

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

SEPT. 26 and OCT. 3, 1930.\*

### I.—GENERAL; PLANT; MACHINERY.

**Spray dryers.** W. KUHLES (Chem. Fabr., 1930, 293—296).—Dryers or evaporators using an atomised spray of liquid or solution are an extension of the principle of film evaporation. The temperature of the air used can be reduced owing to the large surface of contact. The most suitable arrangement has a vertical spray rising to meet a baffle-plate, from which it falls outwards against a countercurrent of heated air passing upwards in an annular exterior zone of the drying chamber. Such an air current has no eddies. Concrete is better than wrought iron as constructional material on account of its lower heat conductivity. In many cases the fine state of division of the product of such a dryer is an advantage. C. IRWIN.

**Adsorption of gases and vapours on active charcoal and recovery of the adsorbed products.** A. FOULON (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 235—237, 281—283).

**Oil firing.** HIND.—See VIII.

### PATENTS.

**Muffle furnace and method of operating same.** W. W. KEMP, Assr. to C. M. KEMP MANUF. CO. (U.S.P. 1,752,800, 1.4.30. Appl., 29.8.27).—The combustion gases are prevented from entering the muffle by pumping air into it, so that the pressure inside the muffle is always slightly higher than that in the combustion chamber. The air is preheated by the waste combustion gases. D. K. MOORE.

**Gas-fired furnaces.** WELLMAN SMITH OWEN ENG. CORP., LTD., and H. W. SOWARD (B.P. 331,635, 9.5.29).—The gas is admitted through independently controlled inlets in the side and one of the end walls and the air downwards through the roof in the form of a number of small jets. Recuperator passages for the air and waste gases are also arranged in the roof structure. B. M. VENABLES.

**Calcining apparatus.** F. GELSTHARP, Assr. to PITTSBURGH PLATE GLASS CO. (U.S.P. 1,754,854, 15.4.30. Appl., 17.5.28).—A rotary, inclined, cylindrical kiln is described for the calcination of green copperas. Among other details, a stationary muller is provided at an intermediate point in the kiln to break up lumps. B. M. VENABLES.

**Drying kilns.** INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 332,491, 2.11.29. Ger., 3.11.28).—Automatic electric controls are described for a drying kiln, used, *e.g.*, for lacquered articles, which must have doors closed during the periodical stoppages of the conveyor. B. M. VENABLES.

**Drying ovens.** S. L. GROOM (B.P. 332,067, 25.7.29).—A conveyor for carrying plate-like objects through a tunnel dryer is arranged so that both runs are effective, provision being made to charge articles at both ends simultaneously, on the top and bottom runs respectively. Hooked carriers may be attached to the conveyor chains, the hooks supporting the articles on the lower run and the conveyor itself on the upper.

B. M. VENABLES.

**Spray-drying apparatus.** W. S. BOWEN (U.S.P. 1,753,915, 8.4.30. Appl., 5.6.29).—The liquid is sprayed centrally in the upper part of the drying chamber or tower and the air is admitted through a false ceiling which is comparatively thick and is provided with apertures situated radially but inclined to the axis of the vessel, the thickness of the roof being necessary to give sufficient tangential motion to the incoming air. B. M. VENABLES.

**[Indirect] heating or cooling of [divided] solid substances.** N. AHLMANN (B.P. 332,226, 16.4.29).—The pulverulent solid substantially fills a container and is subjected to heating or cooling through the walls of the vessel and by means of a stirrer which may be, *e.g.*, in the form of a zigzag tube; an upward current of air or gas is provided to keep the material loose without producing whirling. The heating may be used to bring about chemical reactions between more than one solid substance. B. M. VENABLES.

**Heat-exchange apparatus.** A. D. HARRISON (U.S.P. 1,754,857, 15.4.30. Appl., 10.4.29).—A number of plates, alternately plain and ribbed on both sides to form sinuous passages, are assembled in a press. Packing is applied round the circumference only of the plates, and leakage from around the transfer ports is led out of the press by guard grooves leading to ports. When used for the pasteurisation of milk, the raw milk will first exchange heat with the treated milk in one section of the press and then be further heated by steam or other hot fluid in another section. B. M. VENABLES.

**Heat-exchange device.** G. KRÄNZLEIN, and SAMESREUTHER & Co. G.M.B.H. (B.P. 331,910, 9.4.29. Ger., 10.4.28. Addn. to B.P. 309,445; B., 1930, 798).—The heat exchanger is modified so that electrical heating elements can be placed in the pipes. The pipes are enclosed in two flanged plates and are in thermal contact with them. D. K. MOORE.

(A, C) **Rotary pivoted hammers [for pulverisers].**  
(B) **Mill pulveriser.** J. P. POLSTER, Assr. to ILLINOIS STOKER CO. (U.S.P. 1,753,473—5, 8.4.30. Appl., [A] 14.3.28, [B] 2.5.29, [C] 5.8.29).—In (A) and (C) stirrup-shaped swing hammers for pulverisers are described.

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



In (B) a pulveriser is described having two end-zones mainly for crushing, the beaters of which deflect the material into the centre pulverising zone, which is surrounded by a continuous bull-ring; both inlet and outlet of material is effected through the same openings near the ends, and air inlets may be provided through the ends of the machiné. B. M. VENABLES.

**Pulverising or grinding mill.** D. J. UHLE and J. V. DURNIN (U.S.P. 1,752,314 and 1,752,622—3, 1.4.30. Appl., 22.11., 30.3., and 22.11.28).—Balls are rotated by pusher elements attached by yielding supports to arms on the shaft. This avoids undue strain. By making these pushers rotatable, scoring and wear of the balls are reduced. D. K. MOORE.

**Pendulum roll mill.** F. L. BUCHANAN (U.S.P. 1,754,414, 15.4.30. Appl., 24.9.28).—A rotating drum is provided with liner plates and hollow trunnions, and through the latter and right through the mill extends a bar, which may be hollow and used to supply water, carrying grinding rollers in stirrup-shaped supports. The feed is introduced through separate conduits and screened discharge openings are formed in the ends or central part of the circumference. The mill may be operated wet or dry. B. M. VENABLES.

**Grinding or crushing mills.** G. H. ROBINSON (B.P. 331,877, 10.4.29).—The grinding rolls are fitted in a carriage attached by hinged joint to a carrier fixed to the shaft. Springs behind the carriages force the rolls against the bull-ring. A cone distributor is used for feeding the machine. A suitably shaped plough lifts coarse material from the lower part of the machine back to the grinding rolls. D. K. MOORE.

**Gyratory crushers.** W. W. TRIGGS. From TRAYLOR ENG. & MANUF. CO. (B.P. 332,348, 6.6.29).—A form of seal to prevent dust reaching the mechanism of such crushers is described. B. M. VENABLES.

**Mixing apparatus.** BRIT. CELANESE, LTD. From CELANESE CORP. OF AMERICA (B.P. 331,606, 15.4.29).—A jacketed vessel is provided with downwardly depending fixed stirrers and with moving ones upstanding from a rotating arm, the distance of which from the bottom of the vessel may be varied. The vessel may have the inner shell of chemically resistant material, and may be provided with a lid having inlet and outlet openings, and with a draw-off valve of the type that opens by spring action and is closed manually. B. M. VENABLES.

**Agitation and settling process.** C. P. HOOVER and F. A. DOWNES (U.S.P. 1,752,795, 1.4.30. Appl., 7.8.25).—The top part of a cylindrical vessel is fitted with agitators and serves as a chamber in which the precipitate, by a suitable combination of agitator speed and time factor, is transformed into the best condition for settling. The lower part is made up of a number of superimposed settling compartments with inverted cone-shaped bottoms having central discharge for the sediment, and outlet pipes for the clarified liquor. D. K. MOORE.

**Agitation and settling apparatus.** [Water softener.] F. A. DOWNES, Assr. to DORR CO. (U.S.P. 1,752,789, 1.4.30. Appl., 7.8.25).—The apparatus is

suitable for water softening or other purposes where the weight of solids is small compared with the volume of liquid and the flocculation has to be carefully controlled. It comprises an agitation chamber superposed upon a number of settling chambers. In the former rather more violent agitation may be given in the earlier stages by spacing the stirrers closer in the upper part than in the lower; the settling chambers have a common central feed passage which also forms the outlet for settled sludge to the next chamber below. Of these stirrers only the lowest has a spigot or pump discharge for the sludge, but each has a conical bottom and set of rakes and immediately under the circumference of each bottom is a ring-pipe for clear overflow. The overflow from each compartment is separately controllable by valves or by small adjustment of the height of the ends of the overflow pipes, and the combined overflow may be passed upwards through a filter to a storage compartment situated above the rest of the apparatus; provision is also made for returning sludge to the feed to aid settling. B. M. VENABLES.

**[Shaking-tables for] separation of dry materials.** BIRTLEY IRON CO., LTD., and (A) C. W. H. HOLMES and I. L. BRAMWELL, (B) (HON.) B. L. BOURKE (B.P. 332,291 and 332,583, [A] 27.5.29, [B] 23.1.29).—Shaking-tables with decks pervious to air currents are described, the discharge of material being entirely over one edge. B. M. VENABLES.

**Apparatus for effecting contact between gases and divided solids.** HUMPHREYS & GLASGOW, LTD., Assees. of E. J. BRADY (B.P. 315,868, 10.7.29. U.S., 21.7.28).—The divided solid is allowed to fall through an annular space between two rotating, perforated walls and the gas passes zigzag across the space, the general flow being upward or countercurrent. The perforations slope downwardly towards the annular space and may be in the form of louvres, fingers or wires may be placed in the annular space, the baffles which cause the zigzag passes may be flushed by a liquid, and means may be provided by which the quality of the outgoing gas determines the rate of treatment. *E.g.*, in the removal of hydrogen sulphide from coal gas by iron oxide, a small burning jet of the gas, a ribbon of lead acetate paper, and a photo-electric cell may be used. B. M. VENABLES.

**[Centrifugal] separation of mixtures.** L. P. SHARPLES, Assr. to SHARPLES SPECIALTY CO. (U.S.P. 1,754,774, 15.4.30. Appl., 16.11.27).—Two centrifuges of different types are used in conjunction to separate a mixture of solid and liquid: the first delivers a clear liquid and a sludge, the second delivers dried solid (from the sludge) and a muddy liquid which is returned as original feed. B. M. VENABLES.

**[Centrifugal] extractor.** A. PARTLOW, Assr. to VORCLONE CORP. (U.S.P. 1,753,215, 8.4.30. Appl., 31.3.28).—An under-driven extractor is arranged so that the basket can be easily lifted right out of the casing and, when out, the bottom can be completely opened by swinging two half-doors, one of which carries the female part of a driving boss, the male part remaining on the shaft. B. M. VENABLES.

**Centrifugal machines.** R. M. BERLINE (B.P. 311,248, 7.5.29. Luxembourg, 7.5.28).—A centrifugal



machine for sedimentation is long in comparison with its diameter and is divided into two zones: the first for a preliminary separation without plates, and the second having closely spaced cones or plates producing a more intense effect. B. M. VENABLES.

**Centrifugal machines.** J. L. RUSHTON (B.P. 332,288, 18.4.29).—A self-contained system of forced lubrication for such machines suitable, *e.g.*, for spinning rayon, is described. B. M. VENABLES.

**[Sedimentation and] filtration apparatus [for solid-laden liquids].** C. T. CABRERA, Assr. to ELECTRO DIALYZER CORP. (U.S.P. 1,754,667, 15.4.30. Appl., 26.7.28).—The fluid, such as sewage, is admitted well down on the axis of a cylindrical tank provided with a conical bottom and with rakes to discharge settled sludge; the upper wall of the tank is formed of permeable material through which the partly clarified liquid overflows. These filter members are supported in frames in sections and are double everywhere so that they can be removed section by section for cleaning without stopping the action; the necessity for cleaning is postponed because when the lower part of the filters becomes choked the level of the liquid will rise and pass through a clean, higher surface. There is also no possibility of choked filters causing a material rise of pressure. B. M. VENABLES.

**Filter.** J. T. SHIMMIN (U.S.P. 1,752,758, 1.4.30. Appl., 4.5.25).—In a filter of the continuous rotary-drum type with the filtering medium fastened to the inside of the drum, vacuum assisted by gravity is used to effect filtration and air pressure and gravity to discharge the filter cake. D. K. MOORE.

**Filter.** A. HOPKINS, Assr. to HOPKINS-TULL MACHINERY CO. (U.S.P. 1,754,320, 15.4.30. Appl., 20.12.28).—A number of semi-circular bags are contained in the annular space between two cylindrical walls, the feed entering the bags through a distributing manifold and the filtrate leaving the annular space upwardly. B. M. VENABLES.

**Tipping filter press.** P. W. PRUTZMAN and V. C. BENJAMIN, Assrs. to CONTACT FILTRATION CO. (U.S.P. 1,754,401, 15.4.30. Appl., 4.1.28).—The filter is of the recessed-plate type with filter medium on the lower side only of the cells. The axis of the pile is vertical when in operation, but horizontal for cleaning. B. M. VENABLES.

**Filter-cake loop dryer. Loop dryer [for fabrics].** A. O. HURXTHAL, Assr. to PROCTOR & SCHWARTZ, INC. (U.S.P. 1,755,005—6, 15.4.30. Appl., [A] 24.4.26, [B] 3.7.28).—(A) The dryer is suitable for material that will adhere to a foraminous belt that unwinds from a drum filter or other means of applying the material in a thin layer. The filter drum, belt, and a discharging device are driven by separate electric motors. While in the dryer the belt forms deep U-shaped loops hanging over cross-bars on a link conveyor and at the dry end the loops are straightened out and the dry material is beaten off. In (B) a dryer of similar construction to that described in (A) is adapted to web materials, such as cloth or paper, and a device is added which serves to steady the loops against air currents. B. M. VENABLES.

(A) Process, (B) machine, for treating materials. C. W. VOGT, and VOGT INSTANT FREEZERS, INC. (B.P. 332,577 and 332,593, 23.4.29).—(A) Materials, *e.g.*, ice-cream or edible oils, are subjected to pressure, stirring, heating, or cooling, and, if desired, injection of gas, simultaneously, so as to produce a plastic compound of smooth texture. The resistance to flow or to agitation, being a measure of the plasticity, is caused to regulate the supply of heat-exchanging medium (coarse adjustment) and/or the supply of the material (fine adjustment); usually the greater the resistance the larger is the feed to prevent overfreezing. (B) A machine for above purposes comprises a number of tubular chambers with elongated internal stirrers and external spiral passages for heat-exchanging medium; different media or different quantities of the same medium may be passed round different tubes. External means are provided for producing the pressure. B. M. VENABLES.

**Apparatus for making emulsions or dispersions.** IMPERIAL CHEM. INDUSTRIES, LTD., and R. B. F. F. CLARKE (B.P. 331,928, 13.4.29).—The constituents are sprayed through at least two nozzles so as to impinge on each other or on a common surface; at least one of the nozzles is multiple so that a motive fluid such as steam or compressed air may be used, and partly mixed materials are lifted back into the spray. For example, linseed oil may be sprayed by steam and ammonium oleate solution simultaneously by compressed air. B. M. VENABLES.

**Preparation of emulsions, suspensions, and colloidal dispersions.** P. FREEDMAN (B.P. 332,533, 15.4.29).—The mixture is subjected to vibration by means of piezo-electric crystals energised by electric pulsations, which either may be in direct contact or may transmit their vibrations through another hydraulic medium and flexible dividing wall. B. M. VENABLES.

**Apparatus for production of chemical compounds.** M. PÓLÁNYI and S. VON BOGDANDY (B.P. 303,900, 11.1.29. Ger., 12.1.28).—In order to effect reactions between a liquid or dissolved substance, which cannot be unduly heated, and a gas or vapour, which may have to be heated to produce volatilisation, the liquid is spread in a thin layer on the interior of centrifugal bowl and surrounding container, being kept in circulation by a pump forming part of the centrifuge, while the other constituent is fed to the interior of the bowl and, if necessary, volatilised there by an electric arc or other means. B. M. VENABLES.

**Apparatus for bringing liquids and gas and/or vapours into intimate contact.** S. G. WATSON, D. M. HENSHAW, and W. C. HOLMES & CO., LTD. (B.P. 331,592, 3.4.29).—The apparatus is of the type in which brushes are rotated alternately with fixed partitions in a casing, the liquor being distributed independently of the brushes by a pump for each compartment with suitable multiple outlet. B. M. VENABLES.

**Lining vessels for carrying out operations with liquids and the like.** IMPERIAL CHEM. INDUSTRIES, LTD., and F. H. BRAMWELL (B.P. 331,838, 9.1.29).—A vessel to which an incompressible lining, *e.g.*, of rubber or lead, is to be applied is made with a double (corrodible,



shell, the inner one being perforated, and after the lining is applied a reduced pressure is maintained in the space between the shells, thus preventing creep of the lining.

B. M. VENABLES.

**Evaporating method and means therefor.** INDUSTRIKEMISKA AKTIEB. (B.P. 312,089, 17.5.29. Swed., 18.5.28).—The liquid to be evaporated or solid to be dried is sprayed into a series of evaporating chambers in the presence of air. This air passes through the chambers in series and has its temperature raised step by step so that it takes up more and more moisture. At the end of the series it is further heated by external means such as a steam-coil, additional water being added if desired, and then passes the reverse way through the heating chambers in conducting relation with the evaporators, thus giving up the latent heat of the moisture and causing a corresponding evaporation. Preferably the pressure of the operations is maintained at slightly above or below atmospheric. In some cases, while the evaporating chambers remain in series, some of the heating chambers may be connected in parallel.

B. M. VENABLES.

**Rotary apparatus for cooling liquids.** R. ZWERINA (B.P. 331,812, 5.3.29. Austr., 14.2.29).—The liquid is cooled by flowing through passages in the vanes of a fan. Deflector plates reduce eddying when entering and leaving the vanes.

D. K. MOORE.

**De-aerating water at atmospheric pressure.** W. BOBY, and W. BOBY & Co., Ltd. (B.P. 332,678, 23.5.29).—The water flows countercurrent to live and/or exhaust steam through a series of compartments, being caused to flow under and over baffles and to cascade from compartment to compartment. The steam is regulated so that only enough to raise the water to the b.p. is admitted.

B. M. VENABLES.

**Apparatus for degasifying liquids.** SOC. ITAL. PIRELLI, and L. EMANUELI (B.P. 332,542, 19.4.29).—The liquid, such as insulating oil, is sprayed, after heating if necessary, into a vacuum chamber and discharged therefrom preferably at an increased pressure. A suitable form of spray is described.

B. M. VENABLES.

**Gas-washing apparatus.** PNEUMATIC CONVEYANCE & EXTRACTION, LTD., and W. A. SMITH (B.P. 332,283, 18.4.29).—The gas is subjected to liquid sprays while passing through louver-like baffles; the dust is removed from the apparatus while still in suspension in the liquid, for which purpose the bottom of the apparatus is conical and flushed by sprays.

B. M. VENABLES.

**Filters for gases.** H. WITTEMEIER, Assee. of E. H. SCHÄFER (B.P. 331,754, 7.9.29. Ger., 27.10.28).—A filter composed of a stack of metal plates or grids is arranged so that the plates may be slid out for cleaning one group at a time; a spring holds the remainder of the pack up against the abutment at the discharge side.

B. M. VENABLES.

**Air filters.** I. L. BRAMWELL, C. W. H. HOLMES, and BIRTLEY IRON CO., LTD. (B.P. 332,601, 23.1.29).—A system comprising a number of groups of bag-filters is maintained under reduced pressure by an induced-draught fan, even the inlets of the filters being under slight vacuum; to clean a group by reverse current

without stopping the operation of the others it is necessary only to cut off that group from the fan and open the clean or outlet side to atmosphere.

B. M. VENABLES.

**Separator of liquids and gases, intended particularly for use in gas-compressing plants.** H. W. WILDISH (B.P. 331,704, 18.7.29).—The separator is of the fixed type and has inclined baffles with the outlet above them.

B. M. VENABLES.

**Apparatus and method for testing gases.** R. B. WASSON (U.S.P. 1,753,675, 8.4.30. Appl., 24.10.25).—The sample of gas is held at a constant volume before, during, and after the test and the reduction in pressure due to the absorption of a constituent is determined. The apparatus comprises two concentric chambers, a pump, manometer, and the necessary multi-way cocks. In determining for instance, the carbon dioxide in combustion gases, the inner chamber is completely emptied of gas by pumping caustic liquor covered with a layer of oil into it from the outer chamber; the sample of gas is then drawn into the inner chamber at atmospheric pressure (independent of the pressure of supply) by simply allowing the liquid level to equalise in the inner and outer chambers. The inner chamber is then isolated and the pump used to draw liquid from the inner chamber and deliver it back in the form of a spray which will break through the oil film and absorb the carbon dioxide.

B. M. VENABLES.

**Treatment of fluids with solid substances.** T. T. GRAY, Assr. to GRAY PROCESSES CORP. (U.S.P. 1,753,859, 8.4.30. Appl., 18.1.26).—Fluids such as petroleum products are treated with solid substances which are supported in separate containers within a shell, either above or below the level of the liquid, thus preventing the solid reagents from becoming diffused through the petroleum or mixing with each other. Provision is made for easily handling the containers through a man-hole.

B. M. VENABLES.

**Apparatus for bringing liquids and gas or vapours and gas into intimate contact.** S. G. WATSON, D. M. HENSHAW, and W. C. HOLMES & Co. (B.P. 331,818, 3.4.29).—The apparatus consists of a series of compartments each fitted with stationary brushes through which the liquid and gas pass.

D. K. MOORE.

**Transferring liquefied gases from one container to another.** C. W. P. HEYLANDT (U.S.P. 1,753,785, 8.4.30. Appl., 8.3.29. Ger., 2.3.28).—In the transport or bulk-storage vessel, the liquid is maintained at about its b.p.; when it is required to force liquid out, a small portion is allowed to pass through an evaporator coil and the vapour readmitted to the upper part of the tank.

B. M. VENABLES.

**Control of proportions of components of gaseous mixtures.** W. R. SCHOFIELD, JUN., and R. D. MILNER, Assrs. to LEEDS & NORTHRUP CO. (U.S.P. 1,753,319, 8.4.30. Appl., 7.3.24).—A change in composition and therefore of thermal conductivity of a sample of gas is caused to unbalance a Wheatstone bridge of which two arms are formed of wires (carrying heating currents) having a high temperature coefficient situated in the sample and a standard gas, respectively. The movement of the



galvanometer of the bridge is translated to control, e.g., a valve or damper by known means. The sample and standard gas are situated in adjacent cells and the former is changed continuously by convection only, both the inlet and outlet pipes to the cell being arranged at the same cross-section of the sample pipe and perpendicular to the flow of gas in it. B. M. VENABLES.

**Heating of fluids.** C. W. STRATFORD (B.P. 305,205, 18.1.29. U.S., 2.2.28).—In a still or boiler having a stationary, internal, cylindrical heating surface, the liquid is projected against the heating surface by jets which are evenly spaced and are submerged in the body of the liquid. A portion of the liquid is withdrawn by a pump and continuously circulated through the jets.

B. M. VENABLES.

**[Fractionating] still. Scrubber.** J. A. CAMPBELL (U.S.P. 1,754,605 and 1,754,607, 15.4.30. Appl., [A] 19.4.27, [B] 5.11.28).—In (A) a fractionating column is fed at an intermediate height through an axial column pipe, the trays are perforated, and the downcomers are in the form of sharp-edged froth-breaking trays raised above the plates and provided with pipes leading to near the surface of the plate below. The froth collectors below the feed inlet are preferably larger than those above it. In (B), prior to entering a gasoline absorption plant, natural gas is cleaned by selective absorption in a tower provided with perforated conical trays above which are sharp-edged froth-breaking collectors or trays. It is intended that the crude oil, water, and solid impurities should run down the conical trays, and drains are provided from their apices direct to a separate discharge chamber at the bottom of the tower. The lighter absorption liquid which rapidly becomes saturated with the petrol vapour should form froth and, after being caught in the collectors, run down only one stage at a time to a sump, thence to be pumped either wholly to the top tray or partly to the others.

B. M. VENABLES.

**Gaseous exothermal catalyses.** O. PIETTE, and UNION CHIM. BELGE, SOC. ANON. (B.P. 332,532, 12.4.29).—A number of twin concentric tubes (the lower ends of the larger tubes having closed ends) are buried in the catalyst mass and the gas before the reaction is passed downwardly through the annular spaces, upwardly through the inner tubes, then through the catalyst downwardly, so that the flow of cold gas and reacting gas is concurrent and the temperature of the catalyst more uniform.

B. M. VENABLES.

**Solidification of carbon dioxide or other gases.** H. W. COLE and M. W. McLAREN (B.P. 312,231, 13.5.29 U.S., 22.5.28).—An apparatus is described by which liquid carbon dioxide (or other gas) may be expanded in an entirely closed chamber under carefully controlled pressure and temperature and the snow compressed in that chamber or in an adjacent ram cylinder, so that hard blocks of solid may be obtained. The pressure incidental to the original compression may be used to work the hydraulic ram.

B. M. VENABLES.

**Hygrometers.** W. LUBACH and E. GIESER (B.P. 332,135, 1.10.29).—The hygroscopic element of an electric hygrometer is made of glass, earthenware, or

other material with a glassy surface. An ammeter or electric-discharge lamp may be used as the measuring instrument.

D. K. MOORE.

**Process for extinguishing fires.** O. TREICHEL (U.S.P. 1,771,151, 22.7.30. Appl., 31.10.27. Ger., 15.11.26).—See B.P. 280,543; B., 1928, 628.

**Filter press.** H. JUNG (U.S.P. 1,771,928, 29.7.30. Appl., 1.12.28. Ger., 2.5.27).—See B.P. 322,091; B., 1930, 126.

**Distillation or like columns.** A. KUHN, ASSR. to C. STILL (U.S.P. 1,772,399, 5.8.30. Appl., 15.3.26. U.K., 24.3.25).—See B.P. 239,791; B., 1925, 869.

**Production of a cooling liquid of low f.p.** F. WERNER (U.S.P. 1,771,517, 29.7.30. Appl., 3.11.25. Ger., 23.9.25).—See B.P. 260,760; B., 1927, 65.

**Apparatus for washing, separating, or concentrating minerals and other granular materials.** J. BRASS (B.P. 332,682, 27.5.29).

**Pasteurising apparatus.** P. N. GRØNBORG (B.P. 319,742, 24.9.29. Denm., 27.9.28).

**Tanks for receiving and separating mixed solid and liquid materials.** ASH CO. (LONDON), LTD. (B.P. 311,816, 26.4.29. U.S., 17.5.28).

**Opening and closing of cylinders or capsules for compressed or liquefied gas.** SOC. ANON. D'APPLICATIONS DES GAZ LIQUÉFIÉS (B.P. 318,546, 28.8.29. Fr., 5.9.28).

**Production of foam for fire-extinguishing purposes.** MINIMAX A.-G. (B.P. 320,020, 30.9.29. Ger., 29.9.28).

**Separating particles of different densities** (B.P. 331,841). **Fractional condensation** (B.P. 331,485 and 331,526).—See II. **Preventing corrosion of water-pipes** (B.P. 331,725). **Mercury-vapour boilers** (B.P. 310,880).—See X. **Grinding mills** (B.P. 331,762).—See XIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Ultimate analysis of coal.** F. SCHULZ (Coll. Czech. Chem. Comm., 1930, 2, 486—488).—In the ultimate analysis of coal and coke only very small quantities of nitrogen oxides are formed during the combustion in oxygen, and the error due to their absorption does not exceed 0.1% C.

H. F. GILLBE.

**Extraction of Miike coal [with tetralin and phenol].** K. FUJIMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 201—202 B).—After prolonged extraction with heated tetralin in an atmosphere of carbon dioxide, about 30% of the sample was dissolved. A further 10% of the residue was similarly dissolved by phenol. The residues from these extractions contained rather less volatile matter and were less strongly coking than was the original coal.

C. IRWIN.

**Determination of volatile matter of Japanese coals by means of the high-temperature analytical balance.** T. SOMIYA and S. HIRANO (J. Soc. Chem. Ind., Japan, 1930, 33, 252—254 B).—The change of weight of a number of coal samples in a nitrogen-filled electric furnace over the temperature range 800—1200°



was measured. Higher values were obtained if the coal was heated immediately to at least 800° without pretreatment at 550°, but results by the latter method were the more consistent. In this case all curves became practically straight lines at about 950°, and this temperature is recommended for volatile matter determinations in proximate analysis. C. IRWIN.

**Micro-pyrometer and gas-furnace methods for determination of fusion point of coal ash.** M. C. K. JONES, H. FARMER, J. E. BREWER, and H. C. PORTER (Ind. Eng. Chem. [Anal.], 1930, 2, 325—328).—Results of ash fusion-point determinations on ten samples of bituminous coal and three of anthracite, carried out in different laboratories, are compared and fairly satisfactory agreement is found between results obtained by the micro-pyrometer method and those obtained by the gas-furnace method. Buckwheat anthracites that clinkered in household furnaces using blowers were indicated by the standard gas-furnace test to have highly refractory ash, whilst the micro-pyrometer test indicated in some instances a moderate clinkering tendency.

E. S. HEDGES.

**Coking coal mixed with iron minerals.** N. N. ROGATKIN, L. P. UKHOV, and D. G. JOFFE (J. Chem. Ind., Russia, 1929, 6, 213—214).—When coal is coked with magnetic iron minerals the coke obtained possesses good resistance to pressure and a high reacting capacity.

CHEMICAL ABSTRACTS.

**Purification of spectrum carbon.** G. HEYNE (Z. angew. Chem., 1930, 43, 711—712).—Carbon to be used as carrier for spectrum analysis of powders, solutions, etc. is purified by heating in a current of nitrogen and hydrogen either in a charcoal tube to 2500° for 30 min., or between charcoal blocks electrically; by the latter means a temperature of 3000° may be attained when a shorter heat-treatment is needed. The sample is freed completely from iron and aluminium, and from most of the silicon, calcium, and magnesium.

H. I. DOWNES.

**Calorific value of coconut husks.** S. H. J. WILSON (New Zealand J. Sci. Tech., 1930, 12, 14—15).—A sample of husks from the Cook Islands contained 21.5% of outer skin, 49.6% of fibrous husk, and 28.9% of shell. Duplicate samples of these three materials gave average calorific values of 4113, 4113, and 4382 g.-cal., respectively, and for the whole material, 4192. As a fuel the material is excessively bulky.

E. HOLMES.

**Nomogram for calculating calorific value of gases.** L. J. ROGERS (Gas World, 1930, 93, 119).—Examination of the olefines in low- and high-temperature gases at the Fuel Research Station has shown that their calorific value varies from 2500 to 2200 B.Th.U. per cub. ft.; these figures are used in constructing a nomogram for calculations with mixtures.

R. H. GRIFFITH.

**Water-gas tar.** A. V. SIVOLOBOV (J. Chem. Ind., Russia, 1929, 6, 206).—Phenols, pyridine bases, and carbazole are almost entirely absent; the crystalline products (up to 16%) contain naphthalene (crude, 50%) and a considerable quantity of easily polymerisable unsaturated compounds. The anhydrous tar contains

benzene 0.7, toluene 0.03, crude naphthalene 8, anthracene 1.1, phenanthrene 2, and acenaphthene 0.8%.

CHEMICAL ABSTRACTS.

**Determination of phenol and *m*-cresol in crude phenol oil.** P. K. SAKMIN (J. Chem. Ind., Russia, 1929, 6, 890—892, 966).—The separation of phenols with alkali is described. Fractional distillation affords a fraction of b.p. up to 195°, containing nearly all the phenol, which is determined by a simplified Masselerou procedure; in the fraction, b.p. 195—207°, the *m*-cresol is determined by Raschig's method of nitration. If much is present, however, 80% nitric acid must be employed.

CHEMICAL ABSTRACTS.

**Purification of naphthalene.** I. E. BESPOLOV (Neft. Choz., 1929, 17, 737—738).—Distillation is preferably preceded by removal of oil.

CHEMICAL ABSTRACTS.

**Perm crude oil.** R. A. VIRABYANTZ (Neft. Choz., 1930, 18, 196—201).—The composition is recorded; the sulphur content is high. The results of cracking tests are also given.

CHEMICAL ABSTRACTS.

**Corrosive action of petroleum.** M. BLAGODAROV and V. SHEVLYAKOV (Azerbaid. Neft. Choz., 1930, 106—113).—The copper strip test is satisfactory. Hydrogen sulphide must be removed with alkali before treatment with 90—94% sulphuric acid; otherwise it is oxidised to sulphur. Treatment with hypochlorite may be substituted for that with sulphuric acid.

CHEMICAL ABSTRACTS.

**Decomposition of Surakhani crude oil and its distillates.** I. E. BESPOLOV (Azerbaid. Neft. Choz., 1930, No. 3, 60—73).—Pipe-stills yield more unsaturated hydrocarbons than do shell-stills; deep cracking of mazout increases the yield of aromatic compounds. Vapour-phase cracking favours the formation in the tar of aromatic substances (optimum temperature 650°); the yield of gasoline and kerosene fractions is smaller and of gas and tar larger than in liquid-phase cracking. Lengthening of the cracking time favours the formation of aromatic substances at the expense of unsaturated hydrocarbons. Deep cracking of cracked gas oil yields a tar practically free from saturated hydrocarbons.

CHEMICAL ABSTRACTS.

**True boiling crude analysis [of petroleum crude oils].** G. A. BEISWENGER and W. C. CHILD (Ind. Eng. Chem. [Anal.], 1930, 2, 284—287).—An improved method for determining the quantity and quality of products obtainable from the crude oils is described, and consists in distilling the sample in an efficient true b.p. apparatus, followed by correct blending and inspecting. The method has many advantages: it is inexpensive and rapid, maximum yields of more valuable products can be determined and yields may be correlated with refinery operation, and considerable additional information is obtainable, such as mol. wts. and flash temperatures necessary for pipe-still operations.

E. S. HEDGES.

**Atmospheric cracking of residue from Apsheron crude oils.** V. SHEVLYAKOV and M. VOLF (Azerbaid. Neft. Choz., 1930, 96—100).—Coke (9—19%), gasoline (10%), kerosene (24—30%), and residue of high m.p. (30—40%) were obtained. The gasoline contained



aromatic and unsaturated hydrocarbons, but little naphthenes.

## CHEMICAL ABSTRACTS.

**Crude oil resins.** N. A. VASILIEV and L. V. ZHIRNOVA (Neft. Choz., 1929, 17, 707—712).—After removal of asphaltenes and naphthenic acids the resins from Balakhani-Sabunchi crude oil were separated with silica gel; fuller's earth adsorbed little and caused polymerisation. Vacuum distillation prevents the formation of new resins if carried out in an atmosphere of carbon dioxide. The mol. wts. were 226 and 1112 (silica gel), or 504 and 927 (fuller's earth) for the resins in the lower and higher fractions, respectively. The composition corresponds with the formula  $C_nH_{2n-m}O_p$ , where  $n$  varies from 16 to 69,  $m$  from 8 to 40, and  $p$  from 1 to 3; less than one atom of sulphur is present. Grozni asphalt affords similar resins.

