdered glass, and heated until the glass melts. A plate of metal of lower fusibility than the first is then laid on the glass and melted, causing the glass layer to rise to the top and the two metal layers to become united. C. A. KING.

**Manufacture of iron-chromium alloys.** W. B. D. PENNIMAN and E. J. SHACKELFORD (E.P. 244,413, 3.9.25. Conv., 11.12.24).—See U.S.P. 1,527,088; B., 1926, 363.

**Magnetic alloy.** W. S. SMITH, N. POPPLEFORD, and H. J. GARNETT (U.S.P. 1,622,008, 22.3.27. Appl., 2.5.25).—See E.P. 224,972; B., 1925, 76.

**Increasing the production and phosphoric acid content of Thomas slag.** H. HILBERT (E.P. 247,946, 11.2.26. Conv., 19.2.25).—See G.P. 418,102; B., 1926, 162.

**Electrolytic separation of metallic chromium.** F. W. WÜRKER (E.P. 266,045, 16.11.25).—See F.P. 607,829; B., 1927, 194.

**Solder for aluminium and its alloys.** E. CONTI (U.S.P. 1,619,852, 8.3.27. Appl., 12.9.24. Conv., 21.7.24).—See E.P. 237,224; B., 1925, 811.

**Alloy of lithium and aluminium.** J. CZOCHRALESKI and G. WELTER, ASSRS. to ALLIED PROCESS CORP. (U.S.P. 1,620,081, 8.3.27. Appl., 28.6.20. Conv., 15.2.19).—See E.P. 147,903; B., 1921, 776 A.

**Apparatus for the separation of iron and slag.** W. DIEDRICH (E.P. 267,008, 4.9.26).

**Separation of mineral pulp** (E.P. 264,129).—See I.

## XI.—ELECTROTECHNICS.

**Electric furnace with revolving arc.** G. E. EVRÉINOFF and S. Y. TELNY (Rev. Mét., 1927, 24, 57–63).—An arrangement for producing a revolving arc with direct current consists of a carbon electrode with a graphite pot, in which the charge is melted, as positive electrode. A coil, in series with the arc, surrounds the pot. The electrode *E.M.F.* varies linearly with current strength in the surrounding coil and with the diameter of the pot. The effect of furnace temperature on the *E.M.F.* is investigated. A corresponding arrangement for alternating current is described. Advantages of a revolving over a fixed arc are the greater volume of arc and greater heat-radiating surface in the former case with consequent diminution of local overheating and longer life of refractory linings in the furnace. L. M. CLARK.

**Transformer iron and wattage losses.** EICHENBERG and OERTEL.—See X.

**Potential of zinc-copper alloys.** BAUER and VOLLENBRUCK.—See X.

## PATENTS.

**Galvanic cell.** A. HEIL (G.P. 435,747, 15.5.25).—In an electric cell an alloy of chromium, preferably containing cobalt and nickel, and if desired manganese, is used as the positive electrode. With such an electrode, a cell of size 27 × 22 × 10 cm. will afford a current of 10 amp. for 15–20 hrs. J. S. G. THOMAS.

**Electric cell with constant intensity.** J. PELLINI (U.S.P. 1,621,518, 22.3.27. Appl., 30.6.25. Conv., 22.11.24).—An outer vessel forms the negative element, and is separated by a porous tubular member from a centrally arranged hollow perforated carbon member forming the positive element. The electrolyte placed in the outer vessel consists of an aqueous solution of magnesium sulphate, potassium sulphate, and sodium sulphate, whilst the depolarising liquid contained within the porous member is composed of a slightly acidified aqueous solution of sodium bichromate. The relative proportions of electrolyte and depolarising liquid are chosen so as to maintain a constant voltage of the cell for a predetermined time. J. S. G. THOMAS.

**Electrolytic decomposing cell.** F. LAWACZEK (E.P. 266,803, 30.11.25).—The opposed faces of pairs of closely packed, perforated, co-operating electrodes are electrically insulated from one another by enamel, rubber, varnish, etc., so that gases are evolved during electrolysis only from surfaces behind the plane of opposed faces. A perforated member, *e.g.*, of hard rubber, may be interposed between opposed electrodes, which are clamped together. J. S. G. THOMAS.



**Recovery of the filling material of used dry batteries.** CHEM. FABR. JOHANNISTHAL G.M.B.H., K. VON VIETINGHOFF-SCHEEL, and F. TROSTLER (G. P. 435,748, 7.1.26).—The material is extracted with dilute acids at ordinary temperature and then treated with an aqueous solution of sulphur dioxide at a higher temperature. Finally, recovered graphite is treated with stronger acids in the presence of sulphur dioxide for removal of iron.

J. S. G. THOMAS.

**Manufacture of luminous electrical [neon-] discharge tubes.** R. R. MACHLETT (U.S.P. 1,618,767, 22.2.27. Appl., 24.9.26).—The performance of neon-filled discharge tubes is improved if in their manufacture they are evacuated and heated, then filled with potassium vapour, and a high-frequency current passed through them. Removal of occluded gas is complete.

T. S. WHEELER.

**Electric insulators.** ÖSTERREICHISCHE SIEMENS-SCHUCKERT-WERKE (Aust. P. 104,007, 4.10.23).—Rubber latex treated with vulcanising substances, dyes, fillers, etc. is applied to articles to be covered with insulating material, and allowed to dry.

J. S. G. THOMAS.

**Making magnetic cores.** W. EHLERS, Assr. to GENERAL ELECTRIC CO. (U.S.P. 1,618,818, 22.2.27. Appl., 29.12.24. Conv., 29.1.24).—Magnetic cores for induction coils and the like are prepared by pressing a mixture of pulverised, magnetic material, and a relatively infusible, insulating substance, *e.g.*, porcelain or magnesia, at a temperature above that at which the magnetic material softens, but below the m.p. of the insulating substance. Alternatively a relatively fusible insulating material is employed.

T. S. WHEELER.

**[Preventing deformation of electrodes in] electric accumulators.** C. H. O. LÜBECK (E.P. 242,290, 28.10.25. Conv., 31.10.24).

**Dry battery.** BURGESS BATTERY CO., Assees. of W. B. SCHULTE (E.P. 245,464, 1.1.26. Conv., 2.1.25).

**[Separator for] electric storage batteries.** FOOLPRUFE PATENT ACCUMULATOR CO., LTD., and F. G. BROWN (E.P. 266,797, 26.11.25).

**Electrical treatment of gases etc.** (E.P. 266,983).—See I.

**Treatment of hydrogen sulphide in gases** (E.P. 267,018).—See VII.

## XII.—FATS; OILS; WAXES.

**Improvement in the quality of soaps made with hydrogenated oils.** I. M. HIROSE (J. Soc. Chem. Ind. Japan, 1927, 30, 122–128).—The effect of the addition of castor oil soap to the soap of hydrogenated herring oil is studied. A slight decrease in the drop number is observed, but the lathering power increases remarkably even by the addition of 5% of the soap, and reaches a maximum when 20% has been added. The washing power measured by the indigo method is only slightly affected by the addition of a small amount of castor oil soap, but when more than 15% is added the washing power decreases gradually. The compound soap made with castor oil fatty acid (2–3 pts.), herring

oil soap (90 pts.), and castor oil soap (10 pts.) gives very good results both in the washing and lathering power.

K. KASHIMA.

**Spontaneous heating of oils. Methods of testing.** N. J. THOMPSON (Ind. Eng. Chem., 1927, 19, 394–397).—Clean cotton waste (30 g.) impregnated with the oil to be tested (30 g.) is placed in a horizontal oil-jacketed steel tube allowing access of air to the material. The temperature is maintained at 104.4° until the temperature of the sample exceeds that of the bath by 2°; the temperature of the bath is then raised at the rate necessary just to maintain this difference. The relative tendency of an oil to heat spontaneously is shown by the temperature-time curve so obtained. Contrary to results previously recorded, the heating rate of lard oil is greater than that of cottonseed oil, the erroneous values given by the earlier methods being due possibly to the different accelerating effects of the products of partial oxidation in the two cases. A modified Mackey method using a larger sample (30 g. of oil on 30 g. of cotton) gives results comparable with those of the apparatus here described. In determining the hazard due to the spontaneous heating of oils the test method should be carried out under conditions similar to those under which the oil is to be used. If the presence of a retarding agent is suspected the duration of the test should be suitably extended.

A. B. MANNING.

**Application of thermal analysis [to waxes].** K. ARNDT (Z. angew. Chem., 1927, 40, 314–316).—Thermal analysis may be applied to the study of waxes used in electroplating etc. When cooling curves of these waxes are plotted they show a number of breaks in addition to the first one at the m.p. If, instead of temperature, the rate of cooling is plotted against time, there are strongly marked minima at the temperatures at which the breaks in the original curves occur. By mixing two waxes the minima characteristic of both are reproduced, but at somewhat lower temperatures. The exact positions of the minima vary to some extent with the treatment to which the wax has been subjected; thus, *e.g.*, for wax which has been partly saponified, a much higher m.p. is obtained. Other possible applications are indicated.

M. S. BURR.

**Action of fatty acids on rubber.** SMITH and BOONE.—See XIV.

## PATENTS.

**Manufacture of finely-divided dry soap.** R. L. HOLLIDAY, Assr. to INDUSTRIAL SPRAY-DRYING CORP. (U.S.P. 1,621,506, 22.3.27. Appl., 19.4.26).—Freely-flowing liquid soap stock is sprayed into a current of heated gas, in which the progressively drying soap particles remain in suspension until their moisture content is reduced to the desired value. S. S. WOOLF.

