

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

SEPT. 25 and OCT 2, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Boiling, drying, and heating with high-pressure hot water.** H. STENGER (Chem. Fabr., 1931, 4, 309—310).—The advantages of circulating high-pressure hot  $H_2O$  (e.g., 190°) through a heating system instead of steam are discussed. Any ordinary type of boiler working up to 30 atm. may be used and circulation of the  $H_2O$  through the system is ensured by installing a small rotary pump in the return circuit just before the boiler. As the return  $H_2O$  is at a temp. of only 15—35° below that of the  $H_2O$  in the boiler there is a considerable fuel economy, and as no provision need be made for reducing valves, draw-off cocks for condensed steam, etc., the circulating system is considerably simplified and the labour required in supervision is much reduced. The maintenance of steady temps. is also facilitated and a higher heat efficiency obtained.

A. R. POWELL.

**Detection of phosphate in boiler feed-water.** P. KÖPPEL (Chem.-Ztg., 1931, 55, 539—540).—The presence of a sufficient excess of  $Na_3PO_4$  in  $H_2O$  softened with this salt may be detected by the usual phosphomolybdate test; addition of 10 c.c. of the reagent preheated to 70° to 1 c.c. of the  $H_2O$  gives an immediate yellow ppt. when the  $P_2O_5$  exceeds 30 mg. per litre and a ppt. after 2 min. when less than this quantity is present. Well-softened  $H_2O$  should give the ppt. only after 1—2 min.

A. R. POWELL.

### PATENTS.

**Furnaces.** R. S. COCHRAN (B.P. 351,676, 19.5.30, U.S., 18.5.29).—The bed of the furnace is composed of rails which alternately are stationary and given a reciprocating and rising and falling motion. The outside rails of the moving part form part of a rigid frame which is hung by a number of bifilar suspensions protected as far as possible from the heat. The intermediate rails are attached to only one cross-member of the frame and allowed to slide on the other cross-members; when expansion takes place, these rails are maintained under tension. The surface of both sets of rails is wavy, the pitch of the waves being equal to the stroke.

B. M. VENABLES.

**Pulverulent-fuel furnaces.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 351,768, 16.7.30).—With the object of shortening the flame, that part of the secondary air which is supplied in the neighbourhood of the fuel burner is preheated by burning part of the fuel before it reaches the furnace proper. The apparatus may comprise a hollow and perforated refractory cylinder within the air supply pipe. B. M. VENABLES.

**Removal of dust such as [ash] from plant fired by pulverulent fuel.** CLARKE, CHAPMAN & Co., LTD.,

and W. A. WOODSON (B.P. 351,968, 1.4.30).—The dust is removed by water-operated ejectors through open-ended collecting pipes. B. M. VENABLES.

**Rotary tubular furnace.** E. SCHULZE, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,787,841, 6.1.31, Appl., 9.10.28, Ger., 11.11.27).—The material (e.g., ore, cement) falling from a rotary kiln is received in a funnel-like structure having adjustable walls. The side walls swing about hinges at the top, and the wall upon which the material first impinges is movable bodily and is provided with cooling coils; the opposite wall has a flap door in the lower part for discharge of the material. B. M. VENABLES.

**Heating furnace.** A. W. SMITH and R. B. JONES (U.S.P. 1,790,172, 27.1.31, Appl., 13.5.22).—A regenerative open-hearth furnace is provided with auxiliary currents of preheated air led to points in and at the side of the main ports, which are inlets for the time being, to cause effective mixing of the air and gas.

B. M. VENABLES.

**Heat-treatment furnaces.** HEVI-DUTY ELECTRIC Co., Assees. of W. B. COOLEY (B.P. 352,174, 21.5.30, U.S., 22.5.29).—A furnace having a belt conveyor for the treatment of articles without containers has the belt entirely within the furnace and both runs are supported by rollers which are positively driven to agree with the speed of the belt.

B. M. VENABLES.

**Tunnel kilns.** WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 353,086, 24.4.30).—In a tunnel kiln in which the goods are not permitted to come in contact with the combustion gases, in addition to being heated by radiation the goods are further heated by contact with clean air which has been raised to a high temp. in passages that alternate with the combustion chambers in the roof and walls.

B. M. VENABLES.

**Mercury boilers.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. J. NERAD (B.P. 352,206, 12.6.30, U.S., 13.6.29).—In a Hg boiler the downtake tubes are screened from the fire, but the uptake tubes are divided into a bundle of smaller tubes around and between which good conducting material, solid at the temp. of operation, is placed. Twisted strips may be placed inside the tubes. B. M. VENABLES.

**Heat accumulators.** STEIN-U. THON-INDUSTRIEGES. "BROHLTHAL," and E. and H. POHL (B.P. 352,955, 14.4.30).—The filling of a heat accumulator is arranged in zones having bricks or tiles of exactly similar shape, but the number of passages through the successive zones is in the ratio 1 : 4 : 16 : 64 and so on. The walls are of equal total thickness and the apertures of equal total

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

area. At the junctions of zones the bricks are in the form of De Laval nozzles, and intermediately there may be expansions and contractions producing change of velocity energy into pressure energy and back again.

B. M. VENABLES.

**Chemical heat-storage devices.** A. JUST and M. I. W. J. BYLEVELD (B.P. 351,951, 20.3.30. Holl., 20.8.29).—Such a device (*e.g.*, a foot warmer) is composed of a hermetically sealed rigid vessel containing a salt having negative heat of dissolution and H<sub>2</sub>O (*e.g.*, a 92.5% solution of NaOAc), also at least one solid body (*e.g.*, a few pebbles), and a divided substance (*e.g.*, sand) in sufficient quantity to hold the solid bodies steady unless the vessel is violently shaken. Under these conditions the solution will remain supersaturated when cold under ordinary shocks of transport, and will not lose that property after repeated use.

B. M. VENABLES.

**Drying apparatus, particularly of the continuously operating type.** SOC. ANON. DES ETABL. NEU (B.P. 352,363, 13.11.30. Fr., 13.11.29).—The goods are conveyed through the dryer twice by the same conveyor, on the outside of the upper run and inside of the lower run in succession; they are contained in trucks with foraminous bottoms, and mechanism is described for transferring the trucks from one run to the other.

B. M. VENABLES.

**Apparatus for drying, heating, or cooling.** F. J. WARDEN-STEVENS (U.S.P. 1,787,878, 6.1.31. Appl., 6.9.28. U.K., 18.3.27).—Lump or broken material is allowed to pass downward in a narrow space between two walls which are composed of louvres through which a treating medium enters and leaves. To loosen the material and cause it to flow freely, the louvres are attached to a framework which, when reciprocated, alters the angle of the slats; in addition, cross-bars through the material may be reciprocated up and down.

B. M. VENABLES.

**Drying of materials [tea etc.].** L. N. LLOYD, and PHILLIPS ENG. CO., LTD. (B.P. 351,976, 24.12.29).—A drying chamber is provided with forced- and induced-draught fans and the flow is reversed by a single valve operated at intervals by a funicular arrangement. Means for admixture of fresh air are described.

B. M. VENABLES.

**Rotating drums or cylinders for drying or roasting malt, grain, etc.** PREMIX GAS PLANTS, LTD., and A. DOCKING (B.P. 352,933, 14.3.30).—A rotary cylinder is heated internally by a number of tubular combustion chambers parallel to the axis, which communicate by radial tubes with an annular space in the lining of the kiln.

B. M. VENABLES.

**Drying plant [for road-making materials etc.].** C. G. HODGSON, and MILLARS' MACHINERY CO., LTD. (B.P. 353,458, 23.4.30).—A rotary drum is provided with a combustion chamber adjacent the inlet end, and air, in addition to the gases of combustion, is admitted through ports in the same end after traversing an annular space between the drum and a surrounding shell.

B. M. VENABLES.

**Radiant reading pyrometer.** J. SCHÜNEMANN, Assr. to HARTMANN & BRAUN A.-G. (U.S.P. 1,788,849,

13.1.31. Appl., 16.12.27. Ger., 24.12.26).—In a radiation pyrometer the blackened hot junction is provided with a screen which obstructs the radiation more or less according to the temp. of the surrounding pyrometer body, the change of position of the screen being effected by a bimetallic strip.

B. M. VENABLES.

**Temperature control in chemical reactions.** C. R. DOWNS, Assr. to BARRETT Co. (U.S.P. 1,789,809, 20.1.31. Appl., 11.6.19).—The temp. of an exothermic reaction is regulated by the vaporisation of Hg distributed throughout the zone of reaction, but out of contact with the reagents. The process is applicable to the oxidation of naphthalene to phthalic anhydride.

B. M. VENABLES.

**Binary mixture for absorption refrigerating machines.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 353,806, 13.11.30. Ger., 6.12.29).—The mixture consists of PhMe as the evaporating medium and paraffin oil as the solvent. Part of the difference between condensation pressure and evaporation pressure may be compensated for by the addition of a neutral gas, *e.g.*, A.

D. K. MOORE.

**Refrigerants and absorbents for absorption refrigerating machines.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 351,713, 4.6.30. Ger., 5.6.29).—A refrigerant is chosen having an atm. b.p. close to 100° and a f.p. below 0°, also so that the 50% solution in a suitable solvent has a b.p. at least 10° above that of the pure refrigerant. Examples claimed are: Et isoamyl ether dissolved in paraffin oil, PhMe dissolved in quinoline. The difference in pressure between the absorber and generator can be attained by a reasonable head of liquid and neither mixture attacks steel.

B. M. VENABLES.

**Manufacture of thermal insulating material.** J. A. ROBERTS, R. W. HARDY, and T. HAYMAN (B.P. 352,680, 12.6.30).—Mica is reduced to small flakes and mixed with a paste made by adding boiling H<sub>2</sub>O to a cold solution of flour in such a way that it has become aerated. The mixture is then formed into insulating jackets or other articles and charred by heat.

B. M. VENABLES.

**Sieving or sifting of aggregate material.** LONDON BRICK CO. & FORDERS, LTD., and G. H. C. RACTLIFFE (B.P. 352,155, 10.5.30).—The apparatus comprises a fixed frame, an outer uniformly rotating ring, and an inner ring carrying the screen which rotates with the outer ring but at intervals is held stationary by a catch and then released so that it resumes its normal position with a shock, under the action of springs.

B. M. VENABLES.

**Sorting of coal and other minerals.** W. H. and W. N. BARKER (B.P. 352,796, 2.9.30).—An apparatus of the inclined-plane type is maintained in a humid atm. either by means of a humidifying apparatus within an enclosing casing or by means of currents of humidified air.

B. M. VENABLES.

**Pneumatic separation of materials of different densities.** P. SOULARY, and COMP. DES MINES DE BRUAY (B.P. 352,943, 10.4.30. Fr., 15.4.29. Addn. to B.P. 320,207; B., 1929, 1036).—In an apparatus as described in the prior patent the separating zones are provided

with longitudinal vertical partitions, means are provided for regulating the distribution of air, and the shaking tables are given an upward component of motion by supporting them on leaf springs which are inclined at an angle not exceeding  $55^\circ$  to the horizontal.

B. M. VENABLES.

**Mixing devices.** C. HILDEBRANDT-SØRENSEN (B.P. 353,240, 16.7.30).—A pair of stirrers, eccentric to the vessel, rotate about their own shafts and simultaneously about the axis of the vessel; the lower part of one is in the form of a triangle with twisted apex, which latter runs in an annular channel formed between the sloping wall of the vessel and a conical centre piece, in which channel the discharge ports are placed.

B. M. VENABLES.

**Apparatus for mixing materials.** F. C. OVERBURY, Assr. to FLINTKOTE CO. (U.S.P. 1,789,320, 20.1.31. Appl., 7.12.25. Renewed 16.1.30).—The apparatus comprises a tank in which are a number of vertical, jacketed, cylindrical partitions, spaced from the bottom of the tank except in the case of the last, which is provided with a valved outlet. Each partition is provided with propellers driven by vertical shafts, and some may have heating-cooling coils in addition to the jackets. The flow of liquid is under and over the edges of the partitions and constituents may be added at various points. The apparatus is stated to be suitable for pasty materials.

B. M. VENABLES.

**Mixing machines for plasticising phenol compounds and such like.** D. A. DUCHARME and F. H. BANBURY (B.P. 352,803, 9.9.30. U.S., 31.5.30).—A mixing machine of the Banbury or other type is rendered usable on very sticky compounds by cooling the supply neck and ram or other feeder so that the material is fed in non-sticky condition.

B. M. VENABLES.

**Mixing, dissolving, or dispersing apparatus.** G. C. BAKEWELL, Assr. to TURBO-MIXER CORP. (U.S.P. 1,790,257, 27.1.31. Appl., 20.3.29).—A turbine-like rotor runs near the bottom of a vessel and is surrounded by fixed guide blades, which are again surrounded by plates having a toothed surface, attached to the bottom of the vessel.

B. M. VENABLES.

**Safety mixers.** MIXING EQUIPMENT CO., INC. (B.P. 351,808, 20.8.30. U.S., 28.8.29).—A mixing device suitable for attachment to the rim of a tank of inflammable liquid and embodying an electric motor has the latter pipe-ventilated, the air being drawn from a safe place and delivered in the general direction of the propeller.

B. M. VENABLES.

**Apparatus for making emulsions.** M. G. GREGG, Assr. to FLINTKOTE CO. (U.S.P. 1,789,897, 20.1.31. Appl., 19.6.22. Renewed 27.7.29).—The apparatus comprises two straight pieces of pipe provided with agitators and connected by two return bends to form a closed circuit.

B. M. VENABLES.

**Lixiviation of vegetable, particularly beetroot, slices.** K. PHILIPP, Assee. of A. REIHER (B.P. 352,923, 2.1.30. Ger., 14.1.29).—The lixiviation takes place in a rotary cylinder which is provided with annular end-walls to retain liquid and with lifting vanes which are

inclined so as to work the solid matter countercurrent to the liquid. Washing is completed in an elevator. [Stat. ref.]

B. M. VENABLES.

**Filters.** T. F. MILLER (B.P. 352,038, 6.1.30 and 26.2.30).—A metallic (or other non-fibrous) filter is constructed so as to retain a considerable quantity of filter aid on the edges of the metal. A metallic strip is wound round a central inlet (or outlet) pipe, and the strip is formed with raised portions to act as distance pieces and with central elongated perforations which are longer than the lands left between them, so that some part of the holes will overlap right through all the turns except the outermost which is left unperforated to form the outer wall; the feed tube is correspondingly perforated. Alternate strips may be of different widths (or of the same width but slightly staggered) so that the uneven edges form pockets to retain filter aid, and annular screens may be juxtaposed to the edges for additional support.

B. M. VENABLES.

**Filtering device.** A. E. TOMLINSON (B.P. 353,106, 2.5.30).—The pulp is applied to the inside of an endless belt of filter medium, is squeezed while passing over the pulley, and removed by scrapers or a reverse curve. The cake may be removed from one longitudinal zone of the belt and applied to another for washing or other treatment.

B. M. VENABLES.

**Filtration.** C. T. CABRERA, Assr. to ELECTRO DIALYZER CORP. (U.S.P. 1,789,425, 20.1.31. Appl., 5.5.28).—The apparatus comprises an inclined pervious conveyor running within and without a tank.

B. M. VENABLES.

**Filter press.** P. L. DORNER (U.S.P. 1,788,086, 6.1.31. Appl., 11.5.29).—The press is suitable, e.g., for reconditioning dry-cleaning fluids. It is divided into sections each of which is charged with a different filter aid or absorbent in suspension in clean fluid, after which dirty fluid is passed through all sections in series. The claims mainly refer to an arrangement of pipe-work and valves by which one pump may effect all the operations.

B. M. VENABLES.

**Separation of liquids from fine-grained material.** L. ALTPETER, and GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 352,645, 20.5.30).—A centrifuge of the type comprising upper and lower bowl walls with a hydraulically closed gap between the two for outlet of solid material is provided with a feed-distributing plate and a conical baffle, both rotating with the bowls and with sprays for washing the material while it is on the baffle and bowl walls.

B. M. VENABLES.

**Manufacture of filter blocks of paper pulp or similar material.** F. G. RILEY (B.P. 352,379, 1.5.30).—A press is described for the manufacture of blocks suitable for use in the apparatus described in B.P. 349,262 (B., 1931, 703).

B. M. VENABLES.

**Apparatus for making precipitable the solids held in solution in liquids.** G. W. GANE (U.S.P. 1,789,738, 20.1.31. Appl., 14.6.28. N.Z., 19.7.27).—The liquid is allowed to flow to and fro through a number of horizontal, double-concentric vessels, and at the ends of the inner flows is subjected to jets of steam which either aid or retard the flow.

B. M. VENABLES.

**Recovery of crystallisable constituents from liquors.** R. W. MUMFORD, Assr. to AMER. POTASH & CHEM. CORP. (U.S.P. 1,790,436, 27.1.31. Appl., 14.9.25. Renewed 4.10.30).—The process is described in connexion with the recovery of KCl from Searles Lake brine, which consists mainly of the sulphates, chlorides, carbonates, and borates of Na and K. By evaporation at a high temp. the liquor may be brought to a condition such that, on cooling, KCl and, later, borax will crystallise out, the latter having the greater tendency to remain supersaturated. The cooling is therefore effected first by evaporation under reduced pressure with considerable agitation, H<sub>2</sub>O or KCl wash-liquor being added to replace loss by evaporation; before precipitation of borax begins the liquor is transferred to an indirect cooler having less agitation, in which the remainder of the KCl will grow round the small crystals already formed, while the borax remains in supersaturated solution. B. M. VENABLES.

**Centrifuging salts [ammonium sulphate].** N. V. SILICA EN OVENBOUW MIJ., Assees. of DR. C. OTTO & CO. G.M.B.H. (B.P. 352,828, 29.9.30. Ger., 28.9.29).—The centrifuge is provided with a system of chains in contact with the walls and round the base, the latter being attached to a hood on the bottom of the centrifuge. At the conclusion of the process the raising of the hood and chains breaks up the mass and removes adherent material from the walls, thus facilitating emptying. W. J. WRIGHT.

**Column.** C. B. SCHNEIBLE, Assr. to C. B. SCHNEIBLE, K. F. SCHREIER, and B. B. SCHNEIDER (U.S.P. 1,790,596, 27.1.31. Appl., 16.5.27).—A column for dephlegmating etc. is filled with alternate annular and central plates which do not overlap to any extent under the plates; also not overlapping the edge are a number of vanes inclined to the radial direction. B. M. VENABLES.

**Pipe still.** G. W. WATTS, Assr. to STANDARD OIL CO. (U.S.P. 1,789,267, 13.1.31. Appl., 18.7.25. Renewed 3.10.27).—The heating means comprise a combustion chamber and long flue, preferably after a return bend, and across the latter are a number of banks of tubes. The flow of oil (or other material) is first through a number of tubes in series, and lastly through coils comprising several tubes in parallel; intermediately the oil flows through a smaller number of tubes exposed to direct radiation in the combustion chamber, and at this point a supply of steam may be added. The operations may be effected under pressure; straight tubes are used and cellular connecting headers are described, with means to facilitate cleaning and renewal, and to eliminate return bends and tube sheets. B. M. VENABLES.

**Distillation control.** W. K. LEWIS, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,789,339, 20.1.31. Appl., 31.3.28).—In a line of stills operating on, e.g., petroleum, the bottoms flowing from one of the later stills, usually the one effecting the final removal of gasoline, are subjected to continuous sampling at a uniform rate and the sample is heated with a const. supply of heat. The vapours are rectified with a const. reflux and the temp. of the rectified vapour is caused to vary the conditions of the main distillation

so as to hold the temp. of the rectified sample vapour nearly const. In the control still the const. heat supply is preferably electrical, and the const. reflux may be produced by the evaporation of a const. drip of water. B. M. VENABLES.

**Apparatus for dephlegmating vapours.** W. M. CROSS, Assr. to GASOLINE PRODUCTS CO., INC. (U.S.P. 1,789,670, 20.1.31. Appl., 29.10.23).—A tower containing means for producing a reflux, bubbling trays, and a reboiler is described. B. M. VENABLES.

**Fractionation.** T. DE C. TIFFT and W. MENDIUS, Assrs. to SINCLAIR REFINING CO. (U.S.P. 1,789,059, 13.1.31. Appl., 23.1.28).—A form of tray for a fractionating column is described. No bubbling caps or other apertures are provided. The top of each diaphragm is provided with a single weir extending across a chord, and the edge of each diaphragm is bent down along a chord rather more distant from the axis of the tower and terminates in an adjustable straight edge, so that the depending part dips into the pool formed by the weir on the plate below; the flow of the rising vapour is thus in a zig-zag manner over the weirs and under the depending edges, spray being produced at the latter points. B. M. VENABLES.

**Tubular filters [for gases].** E. W. STRAUS-SCHARINA (B.P. 353,600, 28.5.30. Ger., 28.5.29).—The filter sleeves are subjected to longitudinal shocks during the whole of the time they are in operation. B. M. VENABLES.

**Centrifugal apparatus for separating dust, grit, etc. from flue or furnace gases.** O. B. JACOBSEN (B.P. 352,374, 6.6.30).—In an apparatus as described in B.P. 341,058 (B., 1931, 281), the turbo-blades are supported between conical walls, the angle of the outer one being greater than that of the inner. B. M. VENABLES.

**Apparatus for separating and recovering gases [by adsorption].** O. L. BARNEBEY, Assr. to AMER. SOLVENT RECOVERY CORP. (U.S.P. 1,787,714, 6.1.31. Appl., 29.7.22. Renewed 3.11.28).—A number of separated layers of adsorbent are supported on coarse and fine gauze, or other foraminous 2-ply material, and each layer is provided with embedded means (such as a pipe-coil) for indirect heating or cooling and also for adding steam directly to the adsorbent material. B. M. VENABLES.

**Separation of gaseous mixtures by liquefaction.** M. FRÄNKEL (B.P. 352,570, 16.4.30. Ger., 19.4.29. Addn. to B.P. 246,172. Cf. F.P. 613,755; B., 1927, 513).—The major part of a compressed gas mixture is subjected to cold exchange with a resolved product of low b.p. by means of reversing regenerative cold accumulators, as described in the prior patent, the cold-accumulating substance being metallic and having large surface. The minor part of the gas mixture exchanges cold with the high-b.p. product by means of tubular countercurrent devices which may also be subjected to reversal at intervals, but only for the sake of removing frost. The high-b.p. substance may be compressed to a high pressure while in the fluid state before evaporation in the cold exchanger. B. M. VENABLES.

**Liquefaction of gases.** IMPERIAL CHEM. INDUSTRIES, LTD., and A. W. HARROLD (B.P. 353,618, 6.6.30).—Schemes are described for the recovery by condensation of a gas of high b.p. from an atm. of low b.p., *e.g.*, SO<sub>2</sub> from pyrites gas; the nett amount of cold is provided by the evaporation of a liquid of low b.p., *e.g.*, CO<sub>2</sub>. The simplest scheme comprises compression in one side of an engine, cooling in three heat exchangers the last two of which deliver liquefied desired constituent, expansion in the other side of the engine, and return passage through the third and first exchangers, the second being cooled by the independent fluid.

B. M. VENABLES.

**Liquefaction.** W. L. DE BAUFRE and J. W. DAVIS (U.S.P. 1,786,898, 30.12.30. Appl., 29.12.23).—A scheme for the purification of a gaseous mixture rich in a desired constituent of low b.p. (*e.g.*, He) which has been obtained as residual vapour from a previous liquefaction is described. The gaseous mixture is subjected to cold exchange with a bath of liquefied refrigerant (*e.g.*, N<sub>2</sub>) which has been obtained by compression, heat exchange with itself, partial expansion through an engine, and further cold exchange between the expanded and unexpanded portions; the undesired constituents of the mixture are liquefied out and returned to the original liquefier.

B. M. VENABLES.

**Means for producing and storing non-oxidising gases.** W. J. WILLENBORG (B.P. 352,006, 19.2.30. U.S., 19.2.29).—The gases comprise the exhaust from an internal-combustion engine and are pumped to a storage vessel by a pump cylinder combined with the engine. Electrical controls are provided so that when the gases contain an insufficient proportion of CO<sub>2</sub> they are pumped to waste.

B. M. VENABLES.

**Carrying out catalytic gas reactions.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 353,622, 6.6.30).—The gases prior to passage over the catalyst proper are caused to take up small quantities of the vapour of alkali or alkaline-earth metals by passing them at a moderate temp., *e.g.*, 400° in the case of K, over the heated metal. The process is applicable to the cracking of methane and the synthesis of NH<sub>3</sub>.

B. M. VENABLES.

**Machines for testing the hardness of materials.** W. & T. AVERY, LTD., and A. H. HANCOX (B.P. 353,729, 21.8.30).—The plunger of a machine of the penetration type is loaded by a lever and wts. A device is described by which the latter can be removed and replaced without shock.

B. M. VENABLES.

**Apparatus for measuring the hardness of materials.** A. F. SHORE (B.P. 353,549, 5.5.30).—In a machine of the penetration type, the pointer of the dial that measures the advance of the plunger is triple, the two branches of the tail making an angle equal to the desired penetration so that readings can be obtained with samples of varying height without resetting to zero.

B. M. VENABLES.

**Determination of the m.p. of materials.** G. A. DE GRAAF, ASSR. to EIMER & AMEND (U.S.P. 1,789,098, 13.1.31. Appl., 27.1.28).—The sample (*e.g.*, coal ash) is placed on an electrically-heated strip of Pt or other suitable metal and the temp. is slowly raised by a

motor-operated sliding rheostat. The sample is observed simultaneously by a visual telescope and a radiation pyrometer. Black-body conditions are maintained by an enclosing shell which may also be gastight so that a vac. or inert atm. may be maintained.

B. M. VENABLES.

**Determining the plasticity of [semi-elastic] substances.** A. E. WHITE. From B. F. GOODRICH Co. (B.P. 353,693, 24.7.30).—A motor running at const. speed closes and opens a set of toggles which at one end are loaded by a spring and at the other bear upon a sample of fixed dimensions, *e.g.*, for rubber, a cylinder of length and diam. 1 cm. each. By this means a rigid material will be subjected to a large load and have small deformation, and vice versa for a soft material, so that the range covered is very wide. One dial reads the max. deformation and hence also the load, another dial reads the recovery on removal of the load.

B. M. VENABLES.

**Apparatus for examining crystalline bodies with the aid of X-rays.** N. V. PHILIPS' GLOEILAMP-ENFABR. (B.P. 351,744, 1.7.30. Holl., 15.7.29).—An apparatus suitable, *e.g.*, for distinguishing between natural and cultivated pearls is described, comprising an X-ray tube, a box for the object, and means for observation both by visual fluorescence and photographically. The whole is covered with material impervious to the rays except through the necessary windows, and safety automatic shutters are provided for the latter.

B. M. VENABLES.

**Agitation and settling apparatus.** W. W. TRIGGS. From DORR Co. (B.P. 351,989, 28.3.30).—See U.S.P. 1,752,789 and 1,752,795; B., 1930, 886.

**[Boiler] furnaces.** A. W. BENNIS (B.P. 353,609, 31.5. and 2.10.30).

**Gas-heated apparatus for heating liquids.** H. JUNKERS (B.P. 353,814, 15.12.30. Switz., 17.4.30).

**Liquid fuel burning apparatus.** A. LANSER (B.P. 353,711, 31.7.30. Luxembourg, 4.7.30).

**Refrigerating apparatus.** FRIGIDAIRE CORP., Assees. of H. F. SMITH (B.P. 353,945, 29.4.30. U.S., 13.9.29).

**Intermittently working refrigerating machines of the absorption type.** ARTIEBOLAGET SVEAEXPORT (B.P. 353,963, 12.5.30. Swed., 12.4.30).

**Evaporator for absorption refrigerating machines.** DEUTS. GASGLÜHLICHT-AUER-G.M.B.H. (B.P. 353,746, 3.9.30. Ger., 3.9.29).