## CHEMICAL ABSTRACTS.

**Direct determination of oil in oil-water-soap emulsions.** L. N. MARKWOOD (Ind. Eng. Chem. [Anal.], 1930, 2, 265).—The following method for determining oil in emulsions is based on the fact that emulsions of the oil-in-water type stabilised by soap are resolved by phenol. It has the advantages over the usual technical method that it is direct, can be completed in 20—30 min., and no inflammable solvents are required. About 5 g. of the sample are introduced into a 10-c.c. cylinder, 3 c.c. of 75% phenol solution are added in drops, and the contents of the cylinder gently mixed. The cylinder is then centrifuged and the volume of the oil layer read. The density of the oil is determined in a small pycnometer and the percentage of oil calculated.

E. S. HEDGES.

**Coal from Kuznetski basin, Siberia.** N. M. KARAVAEV and I. B. RAPOPORT (Izvest. Teploekhn. Inst., 1929, No. 8, 15—30).

**Nature and properties of Siberian boghead [coal].** G. L. STADNIKOV (Proc. II. Int. Conf. Bit. Coal, 1929, 1, 625—631).

**Ammonia.** THAU.—See VII. Cresylic acids as flotation agents. LANDOLT and others.—See X.

## PATENTS.

**Manufacture of briquettes, fire-lighters, and other consolidated fuels.** H. ALEXANDER and J. A. GREENE (B.P. 330,295, 6.2.29).—A mixture of a substance which contains the necessary binding agent, *e.g.*, bituminous coal or resinous saw-dust, with anthracite dust, coke dust, etc. is heated or carbonised in moulds provided with an anti-adhesive lining, *e.g.*, wood pulp or graphite. A relatively small quantity of an additional binding material, *e.g.*, water or dolomite, may be added.

A. B. MANNING.

**Manufacture of charcoal fuel briquettes.** C. DEMOULIN (B.P. 332,815, 23.9.29).—Powdered wood charcoal is mixed at about 100° with wood-tar pitch and aqueous (3% solution of) alkali. The resulting emulsion spreads over the solid particles and produces a paste which is then briquetted. Sodium nitrate may also be included.

R. H. GRIFFITH.

**Separation of [coal] particles of different densities by means of [heavy] liquids.** C. W. H. HOLMES,

and BIRTLEY IRON CO., LTD. (B.P. 331,841, 8.3.29).—In the separation of coal by floating it in a heavy liquid, the mixed material is first sprayed with or dipped in paraffin or other liquid that will prevent the heavy liquid (*e.g.*, crude varnish) from wetting it; it is then treated in a liquid of intermediate density, and only the part that sinks is further treated in the heaviest liquid.

B. M. VENABLES.

**Stabiliser for fuel suspensions.** H. G. C. FAIRWEATHER. From AMER. COAL OIL CORP. (B.P. 332,343, 25.5.29).—A suspension of powdered coal, or similar solid material, in a mineral, vegetable, or animal oil, coal tar, etc. is stabilised by the addition of a soap-like substance made by oxidation of a high-boiling oil containing olefines and paraffins; air or oxygen is blown through the hot oil in the presence of a metallic salt of an organic acid, such as manganese oleate, and the reaction product is neutralised and saponified by means of alkali, before addition to the suspension.

R. H. GRIFFITH.

**Coal-carbonising retorts etc.** T. M. DAVIDSON (B.P. 331,906, 8.4.29).—A vertical retort is provided with a close-fitting internal structure, which forms a large number of small pockets close to the walls. The maximum depth of heat transfer through the coal is about 3 in. The retort widens towards the base, and the central structure is movable vertically, through a special lower door, by means of a hydraulic ram.

R. H. GRIFFITH.

**Carbonisation of agglomerates of non-bituminous coals.** G. M. F. F. MASCART (B.P. 331,850, 8.4.29).—In carbonising briquetted coals containing less than 14% of volatile matter, it is possible to use not more than 7% of pitch as binding material if heating is carried out initially at 500° and finally to about 900° during 10 hrs. The amount of unchanged pitch recovered with the tar depends on the initial temperature, decreasing from 60 to 70% at 500° to about half that amount at 750°. The resulting coke is of high bulk density and coherence.

R. H. GRIFFITH.

**Carbonising coal and cracking liquid hydrocarbons.** TRENT PROCESS CORP. (B.P. 313,158, 21.5.29. U.S., 9.6.28).—Coal is continuously carbonised in a vertical retort provided throughout its length with tubular off-takes. The residual hot coke descends into an oil-bath and is there quenched, the vapours produced passing upwards into the retort, where they are cracked and the reaction products escape by way of the tubular off-takes.

R. H. GRIFFITH.

**Wet carbonisation.** D. W. BERLIN and J. A. BRINCK (B.P. 317,401, 13.8.29. Swed., 15.8.28).—A paste of peat or similar substances is forced through the inner of two concentric tubes, and a liquid heating medium moves countercurrent in the other. Means are also provided for transfer of heat from the treated material to the circulating liquid, to which additional heat can be applied as desired. Gas evolved in the apparatus escapes through a valve, and the tubes are constructed to withstand an increase in pressure.

R. H. GRIFFITH.

**Apparatus for low-temperature distillation, drying, etc.** J. Y. JOHNSON. From I. G. FARBEIND.



A.-G. (B.P. 331,879, 11.2.29).—A rotating bundle of inclined tubes is mounted on a water-cooled shaft in such a way that the tubes can expand freely through end-wall borings. Heating is effected by passing gases round the tubes and through the support-plates. Gravity feed and extraction gear are employed to convey material, such as powdered coal, through the apparatus.

R. H. GRIFFITH.

**Gasifying fuels in a molten-ash-type producer, and simultaneously treating ores or residues.** H. J. F. PHILIPON (B.P. 332,366, 15.6.29).—Fuels with a high ash content, such as domestic refuse, schist from coal seams, etc., are gasified in a producer so that the clinker produced becomes thoroughly molten. The nature and quantity of the slag formed is regulated by previous addition of inorganic material, and by controlling the temperature by alterations in the degree of preheating of the blast air. The process can be adapted to working up of cast iron, cement slag, calamine and other ores, and to the recovery of phosphoric acid from phosphate ores. Conditions are adjusted to give a slag of optimum fusibility, and the process is designed to work most satisfactorily with an initial fixed carbon content of about 30%.

R. H. GRIFFITH.

**Destructive hydrogenation of carbonaceous materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,251, 17.1.29).—In order to avoid local overheating, high-pressure hydrogenation of carbonaceous material is carried out in a reaction vessel consisting of several gas-heated vertical tubes. The gases are not allowed to burn on or near the tubes and are previously mixed with effluent gases to lower their temperature before being circulated through the system. The heating media may be 100° hotter than the reaction temperature, provided that they do not exceed 800° and that they are uniform in character.

R. H. GRIFFITH.

**Destructive hydrogenation of solid carbonaceous material.** C. F. R. HARRISON, H. W. STRONG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 332,246, 17.4.29).—Catalysts for assisting liquefaction of coal and similar solid materials are added in the form of a solution (preferably saturated) of a soluble salt so that they are evenly distributed throughout the mass. The amount of liquid added should be insufficient to form a paste with the powdered coal. The impregnated material may be dried and mixed with oil. Nickel nitrate and ammonium molybdate are mentioned as suitable salts.

R. H. GRIFFITH.

**Destructive hydrogenation of brown coal and other oxygen-containing low-grade fuels.** H. W. STRONG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,222, 31.12.28).—The initial material is comminuted and then dried and heated, in two stages if desired, in the presence of a non-oxidising gas, so that not only is drying effected without oxidation, but oxygen compounds already present are partly eliminated as carbon dioxide and water. The product is formed into a paste with a suitable oil or tar and submitted to destructive hydrogenation.

A. B. MANNING.

**Preparation of liquid products from coal or coal-containing materials and liquefaction of such**

**materials.** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 330,223, 31.12.28).—The initial materials are heated with hydrogen under pressure to a maximum temperature (350–550°), with or without catalysts, and the heating is then immediately discontinued so that the mass begins to cool. The gaseous products are drawn off while still hot (e.g., at 325°), and are cooled; the non-condensed gases contain a large proportion of totally oxidised compounds, e.g., carbon dioxide. The residue is then strongly heated with hydrogen under pressure, with or without catalysts, and maintained for some time in contact with hydrogen at the maximum temperature; the gaseous products are drawn off while still hot, cooled, and the non-condensed gases, consisting chiefly of hydrocarbons of high calorific value, are used as fuel. The residue in the reaction vessel may, if desired, be again submitted to an analogous treatment. Substantially the whole of the sulphur present in the initial materials is removed in the first stage of the process as hydrogen sulphide. Molybdenum and iodine, or their compounds, are suitable catalysts.

A. B. MANNING.

**Gas-manufacturing installations.** A. H. LYMN and N. J. BOWATER (B.P. 330,250, 12.2.29).—The installation comprises a carbonisation plant, a water-gas plant, means for mixing the coal gas from the former with the "run" gases from the latter to produce a mixture of suitable quality for use as distribution gas, and means for mixing a portion of the "run" gases and/or a portion of the coal gas with the "blow" gases of the water-gas plant to produce a gas suitable for heating the carbonisation plant. When carburetted water-gas is required to augment the output of the carbonisation plant the water-gas plant may operate alternately to produce carburetted water-gas in one cycle of operations, and blue water-gas for admixture with the "blow" gases to produce heating gas in another cycle.

A. B. MANNING.

**Gas and steam producer generator.** J. BELLAY (U.S.P. 1,753,847, 8.4.30. Appl., 1.8.27).—A water-gas generator is fitted with an annular boiler, which is further heated by all or part of the gas produced, and supplies process steam to the plant.

R. H. GRIFFITH.

**Production of water-gas.** D. TYRER, H. G. WATTS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,221, 31.12.28).—Finely-divided carbonaceous material is completely gasified in steam, or in steam and oxygen, by passing the mixture, at a velocity not exceeding about 10 cm. per sec., and with a stream-line motion, in contact with a highly heated surface. A relatively wide, vertical generator is used, preferably divided into a number of successive zones of temperature, e.g., a preheating zone, a reaction zone, and a hydrocarbon-decomposing zone. The carbonaceous material and the gasifying agent may be introduced together at either the top or bottom of the generator; or the former may be introduced at the top and the latter at the bottom, in which case the rate of gas supply should be less than the rate of settling of the material in the still gas.

A. B. MANNING.

**Manufacture of producer gas.** REID POWER DEVELOPMENT Co., and J. W. HINCHLEY (B.P. 332,007,



30.5.29).—Gas from a lime kiln, essentially carbon dioxide, is passed to a coke-filled producer so that the gas from the latter contains large amounts of carbon monoxide. The quantities of air and gas fed to the kiln and producer are regulated in accordance with the temperatures attained, to give a gas of the required composition. The supply of combustible gas for heating the kiln is controlled by a valve which responds to the producer temperature. R. H. GRIFFITH.

**Purification of technical gases, more particularly coke-oven gases which are to be subjected to decomposition by low-temperature cooling.** GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 307,886, 15.3.29. Ger., 15.3.28).—Deposition of resinous materials during the low-temperature fractionation of coal gas is prevented by preliminary removal of cyclic diolefines. This is effected by treatment with hydrogen, halogens, hydrogen halides, oxygen, etc., or by washing with tetralin or similar high-boiling oils. In the former method a catalyst, such as aluminium chloride for chlorination, may be used, and the latter method is best carried out under increased pressure. R. H. GRIFFITH.

**Removal of sulphur from fuel gases.** S. G. WATSON, D. M. HENSHAW, and W. C. HOLMES & Co., LTD. (B.P. 332,147, 15.10.29).—In purifying crude gases from hydrogen sulphide by means of iron oxide, either dry or as an aqueous suspension, wetting of the solid or dilution of the suspension is prevented by previous drying of the gas. For this purpose a hygroscopic liquid is used such that in subsequent treatment at 35–40° no further deposition of water occurs. R. H. GRIFFITH.

**Production of hydrogen [from methane].** C. F. R. HARRISON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 332,571, 19.4.29).—Thermal decomposition of methane is carried out alternately with the water-gas reaction and the combustion of carbon in a coke-filled chamber. The temperature is initially raised to about 1500° by blowing with air, and the decomposition of methane then proceeds until the temperature has fallen to about 1100°. Between this point and 900° steam is added, after which the cycle is repeated. The different stages can be balanced so that no coke, but only deposited carbon, is consumed. R. H. GRIFFITH.

**Production of acetylene.** R. M. WINTER, P. H. HULL, J. FERGUSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 332,731, 12.7.29).—Methane is mixed with oxygen or air and passed rapidly through a tube at above 1000°, when appreciable amounts of acetylene are formed. The mixture should contain as much oxygen as is possible without explosion, and ignition of the gas must be prevented by the use of a gauze placed before the reaction zone. The yield of acetylene is increased by raising the temperature and by selecting an optimum velocity; a maximum of about 10% is obtainable. R. H. GRIFFITH.

**Manufacture of acetylene and hydrogen from hydrocarbons or gas mixtures containing same.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,057, 12.7.29).—Hydrocarbons etc., preferably under slightly reduced pressure, are passed through one or more

electric arcs and the acetylene formed is wholly or partly separated, part of the residual gas being recirculated after admixture with fresh gas, whilst another part, together with an addition of hydrocarbons or other gases if desired, is treated in another arc or arc system. H. S. GARLICK.

**Vaporisation of heavy hydrocarbons.** COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 316,552, 23.7.29. Fr., 30.6.28).—In order to prevent the formation of hard pitchy and carbonaceous deposits in a vaporiser for heavy oils, heating is carried out in two stages, but in a single vessel. Oil fed to the top of this vessel trickles over heated baffle plates, which are at such a temperature that partial volatilisation occurs but that the residue is sufficiently liquid to flow on to a packing of porous material. The deposit which forms in the lower, hotter zone can then be removed at intervals by combustion. R. H. GRIFFITH.

**Cracking of oils.** C. P. DUBBS (B.P. 331,569, 5.3.29).—Oil is heated to cracking temperature under cracking pressure in a heating zone and directly passed into an adjacent separating or reacting zone where the vapours are separated and dephlegmated. The residual oil is continuously removed without coming into contact with reflux condensate or with fresh oil. In order to obtain the maximum amount of useful fuel oil the polymerisation of the unvaporised residual oil is checked by maintaining the lowest possible level of the latter in the separating zone and by regulating both the rate of withdrawal of the residual oil and the dissipation of heat therefrom. H. S. GARLICK.

**Cracking of hydrocarbons.** J. H. BRÉGEAT (B.P. 331,531, 27.3.29).—Asphaltic, paraffinous, and resinous substances extracted from petroleum, low-temperature distillation tars, acid sludges from petroleum refining, waxes, and natural asphalt are subjected to cracking in a retort at 350–460° with continuous agitation, either with or without the addition of abrasive material. The level of the substance in the retort is maintained substantially constant throughout the treatment and the rate of distillation regulated so that the quantity of distillate collected hourly comprises 5–14% by wt. of the effective capacity of the retort. The distillate is subjected to reflux condensation, and that portion distilling above 300° is returned to the cracking circuit. H. S. GARLICK.

**Manufacture of liquid hydrocarbons and hydrocarbon derivatives.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,558, 7.1.29).—Distillable carbonaceous materials are subjected to destructive hydrogenation under a pressure of 20 atm. or more in the presence of at least three different constituents, one being an oxide of a metal of group II or III or a compound thereof which is solid under the conditions of working, e.g., kaolin or Florida earth, and the other two being oxides, hydroxides, or sulphides of different metals of the type of cobalt, nickel, or metals of group V or VI. H. S. GARLICK.

**Refining system [for hydrocarbon oils].** A. G. PETERKIN, JUN., ASSR. to ATLANTIC REFINING Co. (U.S.P. 1,752,455, 1.4.30. Appl., 8.4.26).—Hydrocarbon vapours are passed upwardly in contact and in interchange



relation with descending liquid reflux oil. Oil fractions are removed from the fractionating zones and a solid purifying agent, *e.g.*, fuller's earth, is suspended in at least a portion of a fraction so removed and the suspension passed in fractionating relation with the ascending vapours.

H. S. GARLICK.

**Purification of gas, particularly such as results from gasification of heavy oils.** C. CHILOWSKY (B.P. 317,332, 13.8.29. Fr., 13.8.28).—Gas is bubbled into a liquid contained in vertical tubes which are provided with means for circulating a cold fluid round them and which establish connexion between a tank beneath them, likewise filled with the bubbling liquid and provided with a means for feeding hot gas thereto and with a collector above them having means for discharging the cooled gas through a valve immersed in the bubbling liquid. Preferably the level of the bubbling liquid is above the top of the ends of the tubes. When the gasification of heavy oils is effected in the presence of steam the vessel in which bubbling takes place is connected by an overflow pipe to a settling vessel, the water necessary for the supply of the gas generator being withdrawn from the settling vessel by a pump through a filter.

H. S. GARLICK.

**Apparatus for lye treatment of petroleum oils.** C. K. PARKER, Assr. to STANDARD OIL Co. of CALIFORNIA (U.S.P. 1,752,350, 1.4.30. Appl., 12.7.26).—The oils are continuously mixed with an immiscible alkaline treating agent, heated and supplied under pressure to an enlarged, horizontal, pressure, settling chamber, through an inlet of gradually increasing cross-sectional area. Oil and lye are separately discharged through an outlet larger than the main section from vertically separated points.

H. S. GARLICK.

**Fractionally condensing (A) composite vapours, (B) petroleum vapours and other vapour compounds.** A. LOSEY, P. GOTTLIEB, and L. L. HAUPT (B.P. 331,485 and 331,526, 25.2.29. U.S., [A] 11.12.28, [B] 10.10.28).—(A) The vapours are passed into fractionating apparatus consisting of a series of compartments in which fuel oil, lubricating stock, distillate, and gas oil are successively condensed in separate compartments by causing the vapours to perform successive limited amounts of work, the resulting vapours being passed into an adjacent compartment and fractionally condensed by heat exchange with a fraction returned under increased pressure. (B) The heated vapours of crude oil are passed through a multistage expansion device, whereby they are fractionally condensed into a series of oil products.

H. S. GARLICK.

**Breaking [water-petroleum oil] emulsions.** J. W. BECKMAN (U.S.P. 1,753,641, 8.4.30. Appl., 21.12.25).—Natural crude oil emulsions containing an emulsifying agent are treated with a medium adapted to sustain the life of, and are inoculated with, living micro-organisms able to destroy such emulsifying agent, the mixture being agitated at intervals during the life action and the temperature maintained at 20–40°. H. S. GARLICK.

**Stabilising absorption system [for gasoline].** J. A. CAMPBELL (U.S.P. 1,754,606, 15.4.30. Appl., 19.5.27).—The vapour-laden gases (casing-head gas) are passed upwardly through a downwardly moving stream

of absorbing liquid, and the vapour-laden liquid is removed from the lower end of the stream and heated to vaporise the absorbed liquids. The lean absorbing liquid is returned to the stream. The lower part of the absorbing stream is maintained at a higher temperature than the upper part, by introducing therein a certain amount of the heated absorbing liquid.

H. S. GARLICK.

**Gasoline filtering system and apparatus.** B. V. STOLL, Assr. to STOLL OIL REFINING Co. (U.S.P. 1,752,709, 1.4.30. Appl., 14.3.29).—Gasoline vapours from a cracking process are condensed, immediately filtered through a decolorising absorbent, and discharged into an alkaline mixer from which it passes into storage through pressure-release valves.

H. S. GARLICK.

**Motor fuel.** H. MENZ, W. STEFFEN, and E. JAAKS-MÜNCHBERG (B.P. 331,966, 30.4.29).—A vaporising fuel comprises 50–55% of a hydrocarbon, preferably hydrogenated, *e.g.*, tetrahydronaphthalene, about 10% of sulphonated vegetable or animal oil soluble in water, and about 35% of distilled water containing 5% of waste sulphite-lye dissolved therein. H. S. GARLICK.

**Manufacture of non-knocking engine fuels of the benzene type.** I. G. FARBERIND, A.-G. (B.P. 303,894, 5.1.29. Ger., 13.1.28).—Benzene (3–7 pts. by wt.) obtained from the destructive hydrogenation of brown coal, tars, or mineral oils are blended with 1–3 pts. of benzene isolated from the destructive hydrogenation of mineral coal, *e.g.*, anthracite.

H. S. GARLICK.

**Treating fuel oils.** A. H. ACKERMAN, Assr. to CATALYTIC CHEM. Co. (U.S.P. 1,754,297, 15.4.30. Appl., 16.2.27. Renewed 8.12.28).—Petroleum coke is ground with a small proportion of a composition comprising anthracene 10 pts., nitrobenzene 4 pts., sulphuric acid 2 pts., caustic soda 1 pt., and naphthalene 200 pts., and the resulting mixture is agitated with fuel oil until dissolved. A product having increased calorific value is claimed.

H. S. GARLICK.

**Coke oven.** H. SCHRÖDER (U.S.P. 1,770,998, 22.7.30. Appl., 28.11.25. Ger., 12.4.24).—See B.P. 254,960; B., 1926, 778.

**Non-knocking motor fuel.** A. C. EGERTON, Assr. to ASIATIC PETROLEUM Co., LTD. (U.S.P. 1,771,169, 22.7.29. Appl., 6.6.27. U.K., 29.7.26).—See B.P. 279,560; B., 1928, 292.

**Valve-reversing devices for intermittent gas producers etc.** BAMAG-MEQUIN A.-G. (B.P. 332,783, 22.8.29. Ger., 27.10.28).

**Recovering oil and grease [from water etc.].** W. G. MORSE (B.P. 332,569, 23.2.29).

**Contacting of gases and solids (B.P. 315,868). Testing gases (U.S.P. 1,753,675). Treatment of fluids (U.S.P. 1,753,859). Still and scrubber (U.S.P. 1,754,605 and 1,754,607).—See I. Activated iron oxide (B.P. 332,259).—See VII.**

### III.—ORGANIC INTERMEDIATES.

**Determination of small amounts of acid in ether.** L. P. HALL (Ind. Eng. Chem. [Anal.], 1930, 2, 244–246).—Formic and acetic acids occur among the products of



decomposition of ethyl ether, but pure ether is found to be neutral. The two-layer method of titration, using phenolphthalein as indicator, is subject to large errors when small amounts of acid are being determined, but a skilled operator can obtain satisfactory results by adding alcohol to keep the solution homogeneous. The ether-alcohol mixture exerts an influence on the colour of the phthalein indicators. The method now recommended is to substitute a sulphonephthalein indicator such as bromothymol-blue, taking care to avoid introduction of carbon dioxide. This indicator may be used satisfactorily also when the ether contains alcohol.

E. S. HEDGES.

**Hygroscopicity of urea.** S. OKA (J. Soc. Chem. Ind., Japan, 1930, 33, 227—228 B).—Hygroscopicity was measured by weight increase in a given time in contact with air of given moisture content. The hygroscopicity of urea was reduced by 25% by pressing into a cake. It was further lessened by melting with 10% of water and rapidly cooling, and further again by melting in absence of water. The last product is hygroscopic only in summer in Japan.

C. IRWIN.

**Determination of the inorganic acidity of crude nitro-products.** I. M. KARYAKIN and A. A. GOLUBEV (J. Chem. Ind., Russia, 1929, 6, 120—121).—Sufficient aniline to combine with the sulphuric acid is added, the salt being collected, washed with benzene or ether, and titrated. Crude nitronaphthalene is first dissolved in benzene; dinitrobenzene and dinitrotoluene are dissolved in aniline.

CHEMICAL ABSTRACTS.

**Preparation of nitrosophenol and of indoles by means of aluminium nitrite.** V. G. GULINOV (J. Chem. Ind., Russia, 1929, 6, 214—216).—A solution of aluminium nitrite is added, with stirring, to an aqueous solution of phenol and aluminium sulphate, stirring being continued for 30 min.; after 18—20 hrs. the precipitate of aluminium *p*-nitrosophenoxide (90—93% of theoretical yield) is collected, washed with cold water, and dried below 40°; alumina is removed by washing with hydrochloric acid (2—3%). The salt is treated with sodium hydroxide solution (5%), the nitrosophenol being precipitated with hydrochloric acid. In the preparation of indophenol for blue a solution of *o*-toluidine (10.7 pts.) in sulphuric acid (*d* 1.70—1.74) is treated at 1—3° with dry aluminium nitrosophenoxide (13.1 pts.) in small portions with stirring, which is continued during 1 hr. After 3 hrs. at 4° the mass is poured on ice and the acid neutralised; the precipitate is collected and washed with cold water. Herz's method (G.P. 199,963 of 1902) for obtaining the sulphur-blue dye is modified. In the preparation of indophenol for green,  $\alpha$ -naphthylamine (14.3 pts.) is dissolved with cooling in sulphuric acid (180 pts.), aluminium nitrosophenoxide (13.1 pts.) being introduced in small portions, with stirring, at 3—5°, stirring being continued for 1 hr. After 2—3 hrs. at 5° the mixture is poured on ice, the precipitate being collected and washed with water at 0°. The preparation of the sulphur-green dye is described.

CHEMICAL ABSTRACTS.

**Crude phenol oil.** SAKMIN. Naphthalene. BESPOLOV.—See II.

## PATENTS.

**Manufacture of methyl alcohol [from carbon monoxide and hydrogen].** G. NATTA (B.P. 330,919, 11.3.29).—Carbon monoxide and hydrogen are passed under pressure (260—300 atm.) over smithsonite, especially smithsonite calcined at 400—450°, at about 400°. The conversion into methyl alcohol is 20%.

C. HOLLINS.

**Removal of water from aqueous formic acid.** H. T. CLARKE and D. F. OTHMER, ASSRS. to KODAK, LTD. (B.P. 318,633, 5.9.29. U.S., 8.9.28).—*n*-Propyl formate is added to the aqueous formic acid, or is formed therein by addition of propyl alcohol. The mixture is heated below the b.p. of water so that an azeotropic mixture of water and *n*-propyl formate distils, the dehydrated formic acid being then separated from the remaining propyl ester by fractional distillation. W. J. WRIGHT.

**Manufacture of arylacetic acids and substitution products thereof.** O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 330,916, 19.2.29).—Aromatic hydrocarbons, and such derivatives as contain no primary or secondary amino-groups, react with mono- or di-halogenated acetic acids or esters at 100—275° in absence of alkali to give arylacetic acids or esters. Chloroacetic acid heated with naphthalene at 165—185° for 3 days yields  $\alpha$ -naphthylacetic acid, m.p. 129°. The preparation of 5-acenaphthylacetic acid, m.p. 187°, b.p. 180°/9 mm.; anthracylacetic acid, m.p. 189°; fluorenylacetic acid, m.p. 148°; 4-bromo- (m.p. 122—128°) and 4-chloro- (m.p. 124°)  $\alpha$ -naphthylacetic acids; ethyl  $\alpha$ -naphthylacetate, m.p. 129—130°; 2-hydroxy- $\alpha$ -naphthylacetic acid, m.p. 106—107°, b.p. 275°/13 mm.; and di- $\alpha$ -naphthylacetic acid, m.p. 216—218°, is also described.

C. HOLLINS.

**Manufacture of sulphonation products of unsaturated hydroxy-fatty acids.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 330,904, 18.3.29).—Air charged with sulphur trioxide is passed over the surface of a well-stirred solution of castor oil or ricinoleic acid in about 3 pts. of an organic solvent (carbon tetrachloride, trichloroethylene). Preferably the hydroxyl group is first sulphonated with 100% sulphuric acid at 30—40°, and the ester is then treated with sulphur trioxide at —10° to —3°. The products are not precipitated by acids or lime.

C. HOLLINS.

**Manufacture of *NN'*-thio-derivatives of amines [*NN'*-disulphides].** IMPERIAL CHEM. INDUSTRIES, LTD., T. BIRCHALL, and S. COFFEY (B.P. 331,016, 18.4.29).—Secondary amines react with sulphur monochloride (disulphur dichloride) in neutral or alkaline aqueous solution or suspension to give *NN'*-disulphides. Products from diethylamine, ethylaniline, and piperidine are described.

C. HOLLINS.

**Manufacture of phenolic compounds.** I. G. FARBERIND. A.-G. (B.P. 308,220, 18.3.29. Ger., 19.3.28. Addn. to B.P. 288,308; B., 1929, 746).—The process of the prior patent is extended to the production of phenols generally, and the yields are improved by addition of copper, nickel, cobalt, or their oxides or nitrates to the silica gel catalyst. Examples are phenol from chlorobenzene and steam over silica gel and copper



oxide at 400°; *p*-cresol from *p*-chlorotoluene over silica gel and nickel at 380°. C. HOLLINS.

**Manufacture of condensation products from aromatic aldehydes and phenols [mothproofing agents].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,893—4, 14.3.29).—Hydroxylated di- and triarylmethanes, valuable as mothproofing agents, are obtained by condensation of (A) a non-hydroxylated aromatic aldehyde with 2 mols. of a *p*-alkylated phenol having a free *o*-position (excluding benzaldehyde) with *p*-cresol, or (B) an aldehyde (excepting hydroxylated aromatic aldehydes) with a 3- and/or 5-halogenated phenol having a free 4-position. The condensations are preferably carried out in presence of acid agents (sulphuric acid, hydrochloric-acetic acid, or zinc chloride). The compounds may be applied to wool, fur, etc. together with a dye. Examples are the products from (A) benzaldehyde-*o*-sulphonic acid and 5-chloro-*p*-cresol or *p*-cresol, (B) formaldehyde and *m*-chlorophenol, benzaldehyde-*o*-sulphonic acid and 2:5-dichlorophenol.

C. HOLLINS.

**Manufacture of 2-aminonaphthalene-3-carboxylic acid [2:3-aminonaphthoic acid].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,941, 20.3.29).—2:3-Hydroxynaphthoic acid or its zinc salt is mixed with zinc oxide or carbonate and ammonium chloride and then heated at 180—200°, preferably in a current of dry air or ammonia, for 3—4 hrs. in a ball-mill. A 70—80% yield of 2:3-aminonaphthoic acid is claimed.

C. HOLLINS.

**Manufacture of oxygenated organic compounds. [Oxidation of aromatic side-chains, naphthalene, etc.]** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 331,100, 28.6.29).—Aromatic hydrocarbons or their halogen derivatives are oxidised in the liquid phase by oxygen or air under pressure at 150—250° in presence of water and an oxidation catalyst (oxide or hydroxide of copper, nickel, cobalt, iron, manganese, cerium, osmium, uranium, vanadium, particularly ferric oxide hemihydrate). The action may be moderated by addition of considerable amounts of carbon dioxide. Examples are the oxidation of toluene to benzaldehyde and benzoic acid, of *p*-xylene to *p*-toluic acid, of *m*-xylene to *m*-tolualdehyde and *m*-toluic acid, of ethylbenzene to acetophenone and phenylmethylcarbinol, of naphthalene to benzoic acid and phthalic acid, of anthracene to anthraquinone, and of *p*-chlorotoluene to *p*-chlorobenzaldehyde and *p*-chlorobenzoic acid.

C. HOLLINS.

**Manufacture of diaminoazoanthraquinones.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,644, 16.3.29).—A 1:4-diaminoanthraquinone is oxidised, *e.g.*, with chromic acid, in an organic acid medium (acetic acid) to give a 4:4'-diamino-1:1'-azoanthraquinone. The product on benzylation and reduction with alkaline hyposulphite yields a 1-amino-4-benzamidoanthraquinone.

C. HOLLINS.

**Manufacture of the 6-sulphonic acids of quinizarin and its homologues.** I. G. FARBENIND. A.-G. (B.P. 308,359, 21.3.29. Ger., 22.3.28).—The boric ester of quinizarin or a homologue (*e.g.*, 2-methylquinizarin) is sulphonated in the 6-position by heating

with oleum at 170—180°, preferably in presence of a little mercuric sulphate. C. HOLLINS.

**Production of hydrogenated phenol compounds.** H. JORDAN, ASST. to CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING) (U.S.P. 1,771,089, 22.7.30. Appl., 20.3.28. Ger., 14.7.26).—See B.P. 274,439; B., 1929, 164.

**Carbazolequinones and process of preparing them.** G. KRÄNZLEIN, H. GREUNE, K. ZAHN, and M. P. SCHMIDT, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,772,317, 5.8.30. Appl., 18.1.27. Ger., 15.1.26).—See B.P. 264,530; B., 1928, 635.

**Acetylene** (B.P. 332,731). **Hydrocarbons** (B.P. 331,558).—See II. **Anthraquinone derivatives** (B.P. 307,306).—See IV. **Activated iron oxide** (B.P. 332,259).—See VII. **Mercury from catalyst sludges** (B.P. 332,106).—See X. **Solvents** (U.S.P. 1,754,417).—See XXII.

#### IV.—DYESTUFFS.

**Fastness of dyestuffs in ultra-violet light. II.** G. A. BRAVO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1930, 8, 184—190).—Results are given of further tests (cf. B., 1930, 453) with dyestuffs of the nitroso-, nitro-, azo-, stilbene, pyrazolone, diphenylmethane, and triphenylmethane groups. T. H. POPE.

**Nitrosophenol and indoles.** GULINOV.—See III. **Dyeing tannins.** UYEDA.—See XV. **Sensitising dyes.** BLOCH and HAMER.—See XXI.

#### PATENTS.

**Manufacture of dyes [for acetate silk] and application thereof.** BRIT. CELANESE, LTD., and H. C. OLPIN (B.P. 330,602, 12.3.29).—*ar*-Tetrahydronaphthylamines are condensed with aromatic nitrocompounds containing reactive halogen, *e.g.*, *ar*-tetrahydro- $\alpha$ -naphthylamine with 1-chloro-2:4-dinitrobenzene or its 6-sulphonic acid, or the  $\beta$ -amine with 1:4-dichloro-2-nitrobenzene, to give yellow dyes for acetate silk. Preferably sulphonic groups are absent.

C. HOLLINS.

**Manufacture of acid wool dyes [from nitronaphthalimides].** I. G. FARBENIND. A.-G. (B.P. 309,552, 11.4.29. Ger., 12.4.28. Addn. to B.P. 299,721; B., 1930, 455).—A nitro-1:8-naphthalimide, or its *N*-alkyl or -aralkyl derivative, is boiled with aqueous-alcoholic sodium hydrogen sulphite or hyposulphite, whereby the nitro-group is reduced and a sulphonic group is introduced. 4-Nitro-*N*-methyl-naphthalimide thus yields a yellow wool dye.

C. HOLLINS.

**Preparation of [vat] dye derivatives.** J. S. WILSON, L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 332,249, 16.1.29).—Halogenated vat dyes (3:3'-dichloro-flavanthrone or -indanthrone etc.) are treated with sulphites to replace halogens with sulphonic acid groups.