**Production of vitamin preparations.** A. W. OWE (E.P. 266,905, 2.3.26).—Vitamin-bearing marine fats are saponified by means of an alkali or alkaline-earth substantially free from water. The soap thus formed is extracted in a practically undissociated condition by lixiviation with a liquid edible fat, in the absence of light and oxygen.

S. S. WOOLF.

**Manufacture of solutions [of fats etc.].** I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. VORM. WEILER-



TER MEER (E.P. 245,129, 21.12.25. Conv., 22.12.24).—Waxes and fats and any admixed dyestuff are dissolved in a solution containing a monoalkyl ether of ethylene glycol, propylene glycol, or butylene glycol.

B. FULLMAN.

**Production of highly oxidised oil suitable for use as emulsifying agent, e.g., in the manufacture of margarine.** K. H. HANSEN (G.P. 396,426, 13.3.23).—A mixture of oil with a high iodine value with oil having an iodine value of less than 10 is treated with air or oxygen at about 190°, but below 200°, until the unsaturated oil is oxidised sufficiently. A small quantity of calcium hydroxide may be added to the product to prevent it from adhering to the walls of the apparatus.

L. A. COLES.

**Production of soaps and detergents.** COLLOIDAL PRODUCTS CO., Assees. of R. M. PETTIT (E.P. 243,735, 25.11.25. Conv., 27.2.25).—See U.S.P. 1,544,103—4; B., 1925, 680.

**Process for separating volatile substances.** E. WECKER (U.S.P. 1,622,126, 22.3.27. Appl., 13.3.24. Conv., 20.3.23).—See G.P. 397,332; B., 1924, 877.

**Preparation of sulpho-aromatic fatty acids** (E.P. 252,212).—See XX.

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

**[Paint] driers.** H. WOLFF (Farben-Ztg., 1927, 32, 1490—1491).—The tensile strength and elasticity of dry detached films of paints made by adding linoleates or resinates of cobalt, manganese, or each of these metals in conjunction with cerium, to a paint base consisting of 68.0% of lithopone, 0.5% of lamp-black, and 31.5% of linseed oil, indicate that the influence of driers on paints etc. must be considered not only from the point of view of induced drying time, but also from that of effect on mechanical properties of the film. The author stresses the significance of the colloid-chemical reactions caused by a drier rather than its chemical composition or metal content. Differences in the degree of dispersion of the metal account for the varying results of substituting resinate for linoleate driers, and for the non-concordant mechanical properties of films made with driers of identical chemical composition, but coming from different sources. The use of cerium in conjunction with cobalt or manganese is recommended.

S. S. WOOLF.

**Oil absorption of lithopone.** E. KLUMPP (Farben-Ztg., 1927, 32, 1491—1492).—A general discussion on lithopones of high and low oil absorption and their suitability for various purposes. The opacity of paints based on lithopone of low oil absorption is gained at the expense of durability, whereas lithopone paints of high oil absorption are durable, but have relatively poor hiding power.

S. S. WOOLF.

#### PATENTS.

**Manufacture of colour lakes.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 265,032, 29.3.26).—A basic dye, whether containing acid groups or not, is treated with components capable of forming complex tungstic acids (except those containing molybdenum) in the presence of an acid more highly dissociated than tartaric acid, such as hydrochloric, sulphuric, nitric, oxalic,

trichloroacetic, or formic acids, such acid not taking part (as does phosphoric acid) in the reaction. The colour lakes may be precipitated in the presence or absence of the usual substrates.

A. DAVIDSON.

#### Manufacture of solutions [for varnishes etc.].

I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. VORM. WEILER-TER MEER (E.P. 252,203, 21.12.25. Conv., 22.12.24).—Resins, cellulose esters, and any admixed dyestuff are dissolved in a solution containing a substantial proportion of a monoalkyl ether of ethylene glycol, propylene glycol, or butylene glycol, with the exception of the monoethyl compound. B. FULLMAN.

#### Synthetic resin and its manufacture.

E. R. LITTMANN, ASSR. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,618,209, 22.2.27. Appl., 25.11.25).—Sodium ethyl, butyl, or other alkyl 3- or 4-nitrophthalate, is treated in solution with copper sulphate, or ferric chloride, or other soluble salt of a heavy metal, when the corresponding heavy metal salt of the alkyl hydrogen nitrophthalate is obtained, in the form of a resinous substance of value as a substitute for natural resins.

T. S. WHEELER.

**Composition and processes for coating or filling surfaces.** F. R., M., and A. HERVÉ (E.P. 253,889, 7.6.26. Conv., 16.6.25).—See U.S.P. 1,615,584; B., 1927, 259.

**Luminescent product.** A. A. GUNTZ (U.S.P. 1,622,379, 29.3.27. Appl., 8.5.22. Conv., 10.6.21).—See E.P. 181,333; B., 1923, 191 A.

**Production of titanium pigments from materials containing titanium.** E. C. R. MARKS. From R. H. MONK and J. IRWIN (E.P. 266,211, 6.8.26).—See U.S.P. 1,542,350; B., 1925, 638.

**Manufacture of varnish.** F. SCHWARTZ, ASSR. to E. GIL-CAMPORRO (U.S.P. 1,621,438, 15.3.27. Appl., 19.3.25. Conv., 26.3.24).—See E.P. 231,457; B., 1926, 373.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Röntgenographic studies with metastyrene.** (Miss) M. HÜNEMÖRDER (Kautschuk, 1927, 106—107).—Between 80° and 105° metastyrene resembles rubber in its elasticity. By repeatedly warming and stretching it is possible to obtain an extension of 3000—4000%. In this condition metastyrene gives an interference X-ray diagram distinctly different from that of the unstretched material, and indicative of incipient orientation of the particles. Extrusion through fine apertures fails to induce any more marked orientation. The behaviour in this respect recalls that of synthetic rubber.

D. F. TWISS.

**Alternating behaviour of fatty acids added to rubber compounds.** W. H. SMITH and C. E. BOONE (Ind. Eng. Chem., 1927, 19, 398—399).—Using a mixture of rubber (100 pts.), sulphur (7—10 pts.), zinc oxide (5 pts.), and fatty acid (1/150 mol.), the tensile strength of the vulcanised product shows an alternating relationship as the homologous series of fatty acids is ascended from formic acid to stearic acid; the curve for the tensile strength of the products containing the acids with an even number of carbon



atoms lies above that for the acids with an odd number. There is no relation between the number of carbon atoms and the final elongation. With the phenyl-substituted fatty acids there is progressive acceleration from benzoic acid to hydrocinnamic acid.

D. F. TWISS.

**Magnesium carbonate and the transparency of vulcanised rubber.** Y. TANAKA (Trans. Inst. Rubber Ind., 1927, 2, 330—344).—Magnesium carbonate, which is used extensively in Japan in the production of transparent rubber goods, varies considerably in its quality for this purpose. Examination of the behaviour of a number of commercial samples shows that the accelerating power and the degree of beneficial effect on the mechanical properties lie in the inverse order to the transparency in rubber. The more transparent grades consist of amorphous particles, whereas those consisting entirely or in part of needle crystals are inferior in this respect. The former have a refractive index approximately equal to that of vulcanised rubber, whilst the latter contain a mixture of particles of diverse refractivities. Satisfactory magnesium carbonate for the production of vulcanised rubber can be selected by examination of the effect on the appearance of a mixture of nitrobenzene and petroleum adjusted to  $n_D$  1.525, or of Canada balsam ( $n_D$  1.52—1.53), or of cedar oil ( $n_D$  1.51—1.52).

D. F. TWISS.

**Volatile oxidation product of balata.** T. R. DAWSON and B. D. PORRITT (Trans. Inst. Rubber Ind., 1927, 2, 345—353).—Venezuelan sheet balata purified by extraction with acetone and separation from clear solution in benzene by the addition of alcohol was exposed to a current of air at the ordinary temperature. The oxidation process followed an auto-catalytic course, and yielded a final product approximating to the composition  $C_{10}H_{16}O_3$ . A small quantity of formic acid also was produced, but no carbon dioxide; on the assumption that one carbon atom in every  $(C_{10}H_{16})_n$  is converted into formic acid the value of  $n$  would be 112.

D. F. TWISS.

**Ageing properties of raw and vulcanised rubber.** G. MARTIN (Trans. Inst. Rubber Ind., 1927, 2, 354—380).—Examination of mixtures with sulphur (90:10) vulcanised to give an elongation of 860% at a load of 1.04 kg./sq. mm., shows no difference in the "ageing" properties of quickly-dried and "matured" rubber from comparable coagula. Sheet rubber, however, ages better than crêpe rubber. Sheet rubber also can be vulcanised over a much wider range than crepe without the development of rapid deterioration in the tensile strength on ageing. With respect to maintenance and tensile strength, evaporated latex is superior to sheet and hard fine Para rubber, and still more so to crêpe rubber; on the other hand, with respect to extension under a given load, hard fine Para undergoes less change than crêpe or sheet rubbers, and these less than evaporated latex. These differences are attributable to the serum substances, the removal or addition of which is shown to have a marked effect. Treatment of rubber with aqueous ammonia removes substances responsible for its satisfactory ageing. After extraction with acetone likewise the ageing properties of rubber are depreciated, but the addition of serum solids in this case has little

beneficial effect, although the re-introduction of the acetone extract is very effective. It is probable, therefore, that more than one substance is necessary for the satisfactory ageing properties of plantation rubber, and that a particular balance of these active non-caoutchouc constituents is of great importance.

D. F. TWISS.

**Rubber solvents.** D. F. TWISS (Trans. Inst. Rubber Ind., 1927, 2, 381—408).—The more important solvents for rubber are reviewed, and the relative behaviour of rubber towards them is considered, *e.g.*, as to rate of swelling or viscosity and transparency of the resulting solutions. Other features of practical importance such as vapour pressure, relative inflammability, and solubility for water and sulphur are also discussed.