**Heating bituminous matter. Distilling apparatus.**—See II. **Pulp-filtering machine.**—See V. **Drying of clay ware etc.**—See VIII. **Granulation of slag.**—See X. **Precipitating particles from gases.**—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Spontaneous electrification in coal-dust clouds.** S. C. BLACKTIN and H. ROBINSON (Safety in Mines Res. Bd., 1931, Paper No. 71, 17 pp.).—Large-scale experiments conducted in the open air have shown that high voltages are produced on well-insulated metal tubes through which a stream of air carrying coal dust

in suspension is passed. The concentration of dust necessary to produce ignition of firedamp is extremely small, and the speed of the air current is well within the limits of mining practice. These effects are obtained only when electrical and atm. conditions are favourable for the accumulation of the electrostatic charges produced by friction, and ducts conveying dust-laden air must possess, therefore, a very high insulation resistance to earth if the electrostatic charge is to be retained on the system. Humidity of the atm. inhibits the production of high voltages on such ducts. A minute leakage to earth is sufficient to eliminate completely the electrostatic charge. Accidental contact of a mine ventilation duct with earth at a number of points, or a single deliberate earth connexion, provides a leakage which will effectively prevent the accumulation of an electrostatic charge on the duct, even though the conditions for electrification may be otherwise favourable.

E. S. HEDGES.

**Evaluation of blast-furnace coke.** A. KILLING (Stahl u. Eisen, 1931, 51, 901—905).—Coke prepared from finely-ground coal (90% through a 3-mm. screen) has a high porosity (over 50%) and a correspondingly low apparent sp. gr. (430 kg. per cu. m.); such coke has given excellent results in the production of Thomas pig iron over a period of several months with an appreciable economy in fuel consumption. The suitability of a coke for blast-furnace purposes does not depend on its combustibility, but is measured by its crushing strength, lightness, and reactivity, the last-named being dependent on its porosity.

A. R. POWELL.

**Coke and charcoal. Effect of ash on the reactivity and combustibility.** Y. OSHIMA and Y. FUKUDA (J. Soc. Chem. Ind. Japan, 1931, 34, 238—240 B).—Graphite, coke, and charcoal when finely ground and extracted with HF and HCl show an increase of effective surface, as measured by adsorption of H<sub>2</sub>O vapour. Ash removal increases the reactivity of coke, but reduces that of wood charcoal, to the oxidation of which ash acts as a catalyst. Charcoal ash is much more finely dispersed than ash in coke. The combustibility in air of different carbonaceous materials when freed from ash is almost identical, the physical structure and state of the C having no apparent influence.

C. IRWIN.

**Production of active carbons by impregnation methods.** J. MILBAUER (Z. Zuckerind. Czechoslov., 1931, 55, 435—439).—Org. materials impregnated with CaCl<sub>2</sub> solution were dried, powdered, and heated in graphite crucibles at const. temp.; the resulting carbons were boiled with HCl, washed, dried, powdered, and tested on molasses solution. No differences in decolorising power resulted from variations of 2:1 to 6:1 in the ratio of CaCl<sub>2</sub> to org. matter, on dry basis. With ratio 4:1 and temp. 500°, the optimum period of heating was in most cases 3 hr. With ratio 4:1 and period 3 hr., the optimum temp. for starch, peat, molasses, cacao shell, and nut shell was 500°, for sawdust 700°, and for horn meal and pure cellulose still higher. Under optimum conditions the most active carbons were obtained from pure cellulose and sawdust.

J. H. LANE.

### Two-step ash-pan for rotary gas producer.

PRADEL (Chem.-Ztg., 1931, 55, 581).—The usual type of ash-pan involves an accumulation of clinker around the edges and an unequal distribution of the fire within the generator. The pan described has sides inclined outwards with a ledge halfway up which delivers the ashes in two stages and obviates this. Works' trials have shown that generators fitted with it need cleaning much less frequently, less poking, and fewer attendants.

C. IRWIN.

### Dilution of straight coal [gas] by producer gas externally generated.

A. L. HOLTON (Gas J., 1931, 195, 213—215).—Data showing the effect of dilution on three qualities of straight coal gas with different diluents are tabulated. Trefois producer gas (B., 1931, 5) has proved a suitable diluent for reducing the calorific val. of horizontal-retort gas from about 600 to 450 B.Th.U. The inclusive cost of producing Trefois gas at the Partington works over a year is estimated at 1.35d. per 1000 cu. ft. of average calorific val. 133 B.Th.U.

A. B. MANNING.

### Dilution of straight coal gas by steaming in intermittent chamber ovens.

G. DIXON (Gas J., 1931, 195, 215).—The ovens were of the inclined type, with three steaming points in the floor of each. The charge was 4½ tons. On a 16-hr. schedule carbonisation was completed in 11½ hr. and steaming was then operated for 4½ hr. prior to discharge. Steam was admitted to the ovens at 26 lb./sq. in. pressure, superheated to 250°. The calorific val. of the straight coal gas was 550 B.Th.U., that of the diluted gas 496 B.Th.U. Steaming raised the yield from 12,727 to 16,178 cu. ft./ton. The net cost of the gas into the holder was 2.67d. per therm.

A. B. MANNING.

### Ignition and propagation velocity in acetylene-oxygen mixtures. Striking back of the welding torch.

RIMARSKI, PLEUS, FRIEDRICH, STREB, NOACK, KEMPER, HOFFMANN, and KONSCHACK (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 52—60; Chem. Zentr., 1931, i, 2418).—With a cooled jet the velocity of inflammation markedly increases with increase in O<sub>2</sub> content; with a jet temp. rising to 400° the velocity change is relatively small (3—10%). The higher is the thermal conductivity of the jet, the lower (40—50°) is the temp. of back-firing.

A. A. ELDRIDGE.

### Hydrogenation of bitumen.

E. H. BOOMER and A. W. SADDINGTON (Canad. J. Res., 1931, 4, 517—539).—Experiments on Alberta bitumen indicate that 75% of the latter may easily be converted into gasoline and oil. A cylindrical autoclave of 1.8 litres capacity and designed for close temp. control and efficient agitation is described.

W. GOOD.

### Hazards in catalytic hydrogenation at elevated temperatures and pressures.

H. ADKINS (J. Amer. Chem. Soc., 1931, 53, 2808—2809; cf. A., 1931, 919).—Commercial-scale hydrogenations require adequate preliminary investigations and properly designed bombs.

J. G. A. GRIFFITHS.

### Determination of olefine, aromatic, and paraffin hydrocarbons in neutral oil from coal tar.

E. B. KESTER and W. D. POHLE (Ind. Eng. Chem. [Anal.], 1931, 3, 294—297).—Methods previously available are

briefly discussed. In the suggested method 100 c.c. of neutral oil are shaken with 300 c.c. of 80%  $H_2SO_4$  for 5 min., set aside for 30 min., and the acid is drained off; after a further 30 min., any additional acid which separates is also drained off. The reduction in vol. is recorded ( $R_1$ ). The oil is then distilled to 5° higher than the end-point of the untreated oil. To the vol. of distillate found a small correction factor is added, computed in earlier runs from the total vol. distilled minus the total vol. of distillate and residuum. The sum of the vol. reduction on distillation, computed to the whole oil basis, and  $R_1$  gives % olefines. 20 c.c. of the distillate are shaken with 60 c.c. of 98%  $H_2SO_4$  for 5 min. and allowed to settle as in the 80%-acid treatment. From the contraction 0.19 c.c. is deducted to allow for the solubility of the paraffins in the acid, the corrected contraction, computed to the whole oil basis, then giving the aromatic content. By this procedure mixed-type compounds, *e.g.*, styrene and indene, are included in the olefines. Satisfactory results are obtained with synthetic mixtures. The results of the analyses of a number of tar neutral oils are tabulated. A. B. MANNING.

**Analysis of gaseous hydrocarbons. A short-cut method.** R. ROSEN and A. E. ROBERTSON (Ind. Eng. Chem. [Anal.], 1931, 3, 284—289).—The method, which has been worked out primarily for mixtures of  $C_2H_6$ ,  $C_3H_8$ , and  $C_4H_{10}$ , consists in distilling the liquefied gas at const. pressure, and comparing the distillation curve, *i.e.*, the curve in which vol. distilled is plotted against temp., with standard curves obtained from known mixtures. Standard curves covering a range corresponding with the composition of certain refinery gases are given. The influence of the presence of  $C_2H_4$ , propylene, and *isobutane* on the form of the standard curves has been studied, and a correction curve for use with mixtures containing those constituents is given. The apparatus and technique are simple; only 200—250 c.c. of liquid  $N_2$  are required for an analysis, which can be completed in 60—75 min. A. B. MANNING.

**Analysis of light oils from low- and intermediate-temperature carbonisation.** E. B. KESTER and C. R. HOLMES (Ind. Eng. Chem. [Anal.], 1931, 3, 292—294).—The crude oil is refined by shaking with  $H_2SO_4$  (*d* 1.84), 70 c.c. of acid per litre of oil, to remove olefines, and is then steam-distilled. The refined oil is fractionated under a column (*cf.* "Gas Chemists' Handbook," p. 402—3) and cuts are taken as follows: (1) to 79°, pre- $C_6H_6$ , (2) 79—81°,  $C_6H_6$ , (3) 81—109°,  $C_6H_6$ -PhMe, (4) 109—111.5°, PhMe, (5) 111.5—137, PhMe-xylene, (6) above 137°, residuum. The aromatic content of each fraction is determined by sulphonation of a portion (20 c.c.) with 3 vols. of 98%  $H_2SO_4$ . To determine the vol. of each aromatic hydrocarbon in those fractions where two are present, 30 c.c. of the fraction are subjected to a distillation test (*loc. cit.*, p. 406), and the % PhMe is found directly from curves obtained with known mixtures. When paraffins are present the % read is modified by multiplying by the % of aromatics in the fraction, the assumption being made that the paraffins are equally distributed throughout the boiling range of the fraction. Satisfactory results are obtained with synthetic mixtures. The results of analyses of light oils from the carbonisa-

tion of two American coals over the temp. range 500—1100° are tabulated. A. B. MANNING.

**Determination of small amounts of benzene vapours in air.** H. F. SMYTH (J. Ind. Hygiene, 1931, 13, 227—230).—The method described previously (B., 1930, 265) is satisfactory for 30 p.p.m. An accuracy of over 85% is claimed for determinations down to this concentration in presence of not more than three times as much PhMe. R. K. CALLOW.

**Determination of softening point as control method for evaluation of paraffin [waxes].** E. DAWIDSON and S. VON PILAT (Allgem. Oel- u. Fett-Ztg., 1931, 28, 261—263).—The range of the components of a paraffin wax may be gauged from the softening point; the smaller the difference between this and the setting point (Shukoff) the sharper has been the fractionation of the wax. Thus waxes of different composition, but with the same setting point, can be differentiated. A modification of the ring-and-ball method for determining softening point is detailed, by which reproducible results are readily obtained. For mixtures such as paraffin wax the softening point is always below the setting point; for pure substances it is slightly above the latter. E. LEWKOWITSCH.

#### PATENTS.

**Manufacture of peat briquettes.** L. RUDEMAN (B.P. 352,542, 11.4.30).—The moisture in the peat is first reduced to 40—60%, then the peat is broken up, mixed with pitch in a cold state, impregnated with phenols, subjected to superheated steam while still being agitated, and finally pressed into briquettes which are waterproof. B. M. VENABLES.

**Manufacture of fuel briquettes.** W. W. WHITE and H. D. PONTET (B.P. 352,444 and 352,556, [A] 3.1.30, [B] 17.3.30).—Carbonaceous briquettes are stoved in a current of non-oxidising gas so as to maintain a clear atm. to permit the transmission of radiant heat, the temp. in (A) being 400—600° and in (B) 500—600°. In (A) the stoved briquettes may be dipped in Na silicate and subsequently subjected to the direct action of a Bunsen flame. To produce a luminous flame, or a heavy ash, a Na salt or Fe (and/or Mn) oxide may be incorporated respectively. In (B) the atm. from the retort may be cooled to recover volatile matter and the inert gas re-used; coke breeze may be incorporated in order to prevent adjacent briquettes from sticking. B. M. VENABLES.

**Coke ovens and the like.** C. STILL (B.P. 351,872, 27.10.30. Ger., 28.10.29).—A tubular apparatus for leading away the gases and vapours from the interior of the charge comprises an upper tube supported by the roof of the oven and a lower tube supported by the charge. The upper tube can be withdrawn from the oven, the lower tube being left in the charge and discharged with the coke. The upper tube opens into a box, for collecting the gas, vapours, and their condensates, which communicates by means of a common collecting pipe for the oven with the collecting main of the battery. A. B. MANNING.

**Coking retort ovens.** KOPPERS Co., Assees. of J. BECKER (B.P. 353,473, 24.5.30. U.S., 28.1.30).—When producing coke requiring a long coking period, *e.g.*,

foundry coke requiring 24–30 hr., the decreased heating rate leads to unequal heat distribution. This is avoided by periodically and alternately supplying fuel gas and air at max. and min. rates which are equally removed from the average rate required for the coking time. The gas and air supplies are automatically controlled.

D. K. MOORE.

**Heating systems for coke ovens.** KOPPERS CO., Assees. of J. BECKER (B.P. 352,383, 3.1.30. U.S., 19.2.29).—A series of retort-type ovens is fired almost entirely with blast-furnace gas, but enough coke-oven gas is added to maintain the calorific val. of the mixture at the max. value of blast-furnace gas alone. The mixture is continuously tested and its thermal val. controls the admixture. [Stat. ref.] B. M. VENABLES.

**Coke-oven doors.** A. L. MOND. From VEREIN. STAHLWERKE A.-G. (B.P. 353,741, 29.8.30).—Heat radiation losses from coke-oven doors are reduced by covering the outside of the doors with a layer of insulating material, e.g., sterchamol brick (analysis given) about 80 mm. thick.

D. K. MOORE.

**Manufacture of coke.** R. A. MOTT, R. V. WHEELER, and NAT. FEDERATION OF IRON & STEEL MANUFRS., LTD. (B.P. 351,546, 27.3.30).—The coal to be carbonised is blended with up to 12% of powdered fusain, material rich in fusain, wood charcoal, low-temp. or high-temp. coke. The particles of the added material should be not more than  $\frac{1}{16}$  in., and preferably not more than  $\frac{1}{100}$  in., in size.

A. B. MANNING.

**Low-temperature carbonisation and distillation of coal, lignite, and oil shale.** "INTERTRUST," COMP. GÉN. DE DISTILLATION À BASSE TEMP. ET MINÈRE SOC. ANON., Assees. of P. BRENU (B.P. 351,552, 27.3.30. Belg., 6.4.29).—The apparatus consists of a succession of distillation chambers, preferably rectangular, each containing a number of vertical retorts rigidly connected with each other and adapted to be lifted bodily with their charges out of the chamber and transferred to a cooling chamber before being discharged. The retorts are provided with central, perforated tubes for leading away the volatile distillation products. Between the distillation chambers are combustion chambers, the hot gases from which can pass into the former and circulate round the retorts therein. An assembling plate provided with holes into which the tops of the retorts fit closes the top of the distillation chamber in a gastight manner, and an outer cover is provided which forms with the assembling plate a collecting chamber for the distillation gases and vapours. This communicates through a seal and a valve with a collecting main. The chambers are directly heated in turn; the hot gases from a directly heated chamber are circulated through the subsequent chambers up to the next directly-heated chamber, so that the temp. of the charges is gradually raised until the corresponding chamber is in turn directly heated.

A. B. MANNING.

**Manufacture of water-gas of regulated nitrogen content.** POWER GAS CORP., LTD., N. E. RAMBUSH, and J. M. BALLENGALL (B.P. 353,522, 26.4.30).—The cycle of operations consists of two up-runs, one lasting for 2 min. and the other for 1 min., one down-run (2 min.), and one blow (1 min.). Precautions are taken to prevent

the admixture of air and gas. A flow-meter on the gas outlet pipe operates a valve controlling the air supply, thus regulating the  $N_2$  content of the gas. A servomotor is provided to operate the valves, some of which are interlocked, and also a delay-action device.

D. K. MOORE.

**Manufacture of producer gas.** K. KOLLER and Z. GALOCSY (B.P. 351,892, 26.11.30. Hung., 28.11.29).—The process is carried out continuously in two stages. In the first stage a solid, liquid, or gaseous fuel is burned with  $O_2$ , air, or mixture thereof, in a combustion chamber which is in direct communication with the gas producer proper. The combustion gases and excess  $O_2$  are mixed with saturated or superheated steam, and in the second stage of the process the mixed gases are passed through the incandescent fuel column of a producer operating with molten slag.

A. B. MANNING.

**Distillation of tar.** BARRETT CO., Assees. of S. P. MILLER (B.P. 351,388—9, 17.1.30. U.S., 26.1.29. Cf. B.P. 347,240; B., 1931, 663).—(A) The tar is heated in an externally-heated still, preferably a pipe-coil still, by indirect contact with hot waste gases, to a temp. above the b.p. of part of the oils contained therein, and the preheated tar is then further distilled by being brought into direct and intimate contact with hot coal-distillation gases. (B) Tar preheated as in (A) is discharged into a vapour box where the lower-boiling constituents are volatilised, the vapours being conducted to suitable condensing means. The residue is then brought in a preheated state into direct and intimate contact with hot fuel gases to effect further distillation of the tar.

A. B. MANNING.

**Apparatus for heating tar or bituminous matter liquefiable by heat.** H. L. DALE (B.P. 352,623, 10.5.30).—The apparatus comprises a horizontal, open-ended, steam-jacketed cylinder surrounded by another cylindrical or prismatic casing with closed ends and so perforated that the material flows longitudinally over the outside and through the inside of the heating jacket. The above apparatus is placed in a larger tank containing the material.

B. M. VENABLES.

**Preparation and treatment of aqueous emulsions of asphalt and the like, particularly for road-making.** METALLGES. A.-G. (B.P. 353,702, 28.7.30. Ger., 16.8.29).—Asphalt, emulsified in  $H_2O$  by the addition of  $Na_3PO_4$ , is flocculated by adding alkaline-earth salts, especially  $CaCl_2$ .

D. K. MOORE.

**Catalytic hydrogenation of hydrocarbon oils.** STANDARD OIL DEVELOPMENT Co., Assees. of R. P. RUSSELL and M. W. BOYER (B.P. 353,792, 16.10.30. U.S., 17.10.29).—Heavy oils are hydrogenated by heating to 400–470° and passing with  $H_2$  under pressure through a drum containing metallic oxides as catalysts. After a time the activity of the catalyst falls, and is restored by passing through the drum a light hydrocarbon oil, e.g., kerosene at above 400°, and  $H_2$  at a pressure above 20 atm.

D. K. MOORE.

(A) Treatment of carbonaceous materials with hydrogen in the presence of catalysts. (B) Refining of liquid carbonaceous materials such as petroleum products and residues, asphalt, etc.



H. D. ELKINGTON. From N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 353,506—7, 25.4.30).—(A) Destructive hydrogenation of materials such as coal, the removal of O from org. materials, *e.g.*, PhOH, and the desulphurisation of petroleum products is effected by heating them to 460° in H<sub>2</sub> at 100 kg. per sq. cm. pressure in the presence of catalysts consisting of colloidal metallic compounds, *e.g.*, Mo oxide, adsorbed by carriers, *e.g.*, pumice, SiO<sub>2</sub> gel, metal hydroxides. (B) For the desulphurisation of petroleum oils the catalyst consists of colloidal Cr compounds adsorbed by active C, with or without colloidal Mo compounds adsorbed by active C.

D. K. MOORE.

**Refining of hydrocarbons.** E. W. ISOM and E. B. PHILLIPS, ASSTS. to SINCLAIR REFINING Co. (U.S.P. 1,786,493, 30.12.30. Appl., 27.3.29).—A solid, absorbent, refining agent is suspended in a crude petroleum stock and the mixture passed rapidly through a heating zone, where it is heated to not above 400° but below cracking temp., and is discharged into a vaporising and digesting zone from which the vapours are removed. A substantial supply of liquid is maintained in this zone with the solid absorbent in suspension at a temp., approx. 326°, such that the residual product discharged therefrom has a Saybolt viscosity of > 100 at 99°. The refined viscous oil product is discharged after remaining in the vaporising and digesting zone for not less than 5 times as long as the raw absorbent stock mixture is in the heating zone.

H. S. GARLICK.

**Treatment of [hydrocarbon] oil.** D. G. BRANDT, ASSR. to DOHERTY RES. Co. (U.S.P. 1,784,087, 9.12.30. Appl., 31.1.22. Renewed 11.7.28).—Oil is pumped through a series of surface condensers, where it is heated to 204—232° by vapours passing in a countercurrent direction, to a dephlegmator maintained under approx. 100 lb. pressure, and thence into a cracking chamber in which is maintained a column of oil. As the mixture of incoming and residual oil passes downwardly through the cracking chamber, part is cracked and part vaporized, the vapours passing up through the oil into the dephlegmator. The residual oil is conducted to the lower drum of a cracking still in which a substantially fixed oil level is maintained and from which it passes rapidly through small tubes, located in a heating furnace, to a vapour-separating drum. The vapours are introduced into the lower portion of the cracking chamber and distributed through the oil therein by means of baffles and trays. As the oil is cracked and the lighter vapours are released, C and tarry matter is liberated and works downwardly over the baffles, finally accumulating in a chamber in the bottom of the cracking chamber, from which it may be drawn off.

H. S. GARLICK.

**Purification of hydrocarbon oils.** C. P. DUBBS, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,787,570, 6.1.31. Appl., 29.6.25. Renewed 5.10.28).—Sulphurous compounds are removed from hydrocarbon distillates by passing the latter through a zone containing aq. Ca(OH)<sub>2</sub>, wherein Ca(OH)·SH is formed as a reaction product. The distillate is passed to a second zone similar to the first and is then returned to the first zone, wherein CS<sub>2</sub> contained in the distillate will react with the above compound. The distillate is thereafter

treated in an independent zone with Ca(OH)<sub>2</sub> and H<sub>2</sub>O to remove H<sub>2</sub>S therefrom.

H. S. GARLICK.

**Sweetening of sulphurous petroleum.** H. H. CANNON and W. W. GARY, ASSTS. to CANNON-PRUTZMAN TREATING PROCESSES, LTD. (U.S.P. 1,789,168, 13.1.31. Appl., 21.1.29).—The oil is treated with a powdered solid comprising an alkaline-earth hydroxide, *e.g.*, Ca(OH)<sub>2</sub>, and PbO, intimately mixed together, to convert the mercaptans into Pb mercaptides, and thereafter the Pb is precipitated from the mercaptides by adding S to the oil.

H. S. GARLICK.

**Treatment of petroleum oils containing sulphur.** G. J. ZISER and J. H. OSMER, ASSTS. to STANDARD OIL COMPANY OF CALIFORNIA (U.S.P. 1,784,215, 9.12.30. Appl., 9.1.26).—The oil is brought into intimate contact with an aerated dil. aq. solution of an ammoniacal Cu salt.

H. S. GARLICK.

**Treatment of crude [petroleum] oil.** K. W. HICKEY (U.S.P. 1,789,233, 13.1.31. Appl., 11.1.29).—A solution of NaHCO<sub>3</sub> (6 lb.) and borax (1 lb.) in H<sub>2</sub>O (8 gals.) is added to crude oil (250 barrels) simultaneously with HCl (14 oz.), to produce a violent reaction which causes precipitation of foreign matter and H<sub>2</sub>O.

H. S. GARLICK.

**Gum inhibitor for hydrocarbons.** W. S. CALCOTT and I. E. LEE, ASSTS. to E. I. Du PONT DE NEMOURS & Co. (U.S.P. 1,789,302, 20.1.31. Appl., 23.8.29).—The use of an acyl derivative of an arylamine of the type R·NH·CO·R', where R is an aryl group which may contain alkyl or alkoxy-substituents and R' is an alkyl, aryl, or aralkyl group, is claimed. Acetanilide is specially mentioned.

H. S. GARLICK.

**Refining of lubricating oils.** J. C. BLACK and W. D. RIAL, ASSTS. to RICHFIELD OIL Co. OF CALIFORNIA (U.S.P. 1,787,505, 6.1.31. Appl., 27.4.27).—Lubricating oil distillate from the vac. distillation of asphalt-base crude petroleum oil is treated with aq. NaOH, under pressure > 1 atm. and at approx. 137°, in quantities sufficient to combine with the emulsifying constituents. The mixture is allowed to separate and the oil is treated with sufficient H<sub>2</sub>SO<sub>4</sub> (*d* 1.83) to combine with all the unsaturated hydrocarbons. After separating the acid sludge the oil is treated at above 93° with a solid absorbent earth in order to absorb all the sulphonic acid constituents. The lighter grades of lubricating oil stocks are separated from the heavier oils by distillation in vac. and are finally clarified and decolorised as desired.

H. S. GARLICK.

**Increasing the viscosity and improving the lubricating properties of oils.** W. FUNGS and E. FRESE, ASSTS. to STANDARD I.G. Co. (U.S.P. 1,787,951, 6.1.31. Appl., 13.6.28. Ger., 25.6.27).—The addition of small quantities (0.25—0.75%) of montan wax bleached by oxidation with CrO<sub>3</sub> or salts thereof in the presence of H<sub>2</sub>SO<sub>4</sub> or org. acids considerably increases the viscosity of mineral oils, whilst the addition of larger quantities gives products of the consistency of petroleum jelly.

H. S. GARLICK.

**Lubrication of textile machinery.** A. E. BECKER, ASSR. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,789,331, 20.1.31. Appl., 20.9.26).—A lubricant that is

easily washed out of the fabric being manufactured comprises a petroleum oil, preferably having a viscosity between 50 and 250 sec. Saybolt at 38°, containing 0.15—1.5% of H<sub>2</sub>O-sol. alkali-metal soap.

B. M. VENABLES.

**Stabilisation of mineral oils and greases, animal and vegetable oils, and fats and soaps.** R. T. VANDERBILT CO., INC., ASSEES. OF P. I. MURRELL (B.P. 353,874, 24.4.30. U.S., 4.3.30).—Small amounts of a phenoxide prepared from a phenylphenol and a base that is itself an antioxidant (*e.g.*, 4-hydroxydiphenyl and diphenylethylenediamine) are added.

E. LEWKOWITSCH.

**Manufacture of concentrated emulsions of oil and water.** C. E. OLIVER (U.S.P. 1,790,070, 27.1.31. Appl., 5.4.26).—Solutions of 10 lb. of rosin in 5 gals. of oil, and 10 lb. of casein in 10 gals. of H<sub>2</sub>O containing 1 lb. of NaOH, having approx. equal surface tension, are mixed together (1 : 1) to form a conc. emulsion, which may be diluted by the alternate addition of H<sub>2</sub>O and oil, preferably in the proportions of 1 : 3. H. S. GARLICK.

**Coke pushers, coke levellers, and door extractors for use in coke ovens.** WELLMAN SMITH OWEN ENG. CORP., LTD., and S. STOCKDALE (B.P. 353,879, 28.4.30).