L. J. HOOLEY.

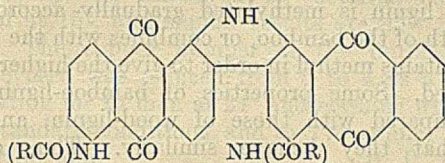
**Manufacture of dyes of the anthraquinone series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,203, 15.4.29).—Aminonitriles of the benzene series are condensed with 4-halogeno-1-aminoanthraquinone-2-sulphonic acids to yield dyes with good solubility and giving even shades. Thus 4-bromo-1-aminoanthra-



quinone-2-sulphonic acid with *m*-aminobenzonitrile gives a reddish-blue, and with 1-amino-4(or 5)-methyl-3-benzonitrile a more greenish dye (cf. B.P. 10,378 of 1914 and 7861 of 1915; B., 1916, 830, 829).

L. J. HOOLEY.

**Manufacture of anthraquinone derivatives [carbazoles of the anthraquinone series].** I. G. FARBENIND. A.-G. (B.P. 307,306, 4.3.29. Ger., 3.3.28).—Hydrolysis of 4:5'-diacetamido- or -dibenzamido- $\alpha\alpha'$ -dianthraquinonecarbazole (annexed formula) under mild

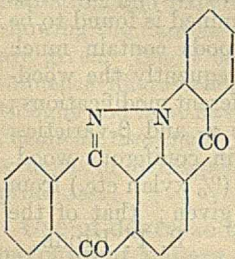


conditions, *e.g.*, with 96% sulphuric acid below 60°, or with 10% alcoholic caustic alkali at 110–115°, removes the acyl group from the 5-nitrogen atom only, giving a reddish-brown vat dye. C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone series.** I. G. FARBENIND. A.-G. (B.P. 307,838, 12.3.29. Ger., 14.3.28).—Carbazoles from  $\alpha$ -amino- or  $\alpha\alpha'$ -diamino-1:1'-dianthraquinonylamines are condensed with halogenoanthraquinones to give vat dyes. Examples are: 5:5'-diamino- $\alpha\alpha'$ -dianthraquinonecarbazole with 2-bromo-4:3-*p*-tolylaziminoanthraquinone (powerful red); 4:4'-diamino- $\alpha\alpha'$ -dianthraquinonecarbazole with 2-chloroanthraquinone (corinth); 5-amino-4'-benzamido- $\alpha\alpha'$ -dianthraquinonecarbazole (cf. B.P. 307,306, preceding) with 4-chloro-1-benzamidoanthraquinone (black-brown). C. HOLLINS.

**Manufacture of vat dyes of the anthanthrone series.** I. G. FARBENIND. A.-G. (B.P. 307,481, 9.3.29. Ger., 10.3.28).—The halogen in mono-, di-, tri-, or tetrabromoanthanthrones is replaced by cyano-groups by heating with cuprous cyanide in benzyl cyanide. The product from 4:10-dibromoanthanthrone is a reddish-orange vat dye. C. HOLLINS.

**Manufacture of compounds of the pyrazolanthrone series and vat dyes therefrom.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,316, 1.5.29).—A 1-halogenoanthraquinone-2-carboxylic acid is condensed with hydrazine, preferably in presence of a diluent such as pyridine, to yield



pyrazolanthrone-2-carboxylic acid, m.p. above 300°, which is converted into the acid chloride and condensed with an aromatic hydrocarbon or halogenated hydrocarbon using an acid condensing agent, *e.g.*, thionyl chloride. By treatment with an acid-binding condensation agent these

derivatives yield dyes probably having the annexed constitution and dyeing cotton red shades from the vat. The pyrazolanthronecarboxylic acids or aroylpyrazolanthrones may be alkylated or aralkylated. L. J. HOOLEY.

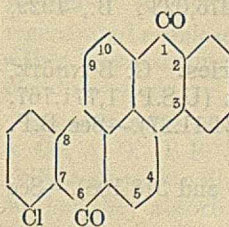
**Manufacture of pink to red [thioindigoid] vat dyes.** I. G. FARBENIND. A.-G. (B.P. 318,595, 6.9.28. Ger., 6.9.28).—A 6-halogeno-3-oxythionaphthen and a

4-methylthionaphthen, halogenated or not in the 6-position (one of these thionaphthens containing a 2-anil group) are condensed to form dyes giving shades faster to light and exposure and nearly as bright as those given by 6:6'-dihalogeno-4:4'-dialkylthioindigo dyes. 6:6'-Dichloro-, 6:6'-dibromo-, and 6'-chloro-4-methyl bithionaphthenindigos are exemplified.

L. J. HOOLEY.

**Manufacture of water-soluble leuco-esters of vat dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,579, 11.3.29. Addn. to B.P. 324,119; B., 1930, 364).—The leuco-compounds of vat dyes are esterified with sulphonyl halides of benzoic acid, preferably in presence of a tertiary base. *m*-Chloro-sulphobenzoic acid reacts, *e.g.*, in pyridine, with leucoindigo, indanthrone, or Caledon-jade-green, to give water-soluble esters which are hydrolysed to vat dyes by heating with alkali. C. HOLLINS.

**Manufacture of vat dyes [from halogenated dibenzpyrenequinones].** I. G. FARBENIND. A.-G. (B.P. 308,617, 23.3.29. Ger., 23.3.28).—Halogenated dibenzpyrenequinones are condensed with suitable amino-compounds (excluding amino-dibenzanthrones and -isodibenzanthrones) in presence of a solvent (nitrobenzene, *o*-cresol), sodium acetate or carbonate, and copper or copper acetate to give vat dyes. The products may be modified by heating with condensing agents. Examples are: bromo-2:3:6:7-dibenzpyrene-1:8-quinone with  $\alpha$ -aminoanthraquinone (dull red), heated with sodium aluminium chloride (red-brown); or with anthranilic acid (claret-red), heated with sulphuric acid (brown-orange); dibromo-2:3:7:8-dibenzpyrene-1:8-quinone with  $\alpha$ -aminoanthraquinone (violet); or with benzamide (violet); chloro-2:3:7:8-



dibenzpyrene-1:6-quinone (annexed formula) with phthalimide, heated with sulphuric acid (violet); or with benzamide (scarlet); tetrachloro-2:3:7:8-dibenzpyrene-1:6-quinone with  $\beta$ -aminoanthraquinone (brown-black); or with  $\alpha$ -aminoanthraquinone (blue-black); or with anthranilic acid (pure green), heated with sodium aluminium chloride or chlorosulphonic acid (brown). C. HOLLINS.

**Manufacture of monoazo dyes [for wool].** I. G. FARBENIND. A.-G. (B.P. 307,303, 4.3.29. Ger., 2.3.28).—*o*-Toluidines or *o*-anisidines, containing in position 4 or 5 an acylamino-group and in position 5 or 4 an alkoxy or (in the case of *o*-anisidines) a methyl group, are diazotised and coupled with 1:8-acylaminonaphtholmono- or -di-sulphonic acids to give red-violet to green-blue level wool dyes fast to alkalis and acids. Examples are: 4-amino-2:5-dimethoxyacetanilide  $\rightarrow$  acetyl-H-acid (reddish-blue); 3-amino-4:6-dimethoxyacetanilide  $\rightarrow$  acetyl-H-acid (violet); 5-acetamido-4-methoxy-*o*-toluidine  $\rightarrow$  acetyl-H-acid (reddish-violet); 4-amino-3-methoxy-6-ethoxyacetanilide  $\rightarrow$  *p*-toluenesulphonyl-H-acid (bluish-violet). [Stat. ref.] C. HOLLINS.

**[Tris]azo dyes and their application to the dyeing of regenerated cellulose rayon.** IMPERIAL



CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 330,607, 13.3.29).—A tetrazotised 3:4'-diaminoazobenzene is coupled with 2 mols. of the same or different coupling components (excepting hydroxynaphthyl aryl ketones and 2:3-hydroxynaphthoic arylamides) chosen so that the resulting dye contains at least two carboxylic or sulphonic groups or one of each. The same dyes may be produced from 3-nitro-4-aminoazobenzenes; they give level shades on viscose silk. Examples are: *m*-nitroaniline  $\rightarrow$  aniline  $\rightarrow$  salicylic acid, reduced,  $\rightarrow$   $\gamma$ -acid (reddish-brown); *m*-nitroaniline  $\rightarrow$  cresidine  $\rightarrow$  Schäffer acid, reduced,  $\rightarrow$  2S-acid (blue); 4-nitro-*o*-anisidine  $\rightarrow$  cresidine  $\rightarrow$  N.-W.-acid, reduced,  $\rightarrow$  2S-acid (greener-blue); 4-nitro-*o*-anisidine or *o*-toluidine  $\rightarrow$  aniline  $\rightarrow$  *o*-cresotic acid, reduced,  $\rightarrow$  phenyl- $\gamma$ -acid (in alkali; yellowish-brown); *m*-nitroaniline  $\rightarrow$  *o*-toluidine  $\rightarrow$  salicylic acid, reduced,  $\rightarrow$   $\gamma$ -acid (yellowish-brown); 3:4'-diamino-5'-methoxy-2'-methylazobenzene, m.p. 164–165°,  $\rightarrow$  1 mol. of  $\gamma$ -acid and 1 mol. of *N*-methyl- $\beta$ -naphthylamine-7-sulphonic acid or 2R-acid (brownish-black), or  $\rightarrow$  1 mol. of benzoyl- $\gamma$ -acid and 1 mol. of M-acid (violet-blue). The last-mentioned diazo component is made by reduction of 3-nitro-4'-amino-5'-methoxy-2'-methylazobenzene, m.p. 163°; 3-nitro-4'-aminoazobenzene, m.p. 208–209°, gives 3:4'-diaminoazobenzene, m.p. 210° (decomp.). C. HOLLINS.

**Vat dyes and process of making same.** B. MAYER and H. SIEBENBÜRGER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,771,802, 29.7.30. Appl., 23.11.26. Switz., 8.12.25).—See B.P. 262,774; B., 1928, 517.

**Manufacture of stable, dry, easily soluble leuco-preparations of vat dyes.** E. BAUDER, Assr. to J. R. GEIGY SOC. ANON. (U.S.P. 1,771,763, 29.7.30. Appl., 8.8.27. Ger., 16.8.26).—See B.P. 276,023; B., 1929, 124.

**Azo dyes of the pyrazolone series.** G. BONHÔTE, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,771,767, 29.7.30. Appl., 20.11.28. Switz., 24.11.27).—See B.P. 301,096; B., 1930, 454.

**Diazo-type prints** (B.P. 316,563 and 318,108).—See XXI.

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

**Photomicrographs of wool fibres: new method.** J. MANBY (J. Text. Inst., 1930, 21, T 231–233).—Mounting media, stains, and methods of illumination are described. A 3% solution of celluloid in amyl acetate, having  $n_D^{20}$  1.4049, is found to be the best medium, whilst celluloid in acetone, and celloidin in a mixture of absolute alcohol and ether, are not so good. Suitable stains are a 5% aqueous solution of acid-fuchsin, and a saturated aqueous solution of picric acid, to both of which 2% of acetic acid is added. B. P. RIDGE.

**Bamboo. II. Bamboo-lignin.** K. SISIDO (J. Cellulose Inst., Tokyo, 1930, 6, 167–170; cf. B., 1930, 812).—Bamboo is specially suitable for a study of lignin formation in vegetable material because the shoots grow extraordinarily quickly. Shoots 5.8 m. long were divided into three parts, each part being then dried and pulverised, and the dry powder extracted exhaust-

ively with alcohol-benzene and dried *in vacuo*. Lignin was removed with 72% sulphuric acid, and its methoxyl number determined by the Zeisel method. The methoxyl numbers for the samples were also calculated on the assumption that this number represents lignin. Both calculated and observed values show a decrease from the base to the tip of the shoots, but good agreement is not found. It is considered probable that in the samples more remote from the base of the shoot the whole of the methoxyl is not fixed to lignin, and it is concluded that the lignin is methylated gradually according to the growth of the bamboo, or combines with the residue which contains methyl in order to give the higher values determined. Some properties of bamboo-lignin have been compared with those of wood-lignin, and it is found that they behave similarly. Thus carbon, hydrogen, ash, and methoxyl contents are similar in amount to those from spruce-wood lignin. Also both spruce-wood and bamboo chips give a green coloration on immersion in concentrated hydrochloric acid, whilst, on separation, the lignin gives a greyish-violet coloration before, and a salmon-pink after, washing with boiling water. This suggests that bamboo-lignin also has a heterocyclic structure. The distribution of oxygen in the lignin as hydroxyl, methoxyl, etc. is discussed.

B. P. RIDGE.

**Suitability of Brazilian woods for pulp manufacture.** E. OFFERMANN and G. A. FELDTMANN (Papier-Fabr., 1930, 28, 461–471).—Experimental digestions of 14 Brazilian woods are described. The highest yields were obtained by the sulphite process, but the sulphate process gave more uniform and more readily bleached pulps. Paper-making trials were not practicable owing to lack of material, and the paper-making properties were judged by microscopical methods. Photomicrographs are given. T. T. POTTS.

**Wood-pentosan. I.** K. NISHIDA and T. TAKAGI (J. Cellulose Inst., Tokyo, 1930, 6, 170–173).—Wood-meals from a number of different coniferous and deciduous trees were extracted with 5% sodium hydroxide solution, the extract was treated with Fehling solution in order to separate pentosan as the copper compound, the latter was decomposed with hydrochloric acid, and the yield and composition of the pentosan were determined. The pentosan content (from the copper compound) of the deciduous woods examined is found to be characteristic, whilst coniferous woods contain much smaller quantities of pentosan. Frequently the wood-pentosan was converted into two different modifications; deciduous woods contain both the  $\alpha$ - and  $\beta$ -varieties, whilst only the  $\alpha$ -type is present in coniferous wood. The composition of the  $\beta$ -pentosan (% xylan etc.) from the deciduous woods examined is given; that of the  $\alpha$ -variety will be given in part II. B. P. RIDGE.

**Hygroscopic moisture of cellulose.** S. OGURI and M. NARA (J. Soc. Chem. Ind., Japan, 1930, 33, 267–270 B).—The water-absorption time curves have been determined for wood, bamboo, and cotton celluloses and for nitrocellulose, exposed to saturated water vapour at various temperatures. The equilibrium (maximum) moisture content depends on the origin of the cellulose, and decreases as the temperature rises; e.g., the



moisture content of standard cotton cellulose is 16.8% and 15.2% (dry basis) at 20° and 30°, respectively, which is less than that of bamboo or wood cellulose, and greater than that of nitrocellulose. The absorption is very slow.

E. LEWKOWITSCH.

**Viscose. XXXIV. Effect of chlorination on the properties of cellulose.** S. MASUDA and J. MURAKAMI (J. Cellulose Inst., Tokyo, 1930, 6, 173—174).—Previous work by the authors has shown that the properties of sulphite-pulp are improved by chlorination of the material at 15°, and similar results are now found for cotton. The copper number of the purified cotton cellulose used was reduced from 5 to 4 as a result of chlorination for 15 min. at 24° and washing with water, under standard conditions, and to 3 by similar chlorination for 30 min.

B. P. RIDGE.

**Imbibition phenomena and viscose silk.** A. WEHRUNG (Cellulosechem., 1930, 11, 170—173).—The difference in behaviour of viscose yarn obtained by variations of procedure is discussed from a theoretical point of view.

T. H. MORTON.

**Beating of [paper] pulp. XII. Improvement of absorbency of blotting paper.** M. NAKANO (J. Cellulose Inst., Tokyo, 1930, 6, 164—167; cf. B., 1930, 813).—Blotting papers of high absorbency are obtained by the use of pulps treated with concentrated sodium hydroxide solutions, and the absorbing velocity of such products is, on the average, about 40% higher than that of paper made from untreated pulp. Hence pulps can be transformed into "freer" states than their natural ones, and this effect is the reverse of that obtained by beating.

B. P. RIDGE.

**Determination of moisture in pulp and paper.** K. G. JONAS (Papier-Fabr., 1930, 28, Fest-u. Auslandsheft, 111—112).—Drying in a stream of dry air at 65° is preferred to oven-drying and distillation methods. Carbon tetrachloride used in distillation methods is held to vitiate results owing to the formation of hydrochloric acid.

T. T. POTTS.

**Examination of half-stuffs and paper with the quartz lamp.** H. G. KLEIN (Papier-Fabr., 1930, 28, 477—480).—The influence of moisture content, action of light, and chemical treatment on the fluorescence of half-stuffs and papers has been investigated, and tables showing the fluorescence colours of a number of samples are given. Slight changes in moisture content do not influence fluorescence greatly, whereas short exposures to light and heat have appreciable effect. The effect of chemical treatment on fluorescence is held to afford a means of distinguishing pulps prepared by the various processes.

T. T. POTTS.

**Use of dextrose in the manufacture of imitation parchment papers.** H. WREDE (Papier-Fabr., 1930, 28, Fest-u. Auslandsheft, 118—119).—The addition of 3% of dextrose to the beater furnish is claimed to improve manufacturing conditions and the quality of the finished sheet. The pulp hydrates more rapidly, less power is required for beating, greater transparency and gloss are obtained, and the sheet lies flat. The pliability of papers may be increased by spraying with dextrose solutions before calendering.

T. T. POTTS.

**Oxide inclusions as cause of working difficulties in copper half-stuffs.** R. W. SCHULZE (Dingler's Polytech. J., 1929, 344, 233—236; Chem. Zentr., 1930, i, 1209).

**Sinkage [of pulp-wood].** SCARTH and JAHN.—See IX.

#### PATENTS.

**Removing marks on wool.** G. J. M. and R. H. M. STEINS (U.S.P. 1,754,338, 15.4.30. Appl., 13.8.27. Belg., 18.8.26).—Wool is soaked at 30—70° in heavy tar oils (b.p. above 100°) and then extracted with a mixture of benzine and light petroleum.

F. R. ENNOS.

**Spools for textile industries.** I. G. FARBENIND. A.-G. (B.P. 311,287, 8.5.29. Ger., 8.5.28).—Metal or other spools are coated with cellulose esters which are resistant to acids and alkalis. The coating consists of one or more cellulose derivatives of fatty acids containing more than 5 carbon atoms, and is applied direct or over a coating of natural or artificial resin.

B. P. RIDGE.

**Manufacture of a cork product.** C. TECCA and C. R. DEL TURCO (B.P. 312,672, 30.5.29. Ital., 30.5.28).—Ground cork is mixed with 20—30 wt.-% of alkali in aqueous solution, and after 10—15 hrs. a further quantity of cork sufficient to neutralise the excess of alkali is added, together with fillers, colouring matter, etc. The plastic mass is moulded, stamped, or pressed and hardened by evaporation of the water.

F. R. ENNOS.

**Production from cellulose hydrates of bodies such as capsules, foils, tubes, etc. which are capable of reversibly swelling.** WOLFF & Co. KOMM.-GES. AUF AKT., and R. WEINGAND (B.P. 316,580, 29.7.29. Ger., 31.7.28).—Such articles, made from regenerated cellulose, are treated with a sucrose solution either before drying or after pre-drying until shrinking commences.

B. P. RIDGE.

**Manufacture of cellulose hydrate films.** KALLE & Co. A.-G. (B.P. 312,076, 16.5.29. Ger., 19.5.28).—Cellulose hydrate films are freely suspended in moist air so that they absorb water, and are afterwards dried by suspending in air under normal atmospheric conditions, whereby their internal stresses become compensated whilst the material remains smooth.

F. R. ENNOS.

**Manufacture of viscose.** F. STEIMMIG, and ERSTE BÖHMISCHE KUNSTSEIDEFABR. A.-G. (B.P. 332,628, 28.2.29).—Organic substances such as multivalent alcohols, sugars, etc., which serve to coagulate viscose and diminish the oxidation of the hydrogen sulphide produced, and sulphites, amides, or imides, which retard ripening, or arsenites, which do not promote ripening, are added to the viscose before spinning. [Stat. ref.]

B. P. RIDGE.

**Manufacture of fibres, ribbons, films, or the like from viscose.** I. G. FARBENIND. A.-G. (B.P. 309,147, 5.4.29. Ger., 5.4.28. Addn. to B.P. 306,971; B., 1930, 74).—An alkali salt of an organic oxy-acid (citric, tartaric, lactic), instead of oxalic acid, is added to the viscose, which is precipitated by means of a solution of sulphuric acid and sodium sulphate.

F. R. ENNOS.



**Manufacture of artificial silk.** L. M. V. GROS-LAFOND, V. M. L. LAFOND, E. M. V. LAFOND-PANSU, and P. BANZET (B.P. 308,267, 19.3.29. Fr., 20.3.28).—Artificial silk threads are dry-stretched on bobbins to an extent sufficient to ensure that they pass beyond their so-called "dead-zone," in which they have no elasticity. [Stat. ref.] B. P. RIDGE.

**Replacing the water in a water-moist cellulose by an organic liquid.** E. H. A. RÖST-GRANDE and E. CORRENS, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,752,596, 1.4.30. Appl., 16.3.27. Ger., 17.3.26).—A water-miscible organic liquid (glacial acetic acid) is forced through a cake of moist cellulose; the acetic acid may then be expelled by means of another organic liquid (benzene) under pressure, which is miscible with the glacial acetic acid but immiscible with water.

F. R. ENNOS.

**Manufacture of artificial yarns, threads, or filaments.** BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 332,527, 21.3.29).—The residual solvent contained in the artificial yarns is extracted by subjecting the material immediately after spinning to the action of a vacuum at 60–70°, the solvent so removed being recovered in a suitable plant. The material is subsequently conditioned while still in the vacuum chamber by the admission of a humidified atmosphere.

B. P. RIDGE.

**Production of cellulose acetate.** A. RUSSELL and J. F. HEGEMAN (U.S.P. 1,752,853, 1.4.30. Appl., 15.2.29).—Cellulose is acetylated with acetic anhydride, acetic acid, and sulphuric acid under a pressure of 50–75 lb./in.<sup>2</sup>, the temperature being gradually raised from about –5° to 40°, and the subsequent hydration is effected by adding sufficient water and acid at 100–200 lb./in.<sup>2</sup>, while maintaining the temperature. F. R. ENNOS.

**Preparation of cellulose acetate.** RUTH-ALDO Co., Assees. of H. L. BARTHELEMY (B.P. 305,096, 28.1.29. U.S., 30.1.28).—Cellulosic material is submitted first to direct oxidation, *e.g.*, by alkali or hydrogen peroxides, persalts, in the presence of moderating agents, *e.g.*, alkali carbonates, silicates, soaps, or sulphuric acid, and afterwards to a softening treatment with at least 50 wt.-% of hot vapours of acetic acid containing a small amount of halogen. The product is then acetylated in at least four stages by additions of acetic acid, acetic anhydride, and sulphuric acid so that in the earlier stages (up to formation of cellulose acetate of acetyl value about 25%), during which a small quantity of halogen or oxidising agent is added to reduce the amount of sulphuric acid required, only slight rises of temperature occur (up to 18–26°), whilst in the later stages the temperature is allowed to rise to 55–65°. The proportion of catalyst (sulphuric acid) in the successive baths is increased progressively, whilst that of the acetic anhydride is reduced until the final stage when the bulk is added. Finally, the acetic ester is partly saponified and the sulphuric esters are decomposed by adding aqueous formic or acetic acid, first alone and later with addition of hydrochloric and hydrofluoric acid equivalent to the quantity of sulphuric acid used as catalyst, the amount of water in the final mixture being adjusted to 5–15% of the total organic acids present.

F. R. ENNOS.

**Cellulose acetate compositions.** E. I. DU PONT DE NEMOURS & Co. (B.P. 306,911, 27.2.29. U.S., 27.2.28).—Carboxylic esters of a phenoxyethyl alcohol, *e.g.*,  $\beta$ -phenoxyethyl phthalate (or laurate) or its methyl derivative, are added as softeners or plasticisers for cellulose acetate. The products are suitable for the manufacture of lacquers, leather-cloth, etc.

B. P. RIDGE.

**Treatment of cellulosic materials and production of cellulose esters therefrom.** BRIT. CELANESE, LTD. From CELANESE CORP. OF AMERICA (B.P. 332,607–8, 17.4.29).—Purified cellulosic materials are more readily esterified if they are treated (A) first with organic anhydrides (acetic anhydride), either alone or mixed with a liquid hydrocarbon, or in vapour form alone or mixed with an inert diluent, and then with an organic acid with or without anhydride (*e.g.*, acetic acid and acetic anhydride), or (B) first with one or more organic acids (*e.g.*, formic or acetic), alone or mixed with inert diluents, and then with one or more organic anhydrides, without or with organic acids, a condensing agent being present in each case, if desired.

B. P. RIDGE.

**Manufacture of cellulose ethers.** DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 306,125, 21.1.29. Ger., 17.2.28).—Cellulose ethers, insoluble in pure solvents but soluble in mixed solvents, such as benzene-alcohol or dichloroethylene-alcohol, are produced by treatment of alkali-cellulose with alkyl halide in the presence of water but without further addition of caustic soda.

B. P. RIDGE.

**Manufacture of cellulose ether esters.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 331,903, 28.3.29).—A cellulose ether containing about  $\frac{1}{2}$  to  $1\frac{1}{2}$  alkyl groups for each  $C_6H_{10}O_5$  molecule, including 0.5–2% of benzyl or substituted benzyl cellulose, is acetylated by means of acetic anhydride in the presence of acetic acid as solvent and a small quantity of a catalyst (0.1–3% of sulphuric acid, or 10–30% of zinc chloride, calc. on the cellulose ether).

F. R. ENNOS.

**Manufacture of soluble cellulose esters of higher organic acids.** G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 330,575, 11.3.29).—Cellulose is esterified by treatment with anhydrides of acids above  $C_7$ , preferably in presence of an aliphatic hydrocarbon sulphonic acid (methanesulphonic acid) and a solvent (carbon tetrachloride, toluene) at moderate temperatures (15–65°). Esterification is facilitated by a pretreatment, *e.g.*, with dichlorohydrin at 90° for 16 hrs., with chloroacetic acid at 80–90°, or with vapours of the appropriate acid. The acid anhydrides exemplified are lauric, palmitic, stearic, and phenylacetic; the esters obtained soften or melt at 65–135° without decomposition.

C. HOLLINS.

**Manufacture of shaped objects from cellulose derivatives.** I. G. FARBENIND. A.-G. (B.P. 315,275, 10.7.29. Ger., 10.7.28).—Cellulose derivatives of naphthenic acid, or of saturated or unsaturated higher fatty acids containing more than 8 carbon atoms, are rolled or calendered at a raised temperature before being shaped. This treatment increases the elasticity of the material and permits of shaping and bending without loss of its pliancy.

B. P. RIDGE.



**Manufacture of paper.** KODAK, LTD., Assees. of G. T. LANE (B.P. 332,743, 22.7.29. U.S., 1.3.29).—Size consisting of a saponified saturated fatty acid of high mol. wt. (e.g., stearic or palmitic acid) and having an iodine value below 40 (preferably below 5), is added to the paper pulp in the beater and precipitated on the fibres by the addition of aluminium or magnesium sulphate, or of potassium or chrome alum. Paper so produced causes no appreciable fogging of a photosensitive emulsion kept in contact with it, and has improved strength and flexibility, a smooth surface, and high resistance to chemicals, moisture, oils, heat, and light.

B. P. RIDGE.

**Cellulose solutions.** L. LILIENFELD (U.S.P. 1,771,460—2, 29.7.30. Appl., 10.5.24. Austr., 5.6., 25.5., and 5.6.23).—See B.P. 217,166 and 216,475; B., 1924, 977; 1925, 37.

**Isolation of benzylcellulose.** E. DORR, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,771,529, 29.7.30. Appl., 20.9.26. Ger., 17.8.25).—See B.P. 265,491; B., 1927, 296.

**Spinning boxes for use in the manufacture of artificial silk etc.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of COMP. FRANÇ. POUR L'EXPLOIT. DES PROC. THOMSON-HOUSTON (B.P. 310,009, 19.4.29. Fr., 19.4.28).

**Treating and utilising the black liquor obtained in the soda-pulp process.** E. HÄGGLUND (U.S.P. 1,772,216, 5.8.30. Appl., 13.7.25. Ger., 16.7.24).—See B.P. 258,035; B., 1927, 165.

**Centrifugal machines (B.P. 332,288). Loop dryer for fabrics (U.S.P. 1,755,005—6).**—See I. Rubberised fabric (B.P. 331,594).—See XIV.

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

**Influence of  $p_H$  on dyeing phenomena.** I. Uneven dyeing of viscose. II. Influence of  $p_H$ , salt, and variations of temperature in the dyeing of viscose with direct dyes. III. Dyeing viscose, cotton, and mercerised cotton in the same bath. IV. Rinsing of dyed cotton. V. Light-fastness of dyed cotton. C. E. MULLIN (Tiba, 1929, 7, 853—867).—The necessity for control of the  $p_H$  in dyeing and rinsing is exemplified, and the effect of addition of sodium chloride and of temperature on shade is discussed.

CHEMICAL ABSTRACTS.

### PATENTS.

**Rapid bleaching of vegetable fibres.** C. G. SCHWALBE and H. WENZL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,754,909, 15.4.30. Appl., 19.1.25. Ger., 15.7.21).—The fibres are rapidly bleached by impregnation with a hypochlorite solution containing 10—30 g. of active chlorine per litre instead of the usual 1 g. per litre, then pressed to remove excess liquor, and shortly afterwards washed; the material may be steamed before, during, or after bleaching.

A. J. HALL.

**Bleaching and stripping dyes from textile fibres and materials.** BRIT. RES. ASSOC. FOR THE WOOLLEN & WORSTED INDUSTRIES, and A. T. KING

(B.P. 332,389, 6.7.29).—Wool materials are treated with a cold or warm solution containing mixtures of sodium bisulphite and sulphite such that the mol. ratio  $SO_2/NaOH=1/1.25-1.35$  (substantially  $2NaHSO_3.Na_2SO_3$ ); stripping is particularly efficient with azo dyes having an  $\alpha$ -naphthol or substituted  $\alpha$ -naphthol component. Examples of dyes not stripped by acidified bisulphite, but stripped by this new process, are Carmoisines WS and L9156K, Benzyl Bordeaux B, and Coomassie Scarlet 9012K.

A. J. HALL.

**Bleaching of fabrics in open width.** F., E., and P. V. BARKER (B.P. 332,718, 3.7.29).—Fabric is suspended in open width in festoons (looped form) on a wagon which can be successively run into kiers and washing chambers where the fabric is subjected first to showers of boiling alkali-liquor, and then to washing, chemicking, souring, and washing liquors.

A. J. HALL.

**[Preparation of] non-aqueous dye-bath.** E. G. SCHLATTER, Assr. to DIP-IT, INC. (U.S.P. 1,754,285, 15.4.30. Appl., 13.3.26).—Solutions of acid, basic, or direct dyes in glycerin are diluted with an organic liquid (e.g., an aliphatic alcohol or acetone) which is a solvent for glycerin but not for the dye.

A. J. HALL.

**Immunising textiles to direct-dyeing dyestuffs.** SOC. CHEM. IND. IN BASLE (B.P. 315,434, 13.7.29. Switz., 13.7.28).—Vegetable fibres are immunised by esterification with maleic anhydride without a catalyst.

A. J. HALL.

**Dyeing shearlings [wool skins] and the like.** J. G. SHOEMAKER, Assr. to A. C. LAWRENCE LEATHER CO. (U.S.P. 1,754,287, 15.4.30. Appl., 4.4.28).—Wool skins or furs are given a resist of molten tallow or wax (preferably a tallow of m.p.  $43^\circ$  is applied at  $49^\circ$ ), mordanted in the usual manner at a moderate temperature, dyed, and washed.

A. J. HALL.

**Vat dyeing.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,563, 7.3.29).—Vat dyes may be vatted by titanous compounds in alkaline medium. Preferably a complex salt, formed, e.g., by addition of sodium formate, lactate, or tartrate to titanous chloride solution, is used, or the alkali and the titanous salt are mixed in presence of glue or other protective colloid.

C. HOLLINS.

**Colouring of wool and other animal fibres [with vat dye sulphonic acids].** J. S. WILSON, L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 332,232, 16.1.29).—Sulphonic acids of vat dyes (indanthrone, Caledon Red BN, Caledon Brilliant Purple RR) are dyed on wool or other animal fibres by a vatting process as distinct from an acid process. The increased solubility given by the acid group appears to aid the dyeing of animal fibres from the vat. The sulphonic acids may be prepared in stable soluble leuco-form.

L. J. HOOLEY.

**Manufacture of evenly-dyed viscose fabrics.** I. G. FARBENIND. A.-G. (B.P. 330,592, 12.3.29. Addn. to B.P. 306,908; B., 1930, 859).—Viscose silk is dyed evenly with the dyes: 5-nitro-*o*-aminophenol  $\rightarrow$   $\beta$ -naphthylamine-5-sulphonic acid, prechromed (green);



4-chloro-*o*-aminophenol-6-sulphonic acid  $\rightarrow$  1-*m*-sulphophenyl-3-methylpyrazolone, prechromed (pink); 1:2:4-aminonaphtholsulphonic acid  $\rightarrow$   $\alpha$ -naphthol-8-sulphonic acid, prechromed (blue); anthranilic acid  $\rightarrow$   $\beta$ -naphthylamine-7-sulphonic acid, prechromed (violet); 5-nitro-*o*-aminophenol  $\rightarrow$  *p*-anisyl-J-acid, zinc compound (violet); 5-nitro-*o*-aminophenol  $\rightarrow$  *p*-phenetyl-J-acid, copper compound (reddish-blue). C. HOLLINS.

**Coloration of materials made of or containing cellulose esters or ethers.** BRIT. CELANESE, LTD., and H. C. OLPIN (B.P. 330,591, 12.3.29).—Acetate silk is dyed with azo dyes containing an *ar*-tetrahydronaphthyl group, preferably having no sulphonic group. Examples are: *ar*-tetrahydro- $\beta$ -naphthylamine  $\rightarrow$  phenol (yellow), or 2:3-hydroxynaphthoic *m*-nitroanilide (orange); *p*-nitroaniline  $\rightarrow$  *ar*-tetrahydro- $\alpha$ - or - $\beta$ -naphthylamine (orange or golden-yellow), *m*-nitroaniline-*ar*-tetrahydro- $\alpha$ -naphthylamine (marigold); *p*-nitro-*ar*-tetrahydro- $\alpha$ -sulphonic acid  $\rightarrow$  *ar*-tetrahydro- $\alpha$ -naphthylamine (orange-red). C. HOLLINS.

**[Photochemical] dyeing and figuring of woven, knitted, and plaited fabrics, leather, and other fibrous material.** W. HAENDEL (B.P. 309,166, 8.3.29. Ger., 7.4.28).—Light-sensitive diazosulphonic acids are applied locally to the material, *e.g.*, by printing, with or without simultaneous application of the coupling components, and the material is then exposed to light and the colour developed. A series of different colours may be successively printed, each being developed before the next printing. The diazosulphonates are made by addition of sodium sulphite to the diazonium chlorides from aniline (with phenol for yellow), benzidine (with resorcinol for red), tolidine (with 2S-acid and  $\beta$ -naphthylamine for blue). The depth of shade may be varied by varying the concentrations or the length of exposure to light. C. HOLLINS.