D. F. TWISS.

**Rubber content of ammoniated latex.** R. O. BISHOP (Malayan Agric. J., 1927, 15, 1—11).—The apparent rubber content of latex preserved with a minimum quantity of ammonia, as indicated by evaporation, or coagulation with acetic acid or alcohol, rises slightly during the early stages of storage and then gradually decreases. The weight of coagulum with acetic acid also varies with the concentration of the acid coagulant and with the proportion of acid in excess of that required for the neutralisation of any ammonia present. Accurate evaluation of the rubber content of latex by coagulation with acetic acid is consequently not simple, and for the ensuring of concordant results the method must be elaborated.

D. F. TWISS.

#### PATENTS.

**Manufacture of rubber-compounding material.** BARRETT CO., Assees. of A. B. COWDERY (E.P. 243,384. 21.11.25. Conv., 22.11.24).—Coal tar is distilled so as to give a residue containing approximately 60% of finely-divided carbon and 40% of hydrocarbons. This product, having  $d$  1.30—1.35 and m.p. above 177°, is used as a compounding ingredient for rubber.

D. F. TWISS.

**Manufacture of rubber.** P. SCHIDROWITZ, and VULTEX, LTD. (E.P. 266,418, 28.10.25. Addn. to E.P. 193,451; B., 1923, 732 A).—Concentrated latex produced by any convenient process is mixed with vulcanising ingredients, *e.g.*, sulphur or polysulphides, accelerators, and with other compounding ingredients, suitable preservatives and preservative colloids, if necessary, also being present. Vulcanisation may be effected by a low-temperature process or under the more customary conditions above 100°.

D. F. TWISS.

**Electric insulators** (Austr. P. 104,007).—See XI.

**Biguanides [as accelerators]** (G.P. 435,668).—See XX.

#### XV.—LEATHER; GLUE.

**Mechanism of tanning. I. Fixation of vegetable tannins by chrome-tanned hide protein.** K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1927, 22, 125—139).—Samples of hide powder treated with anionic, cationic, and mixed chromium compounds respectively, were then subjected to a solution of hemlock bark extract, gallotannic acid, or sulphited quebracho extract. The cationic chrome-tanned powder had a much greater affinity for the vegetable tannins than



either the anionic chrome-tanned powder or the original powder in the usual  $p_H$  range for vegetable tanning, viz. 3–6. Untanned hide powder fixed more tannin than anionic chrome-tanned powder except at high  $p_H$  values. The acidity of the chrome-collagen compound was greatly reduced by the vegetable tannage. The portion of the acid combined with the collagen was completely removed by the vegetable retannage, and acido-groups were also removed from the chromium complex. The latter was accompanied by a loss of fixed chromium. Hide powder freshly tanned with cationic chromium compounds loses chromium when treated with vegetable tannins, but if it is "aged" before the latter treatment there is no loss of chromium. Anionic chrome-tanned powder loses chromium with vegetable retanning, whether "aged" or not. This supports the view that the same protein groups are affected by anionic chromium compounds and vegetable tannins. The increased capacity of cationic chrome-tanned powder for vegetable tannin is attributed to the activation of the basic protein groups, resulting from the breaking up of the closed structure by the reaction of acidic protein groups with chromium complexes. The fixation of vegetable tannin by chrome-tanned powder was practically independent of the  $p_H$  value of the vegetable tannin solution. The Procter-Wilson theory was not applicable in this case. This is attributed to the rigid structure of the chrome-tanned powder. The effects of hydrogen and hydroxyl ions are negligible compared with those on collagen itself.

D. WOODROFFE.

**Action of trypsin on calfskin.** H. B. MERRILL and J. W. FLEMING (J. Amer. Leather Chem. Assoc., 1927, 22, 139–161).—Experiments have shown that all the collagen in calfskin is capable of digestion by trypsin. The rate of digestion increases considerably between 35° and 40°. It is a maximum on the alkaline side of  $p_H$  8.0. Under usual conditions of bating, the action of the trypsin on the collagen is so slight that little or none is lost during that operation. The non-collagen proteins of limed skin constitute 5% of the dry weight of the skin. The quantity of material removed in a few hours from limed calfskin by trypsin increases with the duration of the preceding liming. Marriott's theory of "altered collagen" (B., 1926, 682) and the "protective sheath" theory (B., 1923, 367 A) are discussed. It is shown that the type of curve obtained from the rate of hydrolysis of collagen by trypsin does not agree with these theories, but suggests that the collagen is equally susceptible to the action of the trypsin. The principal function of trypsin in the bating of calfskins is the removal of protein degradation products formed in the lime liquor, of which the most important is probably keratose, the constituent of the interfibrillary substance or solution. The longer the liming, the greater the amount of degradation products to be removed. No elastin is removed from calfskin during the ordinary bating operation. It is probable that its removal is more important in goat-skin bating. Keratose is present in limed skins, and is digested by trypsin, so that it is probably removed by bating. Keratose is precipitated by solutions of  $p_H$  less than 4.1, and it seems probable that the precipitation of such in a skin would impair the finished leather.

D. WOODROFFE.

**Bating action and methods of measuring enzymic activity of natural and artificial bates.** G. HUGONIN (J. Soc. Leather Trades' Chem., 1927, 11, 1–18).—The characteristics of a bated skin are the cleansing of the fibres from interfibrillary substance and from degraded collagen produced in the liming, the "fallen" supple nature, the absence of elasticity and swelling, the porosity to air, the scud is easily raised by scraping the grain, the latter has a feel like oily glass, and most of the lime is removed. The activity of the enzymes present in bates is affected by the temperature of the medium, the enzyme concentration, the deliming agent, neutral salts, hydrogen-ion concentration, and suspended matter which can absorb the enzymes and increase their activity. Pepsin has optimum action at  $p_H$  1.4, does not readily dissolve the elastin, and attacks the grain and flesh sides equally. The optimum effect of trypsin is at  $p_H$  8.0. The liquor should be alkaline to cresol red and acid to phenolphthalein. Tryptic hydrolysis is more drastic than peptic digestion. It hydrolyses proteins to simple polypeptides and amino-acids. Blood serum stimulates tryptic action. Fresh pancreatic extract has only feeble proteolytic activity, but the latter increases after a while and under certain conditions. The pancreas of animals slaughtered in course of digestion becomes active more readily than if slaughtered fasting. The pancreatic extracts of dogs and cattle activate equally well, that of cats only with difficulty. Extraction with boiling water or glycerin water renders it inactive. Unboiled water or water acidified with acetic or lactic acids furnishes an active extract. The addition of amino-acids renders it still more active. The activating agent is enterokinase. Pancreatic trypsin differs in its action from pepsin by forming several compounds titratable with formaldehyde (amino-acids, tyrosine, tryptophan, etc.), by showing a tendency in weak solutions to build up synthetic products, and by not reaching the limiting amount of peptone by a gradual process, but in a series of oscillations. Trypsin acts in liming by dissolving the keratinous layers in contact with the hyaline layer and in bating, where it digests the elastic fibres and the keratose and greases without attacking the collagen. Erepsin is obtained from the small intestine of dogs. It does not act on natural proteins, but acts on albumoses and peptones, and its optimum effect is the same as for trypsin. Pancreatin always contains erepsin. Bating "falls" the skin by acting as a "buffer" of  $p_H$  8.0. Boric acid, ammonium sulphate and chloride, weak organic acids and substances which form them by fermentation, and sodium dihydrogen phosphate may each be used for deliming and "falling" of pelts. Bating usually removes lime, but some artificial bates contain insufficient deliming agent. A preliminary controlled acid deliming is advisable before bating. The pancreatic enzymes are capable of dissolving the elastin fibres during bating, and this property has been suggested for purposes of controlling the digestive power of the enzymes. The speed of digestion of elastin by the enzymes is proportional to the concentration and the time of action. Ammonium chloride (0.5 g./litre) stimulates the enzyme activity at  $p_H$  7.6. Larger quantities have the opposite effect. Solutions of greater concentration than 5% hydrolyse the collagen fibres. Most of the hide substance



dissolved during bating is due to the previous action of the lime liquors on the pelt. Methods have been devised for controlling the effects of bating.

D. WOODROFFE.

**Leather dyeing.** VI. H. SALT (J. Soc. Leather Trades' Chem., 1927, 11, 18—19).—Sheep grains tanned by the one-bath and two-bath chrome tannages, respectively, were dyed with a number of acid, basic, and direct dyes, respectively. The acid and direct dyes produced full shades on both types of leather and the basic dyes weak shades. The two tannages are apparently chemically alike with respect to dyeing properties.

D. WOODROFFE.

**X-Ray examination of the tanning of membranes and tendons.** R. O. HERZOG (Kolloid-Z., 1927, 41, 277).—Polemical (see Katz and Gerngross; B., 1927, 150).

E. S. HEDGES.

#### PATENTS.

**Apparatus for drying leather** (E.P. 266,464).—See V.

**Dyeing leather** (E.P. 247,187).—See VI.

### XVI.—AGRICULTURE.

**Reversion of nitrates in the soil under cultural conditions in Mauritius.** N. CRAIG and F. GIRAUD (Dept. Agric. Mauritius, 1926, Bull. No. 11, 19 pp.).—Laboratory experiments are reported on the extent to which denitrification takes place when organic manures are applied to soil at the same time as ammonium sulphate or sodium nitrate. Addition of large amounts of molasses to a soil in which nitrification normally proceeded fairly rapidly, inhibited nitrification and produced conditions under which ammonical and nitric nitrogen reverted to organic nitrogen. Dried green manure and farmyard manure acted in the same manner, but to a somewhat less extent. Molasses and green manure did not cause an increase in the loss of nitrogen as gas, whereas with farmyard manure a large increase in this loss occurred. The results support and explain the conclusions drawn from previous field experiments with sugar cane on Mauritius soils.