**Pressure gas burners for furnaces.** F. WOLFF, and SELAS A.-G. (B.P. 354,072, 19.8.30).

**Gas-detecting lamps.** R. J. PLUMMER, and CEAG, LTD. (B.P. 353,454, 22.4.30).

**Combustion apparatus for liquid fuels.** T. K. A. NORDENSSON (B.P. 353,457, 23.4.30. Swed., 24.4.29).

**Sorting of coal etc. Mixing phenol compounds. Centrifuging salts. Pipe still. Distillation control. Dephlegmator for vapours. Carrying out catalytic reactions. Determining m.p. [of coal ash etc.].—See I. Impregnating timber.—See IX. Coke-oven effluents.—See XXIII.**

### III.—ORGANIC INTERMEDIATES.

**Synthesis of aliphatic alcohols from mixtures of carbon monoxide and hydrogen.** G. NATTA and M. STRADA (Giorn. Chim. Ind. Appl., 1931, 13, 317—325).—When heated under pressure in presence of compounds of Zn and of alkali metals, mixtures of CO and H<sub>2</sub> containing high proportions of the latter yield almost entirely mixtures of aliphatic alcohols in large part higher than MeOH. The use of catalysts free from elements of groups IV—VIII renders insignificant the production of harmful secondary reactions, especially the formation of CH<sub>4</sub>. At temp. not above 400—420° the formation in the catalysts of alkali salts of fatty acids as intermediate compounds is confirmed. Particularly high catalytic activity is shown by mixtures of ZnO and alkali salts of fatty acids. When reduced by H<sub>2</sub> in presence or absence of ZnO HCO<sub>2</sub>K gives MeOH almost quantitatively; KOAc gives EtOH (from 26.2% of the salt decomposed), Pr<sup>n</sup>OH, and COMe<sub>2</sub> (from 31.8%); PrCO<sub>2</sub>K gives *n*- and *iso*-PrOH. K<sub>2</sub>CO<sub>3</sub> is reducible to HCO<sub>2</sub>K by H<sub>2</sub> in presence of ZnO. Confirmation is thus furnished of the hypothesis that the primary product of these syntheses from CO and H<sub>2</sub> is MeOH, and that the higher alcohols are produced subsequently by interaction of MeOH and CO (in

presence of alkaline catalysts) to form firstly fatty acids (or their alkaline salts). These undergo either reduction or condensation (2 mols. of the acid), with elimination of CO<sub>2</sub>, to ketones or aldehydes, which are then hydrogenated to give alcohols. T. H. POPE.

**Analysis of mixtures of methyl and ethyl alcohols.** L. MARICQ (Bull. Soc. chim. Belg., 1931, 40, 333—337).—The total vol.-% of the alcohols is determined from the density, whilst conditions are described under which oxidation with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and H<sub>2</sub>SO<sub>4</sub> converts MeOH into CO<sub>2</sub> and H<sub>2</sub>O, and EtOH into AcOH. Combination of the results permits the determination of each alcohol. The process is recommended for the determination of MeOH in spirits. A. A. LEVI.

**Recovery of volatile solvents from the viewpoint of modern adsorption methods.** W. HERBERT (Chem.-Ztg., 1931, 55, 577—578, 595—597, 615—617).—The recovery of volatile solvents by condensation alone is usually inefficient and the fire risk is considerable. In the case of methods of recovery involving absorption in a less volatile solvent with, in many cases, formation of a mol. compound, the difficulty is rather in the economical recovery of the absorbed solvent. The Brégeat process, using cresols, is one of the best, but is only advantageous with solvents containing O, as with hydrocarbons etc. no mol. compound is formed. Washing processes are suitable for high and const. vapour concentrations. Capital costs are usually high for small-scale plants. The third class of methods include adsorption on activated C, SiO<sub>2</sub> gel, or Al(OH)<sub>3</sub> gel. The properties of these adsorbents are functions of their enormous surface, activated C having been prepared with a surface of 1250 sq. m./g. or above 30% of that of a sheet 1 mol. thick. Their use is particularly advantageous for the recovery of volatile solvents present only in traces. Vegetable charcoal is heated with ZnCl<sub>2</sub> to 500° or with steam and oxidising gases to 800—1000°. The Bayer adsorption process is described. The use of superheated steam for stripping has been abandoned as no appreciable benefit is obtained. The intermittent working of 2 units alternately is no disadvantage, and the process is now very widely used.

C. IRWIN.

**Volatility of cresols when heated on the water-bath.** N. J. STEKELENBURG (Pharm. Weekblad, 1931, 68, 719—724).—The loss of wt. of cresol, isolated from liquor cresoli saponatus by the Dutch Pharmacopœia method, during heating on the water-bath in Erlenmeyer flasks of various dimensions, has been determined. With short- and wide-necked flasks the loss may amount to 80% within 30 min., and even under good conditions to 2—5%.

H. F. GILLBE.

**Apparatus for experimental pyrolytic production of diphenyl.** A. W. HIXSON, L. T. WORK, H. V. ALESSANDRONI, G. E. CLIFFORD, and G. A. WILKENS (Ind. Eng. Chem. [Anal.], 1931, 3, 289—291).—The influence of temp., pressure, and rate of flow on the production of Ph·Ph when C<sub>6</sub>H<sub>6</sub> vapour is bubbled through a bath of molten Pb has been studied. The optimum temp. is about 740°. Pressures above 1 atm. favour the conversion into Ph·Ph. The % conversion decreases with increasing rate of flow, but less rapidly

under atm. than under higher pressures. The chemical efficiency of the process is high and may reach 98%. With the apparatus described, in which the vapour is bubbled through a column of Pb 17 in. high, the rate of production under atm. pressure and at moderate rates of flow was about 14 g./hr., whilst under higher pressures and with somewhat greater rates of flow the rate reached 50—75 g./hr. A. B. MANNING.

The indanthrone [alkali] melt. V. Effect of water. VI. Catalytic action of the isomeric cresols. T. MAKI (J. Soc. Chem. Ind. Japan, 1931, 34, 249—253 B, 253—257 B; cf. B., 1931, 105).—V. In the fusion of 2-aminoanthraquinone with KOH in absence of PhOH the yield of indanthrone first rises and then falls as the H<sub>2</sub>O present decreases, the max. (15.6%) being reached at 19% H<sub>2</sub>O; there is a continuous fall in alkali-sol. product (alizarin) and rise in PhNO<sub>2</sub>-sol. (2-aminoanthraquinone). In the presence of PhOH (42% of wt. of 2-aminoanthraquinone) similar effects are observed, but the yield is much higher (51.5% max. at 19% H<sub>2</sub>O). As the PhOH is increased in proportion to the 85% KOH, a max. yield (51.6%) is reached at 15% (60% of the 2-aminoanthraquinone) at the expense of the PhNO<sub>2</sub>-sol. product; excess of PhOH acts as a diluent.

VI. *o*- and *p*-Cresols have about equal effects, the max. yields being 51.5% and 48.5% with 17.5% of *o*- and *p*-cresol, respectively (on the 85% KOH), and 44.4% with 25% of *m*-cresol; the amount of alkali-sol. product increases with the proportion of cresols more rapidly than with PhOH. C. HOLLINS.

Analysis of gaseous hydrocarbons.—See II. Syntheses under pressure.—See VII. Glycerin analysis.—See XII. Trinitronaphthalene.—See XXII.

#### PATENTS.

Synthesis of oxygenated aliphatic compounds [from carbon monoxide and steam]. SOC. CHIM. DE LA GRANDE PAROISSE AZOTE ET PROD. CHIM. (B.P. 349,193, 18.3.30. Fr., 28.1.30).—Mixed catalysts containing a rare-earth (except Ce) metal or compound, at least one alkali or alkaline-earth compound, and at least one Cu, Mn, Zn, Cd, Pb, V, Cr, Mo, Sb, Bi, B, Al, W, Si, Ti, Zr, or Th metal or compound, are used, with or without Ce, for conversion of CO and H<sub>2</sub>O into aliphatic oxygenated compounds below C<sub>7</sub>. Commercial Ce(CO<sub>3</sub>)<sub>2</sub> assaying 60% Ce, 30% Di, is dissolved with ZnCO<sub>3</sub> and Ba(OH)<sub>2</sub> in AcOH, Al(OAc)<sub>3</sub> is added, and after addition of NaOH the ppt. is washed with Ba(OH)<sub>2</sub> solution and dried at 100°. This catalyst gives 40% conversion at 350° with a space velocity of 8000. C. HOLLINS.

Production of vinyl halides [from acetylene and hydrogen halides]. J. P. BAXTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 349,017, 12.2.30).—The active C catalyst is activated by preheating with the hydrogen halide at higher temp. (200—600°) than that used for reaction (100—300°). The catalyst may similarly be reactivated. C. HOLLINS.

[Preservation of] oxidisable organic compounds. GOODYEAR TIRE & RUBBER Co., Assees. of A. M. CLIFFORD (B.P. 352,652, 24.5.30. U.S., 27.8.29).—Oxidisable

substances such as rubber, transformer oils, and aldehydes are preserved by incorporating the reaction product of an aromatic amine, a naphthol, and CH<sub>2</sub>O. The reaction products yielded by aniline, *o*-toluidine, and β-naphthylamine with β-naphthol are cryst. and have m.p. 132—133°, 185—186°, and 187—189°, respectively. Their formula is C<sub>10</sub>H<sub>6</sub>(OH)·CH<sub>2</sub>·NHR, where R is an aromatic radical. D. F. TWISS.

Temp. control. Refrigerants.—See I.

#### IV.—DYESTUFFS.

##### PATENTS.

Manufacture of [acid] wool dyes. I. G. FARB-ENIND. A.-G. (B.P. 348,902, 30.5.30. Ger., 17.8.29).—A 2:6-diamino-derivative of a sulphonated secondary amine of the C<sub>6</sub>H<sub>6</sub> series is condensed with a 2:4:6-trinitrobenzene containing a replaceable group (Cl, OMe) in position 1; the diphenylamine first formed loses NaNO<sub>2</sub> on prolonged heating in aq. NaOH, giving yellow-brown to violet-brown acid dyes. Examples of starting materials are: 2:6-diaminodiphenylamine-4-sulphonic acid with 2:4:6-trinitroanisole; 2:6-diamino-*n*-butylaniline-4-sulphonic acid with 1-chloro-2:4:6-trinitrobenzene; 4-chloro-2:6-diaminodiphenylamine-3'-sulphonic acid or 4-(2':6'-diamino-4'-sulpho-anilino)antipyryne with 2:4:6-trinitroanisole. C. HOLLINS.

Manufacture and application of stable diazo preparations. SOC. CHEM. IND. IN BASLE (B.P. 349,339 17.6.30. Switz., 17.6.29).—The diazo compound is precipitated as arylsulphonate in presence of a salt of a metal of group II. Examples are: diazo compounds from 2:5-dichloroaniline or *o*-anisidine with Na 2:7-naphthalenedisulphonate and MgCl<sub>2</sub> or CaCl<sub>2</sub>; *o*-chloroaniline with Na 1:5-naphthalenedisulphonate and MgCl<sub>2</sub>. The products may be used in dyeing with ice colours. C. HOLLINS.

Diazo-type layers.—See XXI.

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

Yolk in New Zealand wool. I. Effect of sheep covers on yolk production. W. G. SUTTON (J. Text. Inst., 1931, 22, τ 365—369).—The production of yolk (wool grease) by the skin is not significantly affected by protecting the sheep from rain etc. by means of light waterproof covers. A method for determining the constituents of greasy wool, which involves extraction with both Et<sub>2</sub>O and H<sub>2</sub>O, is described. B. P. RIDGE.

Sulphur content of some New Zealand wools. D. J. SIDEX (J. Text. Inst., 1931, 22, τ 370—373; cf. A., 1931, 1081).—In the few but strictly comparable cases examined, better spinning wool was higher in S than lower-class wool, and the S content was also in inverse proportion to the coeff. of variation and the contour figure. It therefore appears that there is a correlation between the spinning properties and the S content of a wool. B. P. RIDGE.

Determination of mixtures of textile fibres. P. KRAIS and MARKERT (Rev. Gén. Mat. Col., 1931, 35, 281—285).—The material containing the mixed fibres is treated with a conc. solution of Ca(SCN)<sub>2</sub> for

1 hr. at 70°, whereby viscose-, cuprammonium-, acetate-, and nitro-cellulose rayons and natural silk are quantitatively dissolved, whilst raw or mercerized cotton and wool suffer a loss of wt. of only 2–4%. Thorough stirring during treatment is necessary, and the insol. residue is collected by filtration through a metal sieve, washed, dried, and weighed. Good agreement is obtained between the theoretical values for mixtures of known composition and those found experimentally. When used in conjunction with other known procedures, the method permits the analysis of mixtures containing more than two different fibres.

B. P. RIDGE.

**Chemistry of wood. III. I. Determination of lignin.** L. E. WISE and A. M. FAIRBROTHER. **II. Preparation of lignin from wood.** W. M. HARLOW and L. E. WISE (Ind. Eng. Chem. [Anal.], 1931, 3, 253–255).—I. The U.S. Forest Products Laboratory method, involving hydrolysis of the wood with 72% H<sub>2</sub>SO<sub>4</sub>, gives a uniformly higher lignin fraction from hard woods than does the Ross-Hill procedure using CH<sub>2</sub>O and conc. H<sub>2</sub>SO<sub>4</sub>, but if the material is previously extracted with hot H<sub>2</sub>O and EtOH-C<sub>6</sub>H<sub>6</sub> the results are in approx. agreement. For coniferous woods the Ross-Hill method gives the higher values. This method is, however, the more rapid and convenient.

II. When the procedures described above are followed, both methods give the same yield of lignin from the same sample of wood.

B. P. RIDGE.

**Action of dilute sodium hydrogen carbonate solution on "oxycelluloses" and denitrated cellulose nitrate.** LENZE, METZ, and RUBENS (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 24–26; Chem. Zentr., 1931, i, 2412–2413).—The air-dried material (25 g.) was shaken for 15 hr. with 750 c.c. of 5% aq. NaHCO<sub>3</sub>, the washed residue being 3 times similarly treated. The Cu no. of the product was not that characteristic of cellulose.

A. A. ELDRIDGE.

**Cellulose formate.** G. TOCCO and A. NYSSSENS (Giorn. Chim. Ind. Appl., 1931, 13, 325–330).—Esterification of cellulose by means of HCO<sub>2</sub>H in presence of a dehydrating catalyst under good conditions always yields a cellulose diformate which remains in solution and may be separated in the same way as the acetate. Cellulose diformate is highly stable in dry air, but in ordinary air it begins to decompose after some weeks; decomp. is greatly retarded by immersion in H<sub>2</sub>O or other neutral liquid. Cross and Bevan's hypothesis that cellulose diformate is the mono-substituted ester of a cellulose previously modified by acid is not confirmed. On evaporation of its solution in HCO<sub>2</sub>H, a residue of pure cellulose remains. This behaviour is characteristic of the compound and serves for obtaining a pure cellulose pellicule by evaporation. The solvent properties of H<sub>3</sub>PO<sub>4</sub>, ZnCl<sub>2</sub>, HCO<sub>2</sub>H, readily-sol. thiocyanates, pyridine, etc. for the compound are confirmed. The behaviour of the more important solutions, especially of those in thiocyanates (which follows a cycle of ripening similar to that of viscose) and pyridine, is described.

T. H. POPE.

**Acid values of cellulose fatty acid esters, and rapid analysis of certain cellulose acetates.** T. F. MURRAY, C. J. STAUD, and H. LE B. GRAY (Ind. Eng.

Chem. [Anal.], 1931, 3, 269–273).—Of the more important methods available for the determination of the acetyl value of acetylated cellulose, that due to Eberstadt is considered the most satisfactory, although it requires a long time for the complete saponification of the material. Accurate results for cellulose triacetate are obtained only after a pretreatment with 75% EtOH. A more rapid modification of this method involves dissolution of the ester in pyridine before saponification with NaOH, and the time is then shortened to about 1 hr. This procedure can be used for materials containing 35.8–44.8% Ac, but the ester must be rendered easily sol. in pyridine by previous dissolution in COMe<sub>2</sub> and reprecipitation. The results agree with those given by the slower method. Methods are described for the determination of HCO<sub>2</sub>H and AcOH in the presence of each other and of one or more acids not volatile in steam.

B. P. RIDGE.

**Relation between chemical properties of cellulose nitrate and its suitability for use as a lacquer.** LENZE, METZ, and RUBENS (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 15–22; Chem. Zentr., 1931, i, 2401).—Heating with H<sub>2</sub>O under pressure has a greater action on the cellulose mol. of the product than has treatment with NH<sub>3</sub>, but gives better lacquers.

A. A. ELDRIDGE.

**Pine oil in the cellulose industry.** W. FERMAZIN (Chem.-Ztg., 1931, 55, 613).—Pine oil—used up to the present chiefly for the manufacture of terpeneol—consists principally of the oxidation products of turpentine and has b.p. 208–210°. A sample contained 54% of  $\alpha$ -terpineol, the rest being terpin hydrate, fenchyl alcohol, terpenes, and camphor. A 0.5% addition of pine oil to the size used for an acetate silk yarn improved the strength considerably. A similar effect was produced by an addition of 0.3% to the spinning bath, this proportion being an optimum. Pine oil is a good solvent for nitrocellulose and lacquers prepared with its use are of high quality.

C. IRWIN.

**Oxycellulose.**—See VI. **Stability of cellulose nitrate.**—See XXII.

PATENTS.

**Dry disintegration and deposition of fibres.** F. W. MANNING, Assr. to FILTER FABRICS, INC. (U.S.P. 1,786,669, 30.13.30. Appl., 23.4.28).—Filtering media, e.g., cellulose fibres, asbestos, decolorising C, are deposited in a more or less dry condition on a fabric support from a current of air or other elastic fluid. Thus scrim passes round a cylindrical suction mould revolving at the upper end of a chamber up which the filtering medium is blown by a current of air, deposition being therefore against the force of gravity with the result that only the finely-divided material reaches the fabric. By inserting vertical partitions in the deposition chamber it is possible to apply successive layers of different filtering media. After leaving the suction mould the filter fabric is wound on a suction drum so that steam or air carrying atomised oil may be sucked through the material to promote felting.

D. J. NORMAN.

**Manufacture of rubber-impregnated fibrous material.** G. A. RICHTER and R. B. HILL, Assrs. to BROWN Co. (U.S.P. 1,787,952, 6.1.31. Appl., 27.11.28).

—Absorbent paper is impregnated with an aq. rubber dispersion, *e.g.*, latex, dried, and passed through a bath containing a rubber solvent to swell the rubber. The material is then rolled and heated to volatilise the solvent. Vulcanising agents and accelerators may be present in the solvent bath. The resulting product is less  $H_2O$ -absorbent and has a higher dry and wet strength than before treatment with the solvent.

D. J. NORMAN.

**Making cellulose acetate.** KODAK, LTD. From EASTMAN KODAK Co. (B.P. 351,118, 2.4.30).—Cellulose acetate is obtained in a fluffy form by including in the acetylation bath an inert,  $H_2O$ -immiscible, volatile solvent and subsequently precipitating the cellulose acetate in  $H_2O$  at or above the b.p. of the volatile solvent. The quantity of AcOH in the acetylation bath should be insufficient of itself to dissolve the cellulose acetate formed, complete dissolution being effected by adjusting the proportion of volatile solvent present. Suitable solvents are  $C_2H_4Cl_2$ ,  $CHCl_3$ , and particularly  $C_2H_4Cl_2$ .

D. J. NORMAN.

**Acetylation of nitrocellulose.** H. A. GARDNER and C. A. KNAUSS, Assrs. to H. A. GARDNER LABORATORY, INC. (U.S.P. 1,786,989, 30.12.30. Appl., 12.1.28).—Acetylnitrocelluloses containing 9.6–4.8% N and 20.6–41.2% OAc are obtained by acetylating nitrocellulose containing 1 or more unsubstituted OH groups and about 11% N. The acetylated product is then precipitated with  $C_2H_4Cl_2$  and the excess AcOH recovered by fractional distillation. Acetylnitrocelluloses so obtained are sol. in  $CO_2$  and most of the usual solvents, give solutions of low viscosity and good film-forming properties, and are of low flammability.

D. J. NORMAN.

**Manufacture of nitrocellulose.** A. HOUGH, J. R. DUFFORD, and W. C. LEONHARD (U.S.P. 1,785,030, 16.12.30. Appl., 31.8.26).—Parchment paper, preferably made from  $\alpha$ -pulp, is comminuted and nitrated at 40° with, *e.g.*, an acid mixture containing  $H_2SO_4$  60%,  $HNO_3$  21–22%, and  $H_2O$  18–19%. The nitrated product is then washed, boiled with  $H_2O$  for about 3 hr., and then boiled with successive quantities of very dil. aq.  $NH_3$  until no further yellow colour develops. The product may be finally bleached with alkaline NaOCl solution.

D. J. NORMAN.

**Nitration of cellulose.** L. A. PINCK (U.S.P. 1,784,945, 16.12.30. Appl., 27.6.28).—Nitrating acid is made by passing N oxides, *e.g.*,  $NO_2$  and  $N_2O_4$  as obtained in  $N_2$  fixation and similar processes, into  $H_2SO_4$  until the mol. ratio  $N_2O_4:H_2SO_4$  is 1:1.25–3.0. When the N oxides are practically free from  $HNO_3$  the concentration of the  $H_2SO_4$  may be 95–90%, but when about 15% of the  $N_2O_4$  is replaced by its equiv. of  $HNO_3$  the concentration of the  $H_2SO_4$  may be 95–85%, the presence of  $HNO_3$  serving to increase the degree of nitration at the same time permitting the use of a less conc.  $H_2SO_4$  without any deleterious effect on the cellulose or the nitrated product. Graphs are given showing the degree of nitration obtained with various acid mixtures. Sufficient nitrating acid should be used to give 15 pts. of  $N_2O_4$  per 1 pt. of cellulose, and a reaction time of 4–5 hr. at 25–30° is suitable. D. J. NORMAN.

### [Production of] pyroxylin compound sheets.

FIBERLOID CORP., Asses, of R. G. O'KANE and E. R. DERBY (B.P. 353,045, 9.4.30. U.S., 12.2.30).—Sheets with a smooth, sheeny surface are obtained by extruding a mass of pyroxylin gel containing a solvent through an orifice while under tension, removing part of the solvent by a blast of hot air, and finally while still in a plastic condition subjecting it to progressive transverse stretching.

F. R. ENNOS.

**Manufacture of mixed esters of cellulose.** C. S. WEBBER and C. J. STAUD, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,785,466, 16.12.30. Appl., 2.8.29).—Cellulose triacetate or the hydrolysed acetates containing <44.8% Ac are treated with aromatic org. acids having an ionisation const. above  $1.82 \times 10^{-5}$ , *e.g.*, *o*-, *m*-, or *p*-toluic acids, benzoic, mandelic, and cinnamic acids. The reaction may take place in any suitable medium, *e.g.*,  $H_2O$ , 1:4-dioxan, propionic acid,  $C_2H_4Cl_2$ , at 100–150°, and may be continued until a mixed ester of the desired solubility up to a  $H_2O$ -sol. product is obtained. The prep. of cellulose acetosalicylate (m.p. 248–271°) and acetomandelate is described.

D. J. NORMAN.

### Pigmentation of organic cellulose esters and ethers.

L. CLEMENT and C. RIVIÈRE (B.P. 350,924, 11.2.30).—The preformed pigment and the cellulose derivative are powdered and stirred together in a liquid medium comprising a solvent for the cellulose derivative in admixture with an equal vol. of  $H_2O$ . After a few hr. a large proportion of the pigment becomes fixed on the cellulose derivative and the latter is then filtered off, washed, and dried. The resulting product gives films in which the pigment grains are invisible. Metal powders may be introduced by this process.

D. J. NORMAN.

### Decolorisation of cellulose derivatives.

BRIT. CELANESE, LTD. (B.P. 353,075, 23.4.30. U.S., 13.5.29).—The derivatives are treated, either in suspension or in solution, with a solution of polybasic aliphatic acids or their salts (oxalic, citric, tartaric, etc.).

F. R. ENNOS.

### Manufacture of transparent or translucent films or foil.

A. H. CHVERTON, and F.M., LTD. (B.P. 353,136, 15.5.30).—A mixture of fusel and gallipoli oils is applied by means of rollers to the "casting" surface (drum or band) before deposition of the film material (cellulose acetate solution), whereby a uniform film which is readily stripped off is obtained.

F. R. ENNOS.

### Production of artificial filaments or threads.

BRIT. CELANESE, LTD., H. DREYFUS, E. KINSELLA, J. BOWER, and W. I. TAYLOR (B.P. 352,922, 1.1.30. Addn. to B.P. 300,998, 304,674, and 326,232; B., 1929, 203, 353; 1930, 456).—Solutions of org. derivatives of cellulose are dry-spun into an evaporative medium, the whole or a definite part of which is made to pass through the immediate vicinity of the spinning orifices solely by means of the suction applied to withdraw it from the spinning cell, whilst additional evaporative medium is also introduced into the neighbourhood of the spinning orifices.

F. R. ENNOS.

**Treatment [saccharification] of cellulosic material.** H. DREYFUS (B.P. 353,028, 17.4.30).—Wood

etc., after purification with alkali and pretreatment with mineral and/or org. acid, is esterified with an org. acid anhydride in presence of  $H_2SO_4$ ,  $H_3PO_4$ , or other mineral acid, and afterwards hydrolysed, one or more stages of the treatment being so conducted, *e.g.*, by raising the temp. of reaction, as to degrade the cellulosic material to sugars. F. R. ENNOS.

**Manufacture of artificial horsehair, strips, and ribbons.** G. B. ELLIS. FROM SOC. POUR LA FABR. DE LA SOIE "RHODIASETA" (B.P. 352,414, 7.4.30).—A large number of artificial filaments after spinning are assembled into the desired form and welded together by passing through a solvent or swelling agent, which may contain non-solvent liquids or solids in solution or emulsion (gums, resins, etc.), cellulosic material sol. in the solvent, or colouring matter, at such a speed that they are softened only at the surface. Pigments may also be incorporated in the filaments, if desired, before or after agglomeration. F. R. ENNOS.

**Production of hollow filaments from solutions of cellulose derivatives.** CELLULOSE ACETATE SILK Co., LTD., and H. C. CURTIS (B.P. 353,041, 19.3.30).—Solutions of cellulose derivatives are dry-spun through dies having conical holes which taper to a greater diam. on the outside face of the die, thus: metal thickness of dies 0.4–0.5 mm., holes 0.05–0.07 mm. inside and 0.09–0.11 mm. outside diam. F. R. ENNOS.

**Device for the stretch-spinning [with flowing precipitating liquor] of artificial silk.** BRIT. BEMBERG, LTD. (B.P. 352,862, 6.11.30. Ger., 7.11.29).—Placed inside the cylindrical spinning vessel and ending in its outlet tube is a smooth conical insertion having several vertically arranged series of apertures, which are staggered with respect to one another and serve to equalise sharp variations in concentration of the precipitating liquor. F. R. ENNOS.

**Manufacture of cellulosic filaments having a structure and properties comparable with those of natural wool.** M. J. DASSONVILLE (B.P. 352,692, 18.6.30. Fr., 26.6.29).—By periodic variations in the supply or pressure of the cellulosic solution passing into the spinneret, a noded filament is produced which, immediately after initial solidification, is waved or crimped. Suitable apparatus is described. F. R. ENNOS.

**Manufacture of artificial threads and filaments [of reduced lustre] and products therefrom.** COURTAULDS, LTD., and C. DIAMOND, with, in (c), W. H. GLOVER (B.P. 352,412, 352,610, and 352,611, [A] 7.4.30, [B, C] 5.5.30. [A, B] Addns. to B.P. 338,269; B., 1931, 110).—Solutions of an org. ester of cellulose containing a small proportion of (A) a monohydric aliphatic ester of a higher fatty acid, *e.g.*, amyl stearate, (B) an org. acid ester of a saccharide sol. in org. solvents, *e.g.*, glucose penta-acetate, or (C) either (A) or (B) together with a small proportion of  $TiO_2$ , are dry-spun and treated with aq. soap solution at or near the b.p. F. R. ENNOS.

**Treatment of artificial threads and films made from cellulose esters and ethers.** K. WEISSENBERG and B. RABINOWITSCH (B.P. 352,445, 8.1.30. Ger., 18.2.29).—Finished artificial threads, yarns, or films or cellulose esters or ethers which have been treated

with a neutral org. swelling agent, *e.g.*, 50% aq. dioxan in the case of cellulose acetate, or threads of cellulose acetate spun from solutions containing dioxan, are gradually stretched 200% or more while still in contact with the swelling medium. Previous to, during, or immediately after these operations the threads may be subjected to transverse pressure, *e.g.*, by rolling, and they are finally treated with salt solutions and washed with  $H_2O$ . F. R. ENNOS.

**Devices for treating thread coils or similar annular masses of spun filaments of artificial silk.** M. SCHOENFELD (B.P. 352,771, 7.8.30. Ger., 9.8.29).—The coils etc. are mounted inside a container around an expanding column, which secures them, and after-treatment liquids and gases are passed by suction or pressure through the windings. F. R. ENNOS.