**Manufacture of azo dyes on the fibre [stable diazo preparations].** I. G. FARBENIND. A.-G. (B.P. 306,844, 25.2.29. Ger., 25.2.28).—The compounds of diazo- or tetrazo-compounds with secondary amines (piperidine, dialkylamines) prepared by the method of Baeyer and Jäger (A., 1875, i, 273) or Wallach (A., 1887, 137) are stable in aqueous solution even at 100°, but yield diazo solutions when treated with cold dilute acid. They are used for development of ice-colours; *e.g.*, cotton padded with 2:3-hydroxynaphthoic  $\alpha$ -naphthylamide is treated with an acidified solution of the piperidine compound of diazotised *m*-nitro-*o*-toluidine (for red) or of tetrazotised dianisidine (for blue). C. HOLLINS.

**Manufacture of an azo dye on the fibre [ice colour].** I. G. FARBENIND. A.-G. (B.P. 307,704, 11.3.29. Ger., 10.3.28).—2:3-Hydroxynaphthoic  $\beta$ -naphthylamide is coupled on the fibre with diazotised 2:4-dichloro-6-amino-*m*-cresol methyl ether to give a bluish-red fast to light and to kier-boiling with 0.4% sodium hydroxide solution. C. HOLLINS.

**Production of azo dyes on the fibre [ice colours].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 331,056, 23.5.29. Cf. B.P. 286,274; B., 1929, 675).—

In the production of blue ice-colours from 4-amino-diphenylamines and 2:3-hydroxynaphthoic arylamides, the diazo solution is mixed with excess of (acetic) acid and after development the goods are passed through an alkaline bath (hot aqueous sodium carbonate).

C. HOLLINS.

**Dyeing and printing textile goods etc. [with solubilised vat dyes].** DURAND & HUGUENIN SOC. ANON. (B.P. 310,478, 26.4.29. Ger., 26.4.28).—The development of the shade by oxidation of solubilised vat dyes (B.P. 220,964; B., 1925, 879) printed on textiles is effected by drying in presence of a copper salt, either before or at the time of printing.

A. J. HALL.

**Dyeing and printing with [leuco-ester] vat dyes.** DURAND & HUGUENIN SOC. ANON. (B.P. 319,021, 14.9.29. Ger., 15.9.28).—Development of leuco-ester salts of vat dyes is effected with iron alum, particularly ammonium iron alum. These developers are suitable for all esters on animal fibres and for esters having sufficient affinity on vegetable fibres. They avoid over-oxidation with dyes such as Indanthrene Blue RS, and as they can be used without addition of acid, less water is required in rinsing.

L. J. HOOLEY.

**Production of fast dyeings and prints [with indigosols etc.].** DURAND & HUGUENIN SOC. ANON., Assees. of J. C. LIVINGSTON (B.P. 314,350, 25.6.29. Ger., 25.6.28).—The acid oxidising conditions necessary for developing leuco-ester salts of vat dyes are produced by electrolysis (cf. B.P. 203,681; B., 1924, 982).

L. J. HOOLEY.

**Discharge printing.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,646, 7.3.29. Cf. B.P. 330,563; B., 1930, 901).—A titanous salt, preferably a complex salt such as may be obtained by addition of sodium formate, tartrate, or lactate to aqueous titanous chloride, or, if a simple salt be used, with the addition of a protective colloid such as sulphite-cellulose liquor, is employed as reducing agent in alkaline discharge pastes, especially for vat dyes. The presence of iron is not objectionable.

C. HOLLINS.

**Discharge printing on wool.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,202, 15.4.29).—Wool is dyed with a monoazo dye obtained by coupling diazotised *p*-nitroaniline-*O*-sulphonic acid with a  $\beta$ -naphthylaminesulphonic acid or *N*-alkyl derivative thereof, *e.g.*,  $\beta$ -methylanilinonaphthalene-7-sulphonic acid, and discharge-printed in the usual way.

L. J. HOOLEY.

**Production of printed [cellulose ester] fabrics.** BRIT. CELANESE, LTD. (B.P. 317,455, 16.8.29. U.S., 16.8.28).—Fabrics with warp print effects are obtained by weaving a warp or weft of a treated (*e.g.*, hydrolysed) cellulose ester with an untreated ester and then printing a dye with an affinity for only one of the esters.

L. J. HOOLEY.

**Apparatus for mercerising textile fabrics.** W. MYCOCK & Co., LTD., and W. MYCOCK (B.P. 332,751, 31.7.29).

**Dyeing of acetate silk** (B.P. 330,602). **Trisazo dyes on rayon** (B.P. 330,607).—See IV.



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

**Chamber [sulphuric acid] process.** XX. Empirical formula and nomogram for composition of nitrous vitriol. M. MATSUI (J. Soc. Chem. Ind., Japan, 1930, 33, 248—251B; cf. *ibid.*, 1925, 28, 165 B).—If  $H$  is the percentage sulphuric acid content,  $N$  the content of sodium nitrate in g. per litre, and  $p$  and  $T$  are the nitrous acid tension and temperature, respectively, then  $H = 5.26 \log N - 5 \log p - 10,105/T + 96.710$ . A nomogram is constructed for the solution of this equation within the limits for  $H$ ,  $N$ , and  $T$  met with in practice.

C. IRWIN.

**Manufacture of synthetic nitric acid.** V. I. MALYAREVSKI and V. V. PAPKOV (J. Chem. Ind., Russia, 1929, 6, 849—853).—The operation of the Libinon process under 10—30 atm. pressure is advocated.

CHEMICAL ABSTRACTS.

**Extraction of phosphoric acid from low-grade phosphorites with a mixture of sulphuric acid and ammonium sulphate.** S. I. VOLFKOVICH and L. E. BERLIN (Min. Syr. Tzvet. Met., 1929, 4, 396—407).—The optimal mixture contains 16.6—22.2% of ammonium sulphate, which diminishes the amount of lower oxides extracted.

CHEMICAL ABSTRACTS.

**Change of water-soluble phosphoric acid content of superphosphate during storage in bulk. Effect of temperature.** T. SHOJI, E. SUZUKI and S. HIRABAYASHI (J. Soc. Chem. Ind., Japan, 1930, 33, 204—206 B).—Superphosphate made from Christmas Island rock showed no loss in water-soluble phosphate when stored in bulk in winter when the temperature in the interior of the heap was always below 40°. In summer pyrometers in different positions showed temperatures between 50° and 60°, and it was found that while material on the surface was unchanged, loss of water-soluble phosphate had occurred in the interior in proportion to the temperature reached. Laboratory experiments showed that a slow degradation also occurs at or below 40°.

C. IRWIN.

**Economics and developments of ammonia production.** E. A. THAU (Gas- u. Wasserfach, 1930, 73, 767—775).—A review of the present position concerning by-product and synthetic ammonia, obtained by a variety of methods. Attention is called to the influence of the cost of sulphuric acid on the ammonium sulphate process.

R. H. GRIFFITH.

**Special hydrometer for aqua ammonia.** D. F. FARRAR (Ind. Eng. Chem. [Anal.], 1930, 2, 293).—A hydrometer graduated in percentages of ammonia, ranging from 18 to 28%, with attached thermometer and a chart for temperature corrections has been constructed.

E. S. HEDGES.

**Manufacture of sodium hydroxide from sodium sulphate.** V. I. KURIKOV (J. Chem. Ind., Russia, 1929, 6, 119—120).—A mixture of wood charcoal and barium sulphate is calcined at 1000—1200°, the barium sulphide treated with steam at a temperature above 100°, and the barium hydroxide solution added to boiling sodium sulphate solution.

CHEMICAL ABSTRACTS.

**Pyrogenic decomposition of gypsum.** K. I. LOSEV and S. N. NIKITIN (J. Chem. Ind., Russia, 1929,

6, 169—173).—So-called fusion of gypsum is an optical illusion. Decomposition is noticeable above 1000° and maximal at 1420°; the decomposition temperature may be lowered by addition of silica, alumina, or ferric oxide. Reduction by charcoal is best effected at 1000°.

CHEMICAL ABSTRACTS.

**Manufacture of bleaching powder.** M. N. GURVICH, G. A. DMITRIEV, B. A. SASS-TISOVSKI, and D. V. STEPANOV (J. Chem. Ind., Russia, 1929, 6, 853—858).—The lime was calcined at 1000—1100° and slaked with 3 pts. of water; at the optimal rate of passage of chlorine into milk of lime an easily filterable paste is obtained. The temperature of the external bath should be 14°. The filtered, pressed, and dried precipitate contains 52—55%, and the filtrate 6—7%, of active chlorine.

CHEMICAL ABSTRACTS.

**Identification of barytes, quartz, and silicates.** H. WAGNER (Farben-Ztg., 1930, 35, 2174—2175).—The barium flame test as a means of identifying barytes is not satisfactory. Barytes can be distinguished from gypsum, but not from quartz, by its appearance in polarised light. Adsorption of dyestuff (brilliant-green) is not a successful means of identification. The immersion of the specimen in a refractive medium and examination under a low microscopical magnification is advocated. With phenylthiocarbimide as the medium barytes appears blue and can be clearly identified, although the method may fail under some special conditions.

J. O. CUTTER.

**Manufacture of ferric chloride.** I. E. ADADUROV (J. Chem. Ind., Russia, 1929, 6, 203—204).—A mixture of iron filings (20 pts.) and charcoal (1 pt.) is heated in an iron pipe in a current of chlorine containing a little air. The yield of ferric chloride is 83—92%. Pyrite residues from the manufacture of sulphuric acid may be used (yield 85—92% of the theoretical), provided that the sulphur content does not exceed 1.5%.

CHEMICAL ABSTRACTS.

**Process of decomposing chromite.** Y. KATO and R. IKENO (J. Soc. Chem. Ind., Japan, 1930, 33, 225—226 B).—Methods which have been proposed for the extraction of chromic oxide from chromite are reviewed.

C. IRWIN.

**Preparation of pure chromic anhydride.** V. V. POLYANSKI (Trans. Inst. Pure Chem. Reagents, Sci. Tech. Dept. U.S.S.R., 1929, No. 300, 143—154).—Calcium chromate is prepared by addition of calcium hydroxide paste, followed by calcium chloride solution, to aqueous sodium dichromate; the mixture is concentrated on the water-bath, the precipitate of calcium chromate being collected and dried. A solution in water (2 pts.) is treated with sulphuric acid ( $d$  1.84, 1 pt.), kept on the water-bath, decanted, and this procedure repeated until no more calcium sulphate is precipitated, when chromic anhydride is crystallised. The product is treated with nitric acid,  $d$  1.4, and dried at 60—100°.

CHEMICAL ABSTRACTS.

**Utilisation of the gases produced by distillation of phosphorus in the blast furnace.** E. V. BRITZKE, N. E. PESTOV, and N. N. POSTRIKOV (Min. Syrje Zwet. Met., 1929, 4, 375—387; Chem. Zentr., 1930, i, 1020—1022).—The difficulties encountered in Liljenroth's process, and due to the poisoning of the metallic catalysts



by phosphorus, phosphine, etc., are avoided by using phosphorite, limestone, sylvine, and other natural rocks as catalysts, the phosphoric acid formed by the action of water vapour being absorbed at high temperatures. Phosphite is not produced. Experiments in which a mixture of carbon dioxide and water vapour was passed through a vessel containing molten phosphorus and then through a heated porcelain tube containing the catalyst are described; phosphoric acid fog was precipitated electrostatically. In the absence of a catalyst and at 800° phosphoric, phosphorous, and hypophosphorous acids were obtained, the relative proportions depending on the experimental conditions, together with hydrogen and phosphine. Phosphoric acid free from phosphorous and hypophosphorous acids is formed only in presence of catalysts at about 600°. When the ratio of phosphorus to water is 1:150 the hydrogen contains no phosphine. Oxidation of phosphorus by carbon dioxide proceeds analogously. The gaseous products always contain carbon monoxide and dioxide, even when excess of phosphorus is employed; the solid products consist of oxides of phosphorus and (with deficiency of carbon dioxide) elementary phosphorus. At normal pressure the reaction begins at 800°. For ratios  $P : CO_2 = 1 : 22-15$  the chief reaction is  $P_4 + 8CO_2 = P_4O_{10} + 8CO$ . Phosphine is oxidised by water vapour or carbon dioxide in an analogous manner. When a copper-nickel-pumice (Liljenroth) catalyst was used at 600° the acids contained  $H_3PO_4$  98.75,  $H_3PO_3$  0.91,  $H_3PO_2$  0.34%; with active carbon (Urbain) reaction commenced at 400°. The use of phosphorite (composition recorded) in granular form leads by absorption of phosphoric acid to the production of a compact mass; chalk does not suffer from this disadvantage. The limiting velocity to avoid the collection of phosphorus and phosphine is the greater the smaller are the granules, and falls as the carbonate is converted into metaphosphate. At high temperatures chalk and limestone, at low temperatures active carbon and copper-nickel-pumice, give the best results. In presence of manganese dioxide the activity of the chalk catalyst at 500–600°, but not at higher temperatures, was somewhat increased.

[With M. N. BOLOTIN.] The calcium metaphosphate was converted, in the same apparatus, into  $CaH_4(PO_4)_2$  by treatment with orthophosphoric acid and steam.

A. A. ELDRIDGE.

**Petroleum method of iodine extraction.** O. Y. MAGIDSON (J. Chem. Ind., Russia, 1929, 6, 699–702).—Polemical against Panteleimonov (B., 1929, 812). The presence of nitrites or of considerable acidity does not permit the direct titration of iodine with thiosulphate.

CHEMICAL ABSTRACTS.

**Soluble copper from ores.** SULLIVAN and SWEET; GUGGENHEIM and SULLIVAN.—See X. **Analysis of red lead.** AMPT.—See XIII. **Phosphate fertilisers.** VOLFKOVICH and KAMZOLKIN. **Natural aluminium phosphate.** KIDA.—See XVI.

PATENTS.

**[Catalyst for] oxidation of ammonia.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,728, 17.8.29).—The catalyst comprises platinum-gauze netting

which has been coated with finely-divided rhodium by spraying it with an alcoholic solution of rhodium trichloride and heating in a hydrogen flame or by electro-deposition from a 1% solution of sodium chlororhodate.

A. R. POWELL.

**Regeneration of carbonated ammoniacal solutions.** SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE & PROD. CHIM.), ASSRS. TO L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 306,947, 14.2.29. Fr., 29.2.28).—The solution is preheated in a heat exchanger by means of regenerated solution to about 65°, at which temperature it does not dissociate. It then passes down a plate column in countercurrent to vapours from solution undergoing dissociation and becomes rectified, while at the same time it is heated by regenerated solution in the bottoms of the plates. From the bottom of the column it then passes to a compartment where it is heated by a steam coil, and thence is pumped into the bottoms of the plates in an opposite direction to that of the ascending solution. It is thus again brought to 65°, and passes from the top of the tower to the heat exchanger, where, in its turn, it serves to preheat entering solutions to be regenerated.

W. J. WRIGHT.

**Kilns or furnaces for calcining, burning, or roasting limestone, iron ore, chalk, etc.** C. F. PRIEST (B.P. 332,037, 25.6.29).—In vertical kilns or furnaces the burners are cooled by water-jackets or other means, and project transversely through the wall into the interior, the orifices in the burners being directed vertically downwards. The combustible gases are thus brought into intimate contact with the whole of the material in the kiln.

W. J. WRIGHT.

**Working-up of natural and industrial salt mixtures.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,236, 25.3.29).—Potassium chloride or nitrate or sodium nitrate may be extracted from admixture with other salts, e.g., sodium chloride, by treatment of the mixture with liquid ammonia. A. R. POWELL.

**Production of barium monoxide.** G. T. SHINE (B.P. 331,878, 11.2.29).—Reduction of barium carbonate is effected continuously in a series of chambers of refractory material, placed in an elongated retort setting having a central main flue extending through the upper portion. This flue communicates with inlet ducts at each end, through which producer gas is admitted, and with a central exit for exhaust gases. The chambers are charged at the top and have movable bases for discharging, the charging apertures opening into an air-duct of heat-insulating material through which a current of cold air is caused to pass during the charging of the chambers. By this means gases are carried off, and continuous charging can be effected. W. J. WRIGHT.

**Manufacture of calcium hypochlorite.** R. B. MACMULLIN and J. A. GUYER, ASSRS. TO MATHIESON ALKALI WORKS, INC. (U.S.P. 1,754,473, 15.4.30. Appl., 4.8.26).—Calcium hypochlorite, formed by chlorination of a lime slurry, is salted out by the addition of sodium chloride. The hypochlorite is separated by filtration or other means, and the solution remaining is treated with lime to precipitate basic calcium hypochlorite,



which may be used for making up lime slurry. Part of the solution, after addition of sodium chloride, may be used as a diluent in adding the sodium chloride to the lime slurry, thereby giving a thinner slurry and promoting the chlorination. W. J. WRIGHT.

**Production of clear leach liquors.** A. W. ALLEN (U.S.P. 1,731,450, 15.10.29. Appl., 24.11.25).—Extraction of nitrate from caliche is improved by promoting the formation and distribution of small crystals of sodium chloride, which act as a filter and assist the retention of slime, so that the effluent solution is free from suspended matter. To ensure formation of these crystals, the caliche must be finely crushed so as to restrict and cause more even distribution of the voids between the particles, and to form channels in which the crystals will be deposited from the leaching solution. The solvent is heated out of contact with the charge in order to prevent release of slime, and leaching is effected in an upward direction through a false bottom. W. J. WRIGHT.

**Production of molybdates of alkaline-earth metals and of magnesium.** I. G. FARBENIND. A.-G. (B.P. 331,472, 27.11.29. Ger., 8.12.28).—Roasted molybdenite is made into a pulp with water and milk of lime added at such a rate that the solution always remains only feebly alkaline. Lime may be replaced by baryta, strontia, or magnesia. A. R. POWELL.

**Preparation of highly-activated iron oxide.** G. E. SEIL (B.P. 332,259, 15.4.29).—Iron, or an iron-bearing material such as aniline sludge, is intimately mixed with an alkaline substance such as "fished salt," which consists of potassium and sodium carbonates. The mixture is introduced into a rotary kiln and heated either at 200–400° or, preferably, at 760–1200°. At these ranges of temperature a product of high activity is obtained, whereas at 400–760° only low activity results. The hot mixture is quenched with water and finely ground in a ball-mill while still wet, the ferric oxide and other insoluble material being subsequently filtered off. W. J. WRIGHT.

**Manufacture of aluminium chloride.** A. M. BULEY and H. BLUMENBERG, JUN. (U.S.P. 1,734,200, 5.11.29. Appl., 31.8.26).—Aluminium together with charcoal or other reducing agent, added for the purpose of combining with oxygen present, is introduced into a rotatable cylindrical container having radial blades to promote agitation. The mixture is heated to 700° to melt the aluminium, and chlorine is admitted. Aluminium chloride sublimes and is condensed in another vessel, from which the carbon monoxide or dioxide escapes through an outlet pipe. W. J. WRIGHT.

**Manufacture of titanium compounds [white oxide pigment].** TITANIUM PIGMENT CO., INC., ASSEES. OF L. W. RYAN (B.P. 308,725, 25.3.29. U.S., 27.3.28).—A slightly acid sulphate solution containing 7% Ti is added slowly with vigorous stirring to a solution of oxalic acid at 90–98°, whereby a basic sulphate is precipitated. Calcination of the product at 700–1000° affords a white titania pigment. Addition of a small quantity of phosphoric acid to the oxalic acid solution improves the colour of the calcined oxide. A. R. POWELL.

**Manufacture of solid titanyl and titanate sulphates.** I. G. FARBENIND. A.-G. (B.P. 309,047, 3.4.29. Ger., 3.4.28).—A concentrated titanium sulphate solution is atomised in a Krause dryer by a current of air at 300° and the product mixed with anhydrous sodium, barium, or magnesium sulphates. Alternatively, an organic binder may be added to the solution prior to drying. A. R. POWELL.

**Production of pure sulphur.** W. GLUUD, R. SCHÖNFELDER, and W. RIESE, ASSRS. to BERGWERKSVERBAND ZUR VERWERTUNG VON SCHUTZRECHTEN DER KOHLENTÉCHNIK (U.S.P. 1,771,293, 22.7.30. Appl., 23.7.26. Ger., 6.8.25).—The process described in B., 1927, 521 is claimed.

**Gradual production of gases [e.g., chlorine].** W. E. KEMMERICH (U.S.P. 1,729,043, 24.9.29. Appl., 8.9.24).—The production of definitely determined quantities of chlorine, gradually and uniformly diffused for therapeutic purposes, is effected by causing a suitable chlorine-producing compound to react with acid in such a manner that the chlorine generated causes gradual gravitational contact of unattacked surfaces of the reagents. W. J. WRIGHT.

**Converting hafnium and zirconium salts.** J. H. DE BOER, ASSR. to N.V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,771,557, 29.7.30. Appl., 18.10.24. Holl., 15.11.23).—See B.P. 225,187; B., 1925, 545.

**Extraction of krypton and xenon from oxygen.** A. J. A. BLARINGHEM, ASSR. to SOC. ANON. D'ECLAIRAGE ET D'APPL. ELECTRIQUES (U.S.P. 1,772,202, 5.8.30. Appl., 5.11.24. Fr., 26.12.23).—See B.P. 226,783; B., 1925, 671.

**Calcining apparatus** (U.S.P. 1,754,854). **Solid carbon dioxide** (B.P. 312,231).—See I. **Hydrogen** (B.P. 332,571). **Hydrogen from hydrocarbons** (B.P. 332,057).—See II. **Flux for soldering etc.** (B.P. 314,400). **Nickel for catalytic hydrogen** (B.P. 314,944).—See X. **Fertilisers** (B.P. 332,639, 332,750, 332,803, and 332,864). **Fertilisers and sodium bicarbonate** (B.P. 331,451). **Manures** (B.P. 313,612).—See XVI.

## VIII.—GLASS; CERAMICS.

**Change of quartz into tridymite in silicate blocks in the presence of mineralisers and a partial substitution of quartzites by sand in a silicate bed.** P. P. BUDNIKOV and I. C. SMELYANSKI (Res. Inst., Silicate Ind., Kharkov, 1929, 2, 51 pp.).—Addition of Martin slag assists the transformation of quartz into tridymite. Substitution of quartzite by sand (25%) does not impair the properties of silicate block.

CHEMICAL ABSTRACTS.

**Effect of alumina and silica on some properties of glass.** M. A. BESBORODOV (Glashütte, 1930, 60, 5–9; Chem. Zentr., 1930, i, 1353).—An increase in the alumina and silica contents retarded the devitrification of a boron-silica-alumina-lime-potash-soda glass, and increased the resistance towards water and sodium carbonate. The resistance towards sodium hydroxide and sulphuric acid rises with an increase in the silica content, but falls with an increase in that of alumina.

L. S. THEOBALD.

**Radioactive method for testing the properties of glasses.** O. HAHN and H. MÜLLER (Glastech. Ber., 1929,



7, 380—383; Chem. Zentr., 1930, i, 1355).—The emanating power of Jena glass and of a glass of the composition  $\text{Na}_2\text{O}, \text{BaO}, 6\text{SiO}_2$ , each melted with 0.1% of their weight of a substance containing radium, has been followed with respect to time and humidity of the atmosphere. In dry air the emanating power is small, especially in the case of Jena glass; at 83% humidity this glass remained unchanged whilst the barium glass quickly increased its emanation to 5.7% in 11 months. In saturated air this value rose to 19% in 11 months. The emanation is a measure of the surface development of a glass. The emanating power of Jena glass suddenly increased to a marked extent at 570°. L. S. THEOBALD.

**Acceleration of the melting of glass.** I. I. KITAI-GORODSKI (Keram. i Steklo, 1929, 5, 362—363).—Addition of ammonium salts (1—2%) facilitated the melting of a glass batch. CHEMICAL ABSTRACTS.

**Cement for silica-glass joints.** C. G. MAIER (Ind. Eng. Chem. [Anal.], 1930, 2, 337).—Silver chloride has many disadvantages when used for making detachable joints between silica and glass. A highly satisfactory joint is made by the eutectic mixture of thallous and silver chlorides, containing 60 mols.-% of thallous chloride and melting at 210°. When chilled rapidly the fused mixture solidifies as a fairly flexible, horny mass. For convenience of working it may be cast into sticks in chilled moulds. E. S. HEDGES.

**Oil firing [of ceramic ware]. Report I.** S. R. HIND (Trans. Ceram. Soc., 1930, 29, 149—168).—The results with coal- and oil-firing of the kilns of four firms are compared and discussed. It appears that, considering cost of fuel only, with oil at 90s. per ton, the competitive price of coal ranges between £2 and £3, but there are incidental advantages of oil, such as reduction of spoilt ware, of labour, more rapid firing and cooling, and more correct dimensions of the fired ware. About 24 hrs. can be saved in cooling, with improvement in the brilliance of glost ware. In an appendix a description is given of the fuel oil, of burners for it (all using steam), of viscosimeters, and a diagram showing loss of heat with excess air. B. M. VENABLES.

**Apparent sp. gr. and moisture content of clay.** F. K. CAMERON and R. A. LINEBERRY (Coll. Symp. Ann., 1929, 7, 179—189).—Addition of water to clays (100-mesh) caused no change in apparent  $d$  below 2.5%  $\text{H}_2\text{O}$ , above which balling began to cease and  $d$  to rise. Analogous phenomena occur with barium sulphate and pumice. A larger proportion is required of organic liquids which do not readily wet clay. Plasticity in a multi-component system is due mainly to a high surface tension, which is related to the apparent sp. gr.

CHEMICAL ABSTRACTS.

## PATENTS.

**Making laminated glass.** J. KOLB, Assee. of A. G. WORRALL (B.P. 317,476, 29.7.29. U.S., 17.8.28).—Two sheets of glass cemented by a suitable binder are heated under partial vacuum to soften the binder and are finally pressed together. C. A. KING.

**Laminated glass.** H. A. GARDNER (B.P. 332,234, 11.3.29).—A toluenesulphonamide-aldehyde (*p*-toluenesulphonamide-formaldehyde) resin with nitrocellulose is used as the cement for laminated glass. C. A. KING.

**Manufacture of refractory articles.** HARTFORD-EMPIRE CO., Assees. of P. G. WILLETTS (B.P. 315,856, 7.6.29. U.S., 21.7.28).—A firebrick or refractory made from 55% of white bauxite, 35% of kaolin, and 10% of a white clay is claimed. The fired article has a composition between the following limits: 39.90—34.89%  $\text{SiO}_2$ , 61.39—56.38%  $\text{Al}_2\text{O}_3$ , 0.89—1.11%  $\text{Fe}_2\text{O}_3$ , 2.65—2.72%  $\text{TiO}_2$ , 0.25—0.27%  $\text{MgO}$ , 0.28%  $\text{CaO}$ , and 0—0.06%  $\text{Na}_2\text{O}$ . The batch is fired at such a temperature that the material is almost completely converted into mullite and has a porosity of about 27%.

A. R. POWELL.

**[Tunnel] kilns.** H. M. ROBERTSON (B.P. 305,025, 23.1.29. U.S., 28.1.28).—See U.S.P. 1,683,807; B., 1928, 839.

**Continuous [tunnel] kilns.** H. M. ROBERTSON (B.P. 332,619, 23.1.29).—See U.S.P. 1,737,540; B., 1930, 222.

**Sealing of edges of laminated glass.** L. J. KOLB, Assee. of R. W. HACKETT and A. G. WORRALL (B.P. 312,368, 24.5.29. U.S., 25.5.28).

## IX.—BUILDING MATERIALS.

**Thermal inefficiency of the cement rotary kiln.**

G. MARTIN (Trans. Ceram. Soc., 1930, 29, 143—148).—The paper comprises theoretical thermal data and deductions therefrom, mainly directed to show the transcendent importance of high flame temperature from the point of view of fuel economy. Figures showing the connexion between flame temperature and clinker output are tabulated, covering the range 2966° down to 805°; at the former point the consumption of coal is 6.36 tons per 100 tons of clinker, and at the latter point it is infinite. In a practical test the consumption agreed very well with the observed flame temperature. As a practical standard a fuel consumption of more than 22 tons of standard coal per 100 tons of clinker is considered highly inefficient.

B. M. VENABLES.

**Hydration of Portland cement. Influence of monocalcium phosphate. I—VII.** K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 276—277 B; cf. B., 1930, 769).—Monocalcium phosphate if present in large proportions arrests the hydration of calcium aluminate, which is the principal factor in the setting of cement. Monocalcium phosphate interacts with lime whether originally present in the free state or produced by the hydration of calcium silicate, giving the insoluble bi- or tri-calcium salts. A small proportion of the mono-salt therefore has no injurious effect on setting. C. IRWIN.

**Mixed Portland cements.** S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 192—196 B).—“Neosolidit” is a mixture of 70—75 pts. of Portland cement clinker, 10—15 pts. of calcined granite etc., and 10—15 pts. of siliceous matter containing soluble alumina and ferric oxide. The mixture is ground with 3—5% of gypsum. Chemical analyses are given. The material shows greater contraction on curing than straight Portland cement. On heating, the free lime attains a maximum at 400—600° and then diminishes. The curing process is slow, much free lime remaining after 28 days. C. IRWIN.



**Revised procedure for determination of uncombined lime in Portland cement.** W. LERCH and R. H. BOGUE (Ind. Eng. Chem. [Anal.], 1930, 2, 296—298; cf. B., 1926, 709).—Full details of an improved technique are given. The essential features of the method are the dissolution of the uncombined lime in a hot solution of glycerol and alcohol and the subsequent titration of the dissolved lime with an alcoholic solution of ammonium acetate. Some experimental results are given and the probable errors are represented graphically.

E. S. HEDGES.

**Calcium ferrites and iron cements. III, IV.** S. NAGAI and K. ASAOKA (J. Soc. Chem. Ind., Japan, 1930, 33, 190—192 B, 256—259 B; cf. B., 1930, 769).—III. The compound  $2\text{CaO} \cdot \text{Fe}_2\text{O}_3$  is slowly decomposed by water, yielding calcium hydroxide, but  $\text{CaO} \cdot \text{Fe}_2\text{O}_3$  is not acted on. The strength of the former is considerably decreased by water-curing.

IV. Mixtures of silica, ferric oxide, and lime, containing 10—12%  $\text{Fe}_2\text{O}_3$  and having lime:silica ratios of 2.0—3.0:1, when heated together gave calcium ferrite as the first product, followed in succession by mono-, bi- and tri-calcium silicate. A similar result was obtained if part of the iron was replaced by aluminium. The presence of calcium ferrite or aluminate lowers the temperature of formation of tricalcium silicate by 100—150° as compared with that necessary in ordinary Portland cement.

C. IRWIN.

**Why cannot aluminous cement and Portland cement be used as a mixture?** K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 277—282 B).—*Ciment fondu* and Portland cement when mixed reduce the setting time of both, the shortest time of setting occurring with 70% of Portland cement. It was observed that this effect was more marked with badly-burned Portland cement, which suggested that the lime content of the latter was of importance. Experiments were made with cement to which lime had been added, and the setting of *ciment fondu* was followed microscopically using (1) water, (2) milk of lime; chemical tests were also made on the interaction of water and aluminous cement. It was concluded that the hydration of the latter by water proceeds as follows:  $2(\text{CaO}, \text{Al}_2\text{O}_3) + 11\text{H}_2\text{O} = 2(\text{CaO})\text{Al}_2\text{O}_3 \cdot 8\text{H}_2\text{O} + 2\text{Al}(\text{OH})_3$ , and that this reaction is comparatively slow and reversible. If, however, lime is present the reaction is  $\text{CaO} \cdot \text{Al}_2\text{O}_3 + \text{Ca}(\text{OH})_2 + 7\text{H}_2\text{O} = 2\text{CaO}, \text{Al}_2\text{O}_3 \cdot 8\text{H}_2\text{O}$  and this reaction is rapidly complete and irreversible.

C. IRWIN.

**Lime-alumina cement. II.** S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 196—198 B).—The partial replacement of alumina by ferric oxide reduces the absolute strength but diminishes the deterioration on water-curing and prevents the dusting of clinker. This latter is also an effect of the addition of 0.5—1.0% of borax. Further tests show that the deterioration of lime-alumina cement on water-curing is due to the dissolution of hydrated calcium aluminate. In this quality and in strength it is inferior to alumina cement.

C. IRWIN.

**Laitance of cement mortars and concrete. II.** S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 260—

262 B; cf. B., 1930, 327).—The laitance on 1:2:4 concrete of low water-cement ratio is similar in composition to the cement, but with higher water ratios the lime content is increased relative to the silica. Other samples were found to contain large quantities of sand particles. Laitances from alumina cement or blast-furnace slag cement were found to contain 25—60%  $\text{Al}_2\text{O}_3$  on the ignited sample. C. IRWIN.

**Use of gypsum waste products.** P. P. BUDNIKOV and V. M. LECHYEV (Keram. i Steklo, 1929, 5, 432—435). Cement obtained by heating gypsum at 500—700° has a shrinkage of 1—1.5%; shrinkage is prevented and the setting time reduced by addition of sodium hydrogen sulphate (0.6%) or copper sulphate (0.8%).

CHEMICAL ABSTRACTS.

**Sinkage [of pulp-wood.] I. Mode of penetration of water into logs.** G. W. SCARTH and E. C. JAHN (Canad. J. Res., 1930, 2, 409—424).—The distribution of water in logs floated on a lake is similar to that of living trees. The woods examined have a relatively dry heartwood, becoming wetter in the order jack pine, spruce, poplar, and balsam, the sapwood in all these cases being wet. In birch the heart- and sap-woods are equally wet. The rate of radial penetration increases in the order birch, jack pine, spruce, balsam, poplar, the penetration being very slow, even into sapwood. The advantage of a high proportion of dry heartwood is mainly in the initial buoyancy conferred. The rate of penetration is decreased by density and narrow outer rays. In air-dry logs, saturation of cell-walls precedes penetration of free water. The natural buoyancy of the logs is due to enclosed gases, which, being surrounded by water, can only escape in solution; further amounts of gas are probably formed by fermentation.

T. T. PORTS.

**Electrical conductivity method for determining the moisture content of wood.** A. J. STAMM (Ind. Eng. Chem. [Anal.], 1930, 2, 240—244).—The electrical conductivity of wood varies very greatly with its content of moisture, and a simple, compact, and portable electrical conductivity apparatus based on this fact has been designed for determining the average moisture content of wood. A special feature is the pin-point type of contact. The conductivity measurements can be translated directly into terms of average moisture content, not only when the moisture is uniformly distributed, but also when it is distributed according to any normal drying gradient. Experimental results obtained with 160 specimens of 25 kinds of wood show a mean deviation of 0.6% absolute moisture content and a maximum deviation of 1.7% from the values determined through loss of weight on drying in an oven. Surface moisture temporarily increases the moisture content value, but the normal value is regained after a short time. The usual surface-finishes of wood do not affect the readings.

E. S. HEDGES.

**Pigments for façades.** RASQUIN.—See XIII.  
**Rubber cements.** BUSSE and DOGGETT.—See XIV.

PATENTS.