C. T. GIMINGHAM.

**Relation between the "citric soluble" and "root soluble" nutrients in soils.** G. HASENBÄUMER and R. BALKS (Z. Pflanz. Düng., 1927, B 6, 116—122).—Additions of phosphoric acid, as basic slag or superphosphate, to soil in various proportions, and subsequent extraction with 1% citric acid, gave average figures of 98% of the added  $P_2O_5$  recovered in the case of light soils, and 65% in the case of heavy soils. Corresponding figures for the amounts of added  $P_2O_5$  recovered in rye seedlings, used according to Neubauer's method for determination of available plant nutrients in soils, were 35% and 31% for light and heavy soils respectively. With added potassium, on the other hand, an average of 82% was recovered in the seedlings and 69% by extraction with citric acid.

C. T. GIMINGHAM.

**Influence of drying and heating arable soils on their content of water-soluble phosphoric acid.** A. T. SCHLESING and D. LEDOUX (Compt. rend., 1927, 184, 649—652).—Samples of soil, dried by sun or in an oven, and undried, were washed under standard

conditions with water containing 0.08 g. of calcium nitrate per litre. The washings were repeated until determinations of phosphoric acid gave the same value on washings coming from the dried and the untreated soil. Lebediantzeff's results (B., 1924, 305, 346) have been confirmed on soil from Dangu (dept. Eure) and Villepreux (Seine-et-Oise) taken at depths of 10—25 cm. and 15—25 cm. respectively, but the above method shows differences in the water-soluble phosphoric acid content between the dried and untreated soil about 50 times greater than those found by Lebediantzeff.

R. BRIGHTMAN.

**Relative weights of reacting substances in colloidal flocculations.** J. DUMONT (Compt. rend., 1927, 184, 764—766).—Experiments on the flocculating power of 0.1*N*-solutions of ammonium, sodium, and potassium chlorides, and 0.01*N*-calcium and magnesium chlorides on different specimens of clay soils, before and after separation of the colloidal matter by Schloësing's method (B., 1926, 717), show that the ratio of the weight of flocculant actually participating in the reaction to the weight of gel formed varies with the sample of soil examined and increases with the colloidal content of the sample, though without definite relation to the ratio of slime to colloids in the soil. The limiting amounts of flocculant required are not strictly proportional to the physical composition of the clay, although dependent on the nature and source of the latter. The clay constituents of soils represent either a mixture of electro-negative colloids tending to disperse, or a colloidal complex with amphoteric tendencies which has resulted from the combination of colloids with opposite charges.

R. BRIGHTMAN.

**Manganese deficiency in soils and fertilisers.** O. SCHREINER and P. R. DAWSON (Ind. Eng. Chem., 1927, 19, 400—404).—A description of laboratory and field experiments on the growing of tomatoes in a highly calcareous and slightly alkaline soil (82—92%  $CaCO_3$ , 5% of organic matter, manganese under 0.001%) occurring in Florida, which, with heavy application of inorganic fertilisers, failed to produce a crop unless stable manure was applied to the young plants. With fertilisers alone and without manure the plants failed to blossom, and developed a strikingly characteristic chlorosis indicative of a failure of normal leaf function caused by inadequate chlorophyll synthesis. The addition of from 25—50 p.p.m. of manganese sulphate together with the application of a balanced inorganic fertiliser or peat caused a strong improvement in the growth, colour, and yield of fruit, the plants manifesting a luxuriant growth from the start. Under the conditions prevailing in this type of soil the presence of manganese is indispensable to the normal growth of the tomato plant.

E. H. SHARPLES.

**Manuring of chicory.** J. NUDING (Z. Pflanz. Düng., 1927, B 6, 97—115).—Details are given of manurial experiments with chicory, and the results are discussed from an economic point of view. C. T. GIMINGHAM.

**Sulphite liquor as a spray.** FLEMING and REEDY.—See V.

#### PATENTS.

**Measurement of soil moisture.** SIEMENS & HALSKE A.-G., Assees. of O. WERNER (G.P. 435,229



6.3.25).—A rod- or band-shaped device is made of a porous substance, *e.g.*, blotting paper, and inserted in the soil to a determined depth. From the time taken for the moisture absorbed from the soil to reach a certain height above the surface a measure is obtained of the moisture content of the soil. G. W. ROBINSON.

**Apparatus for measurement of suction force and moisture content of soils.** W. KORNEFF (G.P. 435,815, 1.4.25).—A porous vessel is connected with a non-porous vessel furnished with a thermometer and manometer. The whole apparatus is filled with water. The porous vessel is plunged in the soil and the variations as shown by the manometer give a measure of the changes in the moisture content of the soil.

G. W. ROBINSON.

**Stabilisation of hydrocyanic acid for use as an insecticide.** CHEM. FABR. DR. H. STOLTZENBERG (G.P. 435,714, 3.7.25).—Sulphur dioxide is pumped into a steel receptacle maintained at a low temperature and containing liquid hydrogen cyanide, if necessary under pressure. The receptacle is allowed to attain ordinary temperature. It now contains liquid sulphur dioxide with an admixture of hydrogen cyanide as a liquid phase, and a mixture of the two gases as the gaseous phase. The pressure is used to force out the liquid through a tube, which reaches to the bottom of the receptacle, and is furnished with an atomising jet to produce a fine spray or cloud which rapidly vaporises.

G. W. ROBINSON.

**Insecticidal plant spray.** I. G. FARBENIND. A.-G., Assees. of R. MAY and W. MISCHON (G.P. 435,713, 4.3.23).—The first runnings from benzene distillation, with or without addition of emulsifying agents, have an insecticidal action greater than that corresponding with the amount of carbon disulphide present, and are without the disadvantages of carbon disulphide. The insecticidal effect may be due to constituents such as thiophen, alcohols, and hydrocarbons.

G. W. ROBINSON.

**[Increasing adhesiveness of] insecticidal and like powders.** I. G. FARBENIND. A.-G., Assees. of FARBEN-FABR. VORM. F. BAYER & Co. (E.P. 250,180, 21.1.26. Conv., 3.4.25).—The adhesive properties of insecticidal and fungicidal dusts are greatly increased by intimate admixture with small quantities of a product obtained by treating alkali cellulose with an alkali salt of a halogen-substituted aliphatic acid.

C. T. GIMINGHAM.

**Insecticide and fungicide.** K. S. BOYNTON, Assr. to E.-Z-WAY Co. (U.S.P. 1,621,825, 22.3.27. Appl., 25.7.23).—Sodium sulphide is melted in its own water of crystallisation and sulphur is added to substantial saturation. The mixture of sodium polysulphides thus formed is allowed partly to cool, and is mixed with soap until a paste-like consistency is assumed. 2% of copper sulphate may also be added.

C. T. GIMINGHAM.

**Production of manure by treatment of organic refuse.** A. BAUMGARTEN-CRUSIUS (E.P. 246,484, 21.1.26. Conv., 21.1.25).—Organic refuse is caused to undergo a process of hot fermentation by blowing air through the mass, stacked in loose layers. The mass is maintained above a minimum temperature of 50° (*e.g.*, at about 65°), which ensures destruction of weed seeds and harmful organisms, and causes the fermentation to be

greatly accelerated. The resulting manure may be used at once, or stored under conditions which facilitate the initiation of a second fermentation by renewed aeration.

C. T. GIMINGHAM.

**Manufacture of fertilisers.** G. CLAUDE, Assr. to SOC. L'AIR LIQUIDE (SOC. ANON. POUR L'ETUDE ET L'EXPLOITATION DES PROCÉDÉS GEORGES CLAUDE) (U.S.P. 1,621,311, 15.3.27. Appl., 20.12.23. Conv., 24.1.23. Renewed 5.4.26).—See E.P. 210,399; B., 1924, 530.

## XVII.—SUGARS; STARCHES; GUMS.

### PATENTS.

**Citric acid** (E.P. 266,414—5 and G.P. 431,729).—See XVIII.

## XVIII.—FERMENTATION INDUSTRIES.

**Fermentation in candy and creams.** PAINE and others.—See XIX.

### PATENTS.

**Preparation of citric acid.** B. BLEYER (G.P. 434,729, 19.10.24).—To a 10–15% solution of sucrose, glucose, or maltose, with the addition of nutrient salts (ammonium nitrate, potassium phosphate, and magnesium sulphate), mineral acid or citric acid is added to give a  $p_H$  of 3.3–3.6; the whole is sterilised by heating and then seeded with *Aspergillus*, *Sterigmatocystis*, *Penicillium*, or other related organisms. Gentle warming assists the growth. When germination occurs and short mycellar threads appear, sterilised air is passed in, the liquid is gently stirred, respiratory carbon dioxide being at the same time removed, and acidification commences and continues. For *Aspergillus* the optimum temperature is 15–20° and the maximum yield of citric acid (60–75%) is reached in 5–8 days.

C. HOLLINS.

**Production of citric acid.** A. FERNBACH, J. L. YUILL, and ROWNTREE & Co., LTD (E.P. 266,414—5, 26.10.25).—Solutions of sugars and like substances or mixtures thereof with added nutrient materials are fermented by moulds or fungi, in particular *aspergilli*. Sterilisation of the medium by heat is avoided by the addition of a sufficient quantity of mineral or strong organic acid, so that the hydrogen-ion concentration of the medium is raised, prior to inoculation, to such a point that not only bacteria but yeast and the majority of other fungi are inhibited, whilst the selected organism still functions. The solution is incubated at 10–40°, and is brought as far as possible into contact with the mould. The sugars and fresh nutrients may be added continuously or intermittently with or without the withdrawal of the citric acid liquor.