**Processing of raw cellulosic material.** G. A. RICHTER, AssT. to BROWN Co. (U.S.P. 1,787,953—4, 6.1.31. Appl., 24.9.29).—(A) Pulp rich in  $\alpha$ -cellulose is obtained by digesting cellulosic material with a liquor which is initially acid and is subsequently made alkaline during the cook. Thus spruce chips are digested with  $H_2SO_3$  (5% free  $SO_2$ ) for about 2 hr. at  $120^\circ$  (2 hr. being allowed to reach this temp.), after which the  $SO_2$  is removed by raising the temp. rapidly and opening the relief valve, the pressure being gradually lowered meantime to atm. pressure. The residual  $SO_2$  is then neutralised and the cook completed at about  $168^\circ$  for 3 hr. in an alkaline liquor containing 24–40 g.  $Na_2O$  per litre, 40% of which should be present as  $Na_2S$ . (B) The  $SO_2$  remaining in the digester after the acid cook is oxidised to  $SO_3$  by introducing the requisite quantity of, *e.g.*,  $NaOCl$ ,  $Na_2O_2$ , etc. in order to avoid the formation of thiosulphates on adding an alkaline liquor containing  $Na_2S$ . D. J. NORMAN.

**Preparation of cellulose pulp.** R. B. WOLF, R. P. HILL, and R. S. HATCH (U.S.P. 1,788,555, 13.1.31. Appl., 8.2.29).—Lignocelluloses, *e.g.*, wood chips, are treated with anhyd. liquid  $SO_2$  or a non-aq. solution thereof at  $15$ – $38^\circ$  and  $100$ – $125$  lb./sq. in. for a sufficient time (2–8 hr.) for the  $SO_2$  in conjunction with the moisture in the wood to convert the lignin into lignin-sulphonic acids. The liquid  $SO_2$  is then withdrawn under pressure and the cellulose freed from lignin-sulphonic acids by treatment with  $H_2O$  or alkalis. Under these conditions degradation of the cellulose is avoided, high yields are obtained, and the lignin-sulphonic acids are recovered in an unpolymerised form. D. J. NORMAN.

**Manufacture of chemical pulp or cellulose.** D. E. CABLE, AssT. to OXFORD PAPER Co. (U.S.P. 1,790,260, 27.1.31. Appl., 27.10.27).— $N_2$  or other inert non-oxidising gas which is not absorbed to any great extent by the digestion liquor is used to supply pressure in the digester at the beginning of the cook and to maintain a suitable pressure throughout the digestion period. Pressure-boosting with  $N_2$  is particularly useful in sulphite cooking, especially when air-dry (10%  $H_2O$ ) chips are used, as it facilitates penetration of the chips, reduces the total cooking time, and improves the bleaching qualities of the pulp. The amount of  $O_2$  present in the digester may be reduced to a min. by exhausting

the air from the charged digester and admitting  $N_2$  before introducing the cooking liquor.  $N_2$  of sufficient purity is obtained from the stack at the outlet of the acid-making plant.

D. J. NORMAN.

**Pulp-filtering machine.** E. C. LOETSCHER (U.S.P. 1,787,047, 30.12.30. Appl., 12.9.29).—The stock is fed on to a travelling wire cloth as in the ordinary paper machine, sheet formation being assisted by inclining the wire upwards from the feed box end, providing water sprays about half way up the machine, and applying suction to the underside of the wire for practically the whole of its length. The wire must be suitably reinforced to withstand the suction. D. J. NORMAN.

**Deriving high-grade cellulose from cottonseed hulls.** B. DORNER (U.S.P. 1,789,354, 20.1.31. Appl., 26.10.29).—The cleaned and washed hulls are completely dried at 103–105° to make them brittle and are then humidified just sufficiently long for the cotton fibres to regain their elasticity without any appreciable absorption of moisture by the hull itself. The material is then immediately milled and the cotton fibres are separated from the powdered hull by an air blast. The hulls are more easily powdered if they are soaked in a solution of a metal salt, e.g., 6–8% NaCl solution, before drying.

D. J. NORMAN.

**Manufacture of cellulose and textile fibres from vegetable matter.** C. A. BRAUN (U.S.P. 1,786,890, 30.12.30. Appl., 15.8.27. Fr., 4.7.27).—The action of alkali monosulphite liquors containing free caustic alkali on cellulosic material is considerably accelerated if there is added to the liquor alkali salts of weak acids, e.g., soaps, silicates, borates, aluminates, etc., which by dissociation at high temps. furnish free alkali for dissolving the incrustants and weak acid, the latter, in turn, liberating  $SO_2$  from the monosulphite. A small proportion of  $Na_2CO_3$  is preferably added to maintain a faintly alkaline reaction to the end of the cook. A suitable liquor for 1000 kg. of straw contains, per 2000 litres,  $Na_2SO_3$  85 kg.,  $Na_2SiO_3$  35 kg., NaOH 20 kg., and  $Na_2CO_3$  5 kg. With this liquor a boiling time at 160° of 4 hr. is sufficient as against 5.5–6 hr. with a liquor containing 35 kg. of NaOH and no silicate.

D. J. NORMAN.

**Production of high  $\alpha$ -cellulose fibre.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,790,170, 27.1.31. Appl., 6.5.25. Renewed 23.7.27).—The continuous recovery of heat and soda as applied to a plant producing  $\alpha$ -pulp by digestion of sulphite pulp with alkali, running in conjunction with plant producing kraft pulp, is described.

D. J. NORMAN.

**Preparation of parchment paper stock from corn-stalk pith.** E. R. DARLING, Assr. to CORNSTALK PRODUCTS Co., INC. (U.S.P. 1,790,002, 27.1.31. Appl., 19.10.28).—The cleaned pith is digested with  $H_2O$  under heat and pressure, screened to remove fibrous material, bleached, and beaten to the required degree of hydration at about 57° in an ordinary beater.

D. J. NORMAN.

**Manufacture of coloured and tinted parchment paper and the like.** R. I. COWEN (U.S.P. 1,790,367, 27.1.31. Appl., 6.3.28).—Dyes which are insol. or but slightly sol. in 70%  $H_2SO_4$ , but are sol. in  $H_2O$ , e.g.,

Erie Fast Scarlet (Colour Index No. 326), are suspended in the parchmentsing acid so that they become fixed in the paper during parchmentsing. The full colour of the dye develops during the washing operation.

D. J. NORMAN.

**Production of impregnated paper and the like.** CALLENDER'S CABLE & CONSTRUCTION Co., LTD., and H. HILL (B.P. 352,533, 10.4.30).—The impregnation of paper in reel form is facilitated by embossing nodules in the paper before reeling, to keep the layers slightly apart.

D. J. NORMAN.

**Manufacture of paper and paper board.** L. M. BOOTH (U.S.P. 1,788,790, 13.1.31. Appl., 5.10.28).—When using old rags and old papers in the furnish, decomp. of the material is arrested and the products of decomp. are precipitated in the pulp as fillers by adding hypochlorite and hydrated  $CaO$ , e.g., 2–5 lb. of  $Ca(OCl)_2$  and 1–3 lb. of  $Ca(OH)_2$  per ton of pulp, to the beater.

D. J. NORMAN.

**Manufacture of cigarette paper.** B. SEFERIADIS, and S. C. PATENTS, LTD. (B.P. 352,541, 11.4.30. Addn. to B.P. 325,125; B., 1930, 414).—The resinols etc. used in the prior process are obtainable in varying quantities from any gum or gum resin, natural or synthetic, by maceration with  $CO_2$ , EtOH, liquid hydrocarbons, or mixtures thereof. In making the paper it is advantageous to add 1.5–2% of EtOH to the pulp before running on the machine.

D. J. NORMAN.

**Stencil sheets or stencil paper.** W. W. TRIGGS, From DITTO, INC. (B.P. 352,460, 8.4.30).—Yoshino paper is coated with a solution of an unpolymerised phenol resin (Baekeland's product B) in admixture with shortening agents, e.g., metal soaps, waxes,  $BaSO_4$ ,  $MgCO_3$ , etc., and softeners, e.g., oils, or mixtures of neatfoot oil with butyl phthalate or hydrogenated cottonseed oil, and the synthetic resin is subsequently polymerised (to product C) by heat.

D. J. NORMAN.

**Apparatus for treating [rubbing etc.] fibrous material.** I. TUDOR, and O. TUDOR-HART (B.P. 353,424, 17.1.30).

**Machines for spinning artificial silk.** C. HAMEL A.-G., and E. HAMEL (B.P. 353,803, 5.11.30).

**Filter blocks.**—See I. Lubricant for textiles.—See II. Mineral fibres.—See VII. Artificial wood.—See IX. Paper for roll films. Light-sensitive paper.—See XXI.

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

**Determination of oxycellulose in bleached cotton.** H. HEINRICH (Textilber., 1931, 12, 113–114).—1 g. of the air-dry bleached cotton is boiled with 20 c.c. of 0.6% NaOH for 1 hr. (the vol. of the alkaline liquor is maintained const. by additions of  $H_2O$ ), the liquor is diluted to 50 c.c., and the colour of the extract compared with that of extracts obtained similarly from bleached cottons having known oxycellulose contents. Bleached cotton if satisfactory yields a clear very pale yellow extract, overbleached cotton brown. A. J. HALL.

**Lecithin in the textile industry.** O. MECHEELS (Textilber., 1931, 12, 123–124).—Lecithin (from soya beans) when used in the treatment of textile fibres

should be in the form of a neutral or preferably alkaline emulsion; such emulsions are stable in  $H_2O$  up to  $15^\circ$  hardness. Such emulsions may serve as lubricants for cotton and wool in spinning and as finishing agents in softening the handle of artificial silk materials. In finishing bleached cotton goods a bleached lecithin should be used. Increased depth of shade is obtained by the addition of lecithin to printing pastes, and brighter and more level dyeings (even with vat dyes) are produced by adding lecithin to the dye liquor.

A. J. HALL.

**Modern bleaching, dyeing, printing, and finishing machinery.** P. URMSTON (J. Soc. Dyers and Col., 1931, 47, 215—221).

**Textile sizes.**—See XVII.

## PATENTS.

[Crêping of] textile materials. BRIT. CELANESE, LTD., and W. A. DICKIE (B.P. 351,999 and 352,000, 24.12.29).—Crêpe fabrics are obtained by impregnating with a swelling agent which becomes effective on dilution with  $H_2O$  (A) fabrics woven with tightly-twisted yarns or (B) tightly-twisted yarns which are afterwards woven into fabrics, and then scouring or otherwise treating with aq. liquors. Suitable swelling agents for cellulosic materials include conc. solutions of  $ZnCl_2$  and  $Ca(CNS)_2$ ; org. swelling agents such as  $COMe_2$ ,  $AcOH$ , and  $EtOH$  suitably diluted with  $C_6H_6$  are satisfactory for yarns composed of cellulose esters and ethers, particularly cellulose acetate.

A. J. HALL.

[Crêping of] textile materials. H. DREYFUS (B.P. 352,451, 5.3.30).—Yarns of artificial silk, cotton, silk, etc., which have been sized with a polymerised vinyl compound or an artificial resin together with a softening agent (olive oil), are tightly twisted and incorporated in a fabric; this is then scoured in an aq. soap bath or with a swelling agent for the twisted threads or for the size ( $EtOH$ , xylene dispersed in a sulphonated oil, etc.) without rendering the latter relatively soft or yielding. (Cf. B.P. 348,589; B., 1931, 716.)

F. R. ENNOS.

[Weighting of] animal fibres. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 353,033, 20.1.30).—Wool etc. is treated with an aq. solution of at least 10% of its wt. of an org., non-dyeing, halogenated compound having a mol. wt. above 200 and an affinity for the fibres, e.g., 2:3:6-trichloroanilinesulphonic acid, tetrachlorophthalic acid, chlorinated fatty acids, sulphonic acids of chlorinated paraffin waxes, etc.

F. R. ENNOS.

**Apparatus for treating artificial silk with liquids.** H. A. GILL. From GLANZSTOFF-COURTAULDS G.M.B.H. (B.P. 354,008, 19.6.30).

**Filter press.**—See I. **Stable diazo preps.**—See IV. **Tinted parchment.**—See V. **Coloured rubber.**—See XIV. **Colour photography.**—See XXI. **Insecticide.**—See XXIII.

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

**Nitration process of sulphuric acid manufacture.** M. MATSUI (Proc. World Eng. Cong., 1929, 31, 265—288).—A comprehensive summary of previous

work of the author (cf. B., 1923, 265; 1930, 238, 321, 903).

C. IRWIN.

**Titration of hydrofluoric acid containing hydrofluosilicic acid.** K. KOBAYASHI (Proc. World Eng. Cong., 1929, 31, 399—407).—About 1 g. of the sample is weighed in a covered Pt crucible containing 5 c.c. of distilled  $H_2O$  and poured into a large Pt dish, 10—15 c.c. of saturated aq.  $KNO_3$  are added, and the solution is titrated with  $N-NaOH$  solution and phenol-red until the colour does not fade in 5 sec.; it is then warmed to  $90^\circ$  and titrated to a permanent pink colour. The first figure gives  $HF + H_2SO_4 + \frac{2}{3}H_2SiF_6$ , the second the total acid. This method obviates the use of ice, which is necessary with phenolphthalein. The limit of accuracy is that of the sampling.

C. IRWIN.

**Liquefaction of gases and syntheses under pressure. Synthesis of ammonia and methyl alcohol.** R. E. JORDAN (Proc. World Eng. Cong., 1929, 31, 139—152).—The Claude  $NH_3$  process is described. If 5—8%  $CO$  is left in the  $H_2$  derived from coke-oven gas and the mixture compressed to 1000 atm. a preliminary catalysis converts almost the whole of the  $CO$  into  $MeOH$ . The remaining compressed gases are then available for  $NH_3$  synthesis. By varying the catalyst higher alcohols or hydrocarbons may be obtained in place of  $MeOH$ . The first fraction on liquefaction of coke-oven gas consists chiefly of propylene, and has no present application. The second consists of  $C_2H_4$  and  $C_2H_6$  and forms 1.5—2.0% of the gas. The  $C_2H_4$  is used for the prep. of  $EtOH$ ; it is also possible to prepare instead  $Et_2O$  or  $EtOAc$  using the Ca salt. The synthesis of petrol from  $C_2H_4$  is possible, but not economic at present prices. That of glycol is more attractive if cheap  $Cl_2$  is available. The  $C_2H_6$  may be converted into  $C_2H_4$  with a yield of 80% by controlled oxidation with air. The  $CH_4$  may be decomposed into the elements at  $1150^\circ$  and the C produced is suitable for printing ink. With  $H_2O$  vapour at  $1034^\circ$  and in presence of Cu the reaction  $CH_4 + H_2O = CO + 3H_2$  occurs. The  $CH_4$  may also be chlorinated, or used for the production of cyanides or of  $CH_2O$ .

C. IRWIN.

**Oxometer for the determination of hydrogen peroxide etc.** CHEM. FABR. PYRGOS G.M.B.H. (Chem.-Ztg., 1931, 55, 540).—The apparatus comprises a stoppered cylinder with a lower mark to which the solution to be tested is filled, and a scale graduated in 0.25% steps to 5% starting some distance above the lower mark. The vol. between the latter and the zero of the scale is filled with 50%  $H_2SO_4$  and the solution is then titrated with a  $KMnO_4$  solution supplied with the cylinder, the vol. of the latter used giving directly the %  $H_2O_2$  from the scale reading. The apparatus may also be used for  $Na_2O_2$  and Na perborate.

A. R. POWELL.

**Superphosphate enriched with ammonia.** S. I. VOLFKOVICH, L. E. BERLIN, I. L. HOFMAN, and A. A. IONAS (Udobr. Urozhai, 1930, 2, 556—569).—The preparation of ammoniated superphosphate from raw phosphate (Portland and Saratov),  $H_2SO_4$ , and  $(NH_4)_3PO_4$  is described.

CHEMICAL ABSTRACTS.

**Chemical constitution of bleaching powder.** S. URANO (Proc. World Eng. Cong., 1929, 31, 171—180).



—A mixture of  $\text{Ca}(\text{OCl})_2$  and  $\text{CaCl}_2$  loses the whole of its Cl under action of  $\text{CO}_2$  or of heat alone. The loss of Cl by the action of  $\text{CO}_2$  on bleaching powder is therefore no evidence as to the composition of the latter. Thermochemical tests show that bleaching powder in solution behaves in this respect as a mixture of  $\text{Ca}(\text{OCl})_2$  and  $\text{CaCl}_2$ . Solid bleaching powder, however, is a compound of the two with a heat of formation of 2800 g.-cal. per mol. of  $\text{CaOCl}_2$ . A study of the system  $\text{Ca}(\text{OCl})_2\text{—CaCl}_2\text{—H}_2\text{O}$  showed that a triple point exists at  $48^\circ$  above which no double salt can exist. Between  $48^\circ$  and  $-50^\circ$  double salt can exist in equilibrium with either single salt, and below  $-50^\circ$  it can exist alone. The v.p. of  $\text{Ca}(\text{OCl})_2$  and  $\text{CaOCl}_2$  coincide at about  $47^\circ$ . The reactions between  $\text{CaO}$  and  $\text{Cl}_2$  are therefore (1)  $\text{Ca}(\text{OH})_2 + \text{Cl}_2 = \text{CaOCl}_2 \cdot \text{H}_2\text{O} + 16,500 \text{ g.-cal.}$ ; (2)  $2\text{Ca}(\text{OH})_2 + 2\text{Cl}_2 = \text{Ca}(\text{OCl})_2 + \text{CaCl}_2 \cdot 2\text{H}_2\text{O} + 27,400 \text{ g.-cal.}$  The partial pressures of  $\text{Cl}_2$  calc. by the Nernst formula from the two equations become equal at  $45.3^\circ$ , which is therefore the transition point. Bleaching powder is a double salt with some  $\text{H}_2\text{O}$  of crystallisation. C. IRWIN.

**Utilisation of Korean alunite.** H. TANAKA (Proc. World Eng. Cong., 1929, 31, 289—290).—Alunite  $3(\text{Al}_2\text{O}_3 \cdot \text{SO}_3) \cdot \text{K}_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  is calcined at  $600^\circ$  and treated with hot aq.  $\text{NH}_3$  solution. A mixed solution of  $(\text{NH}_4)_2\text{SO}_4$  and  $\text{K}_2\text{SO}_4$  is obtained and evaporated to crystallisation. The residue, together with 20—40 pts.  $\text{H}_2\text{O}$ , is treated at  $60\text{—}70^\circ$  for 3—5 hr. with  $\text{SO}_2$ .  $\text{Al}_2\text{O}_3$  goes into solution,  $\text{SiO}_2$  remaining undissolved. The solution is heated at  $100^\circ$ , when basic sulphite is precipitated. This is filter-pressed and calcined at above  $900^\circ$ , yielding a pure  $\text{Al}_2\text{O}_3$  almost free from  $\text{Fe}_2\text{O}_3$ . C. IRWIN.

**Determination of the hydrolytic acidity of fuller's earth and its decolorising power.** H. UTERMÖHLEN (Chem.-Ztg., 1931, 55, 625—626).—The hydrolytic acidity of various samples of fuller's earth was determined by the  $\text{NaOAc}$  method, as with soil samples; the results agreed well with those of actual bleaching tests. This quantity is, in the case of a soil, a measure of the absorptive power to OH and Na ions, and the action of fuller's earth on oils is held to be analogous. C. IRWIN.

**Reports of investigations: [Canadian] non-metallic minerals.** R. K. CARNOCHAN and R. A. ROGERS (Canad. Dept. Mines, Repts. Ore Invest., 1929, 159—175).—Experiments designed to improve the quality of asbestos,  $\text{SiO}_2$  sand for glass making, a uraninite ore, mica, and gypsum are recorded. The methods used involved selective crushing, table dressing, and magnetic separation. A. R. POWELL.

**Manufacture of lead suboxide and some of its industrial applications.** G. SHIMADZU (Proc. World Eng. Cong., 1929, 31, 325—344).—Pb lumps about 1 in. diam. are fed automatically into a gas-tight drum with hollow trunnions on which it revolves. Through the trunnion at the feed end passes a compressed-air pipe having a number of branches. The speed of the drum is so arranged that the Pb is in const. motion. Temp. rise occurs owing to friction, but chiefly to the formation of  $\text{Pb}_2\text{O}$ ; the optimum wall temp. is

$140\text{—}150^\circ$ . The exit air passes through bag filters. Air control regulates the temp.  $\text{Pb}_2\text{O}$  as prepared by this process contains 95%  $\text{Pb}_2\text{O}$  and 5% Pb. It is a very finely-divided black powder which is gradually oxidised by air to PbO. It burns rapidly to the same product when lit with a match or agitated with compressed air. PbO can therefore be manufactured without the use of fuel. The rapidly oxidised product contains a little  $\text{Pb}_3\text{O}_4$  and is converted into the latter by igniting at  $400^\circ$  under oxidising conditions.  $\text{Pb}_2\text{O}$  with boiled linseed oil gives a paint of high value, and the PbO and  $\text{Pb}_3\text{O}_4$  prepared from it are exceptionally pure. C. IRWIN.

**Manufacture of oxygen, nitrogen, and the rare gases.** R. E. JORDAN (Proc. World Eng. Cong., 1929, 31, 131—138).—The Claude liquid air process starting with cooled air at 20 atm. divides it into two portions; one is liquefied, the other cooled, by expansion while performing work. The liquid air is fractionated first in a high-pressure column with bottom feed, and secondly in a low-pressure column. The use of Kuhni plates containing spirally wound ribbons of Cu, one of which is flat and one corrugated, further increases fractionating efficiency. The gaseous residue after washing with liquid N contains 37.5% Ne, 12.5% He, 50% N. The Ne and  $\text{N}_2$  are absorbed by coconut charcoal, from which the Ne is recovered by regulated heating. He which is not absorbed is obtained directly. A has b.p.  $-186^\circ$ , and a plate in the column can be found containing up to 4% A in liquid O. This liquid is further rectified, giving an 80% A gas which is freed from  $\text{O}_2$  by burning  $\text{H}_2$ . The Kr and Xe accumulate in the O vaporiser residues. If these are burned with  $\text{H}_2$ , 6000 litres of mixed gas yield 9 litres of Kr and 0.8 litre of Xe. They are absorbed in  $\text{SiO}_2$  gel cooled with liquid N. Kr is liberated by warming to air temp., Xe at  $200^\circ$ . They are purified by redistillation at the temp. of liquid air. C. IRWIN.

**Extraction of [pure] krypton and xenon from liquid air residues.** F. J. ALLEN and R. B. MOORE (J. Amer. Chem. Soc., 1931, 53, 2512—2522; cf. A., 1930, 1508, 989; 1928, 1209).—The compressed liquid air residues are condensed in charcoal at liquid air temp. and then boiled off slowly and passed over hot Cu and Ca, thus avoiding the use of large gas holders. Details of the fractionation are given. J. G. A. GRIFFITHS.

**$\text{CaF}_2$  for furnaces.**—See XI. **Nitroglycerin waste acids.**—See XXII. **Determination of As.**—See XXIII.

#### PATENTS.

**Manufacture of sulphuric acid by the lead-chamber or by the tower process.** E. ROTHAMMEL (B.P. 348,142, 5.2.30).—A portion of the gases from the Glover tower is withdrawn by a suction fan and forced with an injector action into the conduit supplying roaster gases to the system. One or more denitrators followed by one or more nitrators are arranged in front of the Glover tower and nitrosulphuric acid from the Gay-Lussac tower is led through the nitrators, and a portion of the  $\text{H}_2\text{SO}_4$  from these is returned in a closed circuit to the top of the nitrators. In the apparatus succeeding the Glover tower  $\text{H}_2\text{SO}_4$  ( $d$  1.7)

containing  $N_2O_3$  is sprayed under such conditions that the gases flowing through the first section take up  $N_2O_3$  from the acid and give up the gas again to the acid in the second section. Various other modifications of the process are indicated. A. R. POWELL.

**Preparation of phosphoric anhydride and phosphoric acid.** METALLGES. A.-G. (B.P. 352,367, 29.11.30. Ger., 29.11.29).—Molten P is atomised by allowing it to fall on a rotating disc together with a stream of  $H_2O$  while a current of cold air passes upwards round the disc to cool it. Combustion of the P- $H_2O$  spray affords  $H_3PO_4$  in any desired degree of concentration by adjusting the ratio of P :  $H_2O$ . A. R. POWELL.

**Production of sodium carbonate liquor.** H. W. MORSE (U.S.P. 1,791,281, 3.2.31. Appl., 29.4.29).—The mixture of  $Na_2CO_3$ ,  $Na_2SO_4$ , and NaCl obtained from the mother-liquors in the extraction of  $Na_2B_4O_7$  and K salts from Searles Lake brine is leached with small quantities of  $H_2O$  at 30–40°, whereby a solution containing 12%  $Na_2CO_3$  is obtained. A. R. POWELL.

**Production of potassium nitrate, sodium bicarbonate, and compound fertilisers.** SOC. CHIM. DE LA GRANDE PAROISSE AZOTE ET PROD. CHIM. (B.P. 352,307, 4.9.30. Fr., 15.11.29).—In the manufacture of  $KNO_3$  by the interaction of  $NaNO_3$  with KCl, sylvinit is used as the source of KCl, and after the greater part of the  $KNO_3$  has been recovered by crystallisation the mother-liquor is treated with  $NH_3$  and  $CO_2$  to ppt.  $NaHCO_3$ , leaving  $NH_4Cl$  and the remaining  $KNO_3$  in solution; evaporation of this liquor yields a valuable mixed fertiliser. A. R. POWELL.

**Treatment of phosphate material.** ODDA SMELTEVERK A./S., and E. JOHNSON (B.P. 353,160, 27.5.30. Nor., 30.5.29).—After phosphate rock has been decomposed with  $HNO_3$  and  $Ca(NO_3)_2$  crystallised out, the mother-liquor is treated with  $NH_3$  or  $(NH_4)_2CO_3$  under heat and pressure, if desired,  $Ca_3(PO_4)_2$  or a mixture of  $Ca_3(PO_4)_2$  and  $CaHPO_4$  being precipitated and dissolved in  $HNO_3$  together with more phosphate rock. The mother-liquor may be freed from  $HNO_3$  by distillation before precipitation, and the precipitated phosphates may be treated with HCl to remove part of the Ca before dissolving it. The solution of  $NH_4$  salts is evaporated and the product mixed with K salts. W. J. WRIGHT.

**Manufacture of ammonium phosphates.** UNION CHIM. BELGE, SOC. ANON. (B.P. 352,309, 5.9.30. Fr., 7.11.29).—Dry cryst.  $(NH_4)_3PO_4$  is treated with the theoretical quantity of  $H_3PO_4$  containing 50%  $P_2O_5$ , conc.  $HNO_3$ , conc. HCl, or conc.  $H_2SO_4$  to convert the salt into  $(NH_4)_2HPO_4$  or  $NH_4H_2PO_4$  and the mixture is dried. A. R. POWELL.

**Production of ammonium phosphate.** KUNSTDÜNGER-PATENT-VERWERTUNGS A.-G. (B.P. 352,814, 19.9.30. Swed., 5.10.29).—Phosphate rock is leached with  $H_2SO_4$ , insol. matter is removed, and the clear solution neutralised with  $NH_3$ , mono- or di- $NH_4$  phosphate being obtained by recrystallisation. The mother-liquor is utilised in the leaching process, its  $(NH_4)_2SO_4$  content being available instead of a corresponding amount of  $H_2SO_4$ . W. J. WRIGHT.

**Manufacture of triammonium phosphate.** E. URBAIN (B.P. 352,725, 4.7.30. Ger., 8.5.30).— $H_3PO_4$  is passed into the upper part of an absorption tower into which sufficient  $NH_3$  is introduced to give a solution of mono- and di- $NH_4$  phosphates, which is continuously returned to the  $H_3PO_4$  container. The solution is then treated with excess of  $NH_3$  in a cooled mixer, dry cryst.  $(NH_4)_3PO_4 \cdot 5H_2O$  being thus obtained. W. J. WRIGHT.