**Manufacture of cement.** F. S. WATT (B.P. 332,322, 7.5.29).—In a kiln the slurry is either injected through



a central nozzle surrounded by a number of flame jets which impinge on to the atomised slurry, or the respective flow of slurry and fuel is reversed, the object in either arrangement being to cause the spray to be injected into the hottest part of the furnace chamber.

C. A. KING.

**Manufacture of magnesian cement.** J. C. G. SPERNI (B.P. 332,282, 18.4.29).—An oxide of manganese (5–25%) is mixed with a magnesian cement composed of burned magnesite and clay, or the constituents may be calcined together and ground to powder. Sodium silicate may be incorporated in the mixture, and the cement is suitable, when gauged with water, for flooring or moulding into artificial stonework. C. A. KING.

**Production of cellular solid bodies.** J. W. BATTERSBY (B.P. 331,989, 17.5.29).—A solid, colloidal, soluble silicate of the alkali metals still containing about 25% of water is heated at 200–500°. The liberation of water vapour causes a cellular structure and moulded shapes may be formed. Alternatively, the silicate may be initially in the form of powder, and fillers, *e.g.*, coke, asbestos, or pumice, may be incorporated before heating.

C. A. KING.

**Composition for making, maintaining, consolidating, and repairing highways or roads.** "SAPONITE" (B.P. 310, 414, 11.4.29. Fr., 25.4.28).—A consolidating cement is prepared from a mixture of calcareous sand, hydraulic lime, and sodium silicate solution.

C. A. KING.

**Progressive dry kiln and method of drying lumber.** J. B. WELCH (U.S.P. 1,753,974, 8.4.30. Appl., 30.10.25).—The timber is moved progressively through a long chamber and subjected to a gradually increasing temperature, the atmosphere being maintained very moist at the green end by means of steam jets and the air recirculated in a number of zones by means of the convection currents produced by heaters aided by steam aspirators; each circulation is upwards through the lumber towards the dry end and downwards towards the green end.

B. M. VENABLES.

**Preservatives for wood.** GRUBENHOLZIMPRÄGNIERUNG GES.M.B.H. (B.P. 315,860, 21.6.29. Ger., 20.7.28).—Wood is impregnated with a solution at 70° containing 2% of a salt mixture consisting of 40% of ferrous sulphate crystals, 40% of sodium fluoride, 10% of arsenious acid, and 10% of sodium bisulphite. Alternatively, zinc or copper sulphates can be used instead of ferrous sulphate, sodium arsenate instead of arsenious acid, and potassium dichromate instead of the bisulphite.

A. R. POWELL.

**Imitating the grain of wood or the like.** MASA GES.M.B.H. ZURHERSTELLUNG KÜNSTLICHER OBERFLÄCHEN (B.P. 308,371 and 308,373—6, 22.3.29. Ger., 22.3.28).—(A) Printing blocks for intaglio printing are prepared from a photograph of natural wood-grain taken with the aid of ultra-violet rays having a wave-length less than 3700 Å. (B) The softer parts of the wood are first removed by chemical, physical, or mechanical means so as to throw the grain into relief. (C) The wood specimen to be copied is coated or impregnated with a coloured solution, a moistened gelatin paper is applied, and from the impression produced on the paper printing

plates are prepared by any known photo-mechanical process. (D) The specimen is soaked in a solution of a barium salt, then in a sulphate solution, and exposed to X-rays after a light-sensitive plate has been placed behind it. Alternatively, the wood is rubbed with a phosphorescent substance, exposed to the sun for some time, then laid on a photographic plate. (E) The wood is photographed by the aid of light from a quartz mercury-vapour lamp.

A. R. POWELL.

**Bituminous composition.** T. F. KING (U.S.P. 1,771,795, 29.7.30. Appl., 30.8.29. U.K., 3.8.28).—See B.P. 322,431; B., 1930, 178.

**[Reinforced] composition flooring.** H. L. TUCKER (B.P. 332,719, 3.7.29).

**[Sectional] paving and like surfaces.** L. COOKE (B.P. 332,741, 18.7.29).

**Reproducing grain of wood [on printing plates].** MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 308,372, 22.3.29. Ger., 22.3.28).

**Kilns** (B.P. 332,037).—See VII.

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

**Calorific value, heat and gas flow, the physical bases of metallurgical processes.** H. BANSSEN (Stahl u. Eisen, 1930, 50, 668–678).—Diagrams have been constructed to show the heat balance in various stages of iron smelting—the blast furnace, gas producer, acid and basic open-hearth furnaces, converter and sponge iron producer—and the effect of preheating the gas and air is illustrated. A knowledge of the calorific value of the fuel and the rate of flow and heat content of the various gases is shown to be of paramount importance in the correct and efficient operation of the various processes. The resistance of the column of charge in the blast furnace to the flow of gas through the furnace determines to a large extent the output of the furnace, and any factor which increases this resistance reduces the efficiency of the furnace. One of these factors is the deposition of carbon from the flue gases in the upper part of the blast-furnace shaft, and an apparatus is described and illustrated for the determination of the magnitude of this deposition from blast-furnace gas.

A. R. POWELL.

**Substitution of limestone by rock salt in blast-furnace practice.** E. V. BRITKZE, A. N. KRESTOVNIKOV, and I. V. SCHMANENKOV (Min. Ssyrye Zvet. Met., 1929, 4, 359–375; Chem. Zentr., 1930, i, 1031–1032).—The charge of sodium chloride passes partly into the slag as sodium oxide, and partly into the gases as chloride; the proportion of the former (20–70%) increases, but not in simple ratio, with the charge, and is the greater the more finely divided and the more completely mixed are the materials. The reaction is represented by  $2\text{NaCl} + \text{CO}_2 + 2\text{CO} \rightarrow \text{Na}_2\text{CO}_3 (\rightarrow \text{Na}_2\text{O} + \text{CO}_2) + \text{COCl}_2 + \text{C}_2$  (at 800–900°). A slag of optimal composition contained  $\text{SiO}_2$  42.72,  $\text{FeO}$  2.25,  $\text{Al}_2\text{O}_3$  15.46,  $\text{CaO}$  33.76,  $\text{MgO}$  2.56,  $\text{Na}_2\text{O}$  5.04%. The slag, the m.p. of which is depressed by 300°, is vitreous and separates well from the metal. A. A. ELDRIDGE.



**Evaluation and testing of the properties of raw materials and key-products in the iron industry.**

A. WAGNER (Stahl u. Eisen, 1930, 50, 655—668).—The present state of our knowledge of the properties of coal, coke, and iron ore used in the production of iron and steel and of the pig iron produced from various types of ore is reviewed and modern methods of determining the value of these materials for their particular purposes are briefly discussed. Laboratory tests of the coking power of coal and chemical analyses of iron ore do not give sufficient information as to the value of the coke and ore for metallurgical purposes, as no account is taken of their physical condition, which is of considerable importance in determining their behaviour in smelting. Similarly, chemical analysis of pig iron affords little information as to its value for subsequent operations, but, in combination with an examination of its micro- and macro-structure and a determination of its mechanical properties after remelting under standard conditions, the analysis gives a fairly accurate idea of the commercial value of the metal. A. R. POWELL.

**Practical importance of the influence of different cooling conditions on the structure of steel ingots.**

F. LEITNER (Stahl u. Eisen, 1930, 50, 1081—1086).—The effect of varying wall thicknesses of the mould on the structure of plain carbon and nickel-chromium steels has been examined. The results show that a more fine-grained and homogeneous macrostructure is obtained by retarded cooling, and hence thin-walled moulds are to be preferred. Even better structures are produced by retarding the radiation of heat from the mould by covering it with a heat-insulating material or by using a double-walled mould with an annular air-space between the walls. A. R. POWELL.

**Theory of the malleabilising process.** N. G. GIRSHOVICH and E. K. VIDIN (Trans. Inst. Met., Moscow, 1929, No. 4, 3—117).—The mechanism of graphitisation and decarburisation of white cast iron is discussed.

CHEMICAL ABSTRACTS.

**Testing of sands for steel castings.** S. PIÉRARD (Bull. Soc. chim. Belg., 1930, 39, 252—270).—A description of methods of chemical analysis, and of the determination of grain size, binding power, permeability to air, and refractoriness of sands used for casting steel is given. C. W. GIBBY.

**Deoxidisation of steel with aluminium.** C. H. HERTY, JUN., G. R. FITTERER, and G. N. BYRNS (Min. Met. Invest. Carnegie Inst. Tech., U.S. Bur. Mines Co-op. Bull., 1930, No. 46, 1—43).—Ferrous oxide and alumina coalesce slightly to form inclusions which are fusible below the m.p. of iron; inclusions high in alumina tend to group. In small ingots group-segregation of inclusions, undetected by analysis, caused cracking during forging. Equal weights of aluminium and silicon reduce the ferrous oxide content of steel to the same extent, but the reaction with aluminium is the more rapid. CHEMICAL ABSTRACTS.

**Influence of nickel and manganese on the properties of high-speed tool steel.** V. EHMCKE (Arch. Eisenhüttenw., 1930—1, 4, 23—35; Stahl u. Eisen, 1930, 50, 1131—1132).—The hardness of high-speed

tool steel immediately after quenching is decreased by the addition of nickel; steels with more than 5% Ni have a purely austenitic structure, apart from the carbide segregations due to decomposition of the ledeburite eutectic, and their hardness is therefore relatively low, being only 300 Brinell units for the 5% Ni alloy. By subsequent annealing at 600—900° the austenite is converted into martensite and the hardness increases greatly, e.g., to 575 Brinell units for the 5% Ni steel. With increasing nickel content the stability of the austenite becomes greater so that the temper hardness diminishes; a steel with 12% Ni is purely austenitic and cannot be hardened by tempering. Dilatometric tests on nickel high-speed tool steels show that the transformation from austenite into martensite takes place during a critical temperature range on cooling; this starts at 600° in the case of the 3% Ni steel and at 700—900° for the 5—12% Ni steels. In the case of the same steels without nickel a secondary hardening effect occurs at 580°, at which temperature the residual quenching austenite is converted into martensite. At temperatures up to 600—700° the tensile strength and hardness of nickel tool steels are much less than those of ordinary tool steels, but at higher temperatures nickel improves the strength and hardness. Manganese behaves in a similar manner to nickel in decreasing the hardness at the lower temperatures and increasing it above 600°. A. R. POWELL.

**Mechanical properties of copper steels with especial reference to the effect of heat treatment.**

F. NEHL (Stahl u. Eisen, 1930, 50, 678—686).—The addition of more than 0.6% Cu to mild steel increases appreciably the yield point, but tends to make the metal brittle, a tendency which is overcome by the addition of 0.4% Cr. This alloy has a much higher tensile strength and yield point at high temperatures than silicon steel and is more resistant to corrosion. Quenching from above 800° and tempering at 450—550° increases the tensile strength of steels with more than 0.6% Cu (cf. following abstract) without seriously reducing the ductility and resistance to shock. Tables and graphs are given showing the effect of various heat treatments on the mechanical properties of several copper steels after varying mechanical treatment. A. R. POWELL.

**Temper-hardening of steel containing copper.**

H. BUCHHOLTZ and W. KÖSTER (Stahl u. Eisen, 1930, 50, 687—695).—The solid solubility of copper in  $\alpha$ -iron is 0.4% at 0—600° and then increases according to the equation  $\log x = -4125/T + 4.32$  to 3.4% at the eutectoid temperature 810°. On cooling a copper-steel from the  $\gamma$ -solid solution range supersaturated solid solutions of copper in  $\alpha$ -iron having a structure resembling that of martensite may be obtained; these supersaturated solutions as well as those containing 0.6—3.4% Cu quenched from 600—800° undergo age-hardening on tempering at 450—600°, but the separation of the excess copper in a highly dispersed condition and the subsequent coagulation of the particles take place very slowly. When the steel contains both copper and carbon in supersaturated solid solutions both elements separate in a disperse form on ageing at high temperatures, the two reactions taking place independently



of one another. Thus on tempering a quenched steel with 0.05% C and 2% Cu at gradually increasing temperatures the hardness rises sharply from 160 to 210 at 20° during 3 days, then to 220 at 100° due to separation of excess carbon; the hardness then falls rapidly to 150 at 300° owing to coagulation of the cementite, and rises again to 205 at 500—550° owing to precipitation of the copper. The properties of temper-hardened forgings of steel with 0.8% Cu and 0.4% Cr resemble those of a chromium-nickel steel with a tensile strength of 70 kg./mm.<sup>2</sup>; both steels are characterised by the regularity of their mechanical properties throughout large cross-sections and by their stability on prolonged storage after tempering.

A. R. POWELL.

**Ternary system iron-nickel-sulphur.** R. VOGEL and W. TONN (Arch. Eisenhüttenw., 1929—1930, 3, 769—780; Stahl u. Eisen, 1930, 50, 1090—1091).—This ternary system has been investigated by thermal and magnetic analysis within the region nickel-iron-FeS-Ni<sub>3</sub>S<sub>2</sub>. This part of the system includes only one ternary compound, (FeS)<sub>2</sub>Ni<sub>3</sub>S<sub>2</sub>, which forms a continuous series of mixed crystals with Ni<sub>3</sub>S<sub>2</sub> and which decomposes below 520° into a eutectic mixture of FeS and Ni<sub>3</sub>S<sub>2</sub> after undergoing a polymorphic transformation at 615°. In the region studied only three primary crystal phases separate on cooling the liquid, viz., binary iron-nickel solid solution, either in the  $\alpha$ - or  $\gamma$ -crystal phase according to the composition; FeS; and ternary solid solution of Ni<sub>3</sub>S<sub>2</sub> in (FeS)<sub>2</sub>Ni<sub>3</sub>S<sub>2</sub> with excess nickel and iron. The metal solid solution forms eutectics with both sulphide phases. There is a ternary transformation point at 825° at which the liquid containing 38% Fe, 35% Ni, and 27% S reacts with FeS to form a ternary solid solution with 39% Fe, 32% Ni, and 29% S and a binary solid solution with 64% Fe and 36% Ni. Solid solutions rich in nickel and sulphur decompose at 480° forming a ternary eutectoid containing 13% Fe, 27% S, and 60% Ni and consisting of Ni<sub>3</sub>S<sub>2</sub>, (FeS)<sub>2</sub>Ni<sub>3</sub>S<sub>2</sub>, and nickel-iron solid solution with 34% Fe. Ferrous sulphide reacts at 585° with the mixture containing 26% Fe, 46% Ni, and 28% S to form  $\alpha$ -(FeS)<sub>2</sub>Ni<sub>3</sub>S<sub>2</sub> and an iron-nickel solid solution with 57% Fe. Thus at the ordinary temperature the system contains five fields having the composition (a) FeS +  $\alpha$ - (or  $\gamma$ )-Fe-Ni, (b) FeS +  $\gamma$ -Fe-Ni + (FeS)<sub>2</sub>Ni<sub>3</sub>S<sub>2</sub>, (c)  $\gamma$ -Fe-Ni + (FeS)<sub>2</sub>Ni<sub>3</sub>S<sub>2</sub>, (d) (FeS)<sub>2</sub>Ni<sub>3</sub>S<sub>2</sub> + Ni-Fe + Ni<sub>3</sub>S<sub>2</sub>, and (e) Ni-Fe + Ni<sub>3</sub>S<sub>2</sub>. These results elucidate completely the composition of nickel matte and explain the presence therein of free metal similar to the hair-copper found in copper matte. This free metal occurs as leaflets when it has separated directly from the liquid mass and in hair-like crystals when it has been formed by the decomposition of ternary solid solution.

A. R. POWELL.

**Determination of gases in metals, especially oxygen in iron and steel, by the hot extraction method.** H. DIERGARTEN (Arch. Eisenhüttenw., 1928—9, 2, 813—828; 1929—1930, 3, 577—586, 627—635; Stahl u. Eisen, 1930, 50, 911—913).—By heating iron or steel in a graphite crucible in a high-frequency induction furnace or a carbon spiral furnace under a high vacuum at 1550—1650° all the oxygen is evolved as carbon

monoxide no matter in what form it is present in the steel. If more than 0.7% Mn or 0.3% Al is present part of these elements sublimes and reacts with the carbon monoxide, thus causing low results for oxygen; otherwise the method gives exceedingly accurate results. The oxygen content of cast iron lies between 0.002 and 0.014% under normal conditions, but much greater variations occur throughout an ingot than is the case with carbon, manganese, and phosphorus.

A. R. POWELL.

**Determination of silica in the presence of silicon in ferrosilicon.** A. STADELER (Arch. Eisenhüttenw., 1930—1, 4, 1—6; Stahl u. Eisen, 1930, 50, 1132—1133).—The finely-powdered sample (10 g.) is heated in a current of pure dry chlorine in such a way that the temperature rises to 300° in 30 min., 400° in 90 min., and to 500° in 150 min. Heating is then continued for 3 hrs. at 550°. The residue in the combustion boat is extracted with concentrated hydrochloric acid and the insoluble material collected, ignited, and weighed. The loss in weight after treatment with hydrofluoric and sulphuric acids is calculated as silica; the residue from this treatment consists of carborundum which is analysed in the usual way after fusion with sodium carbonate and nitrate. The chlorine is freed from oxygen by passing it over glowing wood charcoal. Blast-furnace ferrosilicon with 11% Si usually contains 0.06—0.10% SiO<sub>2</sub>, 45% ferrosilicon about 0.14—0.26% SiO<sub>2</sub>, and 90% ferrosilicon 1—1.32% SiO<sub>2</sub>.

A. R. POWELL.

**Zinc as a protective coating against corrosion fatigue in steel.** W. E. HARVEY (Met. and Alloys, 1930, 1, 458—461).—The corrosion fatigue limit of uncoated steel in air is the same as that of electroplated specimens in water. With sherardised and galvanised coatings the presence of hard, brittle, intermetallic compounds caused premature failure.

CHEMICAL ABSTRACTS.

**Corrosion tests of iron and steel in distilled, tap, and sea water.** K. HASEGAWA and S. HORI (J. Eng. Assoc., Manchuria, 1930, 7, No. 35, 27 pp.).—The loss by corrosion was largest in distilled, less in sea, and least in tap water. In distilled water the corrodibility of steel increases proportionally to the carbon content; in tap and sea water it is maximal at 1% C. The corrodibility of cast iron is similar to that of medium-carbon steel; wrought iron shows the greatest resistance.

CHEMICAL ABSTRACTS.

**X-Ray examination of commercial galvanised iron by a modified reflection method.** C. W. STILLWELL and G. L. CLARK (Ind. Eng. Chem. [Anal.], 1930, 2, 266—272).—A modified method for obtaining X-ray diffraction patterns by reflection is described, and the method is especially suitable for the examination of the phases present in metals and alloys. It is sufficiently accurate to distinguish between several crystal forms, the lattices of which have already been established. The method has been applied to galvanised iron wire and has served to show that (1) there is no solid solution of zinc in iron adjacent to the steel base; (2) the compound next to the steel base is not FeZn<sub>3</sub>, as formerly supposed, but is Fe<sub>3</sub>Zn<sub>10</sub>, (3) the outer layers of the coating contain the compound FeZn<sub>7</sub>, a solid solution



of zinc in  $\text{FeZn}_7$ , a solid solution of iron in zinc, and a phase with a body-centred cubic lattice the composition of which is unknown. A similar analysis of hot-dipped, Armco, sheet steel made it evident that (a) there is again no solid solution of zinc in iron adjacent to the steel base; (b) there is no evidence of the compound  $\text{Fe}_3\text{Zn}_{10}$ ; (c) the intermediate layers consist of a solid solution of zinc in  $\text{FeZn}_7$  and the body-centred cubic phase, the latter being more concentrated near the surface of the coating; (d) the surface of the coating is pure zinc, the crystals being oriented. The body-centred cubic lattice has not been observed in systems in equilibrium.

E. S. HEDGES.

**Factors governing removal of soluble copper from leached ores.** J. D. SULLIVAN and A. J. SWEET (U.S. Bur. Mines, Tech. Paper 453, 1929, 26 pp.).—The rate of removal of copper sulphate during the leaching of various ores with sulphuric acid has been studied under a variety of conditions. Although the time required to saturate crushed rocks with leach liquor is not a linear function of the particle size, the time of diffusion is almost so; therefore it is advisable to crush the ore reasonably small consistent with the production of a minimum amount of slime before feeding it to the leaching vats. When apparent equilibrium is reached in the vat 15% more of the total copper is retained in the residue at 2–3° than at 20°, but penetration of the leach liquor into the ore particles is somewhat greater and more rapid at the lower temperatures; hence it is recommended that leaching solutions should be added to the vats in the evening and washing carried out in the daytime when the temperature is higher. Alternate wetting and drying of the ore results in complete extraction of the copper in 15–20% of the time required in flood-washing. Tests made on the impregnation of rocks with copper sulphate solution show that the salt is disseminated throughout the rock especially along fractures and cleavage planes; on drying the impregnated rock the crystals of copper sulphate concentrate at or towards the surface, a fact which explains the beneficial action of alternate wetting and drying on the extraction by leaching. A low concentration of salts in the leaching solution does not appreciably improve the rate of diffusion of the copper, but there appears to be some relation between this rate and the viscosity of the solution. Thus 6% sulphuric acid is more sluggish in its action than 0.5% acid and a 4% ferric sulphate solution, which has twice the viscosity of the dilute acid, effects a complete removal of the copper in a time 50% greater than that required by the acid.

A. R. POWELL.

**Acceleration of extraction of soluble copper from leached ores.** M. GUGGENHEIM and J. D. SULLIVAN (U.S. Bur. Mines, Tech. Paper 472, 1930, 30 pp.; cf. preceding abstract).—The effect of alternate wetting and drying tests on the rate of extraction of copper sulphate from rocks previously impregnated with a solution of this salt has been studied. Maximum extraction was obtained by alternately washing for  $\frac{1}{2}$  or 1 hr. and drying for  $\frac{1}{2}$  or 1 hr. In chemical diffusion tests 50% of the total water-soluble copper was removed in 8 hrs. and 90% in 71%, whereas in the alternate

wetting and drying tests the same extractions were obtained in 2 and 17 hrs., respectively. Drying appears to bring the copper more rapidly to the surface, so that the smaller are the pieces of ore the more rapid is the extraction. Rise of temperature and drying the ore in a current of air increase the rate of dissolution of the copper.

A. R. POWELL.

**Effects of silicon on the properties of brass.** I. H. W. GOULD and K. W. RAY (Met. and Alloys, 1930, 1, 455–457).—Silicon (up to 2.8%) forms homogeneous  $\alpha$ -solid solutions with 85:15 brass, but not with 60:40 brass; it forms a homogeneous  $\gamma$ -solid solution with the former at 8.6% and with the latter at 4.2%. All other brasses in the two series show two solid phases. Silicon lowers the f.p. of 85:15 and 60:40 brasses when present in amounts up to 10% and 7%, respectively. A tentative equilibrium diagram is advanced.

CHEMICAL ABSTRACTS.

**Analysis of antifriction alloys.** L. VENIKOV (J. Chem. Ind., Russia, 1929, 6, 209–210).—The alloy is dissolved in nitric acid; the residue after repeated evaporation with nitric acid is dissolved in oxalic acid, antimony trisulphide being precipitated from the solution; tin is determined electrolytically in the filtrate. The antimony sulphide is dissolved in sodium hydroxide solution, a small residue of lead and copper sulphides being dissolved in nitric acid and added to the original solution, in which lead and copper are determined in the usual way.

CHEMICAL ABSTRACTS.

**Nickel casting alloys.** J. McNEIL (Metal Ind., London, 1930, 36, 275–278, 305–306).—The effect of casting temperature and composition on strength and elongation are described. The solubility of lead in molten copper is increased from 35 to 60% by the addition of 2.5% Ni; with 2.5–3% Ni the two metals are miscible in all proportions.

CHEMICAL ABSTRACTS.

**Coarse crystallisation of aluminium.** R. KARNOP and G. SACHS (Metall.-Wirt., 1929, 8, 1115–1118; Chem. Zentr., 1930, i, 1209).—The tendency of aluminium to coarse crystallisation diminishes with increasing content of impurities. Iron is of greater importance than silicon.

A. A. ELDRIDGE.

**Effect of iron on the properties of aluminium.** S. A. POGODIN (Min. Syr. Tzvet. Met., 1929, 4, 616–624).—Addition of iron up to 5.4% increases the hardness, electrical resistance, and corrodibility of aluminium; up to 2.6% it increases the tensile strength and decreases the elongation and compressibility.

CHEMICAL ABSTRACTS.

**Cleaning aluminium.** R. W. MITCHELL (Metal Ind., N.Y., 1930, 28, 171–172).—The solution (of silicate or chromate), 2–6% by wt., should not evolve more than 2 c.c. of gas, nor dissolve more than 1 mg. of metal, per sq. in. of surface at 93–99°. No visible deposit should remain after rinsing, and the sample should not gain in weight.

CHEMICAL ABSTRACTS.

**Solders for soldering aluminium.** M. M. CHRUSHCHOV and M. V. SHAROV (Trans. Inst. Automotive Inst., Moscow, 1929, No. 9, 5–40).—Comparative tests were performed.

CHEMICAL ABSTRACTS.



**Complete analysis of magnesium-aluminium alloys.** S. S. SINGER (Ind. Eng. Chem. [Anal.], 1930, 2, 288).—A rapid method of separation of aluminium and iron from magnesium is based on the fact that iron and aluminium are not precipitated by sodium ammonium phosphate in the presence of tartaric acid, whereas magnesium is precipitated quantitatively. The precipitation of magnesium is greatly accelerated by chilling the solution in ice-water in addition to the usual vigorous stirring and scratching with a glass rod.

E. S. HEDGES.

**Evaluation of stibnite. II. Determination of antimony.** W. N. McNABB and E. C. WAGNER (Ind. Eng. Chem. [Anal.], 1930, 2, 251–256).—Titration of tervalent antimony by bromate, iodine, or permanganate gives practically identical results. The iron present as an impurity in stibnite is in the ferrous condition at the time of titration of the antimony, and affects the result. The permanganate method is recommended and a correction introduced by making a separate determination of the iron, and a method of effecting this is described. The Frankford Arsenal method (Cushman, Ind. Eng. Chem., 1918, 10, 380) gives results which are about 0.5% too low, due to loss of antimony by volatilisation. The authors' evolution procedure described formerly (B., 1929, 286) has been extended to include the iodometric determination of sulphide sulphur and the titration of antimony with permanganate. The analysis is rapid and accurate, and both determinations can be made on a single sample.

E. S. HEDGES.

**Refining of tin.** R. THEWS (Metallbörse, 1929, 19, 2553–2554, 2610–2611, 2666; Chem. Zentr., 1930, i, 1209).—The most undesirable impurities are iron, arsenic, antimony, zinc, and cadmium. Methods for the purification of tin are discussed.

A. A. ELDRIDGE.

**Ternary alloys of lead.** S. J. NIGHTINGALE (Brit. Non-Ferrous Metals Res. Assoc., Develop. Rep. D 3, 1929, 12 pp.).—The properties of lead alloys containing (a) 0.25% Cd and 0.5% Sb, and (b) 0.25% Cd and 1.5% Sn are compared with those of pure lead as regards their utility for the manufacture of piping. The tensile strength of the alloys is 1.62 tons/in.<sup>2</sup> compared with 0.9 ton/in.<sup>2</sup> for pure lead, the ductility 52–55% against 80%, and the fatigue limit 0.74–0.57 ton/in.<sup>2</sup> against 0.18. The alloys are on the whole more resistant to corrosion than is lead, and withstand a considerably higher bursting pressure, so that a lighter pipe can be used for the same service than is the case with lead. The working and soldering properties of the alloys are equally as good as those of lead and taking into account their superior properties are relatively less costly than lead; they do not undergo intercrystalline corrosion or fatigue.

A. R. POWELL.

**Relative [flotation] activity of the various constituents of crude "cresylic acid."** G. L. LANDOLT, E. G. HILL, and A. LOWY (Eng. Min. J., 1930, 129, 351–352).—The order of decreasing activity for a lead-zinc ore was: *m*-, *p*-, *o*-cresol, 1 : 3 : 4, 1 : 4 : 5, 1 : 2 : 4, xylene, phenol, 1 : 3 : 2-xylene. Propylphenol is more active than *o*- or *p*-cresol. CHEMICAL ABSTRACTS.

**Magnesite: its application in assaying.** R. J. B. KETHEL (J. Chem. Met. Min. Soc. S. Afr., 1930, 30, 323–328).—South African magnesite deposits contain an average of 90% MgCO<sub>3</sub> and 5% SiO<sub>2</sub>. For making cupels for gold assaying the material is calcined at 600°, ground to a fine powder, mixed with water, and moulded into bricks which are heated up to 1250° in 3–7 days in reverberatory or muffle furnaces. The calcined magnesite thus obtained is crushed so that 70–75% passes 200-mesh, mixed to a damp powder with a solution of glue or sugar, and pressed into the desired cupel shape in hand or compressed air presses. The cupels are dipped into a 1% solution of sodium silicate and dried for 12 hrs. at 100°, then set aside in a dry warm place to mature. A good cupel should absorb 86–90% of its weight in lead.

A. R. POWELL.

**Influence of atmosphere, temperature, and nature of protection tube on the stability of platinum thermo-elements.** F. STÄBLEIN and J. HINNÜBER (Arch. Eisenhüttenw., 1929–1930, 3, 781–785; Stahl u. Eisen, 1930, 50, 1089–1090).—Prolonged heating of platinum or 10% rhodium-platinum thermocouple wires in air, hydrogen, or coal gas results in a slow but only slight increase in the *E.M.F.* produced against new wire of the same type, but if the wire is enclosed in a protection tube much more serious changes take place which are shown to be due to the volatilisation of impurities from the sheath into the wires. At 1100° contamination is greatest in hydrogen, followed by carbon monoxide and carbon dioxide, and is least in air. Porcelain and refractory masses as protection tubes cause more serious contamination than iron, but fused silica is by far the most satisfactory. That the tube is the cause of the trouble is shown by the gain in weight of the wires and by an increase in their electrical resistance. When a couple is used in coal gas a heavy deposit of carbon is formed on the rhodium alloy wire at 850°, but the platinum wire remains quite bright, although at higher temperatures it, too, becomes covered with carbon.

A. R. POWELL.

**Determination of inhomogeneities in metal specimens of any form by means of X-rays.** E. Z. KAMINSKI and N. Y. SEL'YAKOV (Trans. Phys.-Tech. Lab., Leningrad, 1926, No. 3, 33–37).—Powdered media can be used provided that its absorption coefficient is of the same order as that of the specimen. Two or more powders must be well mixed and of similar coefficients of absorption.

CHEMICAL ABSTRACTS.

**Anodes for the production of electrolytic zinc.** H. R. HANLEY, C. Y. CLAYTON, and D. F. WALSH (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 321, 3–8).—The characteristics of several lead alloys for use as anodes have been examined. Calcium-lead anodes exhibit remarkable lowering of the anode potential, amounting to about 50% below that of pure lead. The alloy became passive on addition of 1% Ag. Thallium (more than 4%) causes remarkable passivity, but the anode potential is only slightly lowered.

CHEMICAL ABSTRACTS.

**Resistance of electrodeposits to corrosion, with special reference to cadmium and zinc.** W. S.



PATTERSON (J. Electroplaters' Dep. Tech. Soc., 1930, 5, 91—106).—The various factors governing corrosion are discussed. Steel is effectively protected against indoor corrosion by zinc or cadmium; the corrosion products of zinc do not affect subsequent corrosion, but cadmium is partly protected by its products. In a highly saturated and polluted indoor atmosphere the corrosion was at first similar for the two metals, zinc subsequently being the less vigorously attacked. In an outdoor atmosphere cadmium was corroded more readily than zinc, owing to the difficulty of formation of a less soluble basic cadmium sulphate. The mechanism of the corrosion is discussed. CHEMICAL ABSTRACTS.

**Pressure die cast aluminium alloy test-pieces.** J. D. GROGAN (Inst. Metals, Advance copy, 1930, No. 530, 18 pp.).

**Rolled gold: its origin and development.** E. A. SMITH (Inst. Metals, Advance copy, 1930, No. 539, 25 pp.).

**Copper in brewing.** STEWARDSON.—See XVIII.

#### PATENTS.

**Apparatus for heat-treatment of metalliferous materials [roasting furnace].** E. M. S. INDUSTRIAL PROCESSES, LTD., R. A. STOKES, and E. G. L. ROBERTS (B.P. 330,930, 20.12.28).—A reverberatory furnace has a hearth formed of a series of troughs of arcuate cross-section over which the material to be roasted is passed by means of a series of paddles worked from water-cooled shafts lying parallel to the direction of the troughs. Discharge from the last trough is effected longitudinally by means of a screw conveyor and the heating gases are passed over the hearth countercurrent to the ore stream. A. R. POWELL.

**Rendering utilisable cupriferos and zinciferous ores.** A. L. MOND. From METALLGES. A.-G. and HOCHOFENWERK LÜBECK A.-G. (B.P. 318,232, 26.6.29).—The hot product obtained from the chloridising roasting of pyrites is cooled to about 100° by spraying it with the minimum amount of water, then air-cooled to below 75°, preferably to 50°, and leached with a small quantity of water or the acid liquor obtained by washing the roaster gases, whereby about 90% of the extractable zinc and only 15% of the extractable copper is removed in the leach liquor. The remaining zinc and the greater part of the copper are removed in subsequent leaches. The first leach liquor is worked up separately from the remainder by known methods. A. R. POWELL.

**[Electric] furnace for reducing metals.** G. C. COX and H. K. MCINTYRE (U.S.P. 1,753,822, 8.4.30. Appl., 5.10.26).—In an electric furnace in which the greater part of the hearth is inclined, the cathode is situated at the bottom of the low portion, and an anode for direct current at a higher level. Auxiliary electrodes for alternating current are provided to maintain the electrolyte at the optimum temperature. C. A. KING.

**Production of metals and alloys by exothermic reduction.** W. B. HAMILTON and T. A. EVANS (B.P. 331,892, 5.2.29).—The incipient heat required to induce reaction in a mixture of ore and a thermo-reducing

agent is provided by a high-frequency induction furnace. The reducing agent may consist partly of an energetically reacting mixture and partly of a slower acting mixture to maintain the heat when propagated. C. A. KING.

**Apparatus for reduction of impurities contaminating molten metal.** L. JONES (U.S.P. 1,753,891, 8.4.30. Appl., 19.7.28).—Molten metal direct from a reduction furnace is filtered through a layer of slag in an open refractory-lined container. An upwardly inclined discharge spout of increasing cross-sectional area is provided at the bottom of the container and the definite quantity of metal maintained therein is covered by a layer of molten slag through which fresh metal falls from a perforated covering disc. The used slag may be run off at intervals as necessary. C. A. KING.

**Annealing furnaces.** C. F. KENWORTHY (B.P. 332,436, 26.8.29. U.S., 14.1.29).—The furnace comprises a chamber opening at the bottom into a container for holding the material to be annealed and mechanical means for moving this container below the chamber and for raising the articles into the furnace; means are also provided for maintaining a non-oxidising atmosphere in the furnace. A. R. POWELL.