C. RANKEN.

**Revivifying carbon used in purifying ethylene.** **Revivifying carbon.** A. A. BACKHAUS, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,619,326—7, 1.3.27. Appl., 27.9.24).—See E.P. 240,126—7; B., 1925, 1007.

**Manure from refuse** (E.P. 246,484).—See XVI.

## XIX.—FOODS.

**Volatile constituents of foodstuffs.** J. KÖNIG and W. SCHREIBER (Z. Unters. Lebensm., 1927, 53, 1–44).—Earlier work has shown that among the substances volatilised when certain foods are heated as in cooking,



may be included carbon dioxide, mercaptan, hydrogen sulphide, aldehydes, and formic acid. By the authors' method all these compounds are determined in one operation by using ammoniacal silver solution as a common absorbent. The material under investigation is heated at 98–100° in an atmosphere of nitrogen for 2 hrs. on a paraffin bath. Four absorption vessels are used containing ammoniacal silver hydroxide solution (3 g. of silver oxide in 100 c.c. of 24% ammonia solution), the third vessel being warmed at 50° and the last serving as a control. Coloured deposits and silver mirrors are formed during the distillation. The contents of the receivers are filtered and systematically analysed. The combined sediment is treated with 5% hydrochloric acid to liberate mercaptan, and then with concentrated acid to liberate hydrogen sulphide, both gases being oxidised by alkaline hydrogen peroxide and determined as sulphuric acid. The residue is treated with ammonia and nitric acid, silver being then precipitated with hydrochloric acid, dried, and weighed. The filtrate after removal of silver is evaporated to dryness, dissolved in nitric acid, and analysed for phosphoric acid. The filtrate from the absorption vessels is boiled (in absence of air) with 3% baryta solution. The precipitate is filtered and decomposed with hydrochloric acid, the carbon dioxide evolved being absorbed in potash. The filtrate is evaporated to dryness with Devarda's alloy, the residue being dissolved in water, acidified with sulphuric acid, and distilled. The formic acid in the distillate is precipitated with mercurous chloride, filtered, dried, and weighed. The residue after distillation is extracted with ether, from which higher organic acids are deposited on evaporation. Details are given of the results obtained for a large number of animal and vegetable materials, the results for coconut oil being entirely negative. H. DOWDEN.

#### Sugar-tolerant yeasts in chocolate-coated creams.

M. B. CHURCH, H. S. PAINE, and J. HAMILTON (Ind. Eng. Chem., 1927, 19, 353–357).—Cultural and microscopical examinations of chocolate-coated creams inoculated with purified yeasts obtained from commercial samples of "burst" chocolates have been made, and it is concluded that the bursting is primarily due to the formation of minute quantities of gas in the cream by the activity of yeasts of high sugar tolerance. The yeasts are of various types, and are either spore-forming or torula-like or a mixture of both types. Experiments on the inoculation of creams with various spore-forming anaerobes gave no evidence of sugar tolerance for these bacteria and no bursting occurred. E. H. SHARPLES.

#### Means for preventing "explosive" or bursting fermentation of chocolate-coated fondant candy.

H. S. PAINE, V. BIRCKNER, and J. HAMILTON (Ind. Eng. Chem., 1927, 19, 358–363).—The fermentation of chocolate-coated creams made from syrups of different composition and consistency and inoculated with highly sugar-tolerant yeasts has been examined. The bursting caused by the presence of these organisms may be prevented by the addition of invertase. This, by inverting a portion of the sucrose present and thereby increasing the sugar solubility, causes an increase in the density and osmotic pressure of the syrup phase of the

fondant, and renders it resistant to fermentation. Fondants containing a syrup phase with a solids concentration of 79% or over caused by the addition of invertase were free from fermentation. E. H. SHARPLES.

**Pectins.** A. MEHLITZ (Kolloid-Z., 1927, 41, 130–146).—A method is described for the fractional filtration of solutions of fruit pectins through membranes graded with respect to the size of the pores, and a description is given of experiments on the determination of the size of the particles in apple pectin by this method. About one third of the particles are greater than 0.6 $\mu$ , about two thirds are between 0.2 $\mu$  and 0.6, and less than 10% are smaller than 0.2 $\mu$ . Apple pectin of  $\eta_{sp}$  3.00 on fractional filtration undergoes a decrease in titration acidity, hydrogen-ion concentration, and viscosity with decrease in the size of the pores of the filter, the change of acidity being affected by the duration of filtering. All fractions gave a positive starch reaction, which was weaker in those passed through the finer filters. A close parallelism was observed between the methoxyl content of the pectin fractions and their ability to gelatinise (cf. Nanji and Norman, B., 1926, 930). E. S. HEDGES.

#### PATENTS.

**Sterilising and preserving milk and similar liquids.** M. BING (G.P. 392,935, 11.5.22).—Fresh milk in a finely-divided state is subjected to the action of ultra-violet rays, treated with hydrogen peroxide, and the excess of the latter decomposed by addition of a small quantity of fresh milk or cream. The product is germ-free, and resembles natural milk in taste.

F. R. ENNOS.

#### Improvement of creams which have undergone certain fermentations, and production of butter.

M. O'CALLAGHAN (E.P. 266,951, 27.5.26).—The cream is neutralised by the addition of a solution of calcium hydroxide or sodium bicarbonate, whereby the nitrogenous matter is precipitated, and heated to the pasteurising temperature. While being centrifuged it is washed with a current of acidified water to remove the flavour due to the alkali and the nitrogenous matter, and then mixed with a current of water or sweet milk, according as it is desired to manufacture butter or reconstituted cream. F. R. ENNOS.

**Extraction of albumin and nutritive salts from the water of potatoes.** R. VOGEL (G.P. 400,414, 13.1.21).—The water is made to froth by whisking or stirring, and the foam skimmed off and dried. F. R. ENNOS.

**Renewal of used brine lake by extraction of its albumin.** GRÄFF & Co. (G.P. 394,993, 19.7.22).—The lake is acidified with hydrochloric acid, the albumin which separates filtered in the usual way, and the filtrate neutralised with sodium hydroxide or carbonate solution. After re-filtering, if necessary, the lake is again ready for use. F. R. ENNOS.

**Treatment of miller's wheat to eliminate the wild garlic it contains.** L. G. PATROUILLEAU (E.P. 260,283, 21.10.26. Conv., 22.10.25).—See F.P. 604,950; B., 1926, 848.

**Mixed salt for nutritive purposes** (G.P. 398,093).—See VII.

**Vitamin preparations** (E.P. 266,905).—See XII.



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

### Furfuraldehyde manufacture from oat hulls.

I. The liquid-solid ratio. H. J. BROWNLEE (Ind. Eng. Chem., 1927, 19, 422—424; cf. B., 1925, 114).—Experiments are described on the yields of furfuraldehyde obtained by steam-distillation of ground oat hulls digested with dilute sulphuric acid under a pressure of 60 lb. (153°) for 5 hrs. in a rotary digester. The acid used was equal to 2.1% by wt. of the hulls and the rate of distillation averaged about 1600 lb. of distillate per hr. from 4500 lb. of hulls. The furfuraldehyde yields in the distillate reached a maximum (10.3%, concentration 5.8%) with a liquid-solid ratio in the initial charge of 0.27:1. These values declined as the liquid-solid ratio was either increased or decreased. The total furfuraldehyde in the system, *i.e.*, yield in distillate and that obtained from the residue, was at a maximum (12.6%) at a ratio of 0.35:1. Steam requirements are discussed, and it is concluded that both from the point of view of increased yields and decreased steam consumption a low initial liquid-solid ratio is desirable. E. H. SHARPLES.

Effect of polarised light on the pharmacodynamic properties of some drugs. D. I. MACHT and J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1927, 16, 106—110).—The action of polarised light of the visible spectrum on tincture of digitalis and on solutions of quinine and cocaine has been examined. The solutions were irradiated simultaneously with polarised and non-polarised light of the same intensity, and even comparatively short exposures (45—60 min.) to polarised light produced a distinct deterioration (10% or more) in their pharmacological activity. This change was not produced to the same degree even by longer exposures to ordinary non-polarised light. The methods used in assaying the solutions are described and the two new methods for testing the pharmacological activity of cocaine are outlined. E. H. SHARPLES.

Effect of ultra-violet and polarised light on mercurochrome. D. I. MACHT and J. H. HILL (J. Amer. Pharm. Assoc., 1927, 16, 110—113).—Exposure of solutions of mercurochrome-220 (1 in 500) to ultra-violet rays ( $\lambda = 1850 \text{ \AA}$ . and upwards) or to polarised light produces no deterioration in the antiseptic or germicidal efficiency tested on cultures of *B. coli*. E. H. SHARPLES.

Colorimetric assay of strophanthus. L. W. ROWE (J. Amer. Pharm. Assoc., 1927, 16, 113—115).—A series of comparative tests on the assay of tincture of strophanthus by the picric acid colorimetric method based on the Baljet reaction (A., 1919, ii, 438) and the Houghton frog method. The total activity being apparently always proportional, though not equal, to the alimentary absorbable activity, if a purification method is not necessary, the colorimetric method, using ouabain, 1 in 25,000, or tincture of strophanthus, U.S.P. X., 1 in 140, as standards, can be used as a preliminary test to a short final check of the activity by the frog method. The colorimetric method is not suitable for the assay of squill, convallaria, or veratrum preparations as judged by one attempt on each. E. H. SHARPLES.