**Removal of calcium chloride liquor adhering to calcium hypochlorite.** I. G. FARBEININD. A.-G. (B.P. 354,093, 11.9.30. Ger., 17.9.29).— $Ca(OCl)_2$  crystals, after centrifuging or pressing, are stirred with a wt. of anhyd.  $Na_2SO_4$  equiv. to the  $CaCl_2$  in the adhering mother-liquor. L. A. COLES.

**Preparation of calcium cyanamide.** N. CARO and A. R. FRANK (B.P. 352,264, 14.7.30. Ger., 18.12.29).—Finely-powdered  $CaC_2$  at least 10% of which passes 75-mesh, is compressed, rolled, or shaped, with the addition, if desired, of suitable solid or liquid agglomerants, into pieces of any size or shape, and these pieces are subjected to the action of  $N_2$  under pressure at a suitable temp. to form  $CaCN_2$ . A. R. POWELL.

**Manufacture of chromium oxide.** J. E. DEMANT (B.P. 353,152, 22.5.30).—Cr or an alloy containing it, e.g., ferrochrome, is roasted in presence of  $O_2$ , or gases containing or evolving  $O_2$ . Fe and Si are removed from the product by treatment with acids or alkalis, or the product may be heated at a high temp. in a reducing atm. to reduce the Fe. W. J. WRIGHT.

**Production of asbestos and mineral artificial fibres.** W. LÜDKE (B.P. 352,681, 13.6.30).—Solutions of sol. silicates, e.g., water-glass, to which are added salts that do not cause immediate coagulation, e.g.,  $NaAlO_2$ , are spun into threads of silicate jelly in suitable coagulating agents, e.g., org. liquids or hot gases. These threads are stabilised by heating with  $H_2O$  or a solution of an electrolyte, e.g.,  $MgCl_2$ ,  $CaCl_2$ ,  $KOH$ , HCl, under pressure. D. K. MOORE.

**Depuration of waste water containing cyanide.** OESTERR.-ALPINE MONTANGES. (B.P. 353,184, 16.6.30. Austr., 24.6.29).—The cyanide is decomposed by bringing the waste water, without previous heating, in counter-current to kiln or other waste gases containing  $CO_2$ , in an irrigation plant, the hydrocyanic acid being suitably extracted from the waste gases. W. J. WRIGHT.

**[Preparation of] substances containing sulphur in a chemically active condition.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of B. W. NORDLANDER (B.P. 353,970, 14.5.30. U.S., 14.5.29).—A mixture (preferably 1:1) of bentonite and  $SeS_2$  is heated at 125–150°, cooled, and pulverised; the product may be used as a vulcanising agent or in the manufacture of therapeutic products, fungicides, skin pastes, etc. L. A. COLES.

**Production of aqueous dispersions or solutions of iodine.** D. MCKIE and A. BENNETT (B.P. 353,892, 21.3.30).—Powdered I is stirred with a dil. aq. solution of a simple  $NH_2$ -compound, decomposed by halogens in the presence of  $H_2O$ , preferably with boiling 0.5–4.0% aq.  $NH_3$ , under conditions such that simultaneous or subsequent oxidation can occur, e.g., in the presence of air or  $O_2$ . L. A. COLES.

Recovering crystals from liquors. Centrifuging salts. Liquefaction of gases. Carrying out catalytic reactions.—See I. Flux for Al etc.—See X. Electrolysis of aq. solutions.—See XI. Fertilisers. Manures.—See XVI.

### VIII.—GLASS; CERAMICS.

Flow tests in small glass tanks. A. L. MARDEN (J. Soc. Glass Tech., 1931, 15, 119—128 T).—The flow through a small glass tank giving rise to cordy glass was confined to a narrow section between bottom and top stagnant layers, in almost a direct line from the charging pot to the outlet. On forcing the glass down to the bottom, the results were more satisfactory if the surface was skimmed occasionally. Flow lines on other furnaces feeding machine blowers are described. C. A. KING.

Investigation of the flow of glass in tank furnaces by the use of barium oxide. E. J. C. BOWMAKER and J. D. CAUWOOD (J. Soc. Glass Tech., 1931, 15, 128—140 T).—The addition of  $\text{BaCO}_3$  to a  $\text{Na}_2\text{O}$ - $\text{CaO}$ - $\text{SiO}_2$  glass for tracing the flow of glass does not sensibly affect a normal working, neither does it provide a quant. measure of distribution complementary to the qual. method of adding  $\text{Co}_2\text{O}_3$ .  $\text{BaO}$  was first observed at the machines in 9—10 hr., which indicates a fast-moving current in the tank, and the actual max. of 20% of the theoretical max. suggests powerful mixing currents. Probable lines of flow are discussed and the results show that the position of a machine is a more important factor than its "pull" in relation to the rate of flow. C. A. KING.

Clay mixtures for glass-melting pots. III. Flow at high temperatures of pot-clay refractories under stress. J. H. PARTRIDGE and G. F. ADAMS (J. Soc. Glass Tech., 1931, 15, 190—208 T; cf. B., 1931, 805).—Under a const. stress, a higher content of  $\text{Al}_2\text{O}_3$  (20—36%) increased the temp. of failure. All specimens containing grog were less resistant, and particularly so when coarser grog was used. Rapid tensile tests proved to be misleading as under prolonged creep tests the more silicious clays withstood the more severe conditions of stress and temp. At temps. as low as  $1300^\circ$  aluminous clays flowed continuously, but silicious clay attained a const. dimension; the former is therefore recommended only for short-period heatings. C. A. KING.

Use of ammonium sulphate as an accelerating agent in glass melting. M. PARKIN, W. J. A. WARREN, and W. E. S. TURNER (J. Soc. Glass Tech., 1931, 15, 153—166 T).—Both the melting and refining processes of glasses containing  $\text{Na}_2\text{O}$  13.1—16.8% and  $\text{CaO}$  8.5—13.8% were accelerated by the addition of up to 10 pts. of  $(\text{NH}_4)_2\text{SO}_4$  per 1000 pts. of sand. The  $(\text{NH}_4)_2\text{SO}_4$  appears to undergo some reaction with the Na salts and to leave residual sulphates accompanied by the escape of  $\text{NH}_3$ , which probably causes a stirring effect.  $(\text{NH}_4)_2\text{SO}_4$  is considered a more effective accelerator than  $\text{Na}_2\text{SO}_4$ . C. A. KING.

Use of ammonium sulphate in glass making. A. E. J. VICKERS [with G. H. WHITING, R. MILLER, and V. BATES] (J. Soc. Glass Tech., 1931, 15, 166—172 T).—Preliminary trials showed that about 0.7% of  $(\text{NH}_4)_2\text{SO}_4$

in a glass batch may increase output to a machine and give a longer working range with a reduced working temp. Glass of more brilliant colour and increased fluidity was made with rapid elimination of seed, and a possible reduction in Na content would produce a more durable glass. C. A. KING.

Influence of manganese oxide on some properties of glass. A. A. CHILDS, V. DIMBLEBY, F. WINKS, and W. E. S. TURNER (J. Soc. Glass Tech., 1931, 15, 172—184 T).—In a parent glass of composition  $\text{SiO}_2$  72%,  $\text{CaO}$  10%,  $\text{Na}_2\text{O}$  18%, the  $\text{Na}_2\text{O}$  was progressively replaced by  $\text{MnO}_2$ . The melting rate was increased distinctly by  $\text{Fe}_2\text{O}_3$  ( $\text{FeO}$ ) up to 2%, but not by  $\text{MnO}$ , which, however, greatly reduced the refining period, though  $>0.5\%$  had no further effect. Fluidity was increased with 2.5—3%  $\text{MnO}$  without further effect up to 5%, but  $\text{MnO}$  was not so effective as  $\text{FeO}$  in this respect. Although glass with 2.5—3%  $\text{MnO}$  is opaque, considerable light is transmitted if 2%  $\text{FeO}$  is present and opacity is not reached with 5%  $\text{MnO}$  + 2%  $\text{FeO}$ . The thermal expansion diminished with increasing  $\text{MnO}$ , but the resistance to corrosion by alkali increased. C. A. KING.

Constitution of glass based on the laws of solutions. O. KNAPP (J. Soc. Glass Tech., 1931, 15, 140—152 T).—The mol. ratio of the oxides does not indicate the properties of a glass, which is considered as a genuine solution of silicates in which, generally, free  $\text{SiO}_2$  is dissolved. The optical consts. and the compositions of glasses indicated according to the highest silicate hypothesis give constitutive rules, and expressions are worked out for the weathering of  $\text{CaO}$  and  $\text{Pb}$  glasses and the tarnishing of the latter for both mono- and di-silicised forms. C. A. KING.

Density of sodium metasilicate-silica glasses. F. WINKS and W. E. S. TURNER (J. Soc. Glass Tech., 1931, 15, 185—190 T; cf. B., 1930, 767).—The densities of  $\text{Na}_2\text{O}$ - $\text{SiO}_2$  glasses containing 50—92%  $\text{SiO}_2$  were determined and compared with the calc. values from known formulæ. An error of  $<0.02$  was obtained by using Winkelmann and Schott's formula if the factors 3.38 for  $\text{Na}_2\text{O}$  and 2.20 for  $\text{SiO}_2$  were applied for glasses containing up to 35%  $\text{Na}_2\text{O}$ . Two formulæ by the Bureau of Standards gave closer values for 50—80%  $\text{SiO}_2$  glasses, but failed with glasses containing  $>85\%$   $\text{SiO}_2$ . C. A. KING.

Heat-absorbing glass. A. F. DUFFON (J. Soc. Glass Tech., 1931, 15, 208—212 T).—The criterion of efficiency hitherto adopted for heat-absorbing glass does not take into account that the glass becomes warm and so transmits heat to the atm. A modified empirical transmission factor  $F = T + (I - R - T) / \{2 + (I + E) / K\}$ , in which  $I$  is the transmission for the solar radiation,  $E$  the emissivity,  $K$  the conductivity, and  $R$  the reflecting power for solar radiation, enables windows of different kinds of glass to be graded. Double-glazing with heat-absorbing glass and ordinary glass which is opaque to radiant heat reduces the heat communicated to a room. C. A. KING.

Transparency of glasses to ultra-violet rays. P. GILARD, P. SWINGS, and A. HANTOT (Bull. Acad. roy. Belg., 1931, [v], 17, 593—602).—Experiments

with industrial glasses having the general composition 5 or 6(SiO<sub>2</sub>,B<sub>2</sub>O<sub>3</sub>),MO,M<sub>2</sub>O, or near, confirm, in general, the previous conclusions (A., 1931, 546) regarding the influence of different ingredients. Fe, Mn, La, and Ce oxides reduce the transparency, whilst Pr and Nd oxides exert little effect. The reduction in the transparency caused by increasing the thickness of the specimen, and by previously irradiating it with a strong beam of ultra-violet light, has been studied.

N. H. HARTSHORNE.

**Hydrolytic stability of chemical glassware.** F. H. ZSCHACKE (Chem.-Ztg., 1931, 55, 580—581).—Standardisation by the boiling H<sub>2</sub>O test is recommended.

C. IRWIN.

**Static rigidity of plastic clays.** C. TERZAGHI (J. Rheology, 1931, 2, 253—260).—Plastic flow is preceded by a gliding flow, having its origin in the highly viscous film of H<sub>2</sub>O surrounding the points of adhesion. The force available for plastic flow is equal to the difference between the total stress and the shearing resistance ("static rigidity"). E. S. HEDGES.

**Canadian non-metallic minerals [SiO<sub>2</sub> sand].**—See VII. **Boundary fields [and glass tubing].**—See X.

#### PATENTS.

**Lehrs for annealing glassware.** BRIT. HARTFORD-FAIRMONT SYND., LTD. From HARTFORD EMPIRE CO. (B.P. 353,245, 19.7.30).—The lehr is of the type in which a metal tunnel is surrounded by an insulated metal casing, the tunnel being supported by heat-insulating blocks which allow of relative and expansional movement.

C. A. KING.

**[Fortifying] treatment of [frosted] glass articles.** R. D. SMITH, Assr. to CORNING GLASS WORKS (U.S.P. 1,791,066, 3.2.31. Appl., 30.4.30).—The articles are washed in an aq. solution containing not more than 750 pts. per 10<sup>6</sup> of a simple Al salt of an aliphatic org. acid, e.g., Al tartrate (cf. U.S.P. 1,772,965; B., 1931, 395).

L. A. COLES.

**Drying of clay ware or kindred products.** W. W. TRIGGS. From HARROP CERAMIC SERVICE Co. (B.P. 352,381, 30.12.29).—The dryer is of the continuous tunnel type. The hot gas enters at the discharge end and by means of baffles is made to flow tortuously for about half the length of the tunnel. It then enters a chamber above the tunnel, passes through a number of ports along the length of the remainder of the tunnel into the tunnel, and is withdrawn at outlet ports opposite to the inlet ports, to be recirculated in the inlet part of the dryer.

D. K. MOORE.

**Production of material for moulding and firing, from raw material containing lime.** B. JIROTKA (B.P. 352,665, 29.5.30. Ger., 29.5.29).—Raw clay pressed into a bar is fed on to a rapidly rotating grinding device and the ground material is thrown off into moulds. Foreign matter, e.g., stones, is thrown off at different velocity and thus separated.

D. K. MOORE.

**Manufacture of porcelain.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of L. E. BARRINGER (B.P. 352,763, 1.8.30. U.S., 10.8.29).—Porcelain made from china clay, ball clay, felspar, and pyrophyllite is less

abrasive and has greater mechanical and dielectric strength than has ordinary porcelain. D. K. MOORE.

**Manufacture of porcelain for use in cooking.** G. FUSCHI and M. KORACH (B.P. 352,723, 3.7.30).—To eliminate the fracture of porcelain owing to the difference of coeffs. of expansion of the mass and the glaze, the moulded articles instead of being glazed are polished before and after burning. Such porcelain may be fired in piles if a layer of non-fusible substance is placed between the articles.

D. K. MOORE.

**Manufacture of refractory products from magnesium silicates.** METALLGES. A.-G., and V. M. GOLDSCHMIDT (B.P. 352,881, 7.7.30. Cf. B.P. 340,487; B., 1931, 350).—Granular Mg silicates the vol. of which remains const. or nearly so on being fired are used in the place of the orthosilicates mentioned previously.

D. K. MOORE.

**[Synthetic] moulded articles such as grinding wheels.** BAKELITE CORP., Assees. of R. S. DANIELS (B.P. 353,019, 17.4.30. U.S., 19.4.29).—A composition for moulding abrasive articles contains 3—10 pts. of abrasive grains and 1 pt. of a phenolic resinoid binder including a finely-divided ingredient (5—35%).

C. A. KING.

**Manufacture of [thin-walled] glass articles.** MACBETH-EVANS GLASS Co., Assees. of T. H. SLOAN (B.P. 353,770, 26.9.30. U.S., 28.9.29).

**Electric glass furnaces.**—See XI.

## IX.—BUILDING MATERIALS.

**Prediction of cement durability.** E. T. CARLSON and P. H. BATES (Eng. News-Rec., 1931, 107, 130—132).—A check study of Merriman's method (B., 1930, 191) of titrating a suspension of cement in sugar solution for CaO confirms to some extent the previous results, but the method is subject to inaccuracy owing to lack of sharpness of the end-point and the time factor. No correlation with actual durability has been proved and the method is not regarded as a reliable accelerated test.

C. A. KING.

**Synthesis of calcium aluminates and their hydration.** VIII. S. NAGAI and R. NAITO (J. Soc. Chem. Ind. Japan, 1931, 34, 159—161).—The strongest cement of the celite type is 4CaO,Al<sub>2</sub>O<sub>3</sub>,Fe<sub>2</sub>O<sub>3</sub> followed by 6CaO,Al<sub>2</sub>O<sub>3</sub>,2Fe<sub>2</sub>O<sub>3</sub>. These cements consist of the ferrite 2CaO,Fe<sub>2</sub>O<sub>3</sub> and the aluminate 2CaO,Al<sub>2</sub>O<sub>3</sub> when the original material has been heated above 1200°.

A. R. POWELL.

**Determination of depth of penetration of mercuric chloride in impregnation of round wood.** M. DREIFUSS and A. STAAB (Chem.-Ztg., 1931, 55, 497—498).—A cylindrical section is cut out of the wood (e.g., a fir mast) and split with a wooden wedge; the split surface is then treated with (NH<sub>4</sub>)<sub>2</sub>S, which causes blackening where the HgCl<sub>2</sub> solution has penetrated. For the quant. determination 100 g. of wood are destructively distilled in an Fe retort, the tarry matter is removed from the distillate with COMe<sub>2</sub>, and the Hg dissolved in HNO<sub>3</sub> and precipitated with H<sub>2</sub>S. The HgS is collected, washed free from HNO<sub>3</sub>, and shaken with CS<sub>2</sub> and I-KI solution and the excess I is titrated with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

A. R. POWELL.

**Trass and sand as additions to cement.** R. GRÜN (Chem. Erde, 1931, 6, 402—415).—A continuation of previous work (B., 1930, 558). Tests were also made with Rhine sand and powdered basalt.

L. J. SPENCER.

**Effect of extractives on the strength of wood.** R. F. LUXFORD (J. Agric. Res., 1931, 42, 801—826).—In certain woods, the extractive contents of which were artificially varied, the compression strength parallel to the grain, the bending strength, and, to a small extent, the shock-resistance increased with the extractive content.

A. G. POLLARD.

**Heat-absorbing glass.**—See VIII. Pig-Fe [slag].—See X.

#### PATENTS.

**Treatment of concrete or the like.** M. GIESE and F. HELE (B.P. 352,676, 6.6.30. Ger., 11.7.29).—The adhesiveness and fluidity of concrete may be improved by submitting it to the action of high-speed rotary beaters.

D. K. MOORE.

**Manufacture of bricks.** A. PAUL, JUN. (U.S.P. 1,791,372, 3.2.31. Appl., 29.5.29).—A mixture of 10—15 pts. of CaO with 90—85 pts. of volcanic ash,  $\frac{1}{10}$ — $\frac{3}{4}$  in. diam., is treated with H<sub>2</sub>O in a closed vessel to retain liberated steam, and the product, after moulding into bricks, is treated with steam under pressure.

L. A. COLES.

**Making coloured granules [for building purposes].** FISKE & Co., INC., and R. L. ATKINSON (B.P. 352,789, 27.8.30. U.S., 13.1.30).—Crushed and screened shale is impregnated with a solution of an inorg. salt to give the required colour and fired in a rotary kiln.

D. K. MOORE.

**Manufacture of artificial wood.** A. DE SAMSONOV (B.P. 353,186, 16.6.30).—Vegetable materials, e.g., reeds, cane trash, etc., having the fibres arranged in one direction, are immersed in a natural or artificial resin and compressed. Fungicides may be incorporated.

C. A. KING.

**Impregnation of timber.** PEASE & PARTNERS, LTD., and G. W. WILLIAMS (B.P. 352,992, 10.4.30).—Timber is impregnated by circulating creosote oil around the wood at above 100° and allowing the oil to cool below 100° after moisture has been expelled. Creosote oil from benzol scrubbers may be used after the benzol-recovery process, and, after cooling, is returned to the scrubbers.

C. A. KING.

**Preservation of wood.** H. BAKER (B.P. 352,650, 23.5. and 2.10.30).—An aq. solution of K<sub>3</sub>Fe(CN)<sub>6</sub> or K<sub>4</sub>Fe(CN)<sub>6</sub>, with or without the addition of crude EtOH, is used as the preservative. The addition of tannic acid renders the wood more resistant to fire and more impervious to salt water.

D. K. MOORE.

**Tubular furnace. Drying plant for roads etc.**—See I. Asphalt emulsions.—See II. Al coating [for wood].—See X. Insecticide.—See XXIII.

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

**Stationary and tilting open-hearth furnaces with mixed gas firing.** H. BANSEN (Stahl u. Eisen, 1931, 51, 989—995).—The effect of varying H<sub>2</sub>O content in the

gas mixture (blast-furnace and coke-oven gas) on the economics of the open-hearth steel process has been investigated over a prolonged period, and the results are reproduced in tables and graphs, which show the effect of varying furnace construction on the operation of the process and the necessity for close control of the gas analysis for the economical running of the furnace.

A. R. POWELL.

**Smelting [iron ores] in the Saar with coke from small chamber ovens.** E. BERTRAM (Stahl u. Eisen, 1931, 51, 883—885).—Coke produced from Saar coal in small chambers 20 in. wide, fired at 1300° for half the usual period, is almost as good as regards size and crushing strength as that produced by the usual process. The new coke gives equally good results in smelting minette, provided that its ignition temp. does not fall below a certain min. value and provided also that the ore and coke are approx. of the same size.

A. R. POWELL.

**Fine structure of various preheated phosphate slags and its relation to the citric acid solubility.** H. SCHNEIDERHÖHN (Arch. Eisenhüttenw., 1931—2, 5, 9—15; cf. B., 1929, 358).—Addition of fluorspar to basic slags in the Thomas converter results in conversion of the citric acid-sol. silico-carnotite into insol. fluorapatite and  $\beta$ -Ca<sub>2</sub>SiO<sub>4</sub> and the proportion of sol. P<sub>2</sub>O<sub>5</sub> falls from the normal (90—95%) to 3—4% with only 1% F in a slag containing about 20% P<sub>2</sub>O<sub>5</sub>. Basic open-hearth slag contains part of the P<sub>2</sub>O<sub>5</sub> as silicocarnotite and part as two other phosphates of unknown composition both of which are relatively insol. in citric acid; addition of CaF<sub>2</sub> to this slag converts all three P<sub>2</sub>O<sub>5</sub> compounds into fluorapatite. Max. solubility of the P<sub>2</sub>O<sub>5</sub> is obtained with the mol. ratio 5CaO : 1P<sub>2</sub>O<sub>5</sub> : 1SiO<sub>2</sub>; a slight excess of SiO<sub>2</sub> does no harm, but with a deficiency of SiO<sub>2</sub> part of the P<sub>2</sub>O<sub>5</sub> is converted into less sol. Ca tetraphosphate and with an excess of CaO the solubility is reduced.

A. R. POWELL.

**Methods of determining sulphur in iron pyrites.** D. P. LIEBENBERG and S. LEITH (J. S. Afr. Chem. Inst., 1931, 14, 47—51).—Comparative tests on the determination of S in pyrites by the HNO<sub>3</sub>-KClO<sub>3</sub>, HNO<sub>3</sub>-Br, and HNO<sub>3</sub>-HCl methods with and without removal of the Fe with aq. NH<sub>3</sub> showed that equally good results were obtained by all the methods when Fe was removed. A more rapid method than the above comprises fusion of 0.5 g. of ore with 5 g. of Na<sub>2</sub>O<sub>2</sub> in a Ni crucible, dissolving the mass in HCl, reducing the FeCl<sub>3</sub> with KI, and treating the solution with BaCl<sub>2</sub> after expelling the I by boiling.

A. R. POWELL.

**Desulphurisation of cast iron by means of soda ash. Harmful effects of sulphur as an impurity in cast iron** (Imperial Chem. Industries, Ltd., 1931, 12 pp.).—Proprietary compounds sold usually in the form of bricks for the desulphurisation of Fe act by virtue of their alkaline content. The brick form is not necessary, as dense soda ash of low dust content is quite suitable. It is placed in the bottom of the ladle and molten metal run on to it, when the evolution of CO<sub>2</sub> produces vigorous agitation. Eventually the alkaline slag is thickened with ground limestone and removed. About 1% (on the wt. of Fe) of Na<sub>2</sub>CO<sub>3</sub> and 0.5% of CaCO<sub>3</sub> is sufficient in

ordinary cases. About 60% of the total S was removed in foundry trials from cast Fe of low S content. The action is complete in a few min. The Mn content of the Fe is not affected, the Si is slightly reduced, and the physical properties of the metal are improved. The use of soda ash for the direct desulphurisation of molten steel is not recommended. The cost of its use for cast Fe is about 1s. 6d. per ton, and it enables cheaper raw materials to be used whilst complying with a rigid specification for the product. C. IRWIN.

**Formation of graphite in cast iron.** H. HANEMANN (Stahl u. Eisen, 1931, 51, 966—967).—Quenching of cast Fe (4—15% C, 0.09% Si, 0.18% Mn, and < 0.03% P and S) from above the eutectic point (1141°) produced a purely ledeburitic structure, and from just below the m.p. a completely grey structure. Complete decomp. of the cementite in the same metal required 1 hr. at 1140° and 6 hr. at 1000°. These results indicate that graphite is present in the Fe immediately after solidification, and is not formed by the decomp. of primary cementite, but separates directly from the liquid metal. A. R. POWELL.

**Case-hardening.** H. MÜLLER (Arch. Eisenhüttenw., 1931—2, 5, 57—62).—Recent work on case-hardening steel with C and N compounds is critically reviewed and in certain respects amplified by further work. CO carburises slowly but evenly without over-carburising the outermost layer; its action may be intensified by increasing the pressure. Hydrocarbons, on the other hand, carburise rapidly and the surface of the metal rapidly becomes converted into carbide which diffuses only slowly inwards unless a high temp. is employed. Nitrogenisation is most effective when pure N<sub>2</sub> is used, but the method is costly and its efficiency depends to some extent on the composition of the steel. Immersion in NaCN baths hardens the surface of steel by absorption of both C and N, but the effect of the latter preponderates. The best case-hardening temp. is 900°, cooling should be rapid, and the hardened articles should be heat-treated below the A1 point (*e.g.*, at 650°) and subjected to a skin-hardening treatment so as to induce a uniform transition from the hard outer layer to the softer core. A. R. POWELL.

**Precipitation hardening of iron by titanium.** R. WASMUHT (Arch. Eisenhüttenw., 1931—2, 5, 45—56).—Ti alone produces precipitation hardening in Fe only when about 6% is present, but in the presence of Si or Ni only 1—3% Ti is necessary. The presence of C reduces the hardening effect owing to the formation of Ti carbides. The hardness of Fe containing 1.3% Mn, 3.2% Si, and 2.7% Ti after quenching from 1250° is 260, but on annealing at 500° it rises rapidly to a max. of 570 in 12 hr. and at 600° to a max. of 510 in 2—3 hr. Lower quenching temps. and higher Ti to Si ratios result in a decreased precipitation hardening. Addition of 2.3% Ti to V2A steel causes an increase in hardness from 280 to 480 on quenching from 1250° and annealing at 500°. The high temp. from which Ti-Si steels must be quenched before hardening produces a coarse-grained structure, but this may be refined without reducing the precipitation hardness by the addition of Mn, Ni, or Cr. Measurements of the changes of hardness, electrical

conductivity, and magnetic properties of Ti steels after various heat treatments indicate that the mechanism of the precipitation hardening is similar to that of the age-hardening of duralumin. Most of the hardened Ti steels retain their hardness and high tensile strength up to 500°, but on prolonged heating above this temp. the steels become soft again. A. R. POWELL.

**Ageing of hardened carbon steel.** S. STEINBERG and W. SUBOW (Stahl u. Eisen, 1931, 51, 911—913).—The hardness of steel with 0.98% C rises slowly over a period of several months when the steel is stored after quenching from 750°. Similar increases in hardness occur when the steel is annealed at 75°, 100°, or 125°, but much more rapidly; in all cases the hardening effect is accompanied by a decrease in vol. which reaches a max. of nearly 0.12% at 125° in 30 min. For practical purposes ageing at 125° for 30 min. is recommended, the bending strength is thereby increased from 145 to 195 and the Rockwell C hardness from 65 to 66. A. R. POWELL.