**Production of pig iron.** M. NEUMARK (B.P. 331,190, 22.3.29).—The fluxes used in the blast-furnace operation are so proportioned that the resulting slag contains >30%  $\text{Al}_2\text{O}_3$  and <10%  $\text{SiO}_2$  and is rich in lime. The resulting iron has a low content of sulphur and phosphorus (cf. F.P. 629,277; B., 1930, 149). A. R. POWELL.

**Pickling process [for iron and steel].** J. C. VIGNOS (U.S.P. 1,736,332, 19.11.29. Appl., 29.8.28).—The metal is pickled in an acid ferrous sulphate bath through which chlorine is passed. A. R. POWELL.

**Prevention of incrustation and corrosion in waterpipes etc.** GROECK WASSERVEREDLUNG GES. M.B.H., Assees. of H. GROECK (B.P. 331,725, 14.8.29. Ger., 14.11.28).—Chromic acid or a soluble dichromate is added to the water of heating or cooling installations so as to form a light, non-adherent deposit instead of the usual hard crust. A. R. POWELL.

**Non-corrodible alloys of iron and articles produced therefrom.** K. M. TIGERSCHÖLD (B.P. 331,414, 2.8.29).—The alloys consist of iron with 15—30% Ni, 0—3% Cr, 0.05—1.5% C, 2.5—10% Si, and 0.2—2.5% Cu. Part or all of the silicon may be replaced by molybdenum and part or all of the nickel by cobalt. Sufficient carbon must be present to give the alloy an austenitic structure. A. R. POWELL.

**Method of soldering [iron-chromium alloys].** H. S. GEORGE, Assr. to ELECTRO METALLURG. CO. (U.S.P. 1,753,509, 8.4.30. Appl., 2.5.27).—Alloys such as stainless steel are soldered by applying a molten solder in conjunction with a piece of sodium to the metal faces. Ordinary silver solders containing about 80% Ag, 13% Cu, and 7% Zn are suitable. C. A. KING.

**Coating metal bodies [e.g., iron] with metal [e.g., aluminium].** W. BAUR (B.P. 331,550, 3.4.29).—The metal is coated with aluminium paint which is allowed to harden and then heated to destroy the varnish



vehicle. The aluminium film is subsequently rolled or hammered into the surface so as to fill up the pores and other surface irregularities.

A. R. POWELL.

**Melting and refining of copper.** C. A. JENSEN. From AMER. METAL CO., LTD. (B.P. 331,353, 7.6.29).—Copper is melted continuously below a layer of slag in a reverberatory furnace, new metal being charged into the flue of the furnace in such a manner that it is preheated practically to the m.p. before it is pushed into the bath of molten metal. The molten metal is tapped from below the slag layer at the opposite end of the furnace.

A. R. POWELL.

**Rendering articles of copper or copper alloys resistant to corrosion and oxidation.** J. LAISSUS (B.P. 308,353, 18.3.29. Fr., 22.3.28).—The articles are packed in a box filled with powdered chromium, tungsten, molybdenum, tantalum, manganese, silicon, nickel, cobalt, vanadium, titanium, uranium, or zirconium, either alone or in the form of alloys, with or without the addition of an inert diluent, *e.g.*, silica or alumina. The box is placed inside a second box which is filled with grey-iron turnings and the whole is heated at just below the m.p. of the articles for a sufficient time to allow the case-hardening element to diffuse into the surface to the desired extent. For electrolytic copper and brass a cementation mixture comprising 65–60% of 80% ferrotungsten and 35–40% of alumina is preferred.

A. R. POWELL.

**Melting down easily fusible and easily oxidisable metals and alloys.** C. BRACKELSBURG (B.P. 331,791, 7.11.29. Ger., 20.8.29).—A rotary tube-furnace heavily heat-insulated is heated by means of an oil or gas flame to a high temperature, the heat is shut off, the material to be melted is introduced, the furnace closed, and the charge allowed to melt by the heat of the furnace.

A. R. POWELL.

**Purifying light-metal [magnesium] alloys.** J. A. GANN, Assr. to DOW CHEM. CO. (U.S.P. 1,754,788, 15.4.30. Appl., 13.7.23).—The molten metal or alloy is agitated with a flux containing magnesium and sodium chlorides. Several treatments are advisable, but the flux from any treatment can be used for an earlier treatment of another batch of metal.

A. R. POWELL.

**Preventing corrosion of metallic surfaces.** H. SUTTON, A. J. SIDERY, L. F. LE BROcq, and C. BRAITHWAITE (B.P. 331,853, 8.4.29).—Magnesium, or its alloys, is protected by immersing it in a neutral or acid solution containing the chromate or dichromate of an alkali metal and an aluminium salt. After drying, the metal is dipped in a solution of lanoline in benzene or coated with lacquer.

C. A. KING.

**Welding [rods for ferrous metals].** W. B. MILLER, Assr. to OXWELD ACETYLENE CO. (U.S.P. 1,754,566, 15.4.30. Appl., 27.1.26).—The rod comprises soft iron, steel, or a chromium-iron alloy coated with a mixture of silica, ferromanganese, ferrosilicon, manganese ore, and a binder. The manganese and silicon may also be added as a manganese-silicon alloy with or without a proportion of magnesium which effectively removes gas from the joints.

A. R. POWELL.

**Welding-rod coating.** W. J. HAMMERS, Assr. to LIVERANCE & VAN ANTWERP (U.S.P. 1,752,793, 1.4.30.

Appl., 9.4.28).—A 2 : 1 mixture of iron oxide and felspar mixed to a cream with a 60% solution of sodium silicate is used for coating welding electrodes.

C. A. KING.

**Manufacture of saline flux coating compositions for use in soldering or welding.** ALLOY WELDING PROCESSES, LTD., Assees. of SOUDURE ELECTRIQUE AUTOGÈNE SOC. ANON. (B.P. 314,400, 26.6.29. Fr., 26.6.28).—A mixture of sodium and potassium chlorides is ground with hydrofluosilicic acid and a solution of lithium chloride is added, followed, after 1 hr., by potassium hydroxide solution to cause peptisation of the precipitated fluosilicates. The mass may be ground in a colloid mill and other crystalloids added, *e.g.*, sodium sulphite or phosphate. The peptised fluosilicates act as protective colloids for the finely-divided crystalloids present in excess of their solubility in water. The mixture is suitable for coating aluminium and other light-alloy welding rods.

A. R. POWELL.

**Flux for welding.** W. REUSS (B.P. 332,048, 3.7.29. Ger., 1.11.28. Addn. to B.P. 296,923; B., 1928, 821).—The flux previously described may contain in addition ammonium and sodium chlorides and sulphuric or other mineral acid. All or part of the sodium chloride may also be replaced by alkali sulphates or nitrates.

C. A. KING.

**Mercury-vapour boilers.** TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 310,880, 26.4.29. U.S., 2.5.28).—In a mercury-vapour boiler the heating surfaces with which mercury is in contact are lined or coated with a non-ferrous metal, *e.g.*, silver, which will be "wetted" by mercury. Heating tubes may be of iron or steel plated with silver or of solid non-ferrous metal or alloy.

C. A. KING.

**Recovery of mercury [from catalyst sludges].** R. RILEY, S. W. ROWELL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 332,106, 21.8.29).—Mercury sludge from acetylene hydration towers is chlorinated in a closed reaction vessel at the ordinary temperature, and the resulting mercuric chloride solution is treated with sodium hydroxide to precipitate mercuric oxide, which is redissolved in sulphuric acid to form mercuric sulphate for use again.

A. R. POWELL.

**Recovery of volatilisable metals [zinc, lead, bismuth, antimony, etc.] as oxides. Treatment of ores, metallurgical products, slags, etc.** F. JOHANNSEN, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,754,169 and 1,754,170, 8.4.30. Appl., [A] 4.1.26, [B] 14.10.26. Ger., [A] 26.5.25, [B] 5.11.25).—(A) After separation of the fine material by screening, the coarse material is passed downwards through a rotating tube-furnace into the middle of which is directed, from an injector at the lower end, a blast of compressed air carrying in suspension a mixture of powdered fuel and fine ore. In this way the latter is injected into the hottest zone of the furnace, thereby preventing caking and losses by dusting. (B) Easily fusible ores and metallurgical products containing volatilisable metals are fed into an inclined, rotating tube-furnace having an upper oxidising zone, and the fuel is introduced in a powdered form into the lower half of the furnace by means of an air blast. In this reaction zone the volatile metals are liberated and



the vapour is converted into oxide as it passes through the upper oxidising zone.

A. R. POWELL.

**[Lead-antimony] bearing metal.** P. KEMP (U.S.P. 1,754,364, 15.4.30. Appl., 11.1.28. Poland, 17.1.27).—The alloy contains 65–77% Pb, 3–14% Sn, 10–27% Sb, 0.7–2.5% As, and 0.5–3.5% Cd or In. The preferred composition is 66.8% Pb, 5% Sn, 25% Sb, 1.2% As, and 2% Cd.

A. R. POWELL.

**Manufacture of [A] lead alloy products and [B] lead yarn.** W. T. BUTCHER, W. CARROTT, and LOCKE, LANCASTER and W. W. & R. JOHNSON & SONS, LTD. (B.P. 330,925 and 330,960, 18.3.29).—(A) Lead alloys containing 4–12.5% Sb are rolled at 100–130° in a heavy rolling mill, which reduces the thickness of the casting by 50% in one pass, whereby the crystal structure is broken down without mechanical disintegration taking place. (B) Lead or lead alloys containing a small percentage of copper are extruded at 150–200° through triangular or polygonal holes in a metal die.

A. R. POWELL.

**Manufacture of nickel in a finely-divided state in particular for the catalytic manufacture of hydrogen from a mixture of methane and water vapour.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 314,944, 31.5.29. Holl., 5.7.28).—A solution of nickel nitrate in which a carrier may be suspended is treated at 40° with a slight deficiency of sodium hydroxide solution, so that the filtrate from the nickel hydroxide is perceptibly green. The carefully washed product is dried and reduced in hydrogen at 350°. A 72% yield of hydrogen is obtained by passing a mixture of methane and steam over the catalyst at 650°.

A. R. POWELL.

**Precipitating system [for metals].** A. G. MCGREGOR (U.S.P. 1,753,015, 1.4.30. Appl., 4.5.28).—In plant for the replacement of metals from solution by other metals, *e.g.*, copper from copper sulphate solution by scrap iron, the solution is circulated through a series of launders containing scrap iron, the newest iron coming into contact with the most dilute liquor. Each launder in turn is cut out of service for discharging and recharging without stopping the process. The precipitated copper and unattacked iron in the launder discharge are separated by washing through a rotary screen.

D. K. MOORE.

**Heat-insulating compositions for safes etc.** A. E. WHITE. From MOSLER SAFE CO. (B.P. 332,356, 11.6.29).—A mixture of a vermiculite material, *e.g.*, jefferisite, kerrite, or maconite, diatomaceous earth, and plaster of Paris is run in between or applied to the walls of the safe.

C. A. KING.

**Removal of carbon impurities from tantalum.** C. W. BALKE, Assr. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,754,453, 15.4.30. Appl., 2.9.26. Renewed 28.2.29).—Powdered tantalum containing small amounts of graphite is purified by heating it, *in vacuo*, with magnesia at a temperature at which the carbon will be oxidised and the resulting magnesium volatilised.

A. R. POWELL.

**Separation of metallic values [precious metals from ores].** E. W. STEVENS (U.S.P. 1,754,408, 15.4.30. Appl., 20.12.29).—The ore pulp is fed through a rotating

circular feeder in the vertical axis of a centrifuge into a Venturi nozzle from which it is ejected into the bowl of the centrifuge. The heavier metal particles settle against the sides of the bowl, whereas the light gangue is caused to circulate in the middle of the bowl by the action of the nozzle.

A. R. POWELL.

**Pen-point alloys.** J. E. WOODWARD, Assr. to KASTENHUBER & LEHRFELD (U.S.P. 1,753,161–2, 1.4.30. Appl., [A] 19.12.27, [B] 22.5.29).—(A) An alloy consisting of 70–85 (80)% Os, 5–15 (10)% Pt, and 5–25 (10)% of a base alloy which contains 65–85 (75)% of a metal of the tungsten group and 15–35 (25)% of a metal of the nickel group is claimed. (B) An alloy containing 75% W and 25 Ni is first formed by gradually adding tungsten to molten nickel at about 1900°. Osmium is then added to the base alloy in the proportion of 80% of osmium to 20% of the latter until the temperature reaches 2400° (the m.p. of the ternary alloy) and the product is finally chilled.

C. A. KING.

**Metallisation of thermally unstable substances, more particularly of organic electrically insulating substances.** SIEMENS & HALSKE A.-G. (B.P. 306,902, 20.2.29. Ger., 27.2.28).—The material is heated at 100–400° and sprayed with the vapour of a metal carbonyl either alone or admixed with hydrogen or nitrogen. [Stat. ref.]

A. R. POWELL.

**Coated metallic articles particularly in electrical apparatus and machinery.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 331,203, 18.3., 9.8., and 22.8.29).—Wires and metallic parts for electrical machinery are coated with resinous products made by the process of B.P. 327,722 (B., 1930, 624), to which, if desired, one or more colouring agents, fillers, or softening agents are added. The dried coating has a high dielectric constant and is highly elastic and pliable.

A. R. POWELL.

**Testing metallic coatings.** K. PITSCHNER, Assr. to AMER. CHAIN CO., INC. (U.S.P. 1,753,301, 8.4.30. Appl., 22.12.26. Renewed 16.5.28).—Plated metal, *e.g.*, nickel or copper on iron, is tested for pinholes, cracks, etc. in the plating by applying a strip of paper treated with ferroxyl solution, made by stirring agar-agar into water, boiling, adding sodium chloride and potassium ferri-cyanide with stirring, and thinning out with methyl or ethyl alcohol. The presence of pinholes in the plating is revealed by brown or blue spots developing on the test-paper.

C. A. KING.

**Alloy [for electrical heating units].** H. J. LORANG, Assr. to F. J. KUNA (U.S.P. 1,753,748, 8.4.30. Appl., 24.12.28).—The alloy contains 1.6% Cu, 1.6% Zn, 6.25% Sn, 21.75 Pb, and 68.75% Sb. The metals are melted in the presence of small quantities of manganese dioxide and borax.

C. A. KING.

**Manufacture of porous metal articles [iron and nickel accumulator plates].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 332,052, 8.7.29).—A porous negative plate for alkaline accumulators is prepared by sintering a mixture of iron powder from iron carbonyl and 15% of colcothar at 600° in an atmosphere of nitrogen and hydrogen, then for 2 hrs. at 1100–1200° in the same atmosphere; the plate has a pore



volume of 81% and a high resistance to breakage. A porous positive plate is obtained by sintering a mixture of 90% of nickel powder and 10% of iron oxide in the same way; the plate has a pore volume of over 76% and is a good carrier for the positive mass in alkaline accumulators.

A. R. POWELL.

**Production of printing plates.** A. R. TRIST (B.P. 331,220, 25.3. and 12.4.29).—A sheet of copper is electroplated with a thin film of chromium and the film is activated by making it the cathode in a 10% acid solution containing 10% of ammonium sulphate. The chromium is then covered with a thin film of electro-deposited nickel and a thin coating of silver bromide emulsion is applied to the nickel surface. The plate is exposed in the usual way and the image developed with a pyrogallol developer. The nickel thus exposed is removed by treatment with alcoholic ferric chloride and the exposed chromium by electrolysis in a mixture of 1 pt. of hydrochloric acid ( $d_1$  1.19) and 7 pts. of glycerin. The clean copper areas are then amalgamated by dipping the plate in mercuric cyanide solution. The plate is then cleaned, the nickel removed from the remainder of the plate by electrolysis in 2% hydrochloric acid, and the amalgamated copper part is plated with silver at 0.5 volt, at which *E.M.F.* silver is not deposited on the chromium.

A. R. POWELL.

**Manufacturing chemically pure aluminium by electrolysis.** K. L. MEISSNER (U.S.P. 1,770,940, 22.7.29. Appl., 1.8.27. Ger., 2.12.26).—See B.P. 299,869; B., 1928, 933.

**Protecting surfaces of aluminium or aluminium alloys.** G. D. BENGOUGH and J. M. STUART (U.S.P. 1,771,910, 29.7.30. Appl., 28.7.24. U.K., 2.8.23).—See B.P. 223,994; B., 1925, 14.

**Coating bodies with tungsten.** J. A. M. VAN LIEMPT, Asst. to N.V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,771,974, 29.7.30. Appl., 2.12.24. Holl., 28.6.24).—See B.P. 236,153; B., 1925, 726.

**Radiators** (U.S.P. 1,753,318).—See I. **Treating ores etc.** (B.P. 332,366).—See II. **Catalyst for ammonia oxidation** (B.P. 331,728). **Kilns** (B.P. 332,037).—See VII.

## XI.—ELECTROTECHNICS.

**Moisture determination of wood.** STAMM.—See IX. **Zinc.** HANLEY and others.—See X. **Manganese compounds and the quinhydrone electrode.** OSUGI and KASHIHARA.—See XVI. **Analysis of air.** HAMILTON.—See XXIII.

### PATENTS.

**[High-temperature] electric furnace.** BRITISH THOMSON-HOUSTON Co., LTD., Assees. of E. HERZOG (B.P. 316,562, 30.7.29. U.S., 30.7.28).—The heating chamber comprises a hollow ceramic resistor, *e.g.*, of zirconium silicate, having a negative temperature coefficient of resistance, in which are embedded ceramic rods of material which is electrically non-conducting when cold but becomes conducting at a lower temperature than that of the furnace walls. Current is supplied to carborundum rods or a resistance wound on a refrac-

tory core within the resistor in order to raise the resistor to the temperature at which the ceramic rods become conducting.

J. S. G. THOMAS.

**Electron-discharge device and getter therefor.** E. A. LEDERER, Asst. to WESTINGHOUSE LAMP Co. (U.S.P. 1,752,747, 1.4.30. Appl., 10.3.26).—A getter for an electron-discharge device consists of a phosphorus compound, *e.g.*, calcium metaphosphate, copper phosphide, or, more especially, phosphorus dinitride, a reducing agent, *e.g.*, powdered aluminium and magnesium, and a binder, *e.g.*, a solution of nitrocellulose in amyl acetate.

J. S. G. THOMAS.

**[Preventing liberation of spray in] electrolysis.** U. C. TANTON (B.P. 332,244, 17.4.29).—Substances, more especially a mixture of cresylic and silicic acids, together with gum arabic if desired, are added to the electrolyte so that a durable froth is formed.

J. S. G. THOMAS.

**Electrolytic cell.** F. LAWACZECK, Asst. to LAWACZECK GES.M.B.H. (U.S.P. 1,771,091, 22.7.30. Appl., 24.8.25. Ger., 1.9.24).—See B.P. 266,803; B., 1927, 338.

**Thermostatic controlling means for electric ovens etc.** N. DENNES, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 332,703, 19.6.29).

**Methods of making selenium cells.** C. ZEISS (B.P. 332,877, 27.12.29. Ger., 7.1.29).

**[Laminated] electrically insulating materials.** H. WADE, From BAKELITE CORP. (B.P. 332,594, 23.4.29).

**Emulsions etc.** (B.P. 332,533). **Degasifying liquids** (B.P. 332,542). **Controlling gas mixtures** (U.S.P. 1,753,319). **Hygrometers** (B.P. 332,135).—See I. **Acetylene and hydrogen** (B.P. 332,057).—See II. **Reducing metals** (U.S.P. 1,753,822). **Metallised insulating substances** (B.P. 306,902). **Coated articles** (B.P. 331,203). **Heating units** (U.S.P. 1,753,748). **Accumulator plates** (B.P. 332,052). **Printing plates** (B.P. 331,220).—See X. **Rubber articles from dispersions** (B.P. 331,563).—See XIV.

## XII.—FATS; OILS; WAXES.

**Selective dissolution of soap.** S. KAWAI (J. Soc. Chem. Ind., Japan, 1930, 33, 244—245b).—Commercial toilet soap was immersed in distilled water for different periods of time and the dissolved soap decomposed with hydrochloric acid. The neutralisation and iodine values and m.p. of the fatty acids obtained were compared with those of the original soap content. The selective effect of dissolution was very marked and is considered to be of importance in the cracking of soap surfaces.

C. IRWIN.

**Fluorescence of olive oil under ultra-violet light.** A. LE R. GLANTZ (Ind. Eng. Chem. [Anal.], 1930, 2, 256—258).—All pure virgin oils exhibit a yellow fluorescence under ultra-violet light, and all refined oils show a characteristic blue fluorescence. By examination of the fluorescence, adulterations of as little as 5% of refined oil in a virgin oil can be detected. The fluorescence of California oils is due to the application of



heat during the pressing and also to the extent to which the olives are pressed. The spectrum analysis of virgin olive oils reveals a characteristic red band at  $669\mu$ , which is lacking in refined oils, but reappears on the addition of chlorophyll to the oil. The blue fluorescence of an oil is independent of its chlorophyll content. The result of adding carotene and annatto showed that the yellow fluorescence of virgin oils can be regained, and it is believed that this property of virgin oils is due to the content of carotene, which is destroyed during the refining. The addition of annatto, but not of carotene, can readily be detected by chemical analysts. E. S. HEDGES.

**Detection of carnauba wax in beeswax.** L. K. WATSON (Amer. Bee J., 1930, 70, 118—120, 146).—The temperature of crystallisation of beeswax from butyl alcohol ( $36-40^\circ$ ) is raised by carnauba wax (but not by tallow or paraffin) proportionally to the amount present. The microscopical appearance of crystals of beeswax (radiating shafts, or flakes) is altered (to minute stars or spherulites) in the presence of carnauba wax.

CHEMICAL ABSTRACTS.

## PATENTS.

**Treating materials** (B.P. 332,577 and 332,593). **Making emulsions** (B.P. 331,928).—See I. **Sulphonated hydroxy-fatty acids** (B.P. 330,904).—See III. **Extracting fat from carcasses etc.** (B.P. 332,637).—See XIX.

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

**Testing wood primers.** H. A. GARDNER and L. P. HART (Amer. Paint & Varnish Manufs.' Assoc., Circ. 368, July, 1930, 343—353).—An accelerated test is described in which the untreated backs of exposed painted panels were subjected to a humidity of 88% at  $46^\circ$  for 6 days. Moisture percolating through the wood causes rapid blistering etc. of incorrectly primed systems. The panels were subsequently removed to a test frame for further exposure, when paint affected by moisture in the early stages of drying usually disintegrate quickly by washing and chalking. The panels were braced to prevent warping. Details of a comprehensive series of tests recently undertaken on these lines on paint systems over various primers on different types of wood are given. S. S. WOOLF.

**Modification of the spinning-film hiding-power method [for paints].** G. S. HASLAM (Ind. Eng. Chem. [Anal.], 1930, 2, 319—322).—A method for measuring the hiding power of paints, regardless of their pigment or vehicle composition, has been developed. The test consists in applying a uniform film to a contrasty panel by spinning on an eccentric spinning device. Eye-grading in north light is the final criterion of hiding. Dry hiding power does not always agree with wet measurements as made on the cryptometer, and some of the exceptions are discussed. E. S. HEDGES.

**The Pfund glossmeter applied to paints and lacquers.** G. S. HASLAM and L. D. GRADY, JUN., (Ind. Eng. Chem. [Anal.], 1930, 2, 346—351).—An apparatus for measuring objective gloss is described.

Data are given to illustrate the adaptability of the apparatus to paint and lacquer problems.

E. S. HEDGES.

**Analysis of red lead.** G. A. AMPT (J. Soc. Chem. Ind. Victoria, 1930, 30, 353—360).—Current methods of analysis are critically examined and the following procedure is advocated: 1 G. of sample is decomposed with 15 c.c. of 5*N*-nitric acid, 115 c.c. of water are added, and the liquid is heated to  $100^\circ$ . 20 C.c. of 0.2*N*-oxalic acid are then added and, after 10 min., the solution is titrated with 0.1*N*-permanganate. It is shown that the concentration of the nitric acid affects the titration and should be kept within specified limits.

H. I. DOWNES.

**Fast pigments for façades.** H. RASQUIN (Farben-Ztg., 1930, 35, 2279—2281).—Exposure tests on aged cement of various pigments in proprietary aqueous-emulsion vehicles are detailed. The suitability of the pigments for exterior painting work is discussed, with comments on their lime-fastness. S. S. WOOLF.

**Constitution of artificial resins.** M. KOEBNER (Chem.-Ztg., 1930, 54, 619).—Published work explaining the constitution of phenol-formaldehyde resins by the formation of chains of phenol groups linked by methylene groups is briefly summarised. Crystalline compounds containing 2 and 3 cresol groups have been discussed previously (cf. Morgan, also Megson and Drummond, B., 1930, 600), and the preparation of chains containing up to 7 members is described. Whilst the less complex compounds are soluble in caustic soda solution, the material containing 7 phenol groups was apparently unaffected thereby, but was subsequently shown to form sodium salts insoluble in water. This loss of solubility has been erroneously attributed to loss of phenolic character by esterification etc., but the elimination of water to an extent made necessary by this assumption is considered unlikely. S. S. WOOLF.

**Rubber micro-sections.** ALLEN.—See XIV. **Foreign materials in resins etc.** GEORGI.—See XVII. **Hop resins.** BAZILEVICH.—See XVIII.

## PATENTS.

**Mills for grinding paints, enamels, inks, and other viscous substances.** DISC BAR MILLS, LTD., and E. A. WHITE (B.P. 331,762, 17.9.29).—The mill comprises a disc rotating against or near to a stationary block. The surfaces may or may not be roughened.

B. M. VENABLES.

**Drying kilns** (B.P. 332,491).—See I. **Cellulose acetate compositions** (B.P. 306,911).—See V. **Titanium pigments** (B.P. 308,725).—See VII. **Laminated glass** (B.P. 332,234).—See VIII. **Coated articles** (B.P. 331,203).—See X. **Adhesive** (B.P. 331,428).—See XV.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Autographic stress-strain curves of rubber at low elongations.** A. A. SOMERVILLE, J. M. BALL, and L. A. EDLAND (Ind. Eng. Chem. [Anal.], 1930, 2, 289—293).—A new test-piece adapted to the autographic registration of stress-strain curves is described, and the effects of variations in curves, speed of testing machine,



temperature, humidity, and loading of carbon black are discussed. E. S. HEDGES.

**Making micro-sections of rubber stocks.** R. P. ALLEN (Ind. Eng. Chem. [Anal.], 1930, 2, 311—313).—A simple method for making thin sections of rubber for microscopical examination is described. For clear observation of highly-loaded gas-black stocks the sections must be less than 1  $\mu$  thick, though somewhat greater thicknesses may be used with lighter-coloured pigments, such as litharge and zinc oxide. The method has been developed for the study of pigments in rubber stocks, but it is further pointed out that rubber is an excellent medium in which to examine finely-dispersed pigments, for it is rigid enough to prevent Brownian movement and sufficiently plastic to be easily deformed, and its refractive index is considerably different from that of most pigments. E. S. HEDGES.

**Viscosity of rubber solutions under the influence of benzoyl peroxide.** G. FROMANDI (Kautschuk, 1930, 6, 177—180).—In the determination of mineral ingredients such as antimony sulphide in commercial rubber articles by heating with a rubber solvent such as dekalin, the addition of about 3% of benzoyl peroxide to the solution after disintegration of the sample followed by further boiling for 1—2 hrs. greatly facilitates sedimentation of the mineral matter. The addition of benzoyl peroxide (1 g.) to a solution of purified rubber (1 g.) in benzene (100 c.c.) causes a greater rate of decrease in the viscosity of the solution than heating at 80°. Exposure of the solution, with or without benzoyl peroxide, to sunlight or ultra-violet radiation accelerates the change, the effect of sunlight for one day being roughly equivalent to that of heating at 80° for 8 hrs. D. F. TWISS.

**Application of the falling cylinder to the measurement of the viscosity of thick rubber cements.** W. F. BUSSE and W. B. DOGGETT (Ind. Eng. Chem. [Anal.], 1930, 2, 314—319).—With many thick cements the time of fall,  $t$ , of cylinders can be expressed by the formula  $dt = k \cdot dx^2(W - \pi r^2 \rho x)^n$ , where  $\rho$  is the density of the cement,  $n$  a constant of the cement, and  $k$  a constant which is independent both of the distance,  $x$ , through which the cylinder falls, and of the weight,  $W$ , but varies with the cement under test and the radius of the cylinder used. A 15% suspension of clay in mineral oil, in contrast with rubber solutions, showed a definite yield value and also anomalous turbulence. Falling-cylinder viscosimeters using steel cylinders have been designed for use in control testing, and simplified equations for their use have been developed. They were found to be satisfactory over a wide range. The thicker cements made from only slightly milled rubber do not follow the law on which the equations given are based, but even in these cases the viscosimeters can be used for control testing, provided that the shearing force at the surface of the cylinder is not greater than the adhesion of the cement to the cylinder surface. The steel viscosimeters have been used in testing asphalts having viscosities up to about 900,000 poises and should be useful in testing other materials. E. S. HEDGES.

#### PATENTS.

**Treatment of rubber and products thereof.** NAUGATUCK CHEM. CO., Assees. of S. I. STRICKHOUSER

(B.P. 332,489, 30.10.29. U.S., 26.11.28).—Surface-cracking of soft vulcanised rubber materials, especially those of high rubber content, is retarded by incorporating an  $\alpha$ -diketone (e.g., 0.5% on the rubber content), such as benzil, phenanthraquinone, or furil.

D. F. TWISS.

**Manufacture of articles of rubber and similar substances from aqueous dispersions containing such substances.** DUNLOP RUBBER CO., LTD., D. F. TWISS, and R. G. JAMES (B.P. 331,563, 7.2.29. Addn. to B.P. 301,100; B., 1929, 105).—Aqueous rubber dispersions in which the particles are negatively charged are treated with formic or acetic acid in the presence of a suitable protective colloid to make the charge on the particles positive, and are then subjected to electrophoretic deposition upon cathodes coated with a paste of litharge, red lead, and glycerin to prevent liberation of hydrogen. A. R. POWELL.

**Manufacture of rubber goods.** ANODE RUBBER CO., LTD. (B.P. 317,435, 4.4.29. U.S., 16.8.28).—The material to be coated is dipped first into rubber latex, then into a solution of a coagulant, and, after wiping, into an aqueous dispersion of rubber. Any desired thickness of rubber may be built up by repeating the second and third operations; the whole is then vulcanised *in situ* or after removing the rubber layer from the base. A. R. POWELL.

**Manufacture of artificial sponges of rubber.** Y. CORNIC (B.P. 314,539, 27.6.29. Fr., 29.6.28).—Rubber latex containing 70—80% of rubber is treated with 1% of an accelerator base (a guanidine derivative), 10—12% of a saturated solution of sulphur in carbon disulphide, 4% of zinc oxide, a suitable quantity of inert filler and/or colouring matter, and 12% of ammonium carbonate. The whole is thoroughly mixed and heated at 140° for 2—3 hrs., whereby the ammonium carbonate decomposes as the rubber vulcanises and a porous spongy mass is obtained. A. R. POWELL.

**Manufacture of rubberised fabric etc.** FIRESTONE TYRE & RUBBER CO., LTD. From FIRESTONE TYRE & RUBBER CO. (B.P. 331,594, 10.1.29).—In the manufacture of rubberised fabric the material is passed through a drying chamber to which inert gas is supplied, and the composition of the gas mixture in the chamber is controlled by passing a portion of it over an electrical resistance coil the resistance of which varies according to the thermal conductivity of the gas mixture. A. R. POWELL.

**Manufacture of sea caoutchouc.** C. A. HOUQUES-FOURCADE (U.S.P. 1,772,218, 5.8.30. Appl., 8.10.27. Fr., 9.10.26).—See F.P. 622,896; B., 1928, 762.

**Rubber-like substance from cashew-nut-shell oil.** M. T. HARVEY, Assr. to HARVEL CORP. (U.S.P. 1,771,785, 29.7.30. Appl., 28.11.25).—See B.P. 262,134; B., 1928, 420.

#### XV.—LEATHER; GLUE.

**Effect of neutral salts on the alkaline swelling of hide powder. I. Sodium chloride.** D. WOODROFFE (J. Soc. Leather Trades' Chem., 1930, 14, 355—358).—The addition of sodium chloride to 0.1N-sodium hydroxide causes a diminution in its swelling power on hide powder. As the amount of added sodium chloride



is increased, the swelling power diminishes to a minimum, after which further additions of salt cause a rapid rise in swelling power.

D. WOODROFFE.

**Stripping effect of fat liquors on dyed leathers.** D. WOODROFFE (J. Soc. Leather Trades' Chem., 1930, 14, 353—355).—Samples of vegetable-tanned sheepskin dyed with different acid dyes were fat-liquored with different commercial fat liquors and soap-oil fat liquors. It was found that all except sulphonated cod oil stripped some of the colour of the dyed leathers, but the amount of stripping depended on the particular fat liquor used, the  $p_H$  values of which were found to differ. Samples of leather dyed with 32 different acid dyes were treated with the above-mentioned fat liquors, one half of each sample being dried out and the other treated with acid before drying. No stripping of the colour was observed from those pieces which had been pretreated with acid. The stripping effect of fat liquors is due to their effect on the  $p_H$  of the dyed leather.

D. WOODROFFE.

**Analysis of bating materials.** A. BOLDIN (J. Soc. Leather Trades' Chem., 1930, 14, 358—363).—Milk concentrated to one half its volume and to which 4% of calcium chloride has been added is used as the substrate. One part of the bating material is extracted with 10 pts. of a solution ( $p_H$  5.3) containing 44.1 g. of potassium dihydrogen phosphate and 2.97 g. of disodium hydrogen phosphate in 5 litres of water. Then 1 c.c. of the milk, 0.2 c.c. of a solution ( $p_H$  5.8) containing 95.5 c.c. of 0.67*M*-potassium dihydrogen phosphate and 2.5 c.c. of 0.6*M*-disodium hydrogen phosphate, 1.0—0 c.c. of boiled enzyme infusion, and 0—1.0 c.c. of untreated enzyme infusion are mixed and kept in a water-bath at 35°; the time taken for an image on a cinematograph film to be seen through the mixture is then compared with that required by a standard product under similar conditions.

D. WOODROFFE.

**Use of calcium hydroxide prepared from marble in the leather industry.** J. OETTINGER (Chem.-Ztg., 1930, 54, 586).—The product obtained by slaking marble lime sufficiently to yield a dry powder contains 96%  $\text{Ca}(\text{OH})_2$ . By its use in liming, a large excess is unnecessary, stains and lime burns are avoided, and the lime pits do not require to be emptied so frequently. This lime does not require to be slaked. It can be used for making lime paints or in liming glue pieces.