Some constituents of Spanish moss. A. W. SCHORGER (Ind. Eng. Chem., 1927, 19, 409—411).—Spanish moss (*Tillandsia usneoides*) contains galactan (less than 1%), araban, xylan, cellulose, and apparently a glucoside. The analyses of the dry material before and after retting, respectively, are: ash 5.51%, 0.54%; nitrogen 1.11%, 0.80%; pentosans 15.68%, 18.13%; non-carbohydrates 16.63%, 19.51%; methoxyl 1.86%, 3.03%; cellulose 46.78%, 38.87%; pentosans in cellulose —, 16.52%. Protein, chlorophyll, a carotinoid pigment, a sterol, and considerable quantities of a wax (saponif. value 41.24) are present in the non-carbohydrates. Methylpentosans and pectin are absent. E. H. SHARPLES.

Effect of vacuum distillation on vanilla extract. C. B. GNADINGER (Ind. Eng. Chem., 1927, 19, 342—344).—Vanilla extracts concentrated under reduced pressures at temperatures below 60° showed a loss in quality, strength of flavour, and aroma varying from 10 to 40% depending on the degree of concentration and the kind of bean. This was due to loss of volatile oils and acids, and not to chemical change or decomposition. At high concentrations the aroma was almost entirely lost. The content of reducing sugars, sucrose, non-sugar solids, vanilla resins, and the lead number were unchanged, and, except with very rapid distillation, there was no loss of vanillin. Anisyl alcohol and piperonal, absent from Mexican and Bourbon extracts, were present in Tahiti extracts both before and after distillation, and also in the distillate. Concentrates made from acetone extracts were inferior to those from alcohol due to the extraction of acrid matter. E. H. SHARPLES.

Studies in the genus *Mentha*. 12. Significance of the presence of  $\gamma$ -methyl-*n*-butyl alcohol in *Mentha piperita*, L., and its identification. S. M. GORDON (J. Amer. Pharm. Assoc., 1927, 16, 130—135).—From an exhaustive examination of 150 lb. of "first runnings" from the distillation of peppermint oil it is concluded that *isobutylcarbinol* is present in American peppermint oil, and that it is formed by simultaneous reduction and oxidation of *isovaleraldehyde*, together with *isovaleric acid*. E. H. SHARPLES.

### PATENTS.

Production of alcohols from methane. G. L. E. PATART (F.P. 593,650, 7.2.25).—Methane mixed with half its volume of oxygen is passed over coke or other combustible fuel at 800—1000°. The gas mixture is preheated to avoid any condensation or any fluctuations in temperature. The gaseous product, consisting of approximately 2 vols. of hydrogen to 1 vol. of carbon monoxide, is then converted into alcohols by the action of suitable catalysts at high temperatures and under pressure. Methane formed as a by-product is separated and utilised in the process. A. B. MANNING.

Catalytic production of methyl alcohol and liquid hydrocarbons. G. L. E. PATART (F.P. 594,121, 23.2.25).—A mixture of equal volumes of illuminating gas and water-gas is passed over a catalyst, consisting, *e.g.*, of zinc oxide and chromium oxide, at 300° and under 150—250 atm. pressure. On cooling, under the same pressure, a liquid product is formed which separates into two layers, the upper



containing hydrocarbons and the lower methyl alcohol. The gaseous product is returned to the initial reaction mixture. In the last hour of the reaction the amount of methyl alcohol produced is scarcely diminished, the pressure having fallen meanwhile to only 34 atm. It is better, therefore, to work at very high pressures (800–1000 atm.). Methane produced in the reaction is separated by refrigeration under pressure and submitted to the process of F.P. 593,650 (cf. preceding abstract). The residual gas mixture consists of hydrogen, nitrogen, and carbon monoxide. It is therefore possible to carry out the process without water-gas, using coal or coke-oven gas only. The liquid product may be used directly for heating or carburetting purposes, or the hydrocarbons may be separated after addition of water, and pure methyl alcohol obtained from the aqueous solution by distillation. A. B. MANNING.

**Synthesis of the higher alcohols.** G. L. E. PATART (F.P. 593,649, 7.2.25).—The hydrocarbon product from the process of F.P. 594,121 (cf. preceding abstract) is passed over the same or another catalyst, the gases and reaction chambers being heated to avoid any condensation or undue temperature fluctuations. The higher alcohols are thereby produced in greater quantity than methyl alcohol. A. B. MANNING.

**Simultaneous production of methyl alcohol and liquid hydrocarbons.** G. L. E. PATART (F.P. 593,648, 7.2.25; cf. preceding abstracts).—Gas mixtures containing hydrogen, carbon monoxide, and ethylene or its higher gaseous homologues, are submitted to the action of the catalysts used in the synthesis of methyl alcohol. Thus a mixture of 23%  $C_2H_4$ , 22%  $CO$ , 49%  $H_2$ , 1.2%  $CO_2$ , 0.5%  $O_2$ , and 4%  $N_2$  was passed over a basic zinc chromate catalyst at  $300^\circ$  under 150–250 atm. pressure; the cooled reaction product formed two layers, of which the upper (about  $\frac{1}{2}$ ) consisted of hydrocarbons of  $d^{15}$  0.735, and possessed the fluorescence characteristic of some petroleum hydrocarbons. The lower layer was almost pure methyl alcohol,  $d^{15}$  0.805, b.p.  $66-68^\circ$ . The process can be applied to gases produced by the cracking of petroleum or other oils. A. B. MANNING.

**Synthesis of the higher aliphatic alcohols, and their separation.** G. L. E. PATART (F.P. 593,447, 9.4.25; cf. preceding abstracts).—Catalysts for the reduction of carbon monoxide or carbon dioxide by hydrogen under pressure at higher temperatures consist of an intimate mixture of alkali or alkaline-earth oxides with other metallic oxides. The former are obtained from the chromates, manganates, molybdates, tungstates, uranates, or vanadates of potassium, sodium, rubidium, barium, etc.; the number of atoms of alkali metal in the catalyst should amount to at least one half, or of alkaline-earth metals one quarter, of the total number of metallic atoms present. *E.g.*, zinc oxide is mixed with potassium chromate or manganese dioxide with neutral barium tungstate, the mixture made into a paste with 10% of a 10–20% dextrin or tragacanth solution, granulated, and dried at a temperature not higher than  $320^\circ$ ; it is freed from dust before use. A suitable gas mixture contains about 50 volumes of hydrogen to 40 volumes of carbon monoxide. During the reduction, which is carried out at  $330-400^\circ$ ,

the carbon dioxide and methane formed are removed by refrigeration under high pressure. The liquid reaction product separates into two layers, an upper layer (about 30%) of  $d$  0.885–0.890 and a lower aqueous layer of  $d$  0.925–0.940. The alcohols in the lower layer are salted out at  $-20^\circ$ , added to the upper layer, and the whole dried and distilled. The main fraction (about 60%) consists of propyl, butyl, and amyl alcohols; hexyl, heptyl, and higher alcohols are also formed. The salt solution contains methyl and ethyl alcohols. The products may be used as a source of the corresponding saturated hydrocarbons, aldehydes, etc., or may be used directly as liquid fuels.

A. B. MANNING.

**Preparation of acetone from acetic acid.** CHEM. FABR. VON. HEYDEN A.-G., and R. FEIBELMANN (G.P. 435,347, 12.2.22).—Acetic acid vapour is passed over cast-iron turnings in an iron tube at  $400^\circ$ , giving acetone and carbon dioxide. The acetone should be removed quickly from the tube by means of a slight suction. The coarseness of the catalyst facilitates the passage of the vapours, and so minimises the decomposition of the acid into methane and carbon dioxide. A good yield of acetone is claimed.

C. HOLLINS.

**Manufacture of *o*-aminoarylpropionic acid, its substitution products or homologues.** I. G. FAR-BENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, UND BRÜNING (E.P. 250,250, 31.3.26. Conv., 6.4.25).—Salts of *o*-aminoarylpropionic acids are obtained by hydrolysis of dihydrocarbostyryl or its substitution derivatives with hot alkali. Thus dihydrocarbostyryl itself, when heated for 12 hrs. at  $150^\circ$  with barium hydroxide and water, gives barium *o*-aminophenylpropionate, which may be converted into the sodium salt.

C. HOLLINS.

**Manufacture of halogenohydrins.** C. L. LONG, F. G. WILLSON, and T. S. WHEELER (E.P. 265,259, 2.10.25).—The production of chlorohydrins from olefines and chlorinated water is not affected by concentrations of hydrochloric acid below which the formation of hypochlorous acid is depressed in accordance with the equilibrium  $Cl_2 + H_2O \rightleftharpoons HCl + HOCl$ . Halogenohydrins are produced by circulating water through a number of devices in some of which it is treated with a halogen, whilst in the others (alternately placed) the halogenated liquid is treated with an olefine at  $0-20^\circ$ . These steps are repeated until the concentration of halogenohydrin in the liquid reaches a value above which the formation of hypochlorous acid is depressed by the accumulated hydrochloric acid. This concentration (of chlorohydrin) should not in general much exceed a normal solution, and is continuously maintained by addition of water and withdrawal of treated liquid. Anhydrous halogenohydrins are obtained from the aqueous solution by conversion of the latter into the hydrocarbon oxide by known means, and treatment with dry hydrogen halide.