**Effect on the properties of a metal [nitrogen-iron alloy] of the combined action of cold-work and disperse precipitation [ageing].** W. KÖSTER (Z. Metallk., 1931, 23, 176—177).—The coercivity of a slowly cooled steel containing N in solid solution rose from 3 to 7 gauss in a smooth curve by subjecting it to an elongation of up to 20% in the tensile machine; on subsequent ageing at 100° for 14 days to cause precipitation of the nitride in a finely-dispersed form a further increase of coercivity was obtained depending on the degree of stretching and reaching a max. with 5% elongation. The abs. increase in coercivity decreased linearly with increasing elongation, although the electrical conductivity of all the specimens was the same, showing that equal amounts of nitride had been precipitated. Subsequent annealing at 300° to redissolve the nitride resulted in the restoration of the original coercivity curve, showing that it is possible to redissolve the nitride without removing the effect of cold-work. In a second series of tests the metal was aged at 100°, then stretched up to 20% elongation; the coercivity curve obtained resembled that obtained with the specimens stretched before ageing, but the actual coercivity values were somewhat higher. Subsequent annealing at 300° reduced the coercivity to the figures obtained in the first test. Hence it appears that the effects of cold-work and of ageing on the coercivity of steel are strictly additive. A. R. POWELL.

**Effects of carbon and iron in blue powder on sherardising.** M. KURODA (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1931, 16, 125—126).—Additions of C, particularly 2%, have a slight beneficial action on the rate of formation of a Zn coating by sherardising. Additions of Fe are disadvantageous, causing decrease in rate of coating and entanglement of Fe particles which lead to local rusting. E. H. BUCKNALL.

**Example of an unstable condition in [nickel-iron] alloys which cannot be stabilised by heating at 1000°.** L. ANASTASIADIS and W. GUERTLER (Z. Metallk., 1931, 23, 189—190).—The ( $\alpha + \beta$ )- $\gamma$  transformation in Ni-Fe alloys with 5—25% Ni is accompanied by hysteresis phenomena which are particularly

marked in the changes which occur in the electrical resistance. In alloys with 15–25% Ni there is a metastable condition which cannot be completely stabilised by prolonged annealing at 1000°. A. R. POWELL.

**Determination of nitrogen in steel and iron alloys.** P. KLINGER (Arch. Eisenhüttenw., 1931–2, 5, 29–32).—The N in Fe alloys containing Cr, W, Ti, V, or Si is not completely recovered as  $\text{NH}_3$  by the dissolution method, and that in Ti, V, or Al alloys is not completely removed by dissociation on heating in vac. at 1100°. Complete expulsion of the N as such is obtained by fusion of the fine drillings with a 1:1 mixture of  $\text{Na}_2\text{O}_2$  and  $\text{Na}_2\text{CO}_3$  in vac.; fusion is carried out in a hard glass tube connected with a heated tube packed with Cu turnings to absorb the  $\text{O}_2$  evolved. The gases are passed backwards and forwards over the Cu until no further reduction in pressure takes place, then freed from  $\text{CO}$ ,  $\text{CO}_2$ , and  $\text{H}_2$  in the usual way, and the  $\text{N}_2$  is measured. A. R. POWELL.

**Formation of hard zinc and iron salts [in hot galvanising].** K. TAUSSIG (Stahl u. Eisen, 1931, 51, 885–886).—The presence of  $\text{FeCl}_2$  on the Fe sheets when they are immersed in the Zn bath leads to the formation of Fe–Zn alloy in the bath by interaction between the Zn and  $\text{FeCl}_2$ ; practically no  $\text{FeCl}_2$  is lost by volatilisation, so that it is essential to wash thoroughly the sheets before immersion in the bath. A. R. POWELL.

**Soil corrosion. Rates of corrosion and pitting of bare ferrous specimens.** K. H. LOGAN and V. A. GRODSKY (Bur. Stand. J. Res., 1931, 7, 1–35).—The extent to which soil conditions are responsible for the corrosion of underground pipes has been investigated by determining the rate of loss of wt. of 1300 specimens removed from 70 test stations. The extent of corrosion depends largely on the character of the soil, and whilst the rate appears to vary from year to year, the general tendency is to decrease with time of exposure. The data do not indicate that any one of the commonly used pipe materials is markedly superior to the others for general use underground, although it seems that certain materials have some advantage in certain locations. Several causes appear to be responsible for the corrosiveness of soils, and it is improbable that a single satisfactory method for determining soil corrosiveness can be developed. E. S. HEDGES.

**Internal stresses in brass tubes.** A. KRÉČEK (Z. Metallk., 1931, 23, 178–185).—Experiments are recorded which show that even small stresses produced by finishing operations may lead to season-cracking in ( $\alpha + \beta$ )-brass; a deformation of 5% in fully-annealed brass or of only 1.5% in brass which has had a low-temp. anneal to remove internal stress may be dangerous. A. R. POWELL.

**Properties of drawn wires and the power consumption in wire-drawing.** W. LINICUS (Z. Metallk., 1931, 23, 205–210).—The effect of drawing conditions and the type of dies used on the mechanical properties of Fe and brass wires has been investigated. A. R. POWELL.

**Behaviour of phosphorus and oxygen in copper-tin melts and alloys.** R. FALKENSTÖRFER (Metall-

Wirts., 1931, 10, 130–131; Chem. Zentr., 1931, i, 2259).—When brass containing Cu 85, Sn 8, Zn 5, Pb 2% is melted in a reducing atm., the strength and extensibility diminish rapidly when the P content exceeds 0.12%. With up to 0.15% P the solid solution is homogeneous, but with more than 0.21% P the phosphide eutectic appears. P is not removed from the heated mass at 1100°, when loss of Zn did not exceed 0.3%. The reducing action of P is obvious when oxidation has occurred during fusion.

A. A. ELDRIDGE.

**Welding of monel metal in the construction of chemical apparatus.** R. MÜLLER (Chem. Fabr., 1931, 4, 310–312).—As  $\text{Cu}_2\text{O}$  is sol. in molten monel metal and renders the alloy brittle and less resistant to corrosion, it is essential that welding be carried out under non-oxidising conditions. In  $\text{O}_2$ - $\text{C}_2\text{H}_2$  welding, the gas should be passed through a solution of  $\text{H}_3\text{BO}_3$  in EtOH and a slight excess of  $\text{C}_2\text{H}_2$  should be present in the gas mixture. In arc-welding, the work should be made the negative pole and the welding rod should be coated with powdered Mg–Mn–Si or Ti–Ca alloy. Good welds are also obtained by welding with at. H, using W electrodes. A. R. POWELL.

**Influence of pressure conditions in the oxy-acetylene welding torch on economy and quality of the weld.** RIMARSKI, PLEUS, FRIEDRICH, STREB, NOACK, KEMPER, HOFFMANN, and KONSCHACK (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 60–64; Chem. Zentr., 1931, i, 2418).—Changes in the mixture ratio in welding torches are due partly to change in pressure owing to rise of temp. and partly to back-pressure. The quality of the weld is unaffected. A. A. ELDRIDGE.

**Reports of investigations: [Canadian] metallic ores.** C. S. PARSONS, J. S. GODARD, A. K. ANDERSON, D. S. HALFORD, and G. B. O'MALLEY (Canad. Dept. Mines, Rept. Ore Invest., 1929, 5–158).—The results of table concentration, ore flotation, cyaniding, and amalgamation tests on numerous samples of Canadian Au, Ag, Sn, Cu, Pb, and Zn ores both simple and complex are recorded, and in most cases suitable methods of treatment are indicated. A. R. POWELL.

**Reports of investigations: [Canadian Mines Laboratory] hydrometallurgical and electro-chemical section.** I. Treatment of titaniferous magnetite from Mine Centre for recovery of iron, titanium, and vanadium. R. J. TRAILL, W. R. McCLELLAND, and J. D. JOHNSTON. II. Separation of copper from molybdenum in molybdenite concentrates. R. J. TRAILL. III. Treatment of zinc concentrates from various Canadian ores. R. J. TRAILL, W. R. McCLELLAND, and J. D. JOHNSTON. IV. Treatment of mixed concentrate from base-metal sulphide ore. W. E. HARRIS, R. J. TRAILL, and H. C. MABEE. V. Use of coking coals in the metallisation of iron ores. W. R. McCLELLAND (Canad. Dept. Mines, Rept. Ore Invest., 1929, 178–185, 186–187, 187–191, 192–199, 199–201).—I. An ilmenite ore containing 48.4% Fe, 12.8%  $\text{TiO}_2$ , 0.9%  $\text{V}_2\text{O}_5$ , and 6.4% Si was heated with coal and coke at 1050° for 5 hr. and the reduced product gave a magnetic product containing 61.2% Fe (58.3% metallic Fe),

17.6% TiO<sub>2</sub>, and 1% V<sub>2</sub>O<sub>5</sub>. Over 50% of this product was dissolved by FeCl<sub>3</sub> solution, leaving 99% of the TiO<sub>2</sub> and 97% of the V<sub>2</sub>O<sub>5</sub> in a residue containing 20.8% Fe, 35.3% TiO<sub>2</sub>, and 2.1% V<sub>2</sub>O<sub>5</sub>. Roasting of this residue with NaCl and Na<sub>2</sub>CO<sub>3</sub> rendered 75% of its V content sol. in H<sub>2</sub>O, and the insol. material is available for the extraction of TiO<sub>2</sub> by heating with H<sub>2</sub>SO<sub>4</sub>.

II. An ore containing 4.6% Cu as chalcopyrite and 2.8% Mo as molybdenite gave only a poor extraction of Cu on leaching with FeCl<sub>3</sub>, but after roasting with S in a reducing atm. over 85% of the Cu was extracted with FeCl<sub>3</sub>; after leaching with dil. HCl to remove Ca and Mg the residue gave a good Mo extraction on roasting and leaching with NaOH solution.

III. The results of a preliminary investigation into the formation of ferrites on roasting ZnS concentrates containing FeS are recorded. The determining factors in ferrite formation are the roasting temp., the Fe content, and the mineralogical nature of the ore.

IV. A mixed sulphide flotation concentrate containing 30% Zn, 5.5% Cu, 3.4% Pb, 19.4% Fe, and 7.5 oz. (Au + Ag) per ton was roasted in a multiple-hearth furnace and the product leached with dil. H<sub>2</sub>SO<sub>4</sub> until the solution contained less than 0.5% of free acid. The solution contained 80% of the Zn and 50% of the Cu; it was purified by treatment with Zn dust to ppt. Cu, then with MnO<sub>2</sub> and CaCO<sub>3</sub> to ppt. Fe(OH)<sub>3</sub>, and filtered. The filtrate contained 120–130 g. of Zn per litre and 2 p.p.m. of Ni which was removed by treatment with 0.01% of HgSO<sub>4</sub>, 0.15% of H<sub>2</sub>SO<sub>4</sub>, and 0.15% of Zn dust; the purified solution was then electrolysed for Zn, the outflowing liquor containing 3.6% Zn and 12.5% H<sub>2</sub>SO<sub>4</sub> being returned to the leaching. The residue from leaching containing 17.4% Zn, 8.3% Cu, and 5.9% Pb, was treated by the Waelz process, whereby 97% of the Zn and over 95% of the Pb were recovered in the fume, leaving a residue containing 0.6% Zn, 11.8% Cu, and 15.6 oz. Au + Ag per ton suitable for smelting for the recovery of Cu, Ag, and Au.

V. A mixture of finely-ground Fe ore and coking coal in a 1:1 ratio was heated at 636–946° for several hr. and the products of destructive distillation were removed continuously so as to maintain a slightly reduced pressure in the furnace. The resulting Fe sponge was of good quality after magnetic separation from the silicious residue.

A. R. POWELL.

**Boundary fields of metallography.** K. SCHRÖTER (Z. Metallk., 1931, 23, 197–201).—For the examination of W powder the material is mixed with 3 times its wt. of Cu powder and the mixture sintered in H<sub>2</sub> in an electric furnace at 1250°. As Cu does not alloy with W the structure of the latter remains unaltered; a section of the sintered mass is embedded in solder, polished in the usual way, and etched with alkaline K<sub>3</sub>Fe(CN)<sub>6</sub> solution. To examine the mechanism of sintering of W bars, samples of the bar at various stages of the sintering process are immersed in molten Cu, which fills up the pores and thus facilitates polishing of a section; in this way grain growth during sintering may be readily followed. The structure of W wires may be examined by coating them electrolytically with Cu, embedding the wire in asphalt, and polishing as usual; the surface Cu is then dissolved in dil. HNO<sub>3</sub> and the remainder is

blackened by (NH<sub>4</sub>)<sub>2</sub>S solution. In this way the structure of the W wire shows up sharply against the black background; a similar device serves for the examination of thin Ni and Ni-Fe alloy wires. Methods for detecting hair-cracks in and the riffling of glass tubing etc. are also described.

A. R. POWELL.

**Effect of iron content of blende on its flotability.**

L. KRAEBER (Mitt. Kaiser Wilh.-Inst. Eisenforsch., 1930, 12, 343–352; Chem. Zentr., 1931, i, 2257).—Max. flotability is reached when the *p*<sub>H</sub> of the H<sub>2</sub>O is 6.0–7.0, the optimal val. being the lower the higher is the Fe content of the blende. The varying rapidity of flotation is not attributable to the Fe content, neither is there a definite relation between the effects of KCN and CuSO<sub>4</sub> and the Fe content.

A. A. ELDRIDGE.

**Effect of small quantities of addenda on the behaviour of soft lead towards acids.**

A. BUCKHARDT (Metall-Wirts., 1931, 10, 181–187; Chem. Zentr., 1931, i, 2258).—Min. strength is rapidly reached by storage for 1 month at 70–200°. Except with alloys containing >0.1% Ag or 0.005% Li, the elongation also falls. On exposure to H<sub>2</sub>SO<sub>4</sub> the metal exhibits no change in strength or elongation. Fe, Sb, Sn, Te, Cu, and Ni decrease, whilst Li, Ag, Zn, Cd, and Bi increase, the corrosion. Tl and Hg have little or no effect. Pb containing Cu, Ni, or Te is least resistant towards HCl, pure Pb being most resistant. Moist Cl<sub>2</sub> causes greater corrosion than HCl.

A. A. ELDRIDGE.

**Determination of lead in slags.** W. WITTER (Chem.-Ztg., 1931, 55, 550).—The slag is decomposed with 1:1 HCl and NH<sub>4</sub>F, the solution boiled to expel SiF<sub>4</sub>, and any sulphide oxidised with KMnO<sub>4</sub>. Pb is then separated as PbS, which is converted into PbSO<sub>4</sub> and the analysis finished by the usual (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub> titration.

A. R. POWELL.

**Heat conductivity, electrical conductivity, and Lorenz value of some light alloys.**

W. MANNCHEN (Z. Metallk., 1931, 23, 193–196).—The thermal and electrical conductivities and the Lorenz values for Al and Mg and for 16 Al and 20 Mg light alloys at 87°, 273°, 373°, and 476° are tabulated. The thermal conductivities of the alloys are all lower than that of the basic metal, but the temp. coeffs. are positive for the alloys and negative for the pure metals. Addition of Mn or Si to Mg results in a considerable decrease in both conductivities; this is explained on the assumption that a limited series of solid solutions is formed, although previous workers have stated that Mn and Si are completely insol. in solid Mg. The Lorenz value for alloys is usually much less than for the pure metals, but in Cu-Al and Mn-Mg alloys the vals. are approx. the same as for Al and Mg respectively.

A. R. POWELL.

**Surface treatment and electroplating of aluminium.**

ANON. (Chem.-Ztg., 1931, 55, 579–580).—Al articles are freed from a coating of oxide by treatment with NaOH solution followed by dil. HCl or H<sub>2</sub>SO<sub>4</sub>-HNO<sub>3</sub> mixture or by treatment with a NaOH-NaCl mixture. Al previously cleaned may be polished by steel balls in revolving drums. After cleaning, Al can be lacquered in the same way as other metals. There is great difficulty in producing a coherent coating on Al by electroplating, and such a coating cannot be relied



on as a complete protection. Articles which are not to be wetted can be electroplated in the same way as other metals, but the oxide surface layer must be removed. Coatings of Fe, Cu, Zn, Cd, Cr, and Ni can be obtained directly and used for the superposition of other metals. Ni may be deposited from a bath containing glycerin with a c.d. of 0.5 amp./sq. dm. Al may be sprayed with other metals by the Schoop process.

C. IRWIN.

**Blast-furnace coke. Welding with  $C_2H_2-O_2$  mixtures.**—See II. Metals and milk.—See XIX.

## PATENTS.

**Furnace [for brazing, annealing, etc.].** (A) C. L. IPSEN and J. L. MCFARLAND, (B) J. L. MCFARLAND, Assrs. to (A, B) GEN. ELECTRIC CO. (U.S.P. 1,789,177 and 1,789,187, 13.1.31. Appl., [A] 29.11.27, [B] 19.4.29).—In (A) a furnace, and in (B) a method of operating it, are described, the object being the annealing or brazing of metals in an explosive atm. The furnace is heated nearly up to the working temp. while filled with air, a non-explosive gas, e.g.,  $N_2$  or  $N_2-H_2$  mixture, is used to sweep out the air, and finally pure explosive gas, e.g.,  $H_2$ , is passed in. In cooling, the reverse process is adopted. The furnace comprises an elongated shell suitably lined and provided with openings at the ends through the bottom for inlet and outlet (respectively) of goods which are placed on trucks and pushed longitudinally through by a ram. Near the inlet a zone is electrically heated, the remainder being for cooling; the zones for the various gases are divided off by transverse partitions which fit closely to the trucks, and it is arranged that, whenever possible, there is a truck in each opening.

B. M. VENABLES.

**Annealing or other heat treatment of metals, more especially iron and steel.** N. G. ELFSTROM (B.P. 352,677, 10.6.30).—The metal is heated in a closed chamber, the atm. of which is provided by blast-furnace, producer, or other fuel gas passing through glowing C heated electrically. Control of the temp. of the C provides either a carburising, inert, or decarburising atm.

C. A. KING.

**Agglomeration of finely-divided ferruginous ores, concentrates, etc.** A. FOLLIET and N. SANDERICHIN (B.P. 352,832, 3.10.30. Fr., 30.10.29).—A mixture of ore and fuel is fed forward in a thin layer and in a certain zone meets penetrating jets of air at 650–800° which promotes combustion.

C. A. KING.

**Treatment of metallurgical slimes.** METALLGES. A.-G. (B.P. 353,212, 27.6.30. Ger., 28.6.29).—Slimes are mixed with solid fuel or other coarse agglomerate which favours the drainage of contained  $H_2O$ , and are then transferred direct to the blast-roasting process.

C. A. KING.

**Conversion of metallic ores.** H. EDWARDS, O. J. PARKER, and R. ARNOT (B.P. 354,098, 13.9.30).—The ore, with or without a preliminary mechanical concentration treatment, is heated at a temp. below that at which sintering occurs with a powdered carbonaceous reducing agent and with a compound which combines with or decomposes  $H_2O$ , e.g., CaO,  $CaC_2$ , or  $CaCN_2$ . Thus an ore containing  $CuCO_3$  in a basic gangue is heated with

coal and  $CaCN_2$  at 400–450° and the product is treated magnetically to separate Fe, leaving a residue from which Cu can be recovered by wet dressing.

A. R. POWELL.

**Moulding processes [for metals] and moulds made thereby.** J. B. DURAND (B.P. 351,592, 3.4.30. Ger., 4.4.29).—Moulds or cores for use in metal casting are made by the process described in B.P. 319,777 (B., 1931, 354) except that artificially produced sand or old sand, produced by grinding used moulds or cores made by the same process, is used. [Stat. ref.]

A. R. POWELL.

**Granulation of liquid slag.** A. L. MOND. From VEREIN. STAHLWERKE A.-G. (B.P. 351,591, 3.4.30).—The liquid slag is run down a channel in which it meets jets of  $H_2O$  directed at various angles into the slag stream and the mixture of hardened slag and  $H_2O$  falls into a centrifugal machine comprising a conical chamber with a rotating central axis carrying arms the length of which increases from top to bottom.

A. R. POWELL.

**Manufacture of sponge iron.** I. RENNERFELT and I. C. S. G. BOHM (B.P. 353,180, 12.6.30).—Finely-divided Fe ore (concentrates) is mixed with approx. the theoretical quantity of solid reducing agent (high-grade charcoal) and heated in coke-oven chambers, reducing gas being admitted during the later stages of reduction. The sponge Fe is then pushed out of the chamber, with protection from the atm.

C. A. KING.

**Production of high-strength grey cast iron.** INTERNAT. NICKEL CO., INC., Asses. of F. B. COYLE (B.P. 352,510, 4.1.30. U.S. 5.1.29).—A charge consisting of >60% of steel, >12% of coke, and scrap Fe is melted and graphitised by adding 2.5–7% of Ni or Si, preferably >5.5% Ni. The tensile strength of the product exceeds 50,000 lb./sq. in.

C. A. KING.

**Manufacture of material suitable for resisting rifle bullets and other projectiles etc.** (SIR) R. A. HADFIELD (B.P. 352,548, 12.4.30).—Steel containing C 0.35–0.6%, Mo or W 0.25–1.5%, Mn 0.25–3%, and Si 0.25–2.5% is annealed, rolled, reheated to 875–900°, quenched, reheated to 530–590°, and cooled slowly to remove strains. The alloy may be tempered from about 650°.

C. A. KING.

**Cobalt high-speed tool steel.** OESTERR. SCHMIDTSTAHLWERKE A.-G. (B.P. 352,951, 14.4.30. Addn. to B.P. 343,508; B., 1931, 448).—Co steels containing not more than 1.5% Cr, preferably <0.5%, are claimed. [Stat. ref.]

C. A. KING.

**Magnetic [iron-nickel] material.** J. E. HARRIS, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,790,704, 3.2.31. Appl., 24.3.30).—Dust for the manufacture of magnetic cores is made by grinding an alloy of 25% Co, 27% Fe, 45% Ni, 3% Sn, and 0.0625% Bi, or an alloy of 25% Co, 26% Fe, 45% Ni, 4% Mo, and 0.25% Pb, until at least 90% passes 120-mesh.

A. R. POWELL.

**Iron-titanium alloys.** W. KROLL (B.P. 352,964, 7.2.30. Ger., 16.2.29).—Alloys with >50% Fe, 0.5–8% Ti, up to either 40% Ni or 8% Mn, and additions of up to 25% Cr, 15% Co, 5% Mo, 3% V, 4% Si, or 25% W are quenched between 700° and 1350° and artificially aged by annealing at 320–600°.

C. A. KING.

**Production of corrosion-resisting ferrous alloys.**

HALL & PICKLES, LTD., and J. SMITH (B.P. 351,532, 26.3.30).—Fe or mild steel containing 0.25–4% Si, 0.1–2% Cu, and 0.1–2% As is claimed. The Cu and As are added when the charge is melted down and the Si as Fe–Si when the metal has been refined. A small amount of Al is added just before casting to deoxidise the charge. A. R. POWELL.

**Joining zinc-aluminium alloys to steel.** H. C. HALL (B.P. 351,977, 25.1.30).—The steel surface is galvanised by the hot-dipping process and after heating to 250° the galvanised surface is covered with a layer of molten Al–Zn alloy containing 70–99% Zn. A. R. POWELL.

**Composition for treating metals [iron to prevent rusting].** E. K. WALLACE, ASSR. TO RUST PREVENTION CHEM. CO. (U.S.P. 1,790,960, 3.2.31. Appl., 7.3.29).—The pickling solution comprises a mixture of 42.5% H<sub>3</sub>PO<sub>4</sub>, 56% H<sub>2</sub>O, and 1.5% casein. A. R. POWELL.

**Continuous copper-melting furnace.** R. P. HEUER (B.P. 351,740, 28.6.30).—The flue gases from the melting furnace are separated into two parts, one of which is used for preheating the air for combustion and the other for preheating the incoming charge to the furnace to a temp. just below the m.p. This is effected by feeding the charge on to conveyor which moves along a circular track in the flue. A. R. POWELL.

**Copper-silicon-zinc alloys.** HIRSCH, KUPFER-U. MESSINGWERKE A.-G. (B.P. 352,639, 19.5.30. Ger., 13.2.30).—Castings of high tensile strength contain Cu 65–80%, Si 2–6%, with one or more of the metals Al, Mn, Co, Fe, Pb, Ni, Cr, Zr, W, Mo, and Ti to the limit of 0.1–3%. C. A. KING.

**Reclaiming and re-using metallic scrap mixtures [copper-tungsten or -molybdenum welding rods].** FORD MOTOR CO., LTD., ASSEES. OF R. H. MCCARROLL and G. VENNERHOLM (B.P. 353,377, 4.2.31. U.S., 4.2.30).—The scrap ends of Cu–Mo or Cu–W welding rods are ground in a disc or ball mill to pass 20–100-mesh and the powder is reduced at 1000° for 15 min. in H<sub>2</sub>. The resulting mixture of W or Mo grains coated with Cu is pressed into electrodes which are sintered until coherent and then used again for welding. A. R. POWELL.

**Reduction of zinciferous materials.** NEW JERSEY ZINC CO., ASSEES. OF E. H. BUNCE (B.P. 353,371, 29.12.30. U.S., 30.12.29).—A mixture of Zn ore and powdered coal is fed continuously into Chilean mills to which tar or pitch is added to bind the mixture, which then passes to a briquetting machine. The briquettes fall on to a conveyor belt which transfers them to a coking furnace, the hot discharge from which is fed automatically to vertical retorts of the gas-fired recuperator type. The exhaust hot gases from the recuperator are mixed with a quantity of unburnt fuel gas and the mixture is used to heat the coking furnace. A. R. POWELL.

**Reduction of zinciferous materials containing lead and other contaminating metals.** L. MELLSHER-JACKSON. From NEW JERSEY ZINC CO. (B.P. 352,005, 3.2.30).—Briquettes of Zn ore and coke are fed downwards through a long vertical retort the lower

half only of which is heated to a temp. at which the Zn distils, whilst the upper half is heat-insulated so that an appreciable length of the briquette column is maintained at 800–900°, whereby the Pb, Bi, etc. which distil in the lower zone are condensed again in the charge and a relatively pure Zn vapour passes to the condensers. A. R. POWELL.

**Refining aluminium, magnesium, and their alloys.** H. C. HALL (B.P. 352,199, 5.6.30).—Molten Al or Al alloy is treated with a tablet, stick, or lozenge of fused anhyd. ZnCl<sub>2</sub> coated with wax, grease, oil, or varnish which prevents it from deliquescing during storage. Mg alloys are treated with MgCl<sub>2</sub> preparations which have been similarly treated. A. R. POWELL.

**Manufacture of aluminium alloys.** S. JUNGHAUS (B.P. 353,891, 29.1.30. Ger., 31.1.29).—Alloys of Al with 5–20% Cu, 1–3% each of W, Mn, and Cr, 0.1–3% Ni, and 0.1–2% Na, K, Li, Be, Mg, Ca, Sr, or Ba are claimed. In addition, 0.1–3% Co, <5% Ag, <10% Cd, and <3% V or Mo may be added. A. R. POWELL.

**Melting flux for purifying aluminium, its alloys and other non-ferrous metals.** F. DUBBERLEY, SEN. and JUN., C. A. DUBBERLEY, and E. H. TYSON (B.P. 353,867, 26.3. and 12.4.30).—The molten metal is treated with a flux comprising (a) 10% K<sub>4</sub>Fe(CN)<sub>6</sub>, 2.5% KOH, 10% Na<sub>3</sub>AlF<sub>6</sub>, 45% CaF<sub>2</sub>, and 32.5% NaCl, or (b) 18% K<sub>4</sub>Fe(CN)<sub>6</sub>, 2% KOH, 48% CaF<sub>2</sub>, and 23% NaCl. A. R. POWELL.

**Metallic [aluminium] protective coating.** S. U. MCGARY, W. F. ROGERS, and S. GILL, ASSRS. TO GILL PRODUCTION CO. (U.S.P. 1,790,643, 3.2.31. Appl., 11.10.29).—Timber or metal articles are protected from corrosion by coating them with a thin sheet of Al (0.0005–0.002 in. thick) which is held on to the article by means of a layer of a plastic adhesive material. A. R. POWELL.

**Lead alloy.** R. J. SHOEMAKER, ASSR. TO S. & T. METAL CO. (U.S.P. 1,791,148, 3.2.31. Appl., 2.8.28).—A Pb alloy containing 0.01–0.03% Li, 0.1–0.3% Ca, 0.25–0.5% Sn, and 0.02–0.1% Al is claimed. A. R. POWELL.