D. WOODROFFE.

**Japanese dyeing tannins.** VII. **Analysis of the dyeing tannins by the cinchonine method.** Y. UYEDA (J. Soc. Chem. Ind., Japan, 1930, 33, 228—231b).—The effect of varying conditions on the determination of tannin by the cinchonine method is examined. To ensure accuracy the solution to be analysed should contain about 0.4% of soluble tannin to which are added 25 c.c. of cinchonine sulphate solution (1.3 g./100 c.c.) for each 0.1 g. of tannin in solution. F. R. ENNOS.

**Problem of tanning and its generalisation.** L. MEUNIER and K. LE VIET (Rev. Gén. Colloïd., 1930, 8, 49—61).—A method for measuring swelling particularly applicable to fibrous structures has been developed; it entails centrifuging the substance so as to determine

the bound liquid and not the capillary liquid. The method has been applied in an examination of the swelling of hide and the effect of some organic phenolic compounds thereon. The amount of swelling is considered to be determined by the presence in the protein molecules of certain lyophilic groups, particularly amino-groups, which exert a polarity comparable with that of the water molecules in the external liquid. When tanning occurs, these groups are transformed irreversibly into less polar groups and the resulting substance is greatly reduced in swelling capacity. The measurement of swelling gives a value for the astringence of the tanning solution. The action of certain tanning agents, such as quinol, depends on the external conditions of oxidation. The theory of tanning suggested is extended so as to apply to general colloid phenomena of a similar nature. The swelling of cellulose and of agar is considered to be due to the presence of hydroxyl groups, and the action of tanning agents is similar to the effect on collagen.

E. S. HEDGES.

**Standard methods (revised) for determining viscosity and jelly strength of glue.** (Ind. Eng. Chem. [Anal.], 1930, 2, 348—351; cf. B., 1924, 394).—Standard methods adopted by the National Association of Glue Manufacturers (U.S.A.), for sampling and preparation of sample, testing of sample, viscosity determination, jelly strength determination, and chilling are given. Some general precautions are emphasised.

E. S. HEDGES.

**Foreign materials in glue etc.** GEORGI.—See XVII.

#### PATENTS.

**Pretreatment of hides for promoting tanning thereof and preparations therefor.** B. QUENDT (B.P. 332,262, 10.1.29).—To accelerate the subsequent tannage, hides ready for tanning are pretreated with a solution of a water-soluble, non-sulphonated condensation product of an arylamine (e.g., aniline, dimethylaniline) with formaldehyde, alone or with a dilute tanning solution, or with a mixture of alum and one or more neutral salts (e.g., Glauber's salt).

D. WOODROFFE.

**Treatment of casein.** P. SCHIDROWITZ, Assr. to J. B. CROCKETT (U.S.P. 1,754,651, 15.4.30. Appl., 8.12.28).—A preparation of casein which will remain fluid when cold is made by heating it with alkali solution and then adding piperidine.

E. B. HUGHES.

**Adhesives.** J. TAYLOR and A. V. KELLER (B.P. 331,428, 29.8.29).—Urea, thiourea, or their derivatives are heated at 90° with paraformaldehyde and water to produce a thin paste; a violent reaction occurs with effervescence and a transparent liquid is obtained which is evaporated at 90° to a syrupy consistency before use as an adhesive.

A. R. POWELL.

**Unhairing and preparing hides for tanning.** C. K. M. M. LE PETIT, Assr. to ROHM & HAAS Co. (U.S.P. 1,772,258, 5.8.30. Appl., 7.7.25. Fr., 18.4.25).—See B.P. 250,907; B., 1926, 1022.

**Adhesive or cement.** R. ARNOT (U.S.P. 1,771,553, 29.7.30. Appl., 3.9.24. U.K., 15.9.23).—See B.P. 225,953; B., 1925, 108.



Cellulose acetate compositions (B.P. 306,911).—See V. Dyeing leather (B.P. 309,166).—See VI. Dissolving glue etc. (B.P. 307,079).—See XVII.

## XVI.—AGRICULTURE.

**Lime additions to friable clay.** W. BRENNER (Bull. Agrogeol. Inst. Finland, 1929, No. 29, 13 pp.; Proc. Internat. Soc. Soil Sci., 1930, 5, 129—130).—Addition to soil of lime in quantities predetermined from titration curves to produce  $p_H$  changes up to 5.6 effected the calculated changes. To bring about  $p_H$  6 in the field, 1.6 times the quantity of lime calculated from laboratory tests was required. It was impossible in the field to bring the  $p_H$  value to 7.0. The most heavily limed areas of this soil rapidly developed further acidity.

A. G. POLLARD.

**Agrogeological studies in the tropics. I. High altitudes of the Oriental tropics.** M. W. SENSTIUS (Soil Res., 1930, 2, 10—56).—Modern conceptions of soil formation are summarised and discussed. Field examination and laboratory analyses of tropical soils from high altitudes are recorded. Although podsoils were not found at high altitudes, evidence is obtained that in these soils the podsolisation process has occurred with minor modifications, which are examined and explained.

A. G. POLLARD.

**Adsorbed bases and unsaturation of Polish sandy soils.** W. BUTOWSKI (Rep. Polish Agric. Exp. Sta., 1928, 2, 195; Bied. Zentr., 1930, 59, 348).—The proportion of adsorbed bases in the soils examined varied with their mechanical composition and increased with the clay content. The plant covering of forest soils increased the adsorbed bases in the upper horizons. The amount of adsorbed bases was less in the B-horizon than in the surface, but increased at still greater depths approaching the parent rock. Variations in the degree of unsaturation were parallel with those of the base content and were greatest in the loams. The genesis of the soils is discussed. Podsolisation is considered to take place in these soils.

A. G. POLLARD.

**Drainage and leaching trials at Peradeniya, 1927-30.** A. W. R. JOACHIM (Trop. Agriculturalist, 1930, 74, 323—330).—Analyses of drainage waters from cropped and uncropped soils in cylinder experiments are recorded. In the leachings nitrates, calcium, magnesium chloride, and potassium occurred in decreasing order of magnitude.

A. G. POLLARD.

**Soil humus.** S. OSUGI and Y. SANO (J. Sci. Soil and Manure; Proc. Internat. Soc. Soil Sci., 1930, 5, 112—113).—Soil humus was fractionated by Woksman's method. The soluble portion is too small to be significant. The carbon, nitrogen, and ash contents of the  $\alpha$ - and  $\beta$ -fractions were examined. No particular relationships between the  $\alpha$ -fractions of rich humus soils or the  $\beta$ -fractions in mineral soils were observed. Schollenberger's chromic acid method for determining humus (B., 1927, 663) is satisfactory if samples of suitable size are used, e.g., 0.01 g. sample of soil with 30% humus, 0.02 g. with 15.20%, and 0.1 g. with 5%.

A. G. POLLARD.

**Reactions of electrodyalysed humus and benton-**

**ite.** A. O. ALBEN (J. Amer. Soc. Agron., 1930, 22, 311—326).—Observations on Tama and Clinton silt loams are recorded; the final  $p_H$  was 2.04. The  $p_H$  of an electrodyalysed soil returned in 60 days to 4.0. The application of electrodyalysis to basic soils is discussed.

CHEMICAL ABSTRACTS.

**Mechanical analysis of soils without acid pretreatment.** L. B. OLMSTEAD and L. T. ALEXANDER (Soil Res., 1930, 2, 68—72).—Modifications of the International method of mechanical analysis are described. In the pretreatment of the sample, the acid decomposition is omitted and, where manganese dioxide is present in sufficient amount to prevent the satisfactory action of hydrogen peroxide, glacial acetic acid is added in quantity slightly in excess of the equivalent of manganese dioxide. Subsequent filtration and washing of the sample is carried out with a Pasteur-Chamberland candle from which the soil is removed by filling the cone of the candle with distilled water and applying pressure. The washed sample is evaporated to dryness on a steam-bath and baked in an oven at 105°. This permits of determinations of "loss by solution" without interfering with the subsequent dispersion of the sample. The sample is shaken with water containing 10 c.c. of 0.5*N*-sodium oxalate and, after separation of the coarser fractions by sieving (up to 300-mesh), the suspension is diluted to 1 litre for analysis by the pipette method for clay (<5 $\mu$ ) and colloid (<2 $\mu$ ). Variations in the amount of sodium oxalate necessary for complete dispersion may be necessary.

A. G. POLLARD.

**Dispersion and mechanical analysis of certain soils high in sesquioxides.** L. T. ALEXANDER (Soil Res., 1930, 2, 72—76).—A sample of bauxite could not be dispersed for mechanical analysis either with sodium oxalate (cf. previous abstract) or with ammonium hydroxide, but was completely dispersed with sodium hydroxide (5 c.c. of 0.6*N*-solution per litre). Lateritic soils of low silica : sesquioxide ratio and small base-exchange capacity may tend to flocculate in sodium oxalate suspensions and are preferably dispersed in sodium hydroxide. This difficulty arises only when the colloid fraction (<2 $\mu$ ) is required separately, the sodium oxalate method being generally suitable when only the clay fraction (<5 $\mu$ ) is needed.

A. G. POLLARD.

**Nitrification in uncultivated soils. I. Various analyses. II. Nitrification as affected by hydrogen-ion concentration.** T. GAARDER and O. HAGEM (Medd. Vestlandets Forstl. Forsøksstat., 1921, 2, No. 4, 172 pp.; 1928, 4, No. 11, 194 pp.).—I. Nitrate production in numerous uncultivated soils is examined. Poor nitrification in many forest and peat soils is traced either to the presence of denitrifying organisms or to the cessation of the oxidation process at the nitrite stage. The moisture content of soil affects nitrification mainly by its influence on soil aeration. The nitrifying power of soil is influenced directly by the soil reaction (but not by its buffer capacity) and by the presence of suitable mineral nutrients for the organisms concerned, and, indirectly, by the numerous factors controlling ammonification. The presence of nitrites in soil interferes with the accuracy of the phenolsulphonic acid method for



determining nitrates. Preliminary decomposition with urea is recommended.

II. The effect of the soil reaction on the nitrification of ammonium sulphate is examined. Nitrifying organisms are active within the  $p_H$  limits 6.0–7.8 (approx.). Exact values are difficult to specify owing to the progressive change in soil reaction occurring during the process. Probably four types of organisms, each active over a specific range of reaction, are present in soils. Nitrification of nitrites occurs in soils with  $p_H$  6.8 to above 9.0; here again several types of organisms are concerned.

A. G. POLLARD.

**Agreement between "Azotobacter values" and results of the seedling method.** H. NIKLAS, H. POSCHENRIEDER, and F. CZIBULKA (Superphosphat, 1930, 6, 111–115).—Comparative investigations of numerous soils by the Neubauer and *Azotobacter* methods for determining available phosphate contents are recorded, and from the values obtained co-ordination tables for the two methods are prepared. In general, agreement between the methods is good, the least satisfactory being the intermediate range of phosphate contents corresponding with Neubauer values of 4–8 mg.

A. G. POLLARD.

**Potassium thiocyanate method for determining soil acidity.** Y. KAMOSITA (J. Imp. Agric. Exp. Sta., Tokyo; Proc. Internat. Soc. Soil Sci., 1930, 5, 103–104).—The colour intensity in the Comber test for soil acidity was standardised by titrating the alcoholic extract with potassium hydroxide solution. Values so obtained for a number of soils were compared with  $p_H$  values, exchange acidity, and exchangeable base content. Soils exhibiting exchange acidity have  $p_H < 5.5$ . Soils with  $p_H < 6.88$  give, with Comber's test, red colorations which increase in intensity with increasing exchange acidity. In soils showing exchange acidity the proportions of exchangeable calcium, and magnesium are smaller than those of iron and aluminium. The reverse is the case where no exchange acidity exists. It is suggested that the red coloration in Comber's test is due to the exchange of ferric ions for potassium ions in the thiocyanate and that exchange acidity is partly the result of the presence of exchangeable iron.

A. G. POLLARD.

**Influence of adsorbed ions on soil reaction.** B. AARNIO (Bull. Agrogeol. Inst. Finland, 1927, No. 22; Proc. Internat. Soc. Soil Sci., 1930, 5, 105–106).—Clays were saturated with various cations by treatment with solutions of the appropriate chlorides and, after washing, the  $p_H$  values of the suspensions were determined. Normal soils saturated with naturally occurring cations had neutral or weakly alkaline reactions. The effect of the uni- and bi-valent cations on the  $p_H$  value was in the order of their hydration values. The hydrogen- and aluminium-clays were much more acidic. Treatment of acid soils with chlorides reduced their  $p_H$  values in every case, the adsorbed hydrogen and aluminium being exchanged and the reaction altered accordingly. In this respect the effect of univalent ions is greater than that of bivalent ions. Extreme alkalinity and acidity in soils is caused by the presence of carbonates (alkali soils) and of hydrolysable iron and aluminium salts,

respectively. Adsorbed hydrogen ions can be removed from clays only by base exchange and not by leaching with water.

A. G. POLLARD.

**Effect of various manganese compounds on the quinhydrone electrode.** S. OSUGI and H. KASHIHARA (J. Sci. Soil and Manure, 1929, 3, 4; Proc. Internat. Soc. Soil Sci., 1930, 5, 104).—The presence of oxides of manganese ( $MnO_2$  and  $Mn_2O_4$ ) in small proportions (0.001% and 0.005%, respectively) caused inaccuracies in  $p_H$  measurements with the quinhydrone electrode. In soils known to contain manganese, however, no significant differences in  $p_H$  values as determined by hydrogen and quinhydrone electrodes were observed.

A. G. POLLARD.

**Use of barium sulphate for clarifying soil suspensions, with particular reference to colorimetric  $p_H$  determinations.** L. D. BAVER and C. J. REHLING (Ind. Eng. Chem. [Anal.], 1930, 2, 338).—Experiments on 19 different types of soils showed that pure barium sulphate containing no soluble barium salts can be used for clarifying soil suspensions without materially affecting their  $p_H$  values. The amount of barium sulphate required depends on the texture and the degree of dispersion of the soil. Clays and silt loams require more than sandy soils.

E. S. HEDGES.

**Report of Committee on soil reaction measurements. I.** D. J. HISSINK (Soil Res., 1930, 2, 77–139).—Detailed results of comparative trials of the quinhydrone electrode method are recorded.

A. G. POLLARD.

**Nitrite production in soils.** G. S. FRAPS and A. J. STERGES (Ind. Eng. Chem., 1930, 22, 863–864).—Considerable proportions of nitrite-nitrogen (up to 360 p.p.m.) were found in certain soils and subsoils. Ammonium sulphate is oxidised to nitrites in these soils, but no nitrate is formed. Addition of calcium carbonate increased nitrite production. Nitrates and nitrites can be extracted from soils with water containing calcium hydroxide and the extract remains stable for at least 7 days.

A. G. POLLARD.

**Adsorption of nitrates by Kuban chernozem.** P. A. KURCHATOV and E. S. KRAMAROVA (Nauch. Agron. Zhur., 1925, 6, 329–325).—The capacity for adsorption of nitrate was higher when the soil originally contained nitrates. Lateritic soils adsorbed large amounts. The humus-free parent material of chernozem adsorbed less than the horizons with the humus.

CHEMICAL ABSTRACTS.

**Fertilising effects of sodium nitrate in paddy fields.** Y. KIDA (J. Sci. Agric. Soc. [Nogaku Kwai Ho], No. 245 and 246; Proc. Internat. Soc. Soil Sci., 1930, 5, 130–134).—Comparison is made of the efficiencies of ammonium sulphate and sodium nitrate used alone and mixed with varying proportions of oil cake.

A. G. POLLARD.

**[Fertiliser trials at Rothamsted.]** (Rothamsted Expt. Sta. Rept. for 1929, 125 pp.).—Efficiency of phosphate fertilisers on grass land is in the order superphosphate > high-solubility basic slag > low-solubility basic slag > mineral phosphate. The high-solubility fertilisers increased the proportion of phosphate in the herbage. Superphosphate increased the potato crop



provided no nitrogen was given, but decreased it in the presence of nitrogen and potash. In cooking tests the colour of potatoes which had received potash fertilisers was superior. In this respect the chloride gave best results, followed, in order, by the sulphate and potash manure salts. In flavour only the best of the potatoes fertilised with potash equalled that of the unfertilised, all others were inferior. Complete fertilisers gave best results with sugar beet, and these were still further improved by dressings of salt. With barley, cyanamide and ammonium chloride again gave as good results as ammonium sulphate, though still inferior to sodium nitrate. Potash reduced the attack of *Fusarium culmorum*. In pot culture chlorides delayed the ripening of barley straw and gave a lower dry weight yield than sulphates. The highest-quality barley was obtained with ammonium chloride and urea. No harm resulted from sowing up to 2 cwt. of cyanamide per acre with the seed, but 4 cwt. per acre was distinctly injurious.

A. G. POLLARD.

**Influence of liming on the dynamics of soil processes.** N. P. REMEZOV (Trans. Sci. Inst. Fertilisers, Moscow, 1930, No. 65, 3—58).—Liming increases the citrate-soluble phosphorus and the nitrates; it is more effective in combination with barnyard manure.

CHEMICAL ABSTRACTS.

**Influence of varying amounts of phosphates on the dynamics of soil processes.** P. N. KOSHELOV (Trans. Sci. Inst. Fertilisers, Moscow, 1930, No. 65, 59—89).—The yield and phosphorus content of plants, and the soil nitrate, were increased by application of raw phosphate.

CHEMICAL ABSTRACTS.

**Tomato, a sensitive and rapid indicator of phosphate deficiency in soils.** L. MAYER (Forts. Landw., 1929, 4, 684; Bied. Zentr., 1930, 59, 348—353).—Tomato seedlings grown in test soils artificially supplied with sufficient nitrogen and potash indicate phosphate deficiency after 8—14 days, *i.e.*, when the first pair of true leaves have developed, by (1) the upward inclination of the seed leaves forming an acute angle, (2) the first pair of true leaves remaining unparted at the tips, (3) the dark green colourings of the upper side and the violet-red colour of the undersides of both true and seed leaves. By allowing plants to continue growth for 2—3 months, the relative phosphate contents of soils may be compared on the basis of the weights of the plants. Exhaustion of phosphate is marked by cessation of growth and the colouring of the undersides of the leaves. Results so obtained agree well with those of Neubauer's and of Wrangell's extraction methods.

A. G. POLLARD.

**Effect of soil type on the availability of finely-ground rock phosphate.** P. O. RIPLEY (Sci. Agric., 1930, 10, 678—689).—Pot trials with barley grown in several soil types are recorded. In all cases finely-ground rock phosphate proved decidedly inferior to superphosphate. Plants can absorb phosphate from soils in which water-soluble phosphorus cannot be detected by customary methods. The growth of plants is not directly proportional to the accumulation of phosphorus in the sap.

A. G. POLLARD.

[Conversion of crude phosphates into concen-

trated fertilisers]. S. I. VOLFKOVICH and V. P. KAMZOLKIN [with others] (Trans. Sci. Inst. Fertilisers, Moscow, 1929, No. 67, 8—134).—Optimal conditions of fineness, temperature, time of reaction, and concentration of sulphuric acid were determined for phosphates from various deposits. Factory tests of the manufacture of precipitated calcium phosphate are also described.

CHEMICAL ABSTRACTS.

**Utilisation of natural aluminium phosphate.** Y. KIDA (Proc. Pan-Pacific Sci. Congr.; Proc. Internat. Soc. Soil Sci., 1930, 25, 137—138).—Methods for converting natural aluminium phosphate into valuable fertilisers are described. The mineral may be decomposed by sulphuric acid and neutralised with ammonia, lime, or tricalcium phosphate. Alternatively, sodium phosphate may be obtained by treating the mineral with hot sodium hydroxide solution. Products of fusion processes, although sometimes suitably soluble, were usually hygroscopic.

A. G. POLLARD.

**Effect of peat and phosphorite on the yield and composition of summer wheat and vetch.** O. K. KEDROW-SICHMAN (Prazy Gory-Garezka Nauk. Tavar., 1928, 5, 86—104; Chem. Zentr., 1930, i, 1200).—The combined fertilisers increase the yield more than does either alone.

A. A. ELDRIDGE.

**Nitrogen content and "quality" of barley.** L. R. BISHOP (J. Inst. Brew., 1930, 36, 352—364).—From the nitrogen content and 1000-corn weight of the barley, the extract which will be obtained with given malting conditions and varieties can be predicted. In addition, the calculation can be made of the amount of permanently soluble nitrogen which will be found in the resulting wort. The nitrogen content is affected chiefly by the soil and season, whilst the influence of variety and artificial manuring is less important. High yields and correspondingly low nitrogen contents are determined on clay soils by drought in March–April and cold weather in May–June, whilst rain and cool weather in July are beneficial. With light soils the rainfall effects are slightly earlier. The presence of organic nitrogenous matter produces excessive nitrification later in the season, which leads to a high nitrogen content and a low yield. Artificial nitrogenous manures applied in moderation in spring produce an approximately proportional increase of yield to the nitrogen absorbed, so that the nitrogen percentage is not markedly affected.

C. RANKEN.

**Phosphoric acid of barley grain.** E. M. CROWTHER (J. Inst. Brew., 1930, 36, 349—351).—The content of phosphoric acid in barley was reduced by the addition of a nitrogenous manure, but was not related to the yield, barley valuation, malt valuation, nitrogen content, 1000-corn weight, diastatic power, cold-water extract, and extract calculated on barley. The different varieties of barley showed a considerable variation in the content of phosphoric acid, which was higher in the winter than in the spring barleys.

C. RANKEN.

**Effectiveness of iodine in the control of smut on oats.** G. M. KARNS (Ind. Eng. Chem., 1930, 22, 864).—Seed infested with smut was treated with a concentrated solution of iodine in carbon disulphide, thorough contact being established in an end-over-end shaker. With



0.5 oz. of iodine per bushel of seed, infestation was reduced to 0.3% (similar to the standard ethyl mercuric chloride treatment), and with 1 oz. of iodine per bushel only 0.04% of infested seed remained. (Cf. Sayre, Ohio Agric. Exp. Sta. Bimonthly Bull., 1928, 13, 19.)

A. G. POLLARD.

**Influence of fallow methods of culture on some physico-chemical properties of plant juices from winter grain.** V. V. KVASNIKOV (Nauch. Agron. Zhur., 1929, 6, 368—379).—The changes in osmotic pressure and hydrogen-ion concentration of the juices from winter rye and wheat under conditions of various methods of fallow have been followed.

CHEMICAL ABSTRACTS.

**Influence of ammonium phosphate on yield and chemical composition of meadow hay.** H. O. ASKEW (New Zealand J. Sci. Tech., 1930, 12, 21—23).—Ammonium phosphate was applied to two soils, a light gravelly loam, well supplied with organic matter, which did not normally respond to phosphates, and to a loam poor in lime and phosphate. Significant increases of yield of hay resulted in both cases, but analysis of the hay indicated that the utilisation of the nitrogen was much higher than that of the phosphoric acid of the fertiliser.

E. HOLMES.

**Influence of phosphoric acid on the cropping power of seed potatoes.** DENSCH (Superphosphat, 1930, 6, 120—123).—Phosphate deficiency in soil not only reduces the crop yield of potatoes but lowers the seed value of the tubers, which in the subsequent season produce smaller crops of lower starch content. Moreover, the smaller crops in the second season are not improved by phosphate manuring. A. G. POLLARD.

**Action of ammonium sulphate and of sodium nitrate on the yield and starch content of potatoes.** O. ENGELS (Forts. Landw., 1930, 5, 97; Bied. Zentr., 1930 59, 358—359).—Ammonium sulphate produced higher crop increases and starch content of potatoes than did sodium nitrate. With each fertiliser there was an economic return.

A. G. POLLARD.

**Composition of a tobacco crop that burns poorly compared with one that burns well.** E. M. BAILEY and P. J. ANDERSON (Conn. Agric. Exp. Sta. Bull., 1930, No. 311, 228—233).—The latter (associated with seasons of high rainfall) contained more potassium and chlorine and less magnesium, nitrogen, phosphorus, and sulphur than the former. Of substances inert as to burning quality, the latter contained more silicon, aluminium, and iron than the former; the reverse holds for manganese.

CHEMICAL ABSTRACTS.

**Manurial effects of pyro- and meta-phosphates.** Y. KIDA (J. Sci. Agric. Soc. [Nogaku Kwai Ho], No. 245 and 246; Proc. Internat. Soc. Soil Sci., 1930, 5, 134—137).—Pyro- and meta-phosphates can be utilised by plants in the same way as orthophosphates. The soluble meta- and pyro-phosphates of the alkalis are the most efficient. Others examined, in order of efficiency, were calcium pyro- > magnesium meta- > magnesium pyro- > calcium meta-phosphates. Pyro- and meta-phosphates of iron and aluminium are inferior to the orthophosphates. The efficiency of these phos-

phates is closely related to their solubility in 2% citric acid solution. On heating to 210—220° monocalcium orthophosphate is converted into soluble monocalcium pyrophosphate, and at higher temperatures to the insoluble metaphosphate. Field trials with heated orthophosphate show a correspondingly decreased efficiency as the temperature of heating exceeds 220°. The fertilising efficiency of superphosphate diminishes when heated above 170—180°. This corresponds to a decrease in water solubility and an increase in ammonium citrate solubility of the phosphate at this temperature. Above 210° the proportion of insoluble phosphate increases steadily.

A. G. POLLARD.

**Ammonia fixation and nitrogen losses in manuring with liquid manure.** S. T. JENSEN (Wiss. Archiv f. Landw., 1930, 3, 161; Bied. Zentr., 1930, 59, 359—360).—To minimise nitrogen losses, liquid manure should be applied to soil when in an open and absorbent condition, in cool quiet weather. Addition of calcium chloride or nitrate to the liquor reduced nitrogen losses by 90%. Gypsum, if used, must be well stirred with the liquor to ensure complete decomposition. Analyses of, and field trials with, the treated liquor confirm the value of the conservation process. A. G. POLLARD.

**Influence of surface configuration, climate, and vegetation on the range of soil types in Chile.** A. MATTHEI (Soil Res., 1930, 2, 57—67).

**Critical considerations of fertiliser practice and crop yields in German agriculture.** ALLEMMEYER (Superphosphat, 1930, 6, 115—120).

**Superphosphate.** SHOJI and others.—See VII.

PATENTS.

**Production of chemical fertiliser.** A. HOLZ and T. VAN D. BERDELL (B.P. 332,750, 30.7.29).—Crude phosphates are treated with sulphuric acid to yield calcium sulphate and phosphoric acid; the calcium sulphate is converted by treatment with ammonium (or potassium) carbonate into the corresponding sulphate and calcium carbonate; and the phosphoric acid is neutralised with part of the calcium carbonate and some calcium hydroxide to yield dicalcium phosphate which, after removal from the mother-liquor, is mixed with concentrated ammonium (or potassium) sulphate solution, and the mixture is crushed and dried.

L. A. COLES.

**Chemical fertilisers.** SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE ET PROD. CHIM.) (B.P. 332,864, 22.11.29. Fr., 23.11.28).—Solutions containing sodium phosphate or the mixture obtained by the double decomposition of sodium phosphate and potassium chloride, with or without the addition of, e.g., sodium nitrate, are treated with ammonia and carbon dioxide to replace the sodium by ammonium with the simultaneous precipitation of sodium bicarbonate; after filtration, the solution is evaporated to yield a mixed fertiliser. The sodium bicarbonate, or the carbonate obtained by calcining it, is used to prepare sodium phosphate from monocalcium phosphate prepared from crude phosphates by the usual method. (Cf. B.P. 331,451; following).

L. A. COLES.



**Manufacture of compound fertilisers and of sodium bicarbonate.** SOC. CHIM. DE LA GRANDE PAROISSE AZOTE ET PROD. CHIM. (B.P. 331,451, 7.10.29. Fr., 20.11.28).—A mixture of potassium chloride and sodium nitrate in any desired proportion is treated with ammonia and carbon dioxide as in the Solvay process, the precipitated sodium hydrogen carbonate is removed, and the solution evaporated to obtain a mixed fertiliser containing potassium and ammonium nitrates and chlorides. A. R. POWELL.

**Manufacture of a concentrated fertiliser material.** W. W. TRIGGS. From ARMOUR FERTILIZER WORKS (B.P. 332,639, 25.4.29).—Phosphatic material which, to increase the proportion of available phosphates, has been calcined in the presence of alkali salts and, if desired, silica and carbonaceous material, is treated with ammonia, water, and carbon dioxide to yield ammonium phosphate, alkali phosphates, and calcium carbonate; after filtration of the solution, the phosphates are recovered together by evaporation for use as a fertiliser or ammonium phosphate may be separated, *e.g.*, by fractional crystallisation. The process may be accelerated by adding finely-divided clay, feldspar, etc. to the calcined phosphates, and the constituents may be so selected that the insoluble residues are suitable for the manufacture of cement. L. A. COLES.

**Simultaneous preparation of fertilisers containing magnesium phosphate and of nitrogenous fertilisers.** F. UHDE (B.P. 332,803, 5.9.29. Ger., 25.10.28).—Solutions of crude phosphates in nitric acid are treated with an alkali or ammonium sulphate, which may be added prior to dissolution, and with sufficient magnesium chloride solution (*e.g.*, residual potash lyes) to convert the phosphoric acid into di- or tri-magnesium phosphate, or, alternatively, the alkali or ammonium sulphate may be partly or completely replaced by magnesium sulphate; after removal of the calcium sulphate, the solution is treated with ammonium or an alkali hydroxide or carbonate to precipitate the magnesium phosphate and to yield a solution which, on evaporation, yields a nitrogenous fertiliser. The calcium sulphate is converted into ammonium sulphate for re-use in the process and into calcium carbonate which, if desired, can be mixed with the fertiliser. L. A. COLES.

**Manufacture of phospho-nitrogenous manures.** SOC. D'ETUDES SCIENTIFIQUES ET D'ENTREPRISES INDUSTRIELLES, Assees. of E. VOITURON (B.P. 313,612, 14.6.29. Ger., 15.6.28).—The acid product obtained by treatment of phosphate rock with nitric acid or with the nitrogen oxides formed by the catalytic oxidation of ammonia is mixed with an alkali or alkaline-earth silicate, *e.g.*, a metallurgical slag, sufficient to neutralise the free acidity. The product is non-hygroscopic and non-corrosive to the usual containers and contains silica in an active form which improves the assimilation of phosphorus and nitrogen by the plant. A. R. POWELL.

**Insecticides and fungicides.** J. STODDART (B.P. 332,638, 25.4.29).—A mixture of finely-divided kaolin with 10% or less of sulphur is heated to vaporise the sulphur and thus to deposit it as a film on the kaolin. Alternatively, a mixture of kaolin and a solution of

sulphur in, *e.g.*, carbon disulphide is heated to expel the solvent. L. A. COLES.

**Disinfecting seeds and the like.** (SIR) G. C. MARKS From E. I. DU PONT DE NEMOURS & Co (B.P. 330,548, 29.11.28).—Dusting powders for seed disinfection comprise an organic mercury compound, with or without sodium thiosulphate, potassium thiocyanate, etc., and 90–98% of diluent (preferably active carbon). Examples of mercury compounds are:  $\beta$ -ethylmercurithiol-naphthalene ( $C_{10}H_7 \cdot S \cdot HgEt$ ), *p*-ethylmercurithiol-benzenesulphonic acid, *o*-ethylmercurithiolbenzoic acid, ethyl mercurichloride, methyl mercuri-iodide, methyl mercurisulphate, ethyl mercuriacetate. Solid alkalis, *e.g.*, sodium carbonate, magnesia, lime, may be added. C. HOLLINS.

**Seed disinfectant.** M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,752,424, 1.4.30. Appl., 15.4.27).—Seeds may be disinfected by dusting with a dry mixture of disinfectants (*e.g.*, mercury compounds) and a wetting-out agent (*e.g.*, soap, saponin, casein). In moist soil the disinfectant becomes evenly distributed over the seed. E. B. HUGHES.

**Keeping cut flowers and the like fresh.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 332,278, 17.4.29).—Small quantities of a silver or copper salt together with a sugar or polyhydric alcohol, and, if desired, another plant nutrient, are dissolved in the water in which the flowers are kept. The total amount of sugar and other nutrients is 10–500 times that of the metallic salt. C. RANKEN.

**Manufacture of concentrated fertiliser.** H. H. MEYERS, Assr. to ARMOUR FERTILIZER WORKS (U.S.P. 1,760,990, 3.6.30. Appl., 13.3.28).—See B.P. 332,639; preceding.

## XVII.—SUGARS; STARCHES; GUMS.

**Steam consumption in [beet-sugar] diffusion.** J. HAMOUS (Z. Zuckerind. Czechoslov., 1930, 44, 409–410).—The heat theoretically required for the diffusion process can be calculated from the heat contents of the materials entering and leaving the battery. For fresh cossettes entering at 5°, and water entering and juice leaving at 25°, the heat required is about 1760 kg.-cal. per 100 kg. of beets, which is increased to 2640–2990 kg.-cal. by radiation and other losses. This heat could be obtained by injection of 4.2–4.7 kg. of steam at 6 atm. or 158°, or 4.3–4.9 kg. at 113° after suitable compression. By insulation the radiation losses can be reduced by one half, thus lowering the consumption of steam at 158° to 3.3–3.8 kg. per 100 kg. of beets. The temperature of the juice leaving the battery should be as low as possible, consistent with efficient extraction, for it can be heated in the juice heaters more cheaply than in the battery and without dilution by injected steam. J. H. LANE.

**Determination of the true sugar content of beets in factory control work.** V. MARES (Z. Zuckerind. Czechoslov., 1930, 54, 442–447).—Attention is called to the error that may arise in sampling beet by means of Stanek's segment rasp, whereby too high a sucrose content may be indicated. Errors arising from the dry-



ing of the pulped material previous to analysis, or from the absorption by the interior of the root of water from the flumes, are of much less importance.

J. P. OGILVIE.

**Determination of sugar in the beet.** J. VONDRÁK (Z. Zuckerind. Czechoslov., 1930, 54, 499–504).—Replying to Kopecky (B., 1930, 634) the author says that error due to the drying of the pulped sample can be reduced to 0.1% or less by preparing a large sample of pulp (at least 1 kg.), and at once weighing it out for analysis.

J. P. OGILVIE.

**Adsorption by insoluble calcium salts in the carbonatation of beet juice.** J. DEDEK and F. DOLAK (Z. Zuckerind. Czechoslov., 1930, 54, 507–513).—Experiments on the elimination of colouring matters from solutions in which different precipitates were produced showed little effect in the case of the formation of calcium oxalate. Calcium carbonate was more effective, but the best result was obtained with calcium phosphate, which adsorbed 7–10 times as much as either of the other two precipitates.

J. P. OGILVIE.

**Influence of carbonatation on the rate of filtration.** V. STANĚK and K. ŠANDERA (Z. Zuckerind. Czechoslov., 1930, 54, 373–382).—The addition of about 1% of lime in the carbonatation of normal beet juice increases the rate of filtration of the scums; with 2.5–3.0% a notable improvement is effected. If juice from which the organic particles have been eliminated be carbonatated after the addition of 0.25% of lime, a cake is formed which in its filtering qualities is similar to that otherwise obtained with 1–3% of lime. Centrifugal pumps have a distinctly unfavourable effect on the rate of filtration of the carbonatated juice transported by them.