B. FULLMAN.

**Preparation of halogenated alcohols.** F. NORD (G.P. 434,728, 29.4.24; cf. Meerwein and others, A., 1925, i, 1239).—Halogenated aldehydes or ketones are reduced by aluminium alkoxides in the presence of aldehydes, such as acetaldehyde and benzaldehyde, in



non-aqueous diluents. Thus an equimolecular mixture of chloral and acetaldehyde, treated in absolute ether with aluminium isoamyloxide, gives 72–87% yield of trichloroethyl alcohol; bromal and benzaldehyde give 92% of tribromoethyl alcohol; *as*-dichloroacetone and acetaldehyde give 45% of dichloroisopropyl alcohol.

C. HOLLINS.

**Production of glycol monoethers.** I. G. FARBEN-IND. A.-G., Asses. of BADISCHE ANILIN- u. SODA-FABR. (F.P. 610,282, 30.1.26. Conv., 24.4.25).—Olefine oxides are treated with alcohols or phenols in the presence of a catalyst, *e.g.*, sulphuric acid. Thus by passing ethylene oxide slowly into methyl alcohol containing 1% of sulphuric acid at 15°, glycol monomethyl ether results in 90% yield. The monoethyl ether is similarly prepared.

C. HOLLINS.

**Production of amide acid sulphates from nitriles.** ROESSLER & HASSLACHER CHEMICAL CO. (E.P. 255,886, 22.7.26. Conv., 22.7.25).—Nitriles the radical of which is not affected by the acid used, are partially hydrolysed by dihydrated sulphuric acid ( $H_2SO_4 \cdot H_2O$ ) to the amide acid sulphates. The reaction may be conducted in an inert liquid such as carbon tetrachloride, and is accelerated by the addition of small amounts of organic or inorganic halides such as sodium chloride, aniline hydrochloride, etc.

A. DAVIDSON.

**Production of esters from amide acid sulphates.** ROESSLER & HASSLACHER CHEMICAL CO. (E.P. 255,887, 22.7.26. Conv., 22.7.25).—The sulphates of organic acid amides (cf. preceding abstract) react readily with alcohols (but not phenols) to form esters thus:— $R \cdot CO \cdot NH_2 \cdot H_2SO_4 + R' \cdot OH = R \cdot CO_2 R' + NH_4HSO_4$ . The preparation of ethyl and allyl formates, benzyl acetate, propyl benzoate, glycol mono- and di-formates, and glyceryl monoformate is described. A. DAVIDSON.

**Preparation of esters.** I. G. FARBENIND. A.-G., Assrs. of M. MÜLLER-CUNRADI (G.P. 434,400, 15.2.24).—An alcohol, together with the ester of a second alcohol, is passed in vapour form over a heated catalyst, *e.g.*, alumina. Thus methyl formate and isobutyl alcohol, led over alumina at 150°, give isobutyl formate, b.p. 98°, and methyl alcohol, the conversion proceeding to the extent of 20–30%. From methyl formate and isopropyl alcohol at 200°, 10% of isopropyl formate is obtained; whilst amyl acetate and methyl alcohol, passed over precipitated silicic acid at 250°, give 10–15% of methyl acetate.

C. HOLLINS.

**Solutions of polymerised formaldehyde, free from paraformaldehyde, in mono- or poly-hydric alcohols.** CHEM. FABR. VON HEYDEN A.-G., Assrs. of R. GEBAUER (G.P. 434,830, 6.5.23).—Solutions of paraformaldehyde or trioxymethylene in mono- or poly-hydric alcohols are heated with zinc oxide, alumina, or aluminium hydroxide, giving a *polymeride* of formaldehyde, m.p. 98°. The product is obtained in tallowy form on cooling, but liquefies at about 60°, and is then miscible with most organic solvents. The solutions may be advantageously used for the production of formaldehyde-phenol resins. The highly concentrated paste finds application as a desiccating agent for animal or vegetable preparations and for the removal of water by dialysis from water-sensitive colloidal solutions.

The paste made with a suitable alcohol and an ointment base is absorbed by the skin with extraordinary rapidity.

C. HOLLINS.

**Production of sulpho-aromatic fatty acids.** G. PETROFF (E.P. 252,212, 18.5.26. Conv., 18.5.25. Cf. E.P. 9160 of 1913; B., 1914, 652).—In the preparation of sulphonylated fatty acids for fat-splitting etc., sulphuric acid and aromatic sulphonic acids are removed from the product of sulphonation of a mixture of aromatic hydrocarbons or phenols or turpentine with unsaturated fatty acids by washing with water and sodium chloride solution etc. The residue is dissolved in benzene, carbon tetrachloride, petroleum, etc., and the sulphonyl-fatty acid extracted with aqueous methyl or ethyl alcohol or acetic or formic acid. *E.g.*, a mixture of oleic acid, naphthalene, and benzene is treated with concentrated sulphuric acid, the reaction product mixed with water, and the upper layer, after washing with sodium chloride or sulphate solution, dissolved in a mixture of petroleum and benzene. The solution is washed with 15% sodium sulphate and 5% acetic acid, treated with 10% ethyl alcohol, and kept for 8–10 hrs. at 50–60°. The alcoholic layer, after washing with light petroleum and evaporation, yields the pure sulphonyl-fatty acid.

B. FULLMAN.

**Heavy metal salts of mercaptosulphonic acids.** W. CARPMAEL. FROM CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING) (E.P. 266,824, 5.12.25).—Mercaptosulphonic acids or their soluble salts are treated in aqueous solution with a soluble heavy metal salt such as potassium bromoaurate, mercuric chloride, silver nitrate, bismuth chloride, etc. Examples are: thiophenol-*p*-sulphonic acid (from sulphanilic acid), 3-amino-thiophenol-6-sulphonic acid (from *m*-nitroaniline-6-sulphonic acid) and its formaldehyde-bisulphite compound, 1-mercaptanaphthalene-4-sulphonic acid,  $\beta$ -hydroxy- $\gamma$ -sulphatopropyl mercaptan (from  $\gamma$ -chloro- $\beta$ -hydroxypropyl hydrogen sulphate and sodium hydrogen sulphide), mercaptomethanedisulphonic acid, and 2-mercaptobenzoxazole-5-sulphonic acid (from *o*-aminophenol-*p*-sulphonic acid and potassium thiocyanate). The heavy metal salts of acylated aminomercaptosulphonic acids (E.P. 234,806; B. 1925, 692) are disclaimed.

C. HOLLINS.

**Preparation of salts of chlorourethane.** W. TRAUBE (G.P. 435,529, 8.6.22).—*N*-Chlorourethane,  $NHCl \cdot CO_2Et$ , b.p. 101–102°/30 mm., prepared by chlorination of urethane in aqueous solution at 15°, is cautiously dissolved in *N*-alkali hydroxide. Evaporation yields the alkali salt; addition of silver nitrate solution precipitates the silver salt. *N*-Chloro-*N*-alkylurethanes are obtained easily by the action of alkylating agents on solutions of the alkali salts, *e.g.*, methyl sulphate yields *N*-chloro-*N*-methylurethane. The alkali salts of *N*-chlorourethane have bactericidal, fungicidal, and insecticidal properties.

C. HOLLINS.

**Manufacture of quinolinic [pyridine-2 : 3-dicarboxylic] anhydride.** SOC. OF CHEM. IND. IN BASLE (E.P. 249,497, 2.3.26. Conv., 19.3.25).—Pure pyridine-2 : 3-dicarboxylic anhydride, m.p. 144–145°, in almost quantitative yield, free from pyridine-3-carboxylic acid, is prepared by heating the acid with acetic anhydride below 100°. Small additions of pyridine, hydrochloric



or sulphuric acid, acetyl chloride, a phosphorus halide, or an acid salt catalyse the reaction and lower the temperature necessary.

B. FULLMAN.

**Preparation of zinc compounds of aromatic biguanides.** I. G. FARBENIND. A.-G., Assees. of K. SCHRANZ (G.P. 435,668, 11.9.24).—Arylbiguanides react with zinc oxide or hydroxide at the ordinary temperature, the former in the presence of water or alcohol, the latter by mere mixing, forming zinc salts which are vulcanisation accelerators. Examples are the zinc salts of  $\alpha$ -phenyl-, o- and p-tolyl-biguanides, m.p. 195–196°, 182–183°, and 196–197°, respectively.

C. HOLLINS.

**Production of pure mercury vapour for inhalation purposes [from cinnabar, mercuric sulphide, etc.].** R. SPUHL (G.P. 435,689, 12.10.24).—The mercury salts are mixed with substances which, in presence of air, combine with the sulphur without the formation of volatile sulphur oxides. *E.g.*, 1 pt. of mercuric sulphide is mixed with 4 pts. of magnesium oxide, 2 pts. of sodium silicate, and 3 pts. of activated iron and the mixture formed into tablets. On heating these, pure mercury vapour is produced. The iron accelerates the reaction.

E. H. SHARPLES.

**[Magnesium] salt of a quinolinecarboxylic acid.** M. L. CROSSLEY, Asst. to CALCO CHEMICAL CO. (U.S.P. 1,618,172, 22.2.27. Appl., 20.9.24).—*Magnesium 2-phenylquinoline-4-carboxylate* is of therapeutic value in the treatment of rheumatism.

T. S. WHEELER.

**Manufacture of organic auro-mercapto-acids and salts thereof.** W. CARPMAEL. From CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING) (E.P. 265,777, 15.2.26).—Auro-mercapto-acids and their salts are prepared by the action of auric salts on mercapto-acids or their salts in the presence of sulphurous acid or a sulphite. Using 1 mol. of sulphur dioxide per mol. of auric salt the mercapto-compounds are completely used up in the formation of auromercapto-compound, no by-product being formed. Examples are given of the preparation of sodium  $\gamma$ -auromercaptoglycerolsulphonate from the mercapto-compound and potassium bromoaurate and sulphurous acid or sodium hydrogen sulphite; and of the preparation of sodium *p*-auromercaptobenzenesulphonate and 4-amino-2-auromercaptobenzoic acid.