**Hard-metal compositions.** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF S. L. HOYT (B.P. 354,028, 9.7.30. U.S., 16.7.29).—A mixture of 100 pts. of WC and 15 pts. of U in the form of fine powder is heated in a C mould at 1500–1600° and a pressure of 1000 lb./sq. in. is applied to compress the mixture into a sintered slug suitable for use, after shaping, as a cutting tool. A. R. POWELL.

**Recovery of refractory metals from ores [zirconium from zircon].** C. V. IREDELL, ASSR. TO WESTINGHOUSE LAMP CO. (U.S.P. 1,791,272, 3.2.31. Appl., 7.12.28).—Finely-powdered ZrSiO<sub>4</sub> is fused with coke, graphite, or charcoal at 1900–2500°, whereby a mixture of SiC and ZrC is obtained and most of the Fe is volatilised. The product is digested with dil. HNO<sub>3</sub> (1:1) to obtain a solution of Zr(NO<sub>3</sub>)<sub>4</sub> and a residue of SiC. A. R. POWELL.

**Purifying or refining non-ferrous metals and alloys.** D. R. TULLIS and P. OAKLEY (B.P. 352,671,

4.6.30).—Vapour of V halide is introduced into a molten metal, e.g., Al, or its alloys. C. A. KING.

**Apparatus for treatment of ores [especially those containing precious metals].** N. LESLIE (B.P. 352,931, 10.3.30. Austral., 11.3.29).—The apparatus comprises a vertical chamber with a conical bottom, an adjustable upcast pipe in the vertical axis of the chamber, and a circulating pipe entering the chamber at the point of the cone and being directed in the style of an injector into the upcast pipe so that by passing compressed air through the jet the contents of the chamber are thoroughly agitated. The apparatus is suitable for amalgamation, cyanidation, or flotation processes. A. R. POWELL.

**Cathodes for the electrolytic refining of metals.** H. WADE. FROM UNITED STATES METALS REFINING Co. (B.P. 352,104, 9.4.30).—Cathodes for the electrolytic refining of Cu consist of thin Cu sheets stiffened by means of horizontal corrugations at the top and bottom and vertical corrugations down the middle. Corrugations and fastening of the suspending loops to the sheet are made in one stamping operation. A. R. POWELL.

**Electrolytic production of metals, particularly magnesium.** A. C. JESSUP (B.P.351,510, 28.2.30).—Electrolysis is carried out in a square tank with a central anode, whereby the circulating currents set up in the  $MgCl_2$  electrolyte are sufficient to cause the deposited Mg on the cathodes to become detached and to be driven into cooled recesses which are provided at the four corners of the tank. The metal is protected against the  $Cl_2$  evolved at the anode by surrounding the latter with partitions which dip just below the surface of the electrolyte. A. R. POWELL.

**Electrolytic production of magnesium.** I. G. FARBENIND. A.-G. (B.P. 351,503, 20.1.30. Ger., 6.2.29).—The bath is provided with a series of plane anodes and cathodes extending nearly across the whole cross-section and separated from one another by non-conducting partitions extending less than half-way down the bath, the partitions being closer to the cathodes than to the anodes and forming a gas-collecting chamber round the anode. The lower sides of the partitions are bevelled upwards away from the anode and the cathodes or extensions thereof are disposed just below these bevelled portions. The bath is replenished with fused  $MgCl_2$  free from suspended C and  $SiO_2$ . A. R. POWELL.

**Electrolytic zinc bath.** G. BIANCO (U.S.P. 1,791,082, 3.2.31. Appl., 20.4.27).—Zn is melted with 1–15% Hg and the granulated alloy is dissolved in a solution containing  $H_2SO_4$ ,  $(Al,Fe)_2(SO_4)_3$ ,  $H_3PO_4$ , and NaOH to give an approx. neutral solution of Zn salts. A. R. POWELL.

**Operation of furnaces for brazing, annealing, etc.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of J. L. McFARLAND (B.P. 353,062, 16.4.30. U.S., 19.4.29).—See U.S.P. 1,789,187; preceding.

**Magnetic materials [nickel-iron alloys].** ELECTRICAL RESEARCH PRODUCTS, Assees. of G. W. ELMEN (B.P. 352,019, 31.3.30. U.S., 2.4.29).—See U.S.P. 1,757,178; B., 1930, 1033.

**Purification of magnesium or magnesium alloys.** C. ARNOLD. FROM DOW CHEM. Co. (B.P. 346,271, 5.12.29).—See U.S.P. 1,754,788; B., 1930, 914.

**Furnaces [for heating leaf springs].** BURDONS, LTD., and J. McQUARRIE (B.P. 353,699, 26.7.30).

**Tubular furnace. Hg boilers.**—See I. Welding electrodes. Separation of foundry sand. Determining K in spaces. Coated Cu articles.—See XI. [Adhesive] reaction products of rubber.—See XIV.

## XI.—ELECTROTECHNICS.

**Electric furnace of fluorite.** A. DAMIENS and L. DOMANGE (Compt. rend., 1931, 192, 1711–1713; cf. B., 1931, 720).—The  $CaF_2$  porcelain being permeable to gases at high temp., the furnace is constructed of pure fluorite in the shape of a tube wound with nichrome wire, and placed in an Fe tube with the intervening space packed with powdered fluorite. The ends are water-cooled, and one is closed with a plate of transparent fluorite; temp. is determined by radiation. Work with  $F_2$  can be carried on at  $1000^\circ$ . C. A. SILBERRAD.

**Synthetic galena as a radio-detector.** W. OGAWA (Proc. World Eng. Cong., 1929, 31, 291–304).— $PbS$  was precipitated from  $Pb(OAc)_2$  acidified with  $AcOH$ , filtered, washed, dried, and fused either alone or with other substances.  $Ag_2S$  and  $Tl_2S$  was found to improve sensitivity, the optimum addition of the former being 7.5%. The effect is associated with reduction to Ag. By moulding with solutions, salts of alkali metals were found to be equally effective. For use with a galena crystal, a sharp needle of electropositive metal or alloy is preferable to Pt. The direction of rectification is determined by the chemical composition of the contact portions of both electrodes, and is of a relative nature. Synthetic galena is a better conductor than the natural crystal, but resistance is increased by addition of Ag. Determinations of thermoelectric powers of galena and other mineral crystals used as detectors give no support to any thermoelectric theory of their action. The author's theory is that rectification is caused by the difference of electron emissions from the pairs of substances which make a couple. This difference must be as large as possible, and the resistance of the electrodes must be great enough to minimise metallic conduction. The emission of electrons from a galena crystal has been demonstrated and other evidence in support of the theory is given. C. IRWIN.

**Electrification in coal-dust clouds.**—See II. Welding of monel metal. Conductivity of light alloys. [Resistance of] Al alloys. Plated Al.—See X.

## PATENTS.

**Electric furnaces and methods for making glass and the like.** J. K. B. RAEDER (B.P. 350,930, 6.3.30).—The charge is fed into a vertical chamber comprising an upper melting zone and a lower refining zone with spaced electrodes projecting into the molten glass at the junction of the two zones. The refining zone is connected at its lower end with a conditioning chamber which permits the escape of gas bubbles from the

molten glass, and is also independently electrically heated. A. R. POWELL.

**Welding electrodes and coatings therefor.** P. S. D. SMITH. From HOLLUP CORP. (B.P. 352,086, 5.4.30).—The sheath comprises a flexible material, *e.g.*, licorice tubes or macaroni, with which is incorporated ground glass as a flux. The tube is split longitudinally so that it can be fitted round the electrode as and when required. The sheaths are specially adapted for covering mild steel welding rods. A. R. POWELL.

**Production of negative electrodes for electric accumulators.** SOC. ITAL. BATTERIE ELETTICHE CELLINO (B.P. 352,604, 2.5.30. Ger., 2.5.29).—Amalgamated Zn is deposited upon a metallic sheet, *e.g.*, of Pb, from an electrolyte containing a colloidal alkaline silicate, *e.g.*, Na silicate and  $\text{HgSO}_4$ , in which a Zn anode is immersed. J. S. G. THOMAS.

**[Preventing loss of electrolyte in] electrical accumulators, polarisation cells, electrolytic rectifiers and condensers.** BRITANNIA BATTERIES, LTD. From ACCUMULATOREN-FABR. A.-G. (B.P. 352,394, 1.4.30).—Gas-discharge tubes lead from the upper part of the accumulator vessel etc. to the bottom of an adjacent protecting vessel partly filled with oil or other liquid or with granular absorbent or porous material, *e.g.*, glass wool, pumice stone, etc. J. S. G. THOMAS.

**Electrolytic purification of aqueous solutions.** J. BILLITER (B.P. 352,109, 11.4.30. Ger., 12.4.29).—The removal of salts from  $\text{H}_2\text{O}$  is effected by electrolysis in a cell divided by diaphragms in such a way that no electro-osmotic flow through the diaphragms occurs. This is effected by the use of a mean p.d. of 6–18 volts per liquid compartment and by weakening or neutralising the membrane potentials by suitably directed liquid movements through the diaphragms, care being taken that the difference of level of liquid in the various compartments is sufficient to drive the liquid by hydrostatic pressure in the direction through the diaphragms which the membrane potential would drive it. A. R. POWELL.

**Electrical precipitation apparatus [for gases].** H. V. WELCH, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,790,961, 3.2.31. Appl., 12.6.29. Austral., 1.8.28).—Apparatus comprising a number of precipitation units with a main flue extending alongside, and means for closing down one or more units of the series and diverting the gas stream to the next following or to the delivery outlet, is claimed. J. S. G. THOMAS.

**Electrical precipitator [of suspended particles from gases].** H. A. WINTERMUTE, Assr. to RESEARCH CORP. (U.S.P. 1,791,338, 3.2.31. Appl., 12.4.27).—Baffles projecting from the collecting electrodes form pockets of "dead" gas along the surface of the electrodes, and means are provided for moving the baffles and for removing adherent precipitated material in the pockets. J. S. G. THOMAS.

**[Controlling the humidity of gases in the] electrical purification of gases, particularly waste furnace gases.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 353,025, 17.4.30. Ger., 19.4.29).—A device indicating the gas temp. is adjustably connected with a lever controlling the moistening device. J. S. G. THOMAS.

**Magnetic separation of materials [iron from foundry sand].** H. H. THOMPSON, A. E. DAVIES, and W. E. BOX (B.P. 352,172, 21.5.30).—The sand is elevated by a bucket elevator passing over an upper magnetic pulley so arranged that the sand is discharged at the top and the magnetic material at a slightly lower level. The buckets are made of Fe which is cut away at the belt side and fingers of Fe protrude from the belt and from the sides of the bucket into the interior thereof. A. R. POWELL.

**Electric-discharge apparatus [with heated electrodes].** GEN. ELECTRIC CO., LTD., Assees. of PATENT TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 353,253, 24.7.30. Ger., 30.8.29).—The shell enclosing each electric-heater winding for heating the separate electrodes is made of a sintered mixture of refractory metal and one or more non-metallic substances of high electronic emissivity. Thus the shell may be made of W (70–85%) and a mixture of BaO and CaO (30–15%). J. S. G. THOMAS.

**[Cold electrodes for] gaseous electric-discharge devices.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of T. E. FOULKE (B.P. 352,756, 28.7.30. U.S., 27.7.29).—"Cold" electrodes are coated with a compound of an alkali or alkaline-earth compound of low work function, *e.g.*, KOH, which is subsequently reduced partly to the oxide and partly to the metal. J. S. G. THOMAS.

**Revivifying used electric luminous discharge tubes.** G. CLAUDE (B.P. 353,135, 15.5.30. Fr., 21.5.29).—Gases absorbed by the envelope and electrodes are driven out by heat. J. S. G. THOMAS.

**[Sealing-in wire for] electric incandescence lamps and electric-discharge tubes.** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 353,335, 15.10.30. Holl., 30.11.29).—A single-crystal W wire is claimed. J. S. G. THOMAS.

**Photoelectric cells.** WESTINGHOUSE LAMP CO., Assees. of H. C. RENTSCHLER (B.P. 353,554–5, 6.5.30. U.S., 6.5.29).—Photoelectric cells comprise in (A), or in (B) in parenthesis, an envelope constructed wholly (wholly or partly) of a material and/or made of such thickness as to be permeable to radiation of wavelength less than about 4500 Å. (3900 Å.), a cathode composed of one of the metals of the Ce group (Th), preferably deposited upon Au and photosensitive only to the same radiation, and an anode. J. S. G. THOMAS.

**Photoelectric cells [sensitive only to ultra-violet radiation].** WESTINGHOUSE LAMP CO., Assees. of H. C. RENTSCHLER and D. E. HENRY (B.P. 353,981, 23.5.30. U.S., 12.6.29).—Cells comprising a cathode of U, preferably deposited upon a film of Au coating the envelope which is transparent to radiation of wave-length about 2900 Å., is claimed. J. S. G. THOMAS.

**Photoelectric cells.** ELECTRICAL RESEARCH PRODUCTS, INC., Assees. of (A) A. R. OLPIN, and (B) G. R. STILWELL (B.P. 352,012 and 352,057, [A] 27.3.30, [B] 29.3.30. U.S., [A] 6.4.29, [B] 18.4.29).—(A) The cell contains an electrode coated with Co the light-sensitivity of which is increased by exposing it in vac. to the action of S vapour in the presence of a trace of  $\text{H}_2\text{O}$  vapour with or without a small quantity of the

vapour of an organic dye, preferably of the cyanine group. (B) Na is used as the light-sensitive metal; after one treatment with S vapour, air is admitted to the cell, which is then again evacuated and a second layer of Na and S is formed on the first.

A. R. POWELL.

**Apparatus for determining the potassium content in spaces containing potassium.** W. KOLHORSTER (B.P. 351,266, 8.7.30. Addn. to B.P. 340,231).—The ionisation set up by the  $\gamma$ -rays emitted by the K are measured by means of an electrical device including a thermionic valve circuit to amplify the current impulses and a rotating impulse relay in the form of a rotative selector as used in automatic telephony.

A. R. POWELL.

**[Manufacture of] piezo-electric substances.** P. FREEDMAN and W. J. RICKETS (B.P. 352,419, 7.4.30).—Preferentially-oriented hemihedral crystals of dielectric material are deposited from a liquid under the combined influence of an electrical field, preferably compounded of direct and alternating components, and high-frequency alternating mechanical stresses. (Cf. B.P. 311,055; B., 1929, 649.)

J. S. G. THOMAS.

**Manufacture of electric insulators.** INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 353,632, 13.6.30. Ger., 13.6.29. Addn. to B.P. 350,429; B., 1931, 767).—The heat treatment (described previously) for hardening the resin consists in contacting the strip with a heated, rotatable drum or roller.

J. S. G. THOMAS.

**Coating of articles [copper wires with insulating compositions].** ELECTRICAL RES. PRODUCTS, INC., Assees. of T. K. COX (B.P. 353,911, 29.4.30. U.S., 8.6.29).—Tinned Cu wire is treated with oleic, stearic, or palmitic acid so as to form a film of Sn salt on its surface. A layer of rubber insulating material is then extruded on the surface of the wire and subsequently vulcanised whereby it adheres strongly to the wire owing to the binding action of the Sn salt.

A. R. POWELL.

**Method of and composition for cleaning and vulcanising materials [used in insulating electrical conductors].** ELECTRICAL RES. PRODUCTS, INC., Assees. of F. S. MALM (B.P. 353,437, 22.4.30. U.S., 30.4.29).—Ends of submarine cables etc. to be joined are washed with a solution composed of a solvent, e.g.,  $\text{CS}_2$  and/or  $\text{CCl}_4$ , a vulcanising agent, e.g., S, and an ultra-accelerator, e.g., Zn butyl xanthate or one of the Zn alkyl xanthate or dithiocarbamate group.

J. S. G. THOMAS.

**[Winding of coreless] electric induction [crucible] furnaces.** ASSOCIATED ELECTRICAL INDUSTRIES, LTD., Assees. of C. S. WILLIAMS (B.P. 353,270, 6.8.30. U.S., 14.8.29).

**Thermo-electric batteries.** J. NEALE (B.P. 353,521, 25.4.30).

**[Grinding of] piezo-electric [quartz] crystals.** ELECTRICAL RES. PRODUCTS, INC., Assees. of R. A. HEISING (B.P. 351,940, 31.3.30. U.S., 2.4.29).

**Manufacture of loaded electrical conductors.** ELECTRICAL RES. PRODUCTS, INC., Assees. of W. M. BISHOP (B.P. 353,472, 24.4.30. U.S., 6.6.29).

**Radiation pyrometer. Safety mixers. Determining m.p. Examining cryst bodies.—See I. Porcelain.—See VIII. Fe-Ni alloy Cathodes for refining metals. Mg. Zn.—See X. Photographic emulsion for electronic discharge.—See XXI.**

## XII.—FATS; OILS; WAXES.

**Glycerin analysis committee report.** J. T. R. ANDREWS (Oil & Fat Ind., 1931, 8, 297—301).—The apparent glycerol content (of conc. glycerins), as determined by the dichromate method, is only 0.3% (about the experimental mean error) lower than that obtained from sp. gr. (Bosart and Snoddy tables) determinations; a bigger discrepancy (1.1% low) between the values from the acetin (I.A.M. method) and the sp. gr. figures is confirmed, and appears to be due to a defect in the acetin method (? loss of glycerol during saponification).

E. LEWKOWITSCH.

**Susceptibility of fats to oxidative rancidity.** D. P. GRETTE and R. C. NEWTON (Oil & Fat Ind., 1931, 8, 291—294).—A weighed amount of fat is distributed over filter paper and heated at 100° in a standardised air current; the volatile products are collected in acid permanganate, the amount of reduction being measured by titration. Great care in manipulative detail is essential for reproducibility.

E. LEWKOWITSCH.

**Absorption and desorption of water from individual soaps.** E. L. LEDERER (Allgem. Oel- u. Fett-Ztg., 1931, 28, 207—210).—The theoretical implications of the experiments of Godbole and Joshi (*ibid.*, 1930, 27, 76) on the adsorption of  $\text{H}_2\text{O}$  by dry soaps are discussed; inverse experiments on the drying out of aq. soap solutions similarly show a two-stage process, part ( $\frac{3}{4}$ ) of the  $\text{H}_2\text{O}$  being loosely held (straight-line desorption), whilst part ( $\frac{1}{4}$ ) is tenaciously retained in the intermicellar spaces. Hence it is to be expected that curd soaps should dry out less readily than cold-process (or soft) soaps. Alcohol-soap solutions behave similarly in type, but the permanation coeff. is so much greater that the soap-alcohol gel must be practically free from retaining spaces.

E. LEWKOWITSCH.

**Drying oils. XV. Oxidation of linseed oil up to gelation.** J. S. LONG and W. S. W. McCARTER (Ind. Eng. Chem., 1931, 23, 786—791; cf. B., 1931, 401).—The gelling time and  $\text{O}_2$  absorption of linseed oil were studied, the following factors being varied: temp., acid val. (admixture of linseed oil acids), complexity (heat-bodied oils and different esters of linseed oil acids), unsaturation, presence of driers and inhibitors. Gelation is shown to occur when a certain degree of complexity or polarity of the mols. is reached, oxidation being a contributory factor, although not reaching completion. Free fatty acids unite with linseed oil, aiding gelation although diminishing  $\text{O}_2$  absorption. Elevation of temp. and presence of driers accelerate one or more reactions leading to gelation (e.g., Pb has a specific associating function), whilst inhibitors merely retard oxidation. The partition of  $\text{O}_2$  between gel and volatile products and the composition of the latter are also recorded.

S. S. WOOLF.

**Coagulation and analysis of stand oils containing tung oil.** H. WOLFF and I. RABINOWICZ (Chem. Umschau, 1931, 38, 213—216).—Erratic results were obtained by the modified (and improved) Scheiber  $\text{SnCl}_4$  method (Farbe u. Lack, 1930, 513) for stand oils and varnishes containing raw and/or bodied tung oil: even 50% mixtures of the latter with rape and arachis oils gave values 43% and 37%, respectively. The coagulate obtained contains other acids (including stearic acid) besides elaeostearic acid.

E. LEWKOWITSCH.

**Re-liquefaction of coagulated tung oil.** R. BÜRSTENBINDER (Chem. Umschau, 1931, 38, 229—230).—Recovery of overheated tung oil may be possible if condensation (cf. B., 1930, 217) has not proceeded too far; alcoholic HCl, raw oil, etc. assist resolution by trans-esterification.

E. LEWKOWITSCH.

**Specifications for oil-extraction solvents.** R. H. FASH (Oil & Fat Ind., 1931, 8, 311—312, 317).—The amount of extract obtained (e.g., from cottonseed) increases with the temp. of extraction. A solvent of pentane type, containing a min. of isopentane, isohexane, and hexane (initial b.p. between 35° and 40°, at least 95% distilling below 55° and not over 85% below 40°), is preferred. Cottonseed extracted at 80° yielded a solid dark resinous material on re-extraction in the usual manner.

E. LEWKOWITSCH.

**Sandal-seed oil.** M. SREENIVASAYA and N. NARAYANA (Proc. XV Indian Sci. Cong., 1928, 160).—The oil (50—55%) thickens in light; at 130—140° S is absorbed and a rubber-like compound is obtained. The oil had  $n_D^{60}$  1.4790,  $d_4^{30}$  0.9304, I val. 130—140, sap. val. 185—195, acid val. 20—25, Hehner val. 96.97, Reichert—Meissl val. 1.5, Polenske val. 0.2—0.3; mol. wt. of fatty acids 288.5.

CHEMICAL ABSTRACTS.

**Some lesser known Brazilian palm oils.** I. F. W. FREISE (Chem. Umschau, 1931, 38, 216—219).—The botanical identity, yields, application, and consts. of the oils from 34 native varieties are tabulated.

E. LEWKOWITSCH.

**Kapok oil and the Halphen test.** H. P. TREVITHICK and W. H. DICKHART (Oil & Fat Ind., 1931, 8, 305, 317).—Halphen tests on mixtures with sesame, olive, palm-kernel oils, etc. were made; in admixture with kapok oil the colour is developed more rapidly and the depth (after 5 hr.) is equivalent to that produced by ten times the proportion of cottonseed oil. With >1% of kapok or >5% of cottonseed oils the colour is too deep for quant. estimation of the contaminant.

E. LEWKOWITSCH.

**Solubility of fats in various solvents. Solubility of (V) camellia oil, (VI) rape oil, in isopropyl alcohol of various concentrations.** K. HASHI (J. Soc. Chem. Ind. Japan, 1931, 34, 224—225 B, 226—227 B; cf. B., 1931, 550).—Solubilities are given for alcohol concentrations from 85% to 100%. Crit. temps. with corresponding oil concentrations rise rapidly with dilution of the alcohol, the values for 100% alcohol being (V) 7.8°, (VI) 29.3°.

C. IRWIN.

**Detecting the ageing of oils, particularly castor oil, by capillary analysis.** A. BOUTARIC and (MLE.)

M. ROY (J. Pharm. Chim., 1931, [viii], 14, 113—123).—The diminution in the surface tension between 0.002N- $\text{Na}_2\text{CO}_3$  and a 1% solution of the oil in  $\text{C}_6\text{H}_6$  forms a more rapid and accurate means of detecting ageing than does the determination of the acid val.; moreover, a very small amount of sample is required. Results obtained with  $\text{Na}_2\text{CO}_3$  are more reliable than those with NaOH, but both are influenced by concentration and the time taken for the drops to form.

T. MCLACHLAN.

**The sulphur monochloride reaction of fatty oils. II. Nature of the reaction product. III. Thermal behaviour of their fatty acids. IV. Evolution of hydrogen chloride.** E. H. HARVEY and H. A. SCHUETTE (Trans. Wisconsin Acad. Sci., 1931, 26, 225—229, 231—232, 233—239; cf. B., 1931, 727).—II. The min. quantity of  $\text{S}_2\text{Cl}_2$  necessary to convert oils into solid products cannot be correlated with the I val. With olive, rape, cottonseed, tung, and cod-liver oils the whole of the S (with corn oil 90%) but only a part of the Cl of the  $\text{S}_2\text{Cl}_2$  added was found in the factice. Substitution as well as addition appears to have occurred.

III. In the reaction under standardised conditions in the presence of a diluent (xylene) the max. temp. rise is greater, and the time required to attain it is less, for the fatty acids than for the respective parent oils.

IV. The amounts of HCl evolved during reaction for 1 hr. (in  $\text{CHCl}_3$ ) cannot be correlated with the Ac or I vals. of the oils, nor are the figures additive for mixtures. Absence of sunlight retards the substitutive reaction.

E. LEWKOWITSCH.

**Sulphonated oils. V. Preparation and properties of the chemically pure alkali acid salts of ricinoleic sulphuric ester and chemistry of acid-salt formation in the sulphuric acid esters of the hydroxy-acids. VII. Properties of commercial sulphonated oils.** K. NISHIZAWA and K. WINOKUTI (Tech. Rep. Tôhoku, 1931, 10, 93—115, 116—130).—V. [With T. KIKUTI.] A full account in German of work published in Japanese (A., 1930, 321; cf. B., 1930, 568; 1931, 307). The formation and the prep. of the salts by treatment of ricinoleic sulphuric ester with alkali salts, including  $\text{Na}_2\text{SO}_4$ , and the properties of the salts (including crystallographic data) and of their aq. solutions (surface tension, viscosity, etc.) are detailed. The acid Na salt tends to form an anhydride (carboxylic) when its alcoholic solution is dried with anhyd.  $\text{Na}_2\text{SO}_4$ .

VII. [With S. IGARASI.] A German translation with full details of the Japanese paper, Part IX (cf. B., 1931, 211).

E. LEWKOWITSCH.

Yolk in wool.—See V.

## PATENTS.

**Manufacture of shaving soaps.** A. C. J. PARENT (B.P. 354,056, 6.8.30. Fr., 28.8.29).—5—10% of non-toxic (e.g., Et) esters of phthalic acid and/or of *p*-aminobenzoic acid is added to the soap. E. LEWKOWITSCH.

**Semi-continuous dry-rendering and meal manufacturing process.** W. B. ALLBRIGHT (U.S.P. 1,791,439, 3.2.31. Appl., 30.3.25).—The high-grade fat is drained off after dry-rendering the material, until the moisture

content of the residue is 20–40%, *i.e.*, greater than the crit. amount (about 16%) below which discoloration occurs, and the material is re-treated until sufficiently dry for the remaining second-grade fat to be easily expressed. E. LEWKOWITSCH.

**Heat treatment of linseed oil [for varnishes, linoxyn, etc.].** E. ASSER, and G. RUTH A.-G. (B.P. 353,949, 30.4.30. Ger., 1.5.29).—Linseed oil is pre-heated to 280° and blown with air while heating is continued up to 310°. E. LEWKOWITSCH.

**Stabilisation of greases etc. Lubricant for textiles.**—See II. Disinfection.—See XXIII.

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

**Turpentine from pines.** T. H. McCOMBS (New Zealand J. Sci. Tech., 1931, 12, 333–337).—*Pinus radiata* and *P. pinaster* produced a first-grade turpentine. Yields and characteristics of the products are recorded.

A. G. POLLARD.

**“Chalking” [of paint films]. V. Practical trials of chalking of oil paints. II.** R. KEMPF (Farben-Ztg., 1931, 36, 1972–1975, 2010–2013).—A further interim report is given after 20 months' exposure of the tests previously reported up to 10 months (B., 1931, 552). Full details of degree of chalking and extent of cracking over the whole period are quoted and graphed. No striking developments occurred in the second 10 months, the general superiority of  $PbCrO_4$  mixtures being maintained. White lead–lithopone mixtures, however, show increased chalking. The tests continue.