J. P. OGILVIE.

**Crystallisation of grape concentrates and syrups.** D. S. GLENN and W. V. CRUESS (Fruit Prod. J., 1930, 9, 248–249).—Grape syrups did not crystallise in the absence of crystals or other solid matter; addition of dextrose caused the crystallisation of dextrose and tartrates. In white syrup crystallisation appears to occur above 70° Balling; in red syrups the crystallisation point is higher.

CHEMICAL ABSTRACTS.

**Charring of sugar in centrifuging.** W. C. BEDDING (Arch. Suikerind. Nederl.-Indië, 1930, 38, 256).—Sugar which was grey was found under the microscope to be permeated with particles of carbon, due to the fact that it had been washed with superheated steam which had been produced from boiler-water containing sugar.

J. P. OGILVIE.

**Variations in the quality of raw sugars.** E. W. RICE (Facts about Sugar, 1930, 25, 290–291).—Raw cane sugars differing greatly in colour showed widely differing characteristics when examined by a number of methods. It is considered that the variations in the nature of the organic impurities cannot be due to fundamental characteristics of the original juices.

J. P. OGILVIE.

**Analyses of Trinidad molasses.** J. A. MACDONALD (Mem. Imperial Coll., Trinidad; Sugar Tech. Ser., No. 2).—Analyses of 20 samples of Trinidad final molasses led to the following observations. The electrometric ash calculated from the conductivity of the 5% solution

agreed within 4% of the value obtained by incineration. The ash figure is of value in judging the exhaustion of a molasses, since certain samples, though having high purities, were nevertheless exhausted, the high purity values being correlated with high ash contents and low reducing sugar contents. The apparent purity value may be quite misleading. The viscosity increases as the crop proceeds. The refractometric solids are closer to the true solids than the degree Brix.

J. P. OGILVIE.

**Determination of the fine grain of molasses and syrups.** K. ŠANDERA and C. SAMAL (Z. Zuckerind. Czechoslov., 1930, 54, 389–394).—Using a method based on the polarisation of a thin layer of the product, the fine grain content was determined in molasses of the 1925–26 and 1929–30 campaigns, and found in every case to be less than 2%. Even heating for  $\frac{1}{2}$  hr. at 110° does not always suffice for dissolving the fine grain of a molasses.

J. P. OGILVIE.

**Application of polarography to sugar-factory analysis.** B. ZIMMERMANN (Z. Zuckerind. Czechoslov., 1930, 54, 394–397).—Heyrovsky's polarographic method (B., 1922, 986) is especially applicable for determining the conditions under which sucrose may be decomposed in the factory or refinery, *e.g.*, in evaporation and boiling. It also gives indications capable of differentiating between the several grades of white sugars.

J. P. OGILVIE.

**Unified conductometric method for determination of ash in refinery syrups.** F. W. ZERBAN and L. SATTLER (Ind. Eng. Chem. [Anal.], 1930, 2, 322–325).—The relation between electrical conductivity and chemical ash of crude sugar solutions is discussed. It is shown that though there is a fairly universal ratio for beet sugars the ratio is very variable for cane sugars. Attempts are made to find a universal formula applicable to any cane product.

E. S. HEDGES.

**Analysis of refined sugars.** B. ZIMMERMANN (Z. Zuckerind. Czechoslov., 1930, 44, 405–409).—Five types of Czechoslovakian refined sugars, viz., cubes, centrifugal pilé, Raffinade gries (coarse castor) Raffinademehl (fine castor), and inland crystals (large grain), representing actual market standards, were analysed by the most recent chemical and physical methods. With a few insignificant exceptions, the arrangement of the five sugars in order of purity was as given above, whether judged by polarisation (99.84–99.75), invert sugar content (0.002–0.041%), ash content (0.009–0.028%), luminescence in solution, tendency to caramelisation on heating, absorption of light, or oxygen-maximum of polarographic curves (cf. following abstract). The moisture contents ranged from 0.029 to 0.046%. Results of screening tests on the last three types are recorded.

J. H. LANE.

**Influence of sugars and non-sugars on the oxygen-maximum of polarographic curves.** B. ZIMMERMANN (Z. Zuckerind. Czechoslov., 1930, 54, 425–432).—The polarographic method of Heyrovsky (A., 1924, 598; 1925, 674) based on electrolysis with the dropping mercury electrode will detect not only incipient caramelisation of sugar (cf. B., 1929, 695), but also the presence of extremely minute traces of molasses. Even at a concentration of 0.00002% in 5% sucrose solutions



the depressing effect of molasses on the oxygen-maximum of the polarographic curves is detectable. The constituents of molasses mainly responsible for this effect are the colouring matters and colloids, which have a measurable action at 0.000002% concentration. Much less active are potassium hexoate, vernine, and adenine, whilst invert sugar, betaine, glutinates, leucine, and potassium formate are without action. Blueing agents in the amounts used with white sugars are also inactive. The polarographic method provides a very delicate means of comparing the qualities of refined sugars.

J. H. LANE.

**Photographic estimation of foreign materials in gums and resins.** E. A. GEORGI (Ind. Eng. Chem. [Anal.], 1930, 2, 331—334).—A photomicrographic method for determining dirt in gums and resins is outlined and the results are shown to be as accurate for practical purposes as those obtained by the usual gravimetric procedure, whilst the cost is less. The method is applicable to glue, gelatin, etc.

E. S. HEDGES.

**Sources of error in quinhydrone  $p_H$  determination of sugar-house products.** B. G. SAVINOV and E. P. KOSHEVEROVA (Nauch. Zapiski Sakh. Prom., 1930, 8, 525—543).—The quinhydrone should be recrystallised twice from alcohol, and 0.01 g. per 15 c.c. of solution is the minimal quantity which should be employed.

CHEMICAL ABSTRACTS.

## PATENTS.

**Dissolution of organic colloids [starch, glue, and gums].** ORANIENBURGER CHEM. FABR. A.-G. (B.P. 307,079, 2.3.29. Ger., 2.3.28).—Carbohydrates and proteins are dissolved in an organic solvent insoluble in water with the addition of a soap dissolved in an organic solvent wholly or partly soluble in water, e.g., an alcohol, ketone, phenol, chlorohydrin, an ester, or dioxan. E.g., a potash-olein soap is dissolved in tetrahydronaphthalene, the solution is clarified with ammonia solution, and powdered glue is stirred into this solvent; the clear viscous product does not solidify.

A. R. POWELL.

**Recovery of nitrogen and acetone from vinasses.** J. GUILLISSEN, ASST. to UNION CHIM. BELGE (U.S.P. 1,772,078, 5.8.30. Appl., 27.8.27. Belg., 21.9.26).—See B.P. 277,932; B., 1928, 345.

## XVIII.—FERMENTATION INDUSTRIES.

**Composition and determination of barley proteins. III.** L. R. BISHOP (J. Inst. Brew., 1930, 36, 336—349; cf. B., 1929, 696).—For each separate variety of barley the weights of the individual proteins are simple regular functions of the total nitrogen content per 1000 corns. The percentage of salt-soluble nitrogen on total nitrogen decreases with increase of the total nitrogen. The percentage of hordein nitrogen shows a corresponding increase so that the glutelin percentage remains constant throughout. The curves for the different varieties are similar in form, but they differ in actual magnitude. Accordingly, the relationship between the quantities of the individual proteins and the total nitrogen is characteristic of the variety. If they are allowed time to reach the natural equilibrium, the same proportions of the proteins are found in samples of developing grain as in mature grain of the same total

nitrogen content per 1000 corns. Development of the proteins is essentially a synthesis of the simple compounds which enter the grain, and the synthesis progresses to a definite equilibrium point which is controlled only by the total nitrogen and the variety.

C. RANKEN.

**Barley proteins. Influence of method of grinding on their determination.** G. HOFMAN-BANG (J. Inst. Brew., 1930, 36, 381—388).—When the barley is ground in a ball-mill with the production of 95% of flour, as measured by a Pfungstädter sieve, the percentage amounts of salt-soluble protein, hordein, and glutelin reach constant values which are unaffected by the heating effect in the ball-mill.

C. RANKEN.

**Numerical expression of the analytical results as basis for the calculation of the brewing value [of barley and malt].** F. KUTTER (Woch. Brau., 1930, 47, 237—241, 245—249, 261—265, 273—279, 283—285, 297—300).—Suggestions are made for assigning such definite numerical values to the results of the analytical examination of barley and malt as may be combined into an equation to give a figure for the brewing value which will vary continuously and not by stages. The optimum amount of protein is considered to be 10%, and the brewing value to fall off with increasing steepness on either side of this. It is claimed that high screening values in malts are associated with high fermentability of the resulting worts. The original must be consulted for details.

F. E. DAY.

**Separation and determination of the bitter resins of hops.** A. S. BAZILEVICH (Nauch. Agron. Zhur., 1929, 6, 415—428).—Resins precipitable with lead acetate are termed "humulids," and others "lupulids." The former are grouped in four, and the latter in three, classes. The hops (10 g.) are shaken for 1.5 hrs. with ether (200 c.c.), and the residue from 100 c.c. of filtrate is dried for 2—3 hrs. at 80° and weighed. The residue after repeated extraction with light petroleum consists of hard resins. For the determination of waxes methyl alcohol is employed as solvent. The amounts of various substances present are influenced by the soil and the method of fertilisation, and they vary in different parts of the plant.

CHEMICAL ABSTRACTS.

**Copper as applied to brewing.** D. M. STEWARDSON (J. Inst. Brew., 1930, 36, 369—381).—A description is given of the application of copper to brewing plant, including mash tuns, backs, fermentation vessels, and racking machines, and it is suggested that copper and gun-metal can be used with advantage to replace iron or galvanised iron for the construction of pulp-washing machines.

C. RANKEN.

**Dry, unpressed wine and natural wine.** J. WERDER and C. ZÄCH (Wein u. Rebe, 1929, 11, 272—276; Chem. Zentr., 1930, i, 1394).—Dry, unpressed wine, but not natural wine, after decolorisation by animal charcoal, shows a blue luminescence in filtered ultra-violet light.

L. S. THEOBALD.

**Detection of fruit wine in grape wine.** C. VON DER HEIDE (Wein u. Rebe, 1929, 11, 251—271; Chem. Zentr., 1930, i, 1394).—Methods are discussed; Brauer's (phosphomolybdic acid) and Seyda's (sodium gold



chloride) methods differentiate, whilst with the sorbitol method 5% of fruit wine in grape wine can be detected.

A. A. ELDRIDGE.

**Detection of fruit wine in grape wine by Werder's sorbitol method.** G. FIESSELMANN (Wein u. Rebe, 1929, 11, 317—326; Chem. Zentr., 1930, i, 1394).

**Barley.** BISHOP; CROWTHER.—See XVI.

#### PATENTS.

**Manufacture of yeast.** G. S. BRATTON, Assr. to ANHEUSER-BUSCH, INC. (U.S.P. 1,732,922, 22.10.29. Appl., 15.4.27).—Yeast is propagated in a solution of sacchariferous materials to which is added a ureide of a reducing sugar.

C. RANKEN.

**Cultivation of micro-organisms.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,235, 11.3.29).—Peat is completely hydrolised by acids, and prior to the inoculation of the resulting solution the humous substances are removed by treating it with non-alkaline adsorbent substances of large superficial area, the  $pH$  being kept within the range 5—8. (Cf. B.P. 318,649; B., 1929, 994.)

C. RANKEN.

#### XIX.—FOODS.

**Measuring the relative plasticity of pastes and doughs.** C. H. BAILEY (J. Rheology, 1930, 1, 429—432). The work-input required to move the blades of a small mixing machine through the material is measured by inserting a watt-hour meter in the motor circuit. The values recorded are particularly useful in the bread-making industry for determining the effect of unit additions of water on the consistency of the dough.

E. S. HEDGES.

**Composition and food value of carob-bean meal and "patay."** C. T. RIETTI (Rev. farm. Buenos Aires, 1929, 71, 633—635; 1930, 72, 3—16).—The meal of *Prosopis alba*, Griesbach, and cake contain, respectively, water 6.67, 9.67; ash 2.70, 6.71; reducing carbohydrates 3.20, 5.70; carbohydrates hydrolysable and soluble in the cold 41.20, 43.98; starch 8.01, 10.40; fibre 10.0, 5.93; pentosans 4.41, 3.49; protein 6.56, 4.37%.

CHEMICAL ABSTRACTS.

**Composition of cottonseed hull bran.** K. S. MARKLEY (J. Amer. Soc. Agron., 1928, 20, 1102—1107).—Comprehensive analytical values are recorded.

CHEMICAL ABSTRACTS.

**Determination of age of eggs by the aid of hydro-ion concentration measurements.** C. SCHWEIZER (Mitt. Lebensm. Hyg., 1929, 20, 312—313; Chem. Zentr., 1930, i, 1240).—The method is untrustworthy.

A. A. ELDRIDGE.

**Determination of crude fibre in cocoa and chocolate.** J. RUFFY (Mitt. Lebensm. Hyg., 1929, 20, 355; Chem. Zentr., 1930, i, 1239).—Matthes and Müller's modification of König's method is preferred to Fellenberg's method but the use of smaller quantities is recommended.

A. A. ELDRIDGE.

**Vitamin content of ethylene-treated and untreated tomatoes.** D. D. JONES and E. M. NELSON (Amer. J. Publ. Health, 1930, 20, 387—394).—Naturally ripened tomatoes are a better source of vitamins-A, -B, and -C than is fruit picked while green and treated with ethylene to develop the red colour, but treatment with

ethylene did not appear to affect vitamins already formed.

CHEMICAL ABSTRACTS.

#### PATENTS.

**Processing cereal grains.** J. W. BECKMAN (U.S.P. 1,753,643, 8.4.30. Appl., 20.3.29).—Cereal grains are treated with alkali and bleaching agent to remove the fat from the outer layer and bleach and sterilise the grain, which is afterwards dried at below 60°.

E. B. HUGHES.

**Preservation of milk and cream.** T. HOFIUS and A. REPPMAN (B.P. 332,641, 26.4.29).—The milk etc. is de-aerated by displacing the dissolved air by carbon dioxide, the latter gas is then displaced by oxygen, and the product kept in that condition until required. Subsequently, the oxygen is displaced by nitrogen immediately before use. Suitable apparatus is described.

E. B. HUGHES.

**Production of stable, homogenised, [milk- or cream-like] emulsions.** C. W. A. KLEINE (B.P. 309,836, 15.4.29. Swed., 13.4.28).—The disperse-phase constituent is introduced into an emulsification machine which is already charged with the constituent which is to constitute the continuous phase. The gradual introduction of the two constituents is continued in the same order and the resulting basic emulsion is well agitated and then immediately homogenised.

C. RANKEN.

**Compositions comprising carbohydrates and acid-reacting substances for incorporation with milk.** G. B. KILGOUR. From F. H. FAULDING & Co., LTD. (B.P. 332,595 and 332,597, 24.4.29).—By intimately mixing small quantities of edible organic acids with relatively large quantities of dextrose and then adding the mixture (in tablet etc. form, if desired) to milk, a digestible humanised milk may be produced.

E. B. HUGHES.

**Dry milk products.** W. R. B. S. GATES, J. TAVROGES, and Cow & Gate, LTD. (B.P. 332,047, 3.7.29).—A laxative milk product is prepared by drying milk in which phenolphthalein has been dispersed or added in alkaline solution.

E. B. HUGHES.

**Preventing rancidity in dried or powdered milk and milk foods or preparations.** W. R. B. S. GATES, J. TAVROGES, and Cow & Gate, LTD. (B.P. 332,669, 14.5.29).—By the addition of a polyhydric alcohol, e.g., glycerin or various glycols, to milk before drying, a milk powder is produced in which the production of rancidity with time is largely inhibited.

E. B. HUGHES.

**Preservation of artificial and natural butter.** J. LONGMAN. From A.-G. F. MEDIZ. PRODUKTE (B.P. 332,806, 9.9.29).—To the butter etc. an unorganised albumin-digesting ferment, preferably in acid solution, is added. E.g., a margarine containing pepsin 0.05%, glycerin 0.06%, and 25% hydrochloric acid (0.1%) is claimed.

E. B. HUGHES.

**Lard product.** J. R. SHIPNER, Assr. to CUDAHY PACKING Co. (U.S.P. 1,755,135, 15.4.30. Appl., 1.6.29).—Lard is refined to remove fatty acids, hydrogenated to an iodine value of 55—60, deodorised, and cooled to a lard-like consistency. It is claimed that the product is neutral in flavour, and is superior to ordinary lard in cooking qualities.

E. B. HUGHES.



**Apparatus for extracting fat and other substances from animal carcasses, slaughterhouse refuse, fish, fish waste, etc.** A. SOMMERMEYER (B.P. 332,637, 24.4.29).—In the continuously operating apparatus described, the separation vessel is arranged by the side of the digestion vessel, a feed pipe connecting the two. A steam and vapour separator is provided in this pipe, and the products condensed are conducted to the separation vessel. E. B. HUGHES.

**Removing lead arsenate from fruit.** A. R. MAAS (U.S.P. 1,754,173, 8.4.30. Appl., 21.12.26).—The fruit is washed or sprayed with a solution of an alkali thiosulphate, with soap or alkali as a wetting agent. E. B. HUGHES.

**Preservation of food products.** E. MILANI (B.P. 332,209, 16.2.29).—Fresh fruit or vegetables are hermetically sealed in the presence of an inert gas in a container which is minutely vented before, or very soon after, the product generates its own gas. C. RANKEN.

**Manufacture of a [pectin-sugar] jelly preparation.** A. LEO (B.P. 331,295, 15.4.29).—Sugar crystals maintained in suspension by an upward current of warm air are sprayed with a 3–5% pectin solution, whereby they become coated with a thin film of pectin. Powdered citric or tartaric acid is coated with a thin film of paraffin wax or palmitic or stearic acid by moistening it with an ethereal solution of the wax or fatty acid. The sugar-pectin powder is then mixed in desired proportion with the acid powder. To prepare a jelly the powder is added to cold fruit juice, whereby the pectin dissolves followed by the sugar; the solution is then heated to melt the film on the acid particles and to cause them to dissolve. On cooling, the mass sets to a jelly. A. R. POWELL.

**Heat-exchanger** (U.S.P. 1,754,857). **Treating materials** (B.P. 332,577 and 332,593).—See I.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Acid in ether.** HALL.—See III. **Tobacco crops.** BAILEY and ANDERSON.—See XVI.

### PATENTS.

**Manufacture of substituted ureas and thioureas.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,583, 10.1.29).—Substances of the "Bayer 205" type (cf. B.P. 9472 of 1914; B., 1914, 825), but containing one or more heterocyclic residues, have similar destructive action on blood parasites. The condensation product from 1 : 4 : 6 : 8-naphthylaminetrisulphonic acid and 4-chloro-6-nitroquinazoline is reduced, the amine so obtained is treated with 2-nitro-*p*-toluyl chloride, and the product is reduced and phosgenated; or the amine may be condensed with a second mol. of 4-chloro-6-nitroquinazoline, reduced, and phosgenated; or the amine may be phosgenated directly. Similarly 2 : 4 : 6 : 8-naphthylaminetrisulphonic acid is *m*-nitrobenzoylated, the reduced product is condensed with 10(*ms*)-chloro-1-nitroacridine, reduced, and phosgenated. C. HOLLINS.

**Manufacture of salts of higher homologues of polyhydroxybenzenes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,519, 6.3.29).—The causticity or unpleasant taste of octyl- and higher alkyl-

resorcinols etc. is removed by their conversion into salts with alkalis or organic bases. The hexamethylene-tetramine salts of 4-octylresorcinol, decomp. 118°, 4-decylresorcinol, decomp. 120°, 3-decylpyrocatechol, and decylpyrogallol, decomp. 140°, and the piperazine, anhydrosarcosine, and betaine salts of decylresorcinol (the last two having m.p. 76° and 80°, respectively), are described. Sodium decylresorcinol is salted out by calcium chloride solution. C. HOLLINS.

**Manufacture of 3 : 3'-dichloro-5 : 5'-diacetamido-4 : 4'-dihydroxyarsenobenzene.** I. G. FARBENIND. A.-G. (B.P. 330,862, 25.10.29. Ger., 25.10.28. Addn. to B.P. 296,327; B., 1928, 837).—3 : 3'-Dichloro-5 : 5'-diamino-4 : 4'-dihydroxyarsenobenzene is acetylated by addition of acetic acid to an aqueous solution of the sodium salt at 15–35°. C. HOLLINS.

**Antiseptic composition.** V. LEONARD, Assr. to SHARPE & DOHME, INC. (U.S.P. 1,771,895, 29.7.30. Appl., 19.1.28).—See B.P. 299,522; B., 1928, 944.

**Manufacture of aromatic arsenic compounds containing an isoxazine ring.** G. NEWBERRY, Assr. to MAY & BAKER, LTD. (U.S.P. 1,771,307, 22.7.30. Appl., 27.6.27. U.K., 8.7.26).—See B.P. 280,613; B., 1928, 211.

**Chlorine** (U.S.P. 1,729,043).—See VII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Optical and photographic properties of sensitising and desensitising dyes of the cyanine and related types.** O. BLOCH and F. M. HAMER (Phot. J., 1930, 70, 374–390).—The absorption and sensitising curves of dyes of the  $\psi$ - and *iso*-cyanine type derived from a number of heterocyclic ammonium iodides, and of corresponding *p*-dimethylaminostyryl and *p*-dimethylaminocinnamylidene derivatives, and *p*-dimethylaminoanils are determined and compared (cf. A., 1930, 930). In general, the absorption bands of the styryl compounds are nearer the blue end of the spectrum than are those of the corresponding anils; in both cases they are usually single and broad in contrast to the narrow double bands of the cyanines. All the cyanines are photographic sensitisers; the styryl compounds with one exception are much weaker, and the two cinnamylidene derivatives have but little photographic activity. The anils are powerful desensitisers, comparable with, and in most cases superior to, safranin, but are not recommended for technical application as they give decomposition products which attack the latent image (cf. Hamer, B., 1929, 1032). The following are prepared by interaction of the appropriate quaternary salt with *p*-dimethylaminobenzaldehyde, *p*-dimethylaminocinnamaldehyde, or *p*-nitrosodimethylaniline in alcoholic solution in the presence of piperidine: 4-*p*-dimethylaminostyrylquinoline ethiodide, m.p. about 124°; tetramethyl-*pp'*-diamino-2 : 4-distyrylquinoline ethiodide, m.p. 243° (decomp.); 4-methyl-2-*p*-dimethylaminostyrylthiazole ethiodide, m.p. 249° (decomp.); 1-*p*-dimethylaminostyrylbenzthiazole ethiodide, m.p. 255–257° (decomp.); 2-*p*-dimethylaminostyryl-3 : 3-dimethylindolenine ethiodide, m.p. 222° (decomp.); 1-*p*-dimethylaminostyrylbenzoxazole ethiodide, red and blue dichroic crystals, m.p. 248–249° (decomp.); *p*-dimethylaminocinnamylidene- $\alpha$ -picoline



*ethiodide*, m.p. 209°; 1-*p*-dimethylaminocinnamylidene-methylbenzthiazole *ethiodide*, m.p. 220°; the *p*-dimethylaminoanils of  $\beta$ -naphthaquinaldinealdehyde *ethiodide*, m.p. 222° (decomp.), *quinoline-4-aldehyde ethiodide*, m.p. 174°, 4-methylthiazole-2-aldehyde *ethiodide*, m.p. 227° (decomp.), and of benzthiazole-1-aldehyde *ethiodide*, m.p. 240°; and the di-*p*-dimethylaminoanil of *quinoline-2:4-dialdehyde ethiodide*, m.p. 217° (decomp.). H. A. PIGGOTT.

**"Halogen value" of silver iodide emulsions, its determination and photographic interpretation.** H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1930, 2, 111—117).—Silver iodide emulsions were prepared by different methods in excess of potassium iodide, thoroughly washed, and then broken down with dilute nitric acid. In all cases, contrary to results obtained with the other silver halides, iodide ions, but no silver ions, were detected in the filtrate, the precipitate being free from silver ions. Emulsions prepared in presence of excess of silver ions gave, after breakdown with nitric acid, a filtrate containing appreciable amounts of silver ions. The amount of iodide in the filtrate depends on the type of emulsion and particularly on the grain size, and appears to be inversely proportional to the latter. Some of the known properties of silver iodide emulsions, *e.g.*, sensitisation with dyes, are discussed in the light of these results.

J. W. GLASSETT.

**Nomenclature of "silver and halogen values" in photographic layers.** H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1930, 28, 118—120).—A system of nomenclature, based mainly on the authors' work, is suggested for the characterisation of photographic layers.

J. W. GLASSETT.

**Solarisation. I. Effect of bromide ions on solarisation. II. Dependence of solarisation on method of preparation of emulsion.** H. ARENS (Z. wiss. Phot., 1930, 28, 91—98, 98—110).—I. Experiments on a series of commercial plates and films showed that bromide ions were effective in increasing solarisation only when present during exposure. When the bromide ions were removed by washing in silver nitrate, followed by sodium chloride to destroy excess of silver ions, no solarisation was exhibited. Similarly treated layers of peptised silver bromide showed a very much reduced solarisation. The effect, however, reappeared when the treated plates were bathed in potassium bromide solution. The inhibition of solarisation exhibited by nitrate ions is destroyed in presence of bromide ions.

II. The solarisation properties of various silver bromide layers prepared from silver bromide precipitated in presence of excess of silver, equivalent proportions, and excess of bromide, and afterwards peptised in gelatin solutions with varying additions of potassium bromide, have been investigated. The silver body solarised less than the bromide body even if equal quantities of potassium bromide were present in the finished emulsion. Contrary to previous experiments, increased quantities of potassium bromide at peptisation decreased the solarisation, the minimum of the curve of the solarisation range being raised. Furthermore, an increase in bromide content caused a displacement of the solarisation range towards the shorter exposures,

whilst the sensitivity of the foot of the curve was decreased. The difference in solarisation of the silver and bromide bodies cannot be correlated with differences in grain-size distribution. The silver, equivalent, and bromide bodies peptised in presence of ammonia solarised strongly, the effect being increased with increase in ammonia content.

J. W. GLASSETT.

#### PATENTS.

**Developing kinematograph films sensitised with dichromated gelatin.** M. G. M. G. BIZOT (B.P. 332,195, 10.4.29. Fr., 11.10.28).—The impressed films are dipped for a short time first in cold water, and then in cold water containing sodium hypochlorite. They are then washed in hot water so as to remove all the unimpressed gelatin, the impressed gelatin being dyed with a dye such as methylene-blue, or so as to remove part of the unimpressed gelatin, in which case the impressed and the remainder of the unimpressed gelatin are dyed, either to a different extent or by means of different dyes.

W. J. WRIGHT.

**Production of diazo-type prints.** KALLE & Co. A.-G. (B.P. 316,563 and Addn. B.P. 318,108, [A] 30.7.29. Ger., 30.7.28; [B] 17.8.29. Ger., 27.8.28).—The resistance of diazo-types to water is improved, where the azo dye contains sulphonic or carboxylic groups, by adding to the layer at any convenient stage (A) an arylated diguanidine or (B) an arylated guanidine (diphenylguanidine). Examples are: diazotised 5-dimethylaminoanthranilic acid, phloroglucinol, phenyldiguanidine, tartaric and boric acids (black); diazotised *p*-aminodiethylaniline-*m*-sulphonic acid, phloroglucinol, resorcinol, 3-chloro-*o*-tolyl diguanidine or diphenyl- or di-*o*-tolyl-guanidine, tartaric and boric acids (black).

C. HOLLINS.

**Films [with filter mask] for use in colour photographic processes.** KODAK, LTD., Assees. of P. FAVOUR (B.P. 318,238, 15.7.29. U.S., 31.8.28).

**Paper** (B.P. 332,743).—See V. **Photochemical dyeing etc.** (B.P. 309,166).—See VI. **Imitating wood-grain** (B.P. 308,371 and 308,373—6).—See IX. **Printing plates** (B.P. 331,220).—See X.

#### XXII.—EXPLOSIVES; MATCHES.

**Nitrostarch [explosives].** A. SCHRIMPF (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 273—278).—Two kinds of nitrostarch explosives are used in America. For one kind, nitrostarch is mixed with sodium nitrate and an oil, a typical composition consisting of nitrostarch 50%, sodium nitrate 47.5%, impregnating oil 1.5%, and sodium bicarbonate 1.0%. This explosive has a high brisance and is readily detonated; it is suitable for chamber blasts and quarrying limestone or granite. The other type contains a large amount of ammonium nitrate mixed with the nitrostarch, together with oxidisable materials, such as coal dust, trinitrotoluene, and aluminium, and a small amount of oil. A representative explosive consists of ammonium nitrate 73.0%, nitrostarch 15.0%, trinitrotoluene 3.0%, coal dust 2.0%, aluminium 6%, mineral oil 0.5%, and zinc oxide 0.5%. Explosives of this type have a lower brisance and are more difficult to detonate; they also give rise to larger amounts of gaseous products. Some



of these compositions are used in fiery mines. Investigations on the sensitising of ammonium nitrate by nitro-starch, made by the Pittsburgh experimental station, are described. W. J. WRIGHT.

**Universal explosive.** A. STETTACHER (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 278—279).—"Penthrinit," consisting of mixtures of pentaerythritol tetranitrate (penthrinit) and nitroglycerin or dinitroglycerol in the proportions of 10—70% and 90—30%, respectively, is superior as regards efficiency and detonation velocity to blasting gelatin. In order to obtain uniform distribution of the penthrinit, about 1% of collodion cotton is added. Both normal and gelatinised penthrinit can be moderated by means of camphor or centralite. Mixtures of 80:20 or 50:50 penthrinit with collodion cotton and camphor or centralite are less sensitive to shock, as tested by the falling weight, than is penthrinit alone, and the latter is less sensitive than penthrinit. Penthrinit suffers no loss of efficiency or reduction in detonation velocity on storage. "Ammonium penthrinit," consisting of penthrinit 40%, nitroglycerin 10%, vaseline 1.7%, and ammonium nitrate 48.3%, equally retains its original strength. W. J. WRIGHT.

**Determination of centralite in double-base, smokeless powders.** H. LEVENSON (Ind. Eng. Chem. [Anal.], 1930, 2, 246—247).—The centralite (diethyl-diphenylurea) in smokeless powders made from nitro-cellulose and nitroglycerin may be determined directly by the following method. 5 G. of the powder are extracted with ether for 24 hrs., and the extract is evaporated. The liquid residue is transferred to a 250-c.c. flask with 50 c.c. of alcohol, 25 c.c. of standard bromide-bromate solution are added, the mixture is cooled to 20°, and 5 c.c. of hydrochloric acid are added. The flask is stoppered and the contents are shaken, and exactly 30 sec. from the time of adding the acid 10 c.c. of 15% potassium iodide are added. The liberated iodine is titrated with sodium thiosulphate. The bromide-bromate solution should contain 5.568 g. of potassium bromate and 30 g. of potassium bromide in 1 litre and should be standardised in the presence of alcohol and 30 sec. after adding the acid as described above. E. S. HEDGES.

**Action of volatile solvents on cellulose nitrate powders.** J. P. LEHALLEUR (Ann. Acad. Brasil. Sci., 1929, 1, 149—150).—Disintegration, due chiefly to surface denitration, is greater with powders prepared with volatile solvents. CHEMICAL ABSTRACTS.

#### PATENTS.

**Manufacture of priming compositions.** H. RATHSBURG and E. VON HERZ (B.P. 310,509, 18.3.29).—Priming compositions containing lead styphnate are sensitised by addition of guanynitrosoaminoguanyl-tetrazene, the amount of this latter compound being 0.5—15% of the total composition. W. J. WRIGHT.

**Method of loading explosives.** F. OLSEN and C. J. BAIN (U.S.P. 1,752,391, 1.4.30. Appl., 14.6.29).—In filling projectiles or moulds with mixtures of ammonium nitrate and an explosive such as trinitrotoluene, a predetermined amount of the latter, after melting, is first poured in, and pellets of ammonium nitrate larger than 10-mesh are added until the liquid just covers them. The

unheated pellets absorb heat from the molten explosive, and thereby accelerate the setting of the mixture.

W. J. WRIGHT.

**Manufacture of smokeless powder.** C. R. BORLAND, Assr. to AMER. POWDER CO. (U.S.P. 1,752,881, 1.4.30. Appl., 1.3.24).—In the process of hardening grains of nitrocotton for smokeless powders by means of a liquid solvent, the latter is introduced as a spray of such fineness that the particles of the solvent have a smaller volume than the grains of the nitrocotton. A special type of atomiser is used. The granular mass of nitrocotton is caused to move in such a manner as to uncover all the grains successively and to remove those already treated on the surface. W. J. WRIGHT.

**Manufacture of a tetrazene explosive.** H. RATHSBURG (B.P. 308,179, 18.3.29. Ger., 16.3.28).—The preparation of tetrazene by treating an aqueous solution of an aminoguanidine salt, such as the nitrate, with sodium nitrite solution is accelerated, and the yield is increased if the reacting solutions are heated to 25—30° before being mixed. During mixing, the temperature is allowed to reach 40—50° by suitably insulating the reaction vessel, and the reaction is controlled by cooling the vessel. W. J. WRIGHT.

**Explosive and solvent therefor.** T. L. DAVIS (U.S.P. 1,754,417, 15.4.30. Appl., 8.11.27).—Aliphatic nitroamines are solvents for nitroguanidine, having the property of converting it into a non-crystalline mass. Being soluble in water and organic solvents, they may be used in the preparation of nitrocellulose-nitroguanidine explosives, and as they contain a nitro-group they increase the power of such explosives. W. J. WRIGHT.

**Manufacture of [strip] matches.** J. G. NEWMAN (B.P. 332,700, 17.6.29).

#### XXIII.—SANITATION; WATER PURIFICATION.

**Industrial analysis and recording of carbon dioxide and oxygen in air.** W. F. HAMILTON (Ind. Eng. Chem. [Anal.], 1930, 2, 233—237).—The quantitative analysis of gaseous mixtures by measurements of thermal conductivity of the gases is discussed, and the application of this method has led to the construction of an apparatus for the continuous recording of the percentages of carbon dioxide and oxygen in air. Quartz-protected platinum spirals are used both for heating elements and for the measurement of temperature. An improved thermal conductivity gas-analysis unit is described, and the effects of varying gas composition, temperature, current, and flow rate are indicated. The time lag is shown to be constant and independent of the magnitude of change of composition of the gas, providing a constant rate of flow is maintained. The successful operation of the apparatus depends on the maintenance of a Wheatstone bridge circuit which is only slightly unbalanced and the adjustment of the cells to a point where the rates of heat dissipation from the reference and analysis spirals are nearly equal. E. S. HEDGES.

#### PATENTS.

**Purification of water.** O. and R. ADLER (U.S.P. 1,771,518, 29.7.30. Appl., 9.7.26. Czech., 15.7.25).—See B.P. 286,338; B., 1928, 350.

**Water softener** (U.S.P. 1,752,789). **Filtration apparatus** (U.S.P. 1,754,667).—See I.