B. FULLMAN.

**Manufacture of basic bismuth salts of arylarsinic acids.** R. W. E. STICKINGS, and MAY & BAKER, LTD. (E.P. 266,820, 5.12.25).—For the precipitation of basic bismuth salts in good yield and in a form suitable for purification, an alkali bismuthyl tartrate is heated with considerable excess of an arylarsinate in aqueous solution. The precipitate should be filtered off without delay in order to prevent gel formation. The products combine the therapeutic properties of bismuth and arsenic compounds, and have low toxicity.

C. HOLLINS.

**Production of concentrated acetic acid from dilute acid.** H. SUIDA (U.S.P. 1,621,441, 15.3.27. Appl., 27.2.25. Conv., 8.3.24).—See E.P. 230,447; B., 1925, 827.

**Manufacture of organic lead compounds.**

T. MIDGLEY, JUN., Asst. to GENERAL MOTORS CORP. (U.S.P. 1,622,228, 22.3.27. Appl., 19.5.23).—See E.P. 216,083; B., 1925, 299.

**Manufacture of barbituric acid.** F. BOEDECKER. Asst. to J. D. RIEDEL A.-G. (U.S.P. 1,622,129, 22.3.27. Appl., 23.9.24. Conv., 10.10.23).—See E.P. 223,221; B., 1925, 970.

**Citric acid** (G.P. 434,729 and E.P. 266,414–5).—See XVIII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Turbidimetry and grain size.** F. F. RENWICK (Phot. J., 1927, 51, 185–193).—The properties of turbid media are considered in general, and a turbidimeter is described which determines the thickness of a medium which will just obscure a given test object. The instrument consists of a graticule immersed diagonally across a cell filled with the turbid liquid to be examined, and illuminated from the rear by diffuse light. The position of the last clear line of the graticule that can be resolved is measured and is used as a basis for calculating the obscuring thickness. Using this instrument, it is shown that the thickness of the layer of a turbid liquid which will just obscure the test pattern is inversely proportional to its concentration, or directly proportional to the volume containing unit mass of the suspended material. In the case of a silver bromide emulsion, the slope of the line connecting obscuring thickness with the volume of suspension containing unit mass of silver bromide varies over a wide range with change of size of the emulsion grains. Slow emulsions of intermediate grain size have the maximum covering power. The specific turbidity of a suspension is defined as the maximum area of a given contrast-pattern which can just be obscured by 1 g. of the suspension. If two emulsions of differing average grain characteristics are compared in the manner described, they differ considerably in their specific turbidities. Suspensions adjusted in dilution to have equal obscuring thicknesses have the same obscuring thickness after admixture, no matter how widely they differ in specific turbidity, or in what proportions they are mixed. Formulæ are given for determining the obscuring thickness of mixtures with known individual values, and for the calculation of the specific turbidity of a mixture of two different grain types, if the connexion between specific turbidity and grain size were known. There is a marked difference between the obscuring power of a gelatin-silver bromide layer in the wet and dry state, and the ratio of their turbidities varies greatly with grain size. The descending order of turbidity ratio does not strictly correspond with that of diminishing speed, and it is not certain whether it corresponds with their average grain sizes. A second turbidimeter is described, the principle of which depends on the use of a comparison wedge of diffusing medium such as a gelatin wedge of bismuth oxychloride or silver iodide.

W. CLARK.

### PATENTS.

**Production of photographic plates, films, and the like for indirect three-colour photography,**



E. A. LAGE (E.P. 266,468, 28.11.25).—A film, coated with emulsion suitable for the red partial image, is squeezed on to a support provided with a coating of emulsion suitable for the blue partial image, the emulsion surfaces being in contact, after which a coating of emulsion suitable for the yellow partial image is applied. After exposure, the film, together with the final coating, is stripped, and the two parts are developed separately, and dried. One or both negatives are then stripped from the film. In this way three partial negatives are obtained for printing. Alternatively, one or other of the partial images on the film which has been separated from the support is rendered translucent by bleaching, the unbleached negative is printed, and the bleached image is then redeveloped. W. CLARK.

**Photographic prints in colours.** G. P. J. SCHWEITZER (E.P. 249,530, 17.3.26. Conv., 18.3.25).—Coloured photographic images are produced by dyeing a plate carrying a silver image, and then attacking the silver with solutions capable of freeing, in this attack, a reagent which bleaches the dye by oxidation. A solution containing sodium bromate and sulphuric acid is suitable. Alternatively, a solution capable of reacting with silver halide to free a reagent which bleaches the dye is used. Such a solution contains iodic acid, sulphuric acid, and iodine, and it is used on a developed but unfixed plate. The dye is bleached according to the amount of silver or silver halide present. W. CLARK.

## XXII.—EXPLOSIVES; MATCHES.

**Propagation of detonation across an air-gap between two cartridges of explosive.** G. ST. J. PERROTT and D. B. GAWTHROP (J. Franklin Inst., 1927, 203, 387—406).—Experiments with a Mettegang recorder show for explosives detonating at about 1700 m./sec. that the velocity of propagation across an air-gap between two cartridges is at first much higher than the rate of detonation of the explosive and falls off regularly until the second cartridge fails to detonate. When the rate is about 3000 m./sec. the velocity across the air-gap is about the same over an interval of  $4\frac{1}{2}$ — $7\frac{1}{2}$  in. from the end of the initiating cartridge. With a rate of detonation as high as 4800 m./sec. the velocity in the air-gap falls off linearly as the gap is increased from  $1\frac{1}{2}$  to 27 in. The decrease is slow from 27 to 48 in., and the second cartridge will not always detonate at 48 in. Explosives containing ethylene glycol dinitrate propagate across a greater air-gap than do similar explosives containing nitropolyglycerin, and maintain a higher average velocity across the air-gap. Photographs on a moving film confirm the above results. When the explosives are confined in glass tubes the speed of the disturbance is considerably greater than the rate of detonation of the explosive, and is maintained for a longer distance. A luminous phenomenon is propagated through the air-gap, and is in some way related to the disturbance effecting detonation in the second cartridge. R. A. MORTON.

### PATENT.

**Explosive.** A. SEGAY (U.S.P. 1,620,696, 15.3.27. Appl., 30.4.23. Conv., 26.5.22).—See E.P. 201,791; B., 1923, 999 A.

## XXIII.—SANITATION; WATER PURIFICATION.

**Hufschmidt's process for regenerating permutit filters.** K. HOFER (Glückauf, 1926, 62, 1396—1398; Chem. Zentr., 1926, II, 2993).—The addition to an existing permutit plant of rinsing tubes and a pump for circulating the regenerating solution resulted in a considerable saving of sodium chloride and decreased the time of regeneration from 9 hrs. to  $\frac{1}{2}$  hr., whilst the addition of a gravel filter and a froth trap reduced the loss of permutit to practically nil. L. A. COLES.

### PATENTS.

**Apparatus for preparing lime water for use in water-purifying plants.** H. MORGENSTERN and W. HAGEN (G.P. 435,722, 13.1.25).—Water is added to lime suspended at the bottom of a funnel-shaped vessel, through a pipe which passes downwards through the liquor and then bends round and is connected with a length of wider upright tube, a suction pipe dipping into the suspension being attached at the point of connexion of the two tubes. L. A. COLES.

**Filtration process.** H. W. BLAISDELL, Assr. to BLAISDELL FILTRATION Co. (U.S.P. 1,614,947, 18.1.27. Appl., 12.12.21).—At intervals between the cleanings of a filter, a reverse flow is allowed at a low rate insufficient to remove sediment on the bed, but sufficient to cause relative re-arrangement of the material and sediment. W. T. LOCKETT.

**Reconditioning zeolite water softeners.** W. J. KENNEY, Assr. to ZEOLITE ENGINEERING Co. (U.S.P. 1,617,288, 8.2.27. Appl., 16.3.25).—To facilitate the reconditioning of zeolite water softeners means are provided to cause a portion of the liquid discharged from the treating reservoir during regeneration to be passed through a salt pot and recirculated through the reservoir without placing the interior of the salt pot either under the pressure of the dispensing system or that of the water supply main. W. T. LOCKETT.

**Improving the odour of phenols and hydrocarbons used for disinfectants.** ZEICHE M. STINNES (G.P. 435,692, 23.10.24).—To the phenols or hydrocarbons, with or without the addition of solutions of soap or other emulsifying agents, are added ethers or esters in sufficient amount to mask the odour of the disinfectant, the bactericidal power of which is thereby unaffected. E. H. SHARPLES.

**Germicidal agent.** I. G. FARBERNIND A.-G., Assees. of M. MÜLLER-CUNRADI and K. VIERLING (G.P. 435,755, 4.3.24).—A mixture of alcohol and a butyl alcohol such as, e.g., *n*- and *iso*-butyl alcohol, suitably diluted with water, is used. E. H. SHARPLES.

**Disposal of sewage.** C. P. DERLETH, Assr. to CELITE Co. (U.S.P. 1,617,014, 8.2.27. Appl., 7.2.22).—In the activated sludge sewage disposal processes to facilitate the purification of the sewage, the settlement of the sludge in the settlement tanks, and the filtration of the surplus sludge in filter presses and the like, a quantity of diatomaceous earth is added to the sewage or the mixed liquor prior to aeration. W. T. LOCKETT.

**Disinfectant composition.** G. G. GORDON (U.S.P. 1,619,861, 8.3.27. Appl., 17.12.26. Conv., 29.6.25).—See E.P. 258,678; B., 1926, 998.