S. S. WOOLF.

**Evaluation of “chalking” of paint [films].** H. WOLFF and G. ZEIDLER (Farben-Ztg., 1931, 36, 1930–1932).—The loss of wt. during 17 months and the degree of “chalking” (by Kempf's method) of films of white and grey paints based on basic sulphate and basic carbonate white lead, with and without  $Pb_3O_4$  priming, were determined. Grey paints, in general, lose less wt. than do white. Chalking is shown to be a surface effect only, not necessarily paralleled by the loss of wt. which is a function of the whole film, as is illustrated by the differences between the results with and without the  $Pb_3O_4$  undercoat. Results on a further series of mixed-pigment paints are also quoted. Kempf's “stamping” method for measuring chalking is approved, in general, and its application to the measurement of freedom from “tack” is indicated. The integration of the chalking curve as recommended by Kempf is, however, considered unsound, since the chalking process is not regular and false evaluation results from integration.

S. S. WOOLF.

**Difficulties in the manufacture of oil and lacquer colours.** R. BÜRSTENBINDER (Farbe u. Lack, 1931, 122).—Faults in ready-mixed paints or paste colours may be traced to inaccurate additions of constituents or to unsuitable combinations of dry colours. The breaking down of paint films is frequently due to faulty drying of the media, and the rough matt surface given by some lacquers can be attributed to a reaction with the plasticiser. In the case of mixed pigments, cloudiness of the paint film and variations in tone are generally the result of a reaction between the colours themselves

and/or the medium, or to a chemical change in one of the constituents during drying. F. C. HARWOOD.

**Test for predicting the durability of varnishes. [The photochemical embrittling test.]** J. H. WILSON (Bur. Stand. J. Res., 1931, 7, 73–83).—The film of varnish is exposed to an intense source of light for a definite time, after which it is bent double at a prescribed temp. over a rod of fixed diameter. The more durable is the varnish, the longer it may be exposed to the light before it will crack on bending. A number of advantages over the methods commonly in use are given, particularly in simplicity and speed. The test is not sufficient for pigmented coatings which also fail by chalking, fading, loss of gloss, etc. E. S. HEDGES.

**Rosin in lacquer and oil colours.** R. SCHWARZ (Farbe u. Lack, 1931, 123–124).—For use in lacquers rosin is heated with tung oil to 275–300°, followed by an addition of CaO or ZnO etc. at about 250°. The CaO required for hardening is calc. from the acid val. of the rosin, and must be freshly slaked, finely powdered, and free from  $CaCO_3$ , or the rosin boilings become mouldy and lumpy. Addition of a small quantity of a solution of rosin in  $C_6H_6$  or turpentine to a tung oil lacquer remedies polymerisation. Resin esters formed by the addition of approx. 10% of glycerin to the rosin give a more durable lacquer film. F. C. HARWOOD.

**Cellulose nitrate for lacquers. Pine oil.**—See V.  **$Pb_2O$ .**—See VII. **Drying oils.**—See XII. **Colours for rubber.**—See XIV.

### PATENTS.

**Polishing [of cellulose lacquer surfaces].** BRIT. CELANESE, LTD. (B.P. 352,547, 12.4.30. U.S., 26.4.29).—A polish for surfaces composed of an org. derivative of cellulose and a synthetic resin consists of an abrasive, a diluent ( $C_6H_6$ ), and a liquid which has a softening effect on the surface. For resins made from  $CH_2O$  and substituted sulphonamides the latter are used as softening liquids; for phenol- $CH_2O$  resins Et phthalate, diphenylpropane, or triacetin may be used. D. K. MOORE.

**Manufacture of plates, section pieces, etc. from phenol-formaldehyde artificial resins.** DR. F. RASCHIG GES. M.B.H., Assees. of DR. F. RASCHIG CHEM. FABR. (B.P. 353,870, 22.4.30. Ger., 20.4.29).

**Mixing phenol compounds.**—See I. **Pigmented cellulose esters etc. Stencil sheets.**—See V. **Grinding wheels.**—See VIII. **Linseed oil.**—See XII. **Reaction products of rubber [as lacquer]. Vulcanisation moulds.**—See XIV.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Colours used in the rubber industry.** G. F. THOMPSON and E. V. BRATBY (J. Oil Col. Chem. Assoc., 1931, 14, 218–241).—A review of some of the desired characteristics in colours for rubber is followed by detailed reference to particular materials or types.

D. F. TWISS.

**Action of titanium dioxide in rubber mixings.** R. DITMAR (Caoutchouc et Gutta-Percha, 1931, 28, 15,640–15,643).—The methods used for preparing amorphous

TiO<sub>2</sub> are reviewed. Experiment shows that rubber containing TiO<sub>2</sub> and S<sub>2</sub>Cl<sub>2</sub> has a good white colour, but the pigment tends to soften the rubber and a slightly greater degree of vulcanisation is necessary. In heat-vulcanisation, TiO<sub>2</sub> resembles ZnO in activating org. accelerators, but has the additional advantage of being non-toxic. The softening influence of TiO<sub>2</sub> in larger proportions is effective in preventing scorching of rubber mixtures during the preliminary milling operations.

D. F. TWISS.

#### Increase of the resistance of vulcanised [rubber] products to wear by tin and cadmium selenides.

R. DITMAR and C. H. PREUSSE (Caoutchouc et Gutta-Percha, 1931, 28, 15,639—15,640).—In rubber vulcanised with S<sub>2</sub>Cl<sub>2</sub> or with S in the absence of an org. accelerator, CdSe and SnSe have neither reinforcing nor accelerating influence. In the presence of most org. accelerators, however, vulcanised products containing either of these selenides show marked reinforcement and increased resistance to abrasion.

D. F. TWISS.

#### PATENTS

#### Manufacture of natural rubber and artificial rubber-like masses.

A. CARPMAEL. From I. G. FARBENIND A.-G. (B.P. 352,549, 12.4.30. Cf. B.P. 333,941; B., 1930, 1017).—Aliphatic aldehydes are condensed with  $\alpha$ -naphthylamines or their substitution derivatives in the presence of H<sub>2</sub>O-sol. alcohols and acids, and the primary resin-like products are treated with H<sub>2</sub>O, preferably with heating. The products, which are yellowish inodorous powders, sparingly sol. in EtOH, are used as antioxidants for rubber and do not interfere in any way with the normal course of vulcanisation.

D. F. TWISS.

#### Manufacture of rubber or similar material.

DUNLOP RUBBER CO., LTD., ANODE RUBBER CO., LTD., and E. W. MADGE (B.P. 351,937, 31.3.30).—Porous or microporous articles of vulcanised rubber are obtained by adding to compounded latex one or more reagents which are substantially non-coagulants, but which effect coagulation on the application of heat. NH<sub>4</sub> salts of strong acids act in this way in latex containing ZnO. Coagulation or gelling is then induced by heat in one or more operations such as moulding, spreading, or dipping. Vulcanisation is effected under conditions preventing the escape of the dispersion medium from the pores of the product.

D. F. TWISS.

#### Manufacture of rubber.

IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 352,781, 21.8.30. U.S., 22.8.29).—Rubber articles are protected against the effects of heat as well as of O<sub>2</sub> by incorporating a compound of the formula NH<sub>2</sub>·R'·NH·R''·Y, where R' and R'' represent aromatic residues and Y represents H, OH, or NH<sub>2</sub>. The aminodiphenylamines and *N*-aminophenyl-naphthylamines are typical.

D. F. TWISS.

#### Manufacture of rubber and the like. [Anti-ageing compounds.]

IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 349,391, 29.8.30. U.S., 30.8.29).—Tetraaminobenzhydrols, carrying, if desired, nuclear alkyl or alkoxy or *N*-alkyl or -aralkyl substituents, are added to a rubber mix or applied to vulcanised rubber as antioxidants.

C. HOLLINS.

**Manufacture of goods of rubber or similar material.** DUNLOP RUBBER CO., LTD., ANODE RUBBER CO., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 351,938, 31.3.30. Addn. to B.P. 327,451; B., 1930, 626).—Using the method of procedure described previously an aq. dispersion of rubber containing disintegrated org. material such as ground rubbered cotton waste, cork powder, or leather dust, is converted into a crumb-like coagulum by the addition of compounding ingredients of the type already indicated. The crumb-like compositions may be shaped as such or dried and shaped, *e.g.*, by moulding, with or without previous milling.

D. F. TWISS.

**Manufacture of coloured rubber masses.** A. CARPMAEL. From I. G. FARBENIND A.-G. (B.P. 352,506, 10.4.30).—An alkaline-earth salt of a sulphonated azo dye prepared from a nitroaniline and an acetoacetic arylyde, *e.g.*, the Ca salt of the azo compound derived from 5-nitro-2-aminobenzoic acid and acetoacetic-2-anisidide-sulphonic acid or from *o*-nitroaniline-4-sulphonic acid and acetoacetic-2-anisidide, is used exclusively or in part as a colouring agent for manufactured rubber.

D. F. TWISS.

#### Manufacture of reaction products of rubber.

DUNLOP RUBBER CO., LTD., S. A. BRAZIER, E. H. HURLSTON, and E. V. BRATBY (B.P. 352,080, 5.4.30).—The products obtained by treating rubber with an org. nitroso-compound, *e.g.*, *p*-nitrosodimethylaniline, in substantially greater proportions than those used for satisfactory acceleration of vulcanisation have strong adhesive properties and can be applied as a lacquer or bonding agent for rubber to rigid impermeable surfaces such as of metal.

D. F. TWISS.

#### Process and compounds for treating rubber.

ROESSLER & HASSLACHER CHEM. CO. (B.P. 352,746, 18.7.30. U.S., 2.8.29).—The hexamethylenetetramine benzyl ester of a mercaptothiazole, obtained by the interaction of the additive product of a benzyl halide and hexamethylenetetramine with a metal salt of a mercaptothiazole, is applied as an accelerator of vulcanisation. The product from mercaptobenzthiazole has m.p. 140°.

D. F. TWISS.

#### Vulcanisation accelerators [for rubber] and application thereof.

IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, W. J. S. NAUNTON, and W. A. SEXTON (B.P. 352,165, 17.5.30).—Vulcanisation is accelerated by a compound of the formula R' <math>\begin{matrix} \text{N} \\ \diagup \quad \diagdown \\ \text{S} \end{matrix}> \text{S} \cdot \text{S} \cdot \text{C} <math>\begin{matrix} \text{N} \\ \diagup \quad \diagdown \\ \text{S} \end{matrix}> \text{R}''</math>, (R' and R'' being aromatic residues) with or without the additional presence of a basic org. accelerator such as diphenylguanidine. The products are obtained by the action of a compound R <math>\begin{matrix} \text{N} \\ \diagup \quad \diagdown \\ \text{S} \end{matrix}> \text{S} \cdot \text{Cl}</math> (R being an arylene radical) on a 2-mercaptoarylenethiazole. They are free from tendency to prevulcanisation.

D. F. TWISS.

#### Vulcanisation of rubber and rubber-like substances.

IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, W. J. S. NAUNTON, and W. A. SEXTON (B.P. 352,133, 26.4.30).—The reaction products of 2-mercaptoarylenethiazoles and a halogenated benzoquinone, *e.g.*, chloranil, containing a reactive halogen atom, accelerate vulcanisation without inducing scorching during



the preliminary milling operations. They are preferably used in conjunction with a basic org. accelerator, e.g., diphenylguanidine. D. F. TWISS.

**Vulcanisation moulds.** BAKELITE CORP., Assecs. of G. W. CROSBY (B.P. 352,519, 6.3.30. U.S., 7.3.29).—A mixture of a thermo-setting phenolic resinoid (approx. 40%) with finely-divided mica (5–60%), the balance, if any, consisting of org. or inorg. fillers such as wood flour, asbestos, or Al or other metallic powder, is used to form the contact surface in a mould for the vulcanisation of rubber. D. F. TWISS.

**Determining plasticity.**—See I. **Rubber-impregnated fibres.**—See V. **Substances containing S.**—See VII. **Coated Cu wires.**—See XI.

## XV.—LEATHER; GLUE.

**Behaviour of leather towards free sulphuric acid.** RIMARSKI, PLEUS, FRIEDRICH, STREB, NOACK, KEMPER, HOFFMANN, and KONSCHACK (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 84–88; Chem. Zentr., 1931, i, 2427).—Immerheiser's method is unacceptable. In Paessler's method with ox or calf leather the value of the acid found diminishes with diminution of the concentration of the  $H_2SO_4$ . Calf leather takes up about 50% more  $H_2SO_4$  than does ox leather. The extraction loss of the leather and the N content of the Procter extract fall with decreasing  $H_2SO_4$  concentration.

A. A. ELDRIDGE.

**Testing of leather preservatives and impregnating materials.** RIMARSKI, PLEUS, FRIEDRICH, STREB, NOACK, KEMPER, HOFFMANN, and KONSCHACK (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 89–92; Chem. Zentr., 1931, i, 2427–2428).—The composition of commercial materials is recorded. To test whether the impregnating material causes any change in the leather the former is removed and the total N,  $H_2O$ -sol. N, and extraction loss of the latter are compared with values obtained for untreated leather. A. A. ELDRIDGE.

PATENTS.

**Apparatus for manufacturing pellet glue.** T. K. LOWRY (B.P. 353,881, 28.4.30).

**Insecticide.**—See XXIII.

## XVI.—AGRICULTURE.

**Secular and seasonal changes in soils.** J. S. BURD and J. C. MARTIN (Hilgardia, 1931, 5, 455–509).—The soils examined were maintained under controlled conditions. If water is withheld between seasons losses of N from low-fertility soils under continuous cropping are very small and some gain in N may occur. Under similar conditions substantial increases in N occur in fallowed uncultivated soils. The C:N ratio of continuously cropped soils remains the same as when prolonged fallow periods intervene between crops. The total concentration of the soil solution is reduced by cropping and usually, but not invariably, increased by fallowing. The  $[SO_4^{--}]$  tends to increase in both fallow and cropped soils.  $NO_3^-$  increases in fallow and falls in cropped soils. A significant decline in the  $[K^+]$  occurs in many cropped soils. After depletion by cropping the subsequent increase in the concentration of the soil

solution may be fairly rapid. Whatever their previous history, cropped soils lose more  $NO_3^-$  than is absorbed by the crops, the loss being very heavy in high- $NO_3^-$  soils. The cropping of soils after prolonged fallowing results in a rapid decline in total concentration and a corresponding reduction in crop yield. A. G. POLLARD.

**Characteristics of an eroded soil.** G. W. MUSGRAVE and H. DUNLAVY (J. Amer. Soc. Agron., 1931, 23, 245–252).—Eroded soil exhibited a downward trend of moisture and relative  $H_2O$ -holding capacity, loss of org. matter, decrease in colloids and moisture equiv., and a lower percentage of clay. CHEMICAL ABSTRACTS.

**Podsollic process in soils.** T. J. DUNNEWALD (J. Amer. Soc. Agron., 1931, 23, 259–265).—Laboratory experiments indicate that org. matter is not concerned in the podsollic accumulation of  $SiO_2$ , but is concerned in the translocation of bases and sesquioxides. Extraction of the bases and formation of acid conditions appear to be necessary preliminaries to deposition of  $Fe_2O_3$  and  $Al_2O_3$  in subsoil. Timbered and grass soils are compared. CHEMICAL ABSTRACTS.

**Humus formation and destruction by certain higher filamentous fungi in forest soils.** R. FALCK (Forst-Archiv, 1930, [16]; Bied. Zentr., 1931, 60A, 252–253).—The action of fungi on the lignin and cellulosic matter of beech leaves may result in a "corrosion" process ultimately yielding humus, or a destructive process characterised by the production of sugars and org. acids, notably  $(CO_2H)_2$ . The formation and maintenance of forest humus is examined in the light of these facts. A. G. POLLARD.

**Variability of the buffer value of soils.** S. GOY [with P. MÜLLER and O. ROOS] (Z. Pflanz. Düng., 1931, 21A, 257–270).—The buffer power of a soil is dependent on the proportion of sol. basic or neutral salts present, and values recorded by different methods for its determination vary accordingly. Soils highly saturated with bases have lower buffer capacities than those deficient in bases. The reduction of the buffer powers of soils by liming is due to the resultant increase in particle size. The utilisation of these results in the quant. examination of the effects of CaO on the physical properties of soils is discussed.

A. G. POLLARD.

**Base-exchange property of organic matter in soils.** W. T. McGEORGE (Arizona Agric. Exp. Sta. Tech. Bull., 1930, No. 30, 181–213).—The exchange capacity of highly org. soils is a linear function of their C content, but bears no relation to the N content or C:N ratio. Base exchange in highly org. soils takes place in chemically equiv. proportions. Loss of exchange capacity following treatment with  $H_2O_2$  is a linear function of the amount of org. matter destroyed. Digestion with  $H_2O_2$  does not affect the exchange capacity of natural bentonites, but reduces that of synthetic zeolites. Lignin, lignohemicellulose, and lignocellulose function as exchange compounds and each has a definite equiv. exchange capacity. Tervalent ions (e.g.,  $Al^{+++}$ ) cannot enter the org. exchange complex by replacement of mono- and bi-valent ions. Lignin-like substances suffer a partial loss of exchange capacity

by leaching with  $\text{AlCl}_3$  solution. This is regained by treatment with a solution of a hydroxide or of a salt yielding OH ions by hydrolysis. A. G. POLLARD.

**Organic compounds associated with base-exchange reactions in soils.** W. T. McGEORGE (Arizona Agric. Exp. Sta. Tech. Bull., 1931, No. 31, 215—251).—The base-exchange capacity of soil lignins and "ligno-humates" varies with soil type. The aq.-alkali-sol. ligno-humate has a capacity of 382 milliequiv. and the alcoholic-alkali-sol. portion 116 milliequiv. per 100 g. Leaching with HCl increases the capacity of lignin and ligno-humates, probably by hydrolysis. "Lignin acid" and "ligno-humic acid" (H-saturated lignin and ligno-humate) are, respectively, di- and tetra-basic. The amount of base absorbed from an acetate solution by lignin is equal to that necessary to neutralise the lignic acid. The degree of ionisation of the Na and K compounds of the org. complexes is relatively high and that of Ca, Ba, and H compounds low. Xylan has a slight and synthetic humus made from xylan a considerable exchange capacity. Xylan, lignin, and ligno-humates absorb colour from basic dyes and the colour is replaceable by the base of a neutral salt. Dry-ground lucerne has a definite exchange capacity which is not easily destroyed by  $\text{H}_2\text{O}_2$  and increases considerably during the spontaneous decomp. of the material. The lignin content of the org. matter of highly org. soils is a linear function of the base-exchange capacity. No similar relationship exists with the cellulose or hemicellulose. A. G. POLLARD.

**Dispersion of soils by a supersonic method.** L. B. OLMSTEAD (J. Agric. Res., 1931, 42, 841—852).—The mechanical dispersion of soil in  $\text{H}_2\text{O}$  is produced by means of supersonic waves. The degree of dispersion is of the same order as that obtained in the rubbing method, but is more quickly reached. By neither method is the extraction of colloidal matter complete, small and decreasing amounts being obtained by successive treatments. A. G. POLLARD.

**Determination of the total surface area of soils, clays, and similar substances. III. Effect of the salt content on the water absorption of soils and clays.** H. KURON (Z. Pflanz. Düng., 1931, 21A, 271—300; cf. B., 1931, 37).—Both the capillary-bound and the adsorbed  $\text{H}_2\text{O}$  of clays etc. can act as solvent for salts. In the system Na-clay—NaCl— $\text{H}_2\text{O}$ , all the bound  $\text{H}_2\text{O}$  has solvent powers only when the NaCl concentration is high. In the system Na-clay— $\text{Na}_2\text{CO}_3$ — $\text{H}_2\text{O}$  there is a positive adsorption of NaOH by the clay. The nature of the adsorption isotherm of a Na-clay is not affected by the addition of Na salts. In the systems Ca-clay—NaCl— $\text{H}_2\text{O}$  and Ca-clay— $\text{Na}_2\text{CO}_3$ — $\text{H}_2\text{O}$  the initial exchange reaction is followed by increased adsorption. Changes in the  $\text{H}_2\text{O}$ -absorbing power of clays saturated with Ca, Mg, or H brought about by the addition of increasing amounts of  $\text{Na}_2\text{CO}_3$  may be utilised for the indirect determination of the base-adsorptive capacity. The application of such a method to soils rich in  $\text{CaCO}_3$  is noted. A. G. POLLARD.

**Determination of total carbon [in soils].** E. WINTERS and D. C. WIMER (J. Amer. Soc. Agron., 1931,

23, 280—285).—The loaded boat is moved magnetically into the centre of the furnace after the flow of  $\text{O}_2$  has been started. The error is  $\pm 0.55\%$ .

CHEMICAL ABSTRACTS.

**Plant-soluble phosphoric acid in soils in relation to geological soil structure.** L. NEUBERGER (Kunstdünger u. Leim, 1931, 28, 77—78; Chem. Zentr., 1931, i, 2383).—Degrees of P requirement are computed.

A. A. ELDRIDGE.

**Determination of the potash requirement of soil by means of *Aspergillus niger*.** H. NIKLAS, H. POSCHENRIEDER, and G. VILSMEIER (Arch. Pflanzenbau, 1930, A, 5, 152—165; Bied. Zentr., 1931, 60A, 272).—Results of the *Aspergillus* method were in good general agreement with those of the Neubauer test, but there is some divergence in soils of low K content. Repeated use of *Aspergillus* cultures on K-deficient soils leads to a decreased development of mycelium and *vice versa*. Relationships between the wt. of mycelium produced and the K assimilated are not always strictly regular. It is suggested that after the initial growth the more difficultly-sol. K may be assimilated. A. G. POLLARD.

**Effect of superphosphate on the reaction, degree of saturation with bases, and buffer power of acid mineral soils.** L. SCHMITT (Superphosphat, 1930, 6, 219—227; Bied. Zentr., 1931, 60A, 257—258).—Addition of superphosphate to soil produces a temporary increase in acidity which, however, quickly returns to the initial value even in definitely acid soils. The duration of the acid condition is insufficient to incur plant injury. Continuous applications of superphosphate during 29 years had no unfavourable effect on soil reaction, degree of saturation with bases, or the buffer capacity. A. G. POLLARD.

**Solubilisation of plant nutrients by drying and other treatments of soil.** F. ZUNKER (Kulturtechniker, 1930, 6, 591—603; Bied. Zentr., 1931, 60A, 255).—The increased solubility of mineral soil constituents during drying is ascribed to the reduced adsorptive power per unit surface area of the particles. Relationships between the adsorptive power of soils, changes in concentration of the adsorbed and capillary fluids, and the effects of humic acid are discussed. A. G. POLLARD.

**Explosive properties of fertiliser salts containing (1) calcium nitrate, (2) ammonium nitrate and various amounts of oil.** HAID, GOETZE, SELLE, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 108—113, 113—115; Chem.-Zentr., 1931, i, 2381).—The stability of  $\text{Ca}(\text{NO}_3)_2$  depends on the  $\text{H}_2\text{O}$  content; the fertiliser containing 13.2%  $\text{H}_2\text{O}$  is incompletely decomposed in the Pb block test even when the oil content reaches 4%, and it is not detonated by shattering explosives or direct heating. Technical urea— $\text{Ca}(\text{NO}_3)_2$  behaves similarly. Nitrophoska and Leuna saltpetre, when oiled, are not explosive, whilst the explosibility of  $\text{Ca NH}_4$  saltpetre is increased by the presence of oil. A. A. ELDRIDGE.

**Fertilisers and soils in relation to Concord grapes in south-western Michigan.** N. L. PARTRIDGE and J. O. VEATCH (Michigan Agric. Exp. Sta. Tech. Bull., 1931, No. 114, 42 pp.).—Characteristics of the vine soils

are recorded. Application of N fertilisers affected vines 15 ft. (but not at 25 ft.) from the margin of the fertilised area. Variations in the thickness of the humus layer of the surface soil had more influence on the growth and productivity of vines than any other soil factor. Vines weaker at the beginning of a fertiliser trial remained relatively weaker throughout whether fertilised or not. Application of  $(\text{NH}_4)_2\text{SO}_4$  or  $\text{NaNO}_3$  increased the wt. of prunings more than that of matured fruit. The effects of the two fertilisers were similar. Increased growth and productivity followed applications of either superphosphate or KCl with N. Use of both P and K with N gave little additional yield. Increased production following use of fertilisers was primarily the result of actual growth increase, vines of similar vigour yielding similar crops irrespective of the fertiliser used. The response to fertilisers was more marked in weak than in vigorous vines and differences in yield between individual vines is thus reduced considerably. A. G. POLLARD.

**Black cotton soil under different fertiliser treatments.** R. D. REGE (Proc. XV Indian Sci. Cong., 1928, 50—51).—Comparative experiments are described.

## CHEMICAL ABSTRACTS.

**Nitrification of stall manure in arable soils.** VI, C. BARTHEL and N. BENGTON (Centralanst. Versuchswesen Geb. Ackerb. bact. Abt. Medd., 1930, No. 381, 18 pp.; Bied. Zentr., 1931, 60A, 254—255).—The rates of nitrification of the N-constituents of bacterial tissue by pure cultures of various organisms are recorded. Simpler amino-acids are nitrified more quickly and more completely than the more complex. Differences between this type of nitrification and that of cattle manure (50% of the N of which is calc. to be bacterial matter) are ascribed to the presence of colloidal matter which retards bacterial activity, and to the less favourable C : N ratio of the org. matter. Moreover, the varied decomposability of the C compounds of manure introduces a time factor into the action of the bacteria.

A. G. POLLARD.

**Effect of application of manurial salts on the suction force of soil.** H. E. STEINER (Fortsch. Landw., 1930, 5, 550—552; Bied. Zentr., 1931, 60A, 258).—Manuring with 40% K salts and with  $\text{NaNO}_3$  increased the suction force of soil, the effect increasing with falling soil-moisture content.

A. G. POLLARD.

**Treatment of lime-induced chlorosis with iron salts.** J. P. BENNETT (California Agric. Exp. Sta. Circ., 1931, No. 321, 12 pp.).—Methods of spraying, soil treatment, and injection are described.

A. G. POLLARD.

**Effects of various plant foods on growth activities and development of oats.** C. K. McCLELLAND (J. Amer. Soc. Agron., 1931, 23, 304—311).—P increased the number of tillers, heads, and spikelets and the yield and the seed size of oats; it caused early tillering and growth. N behaved similarly; K rather restricted the tillers, heads, and yield.

## CHEMICAL ABSTRACTS.

**Diseases of grain and their control.** W. W. MACKIE (California Agric. Exp. Sta. Bull., 1931, No. 511, 87 pp.).—Methods and formulæ for chemical treatment of seed are recorded.

A. G. POLLARD.

**China aster seed treatment and storage.** W. O. GLOYER (New York State Agric. Exp. Sta., Tech. Bull., 1931, No. 177, 41 pp.).—For the control of fungal diseases, aster seed is soaked for 30 min in  $\text{H}_2\text{O}$  at  $38^\circ$ , drained, and steeped in 1 : 1000  $\text{HgCl}_2$  solution for a further 30 min. at  $38^\circ$ , washed with  $\text{H}_2\text{O}$ , and spread out to dry for 24 hr. Org. Hg compounds were less efficient.

A. G. POLLARD.

**Phosphate slags. Soil corrosion.**—See X.

## PATENTS.

**Fertiliser manufacture.** IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 352,739, 14.7.30. U.S., 15.7.29).—Phosphate rock is partly decomposed with  $\text{H}_2\text{SO}_4$ , further decomp. being then effected by addition of  $\text{HNO}_3$ . Loss of the latter through evolution of nitrous fumes is thus avoided.

W. J. WRIGHT.

**Conservation of unstable chemical fertilisers by coating.** A. MENTZEL (B.P. 352,258, 11.7.30. Ger., 19.7.29).— $\text{NH}_4\text{HCO}_3$  crystals are coated with  $\text{NH}_4\text{Cl}$  by subliming the latter on to the crystals, which are kept cool and stirred during the condensation. Urea may be used instead of  $\text{NH}_4\text{Cl}$ , but sublimation must then be effected in vac.

A. R. POWELL.

**Manufacture of manures.** J. I. BRONN, and CONCORDIA BERGBAU A.-G. (B.P. 352,476, 4.4.30).—Waste  $\text{CaCl}_2$  liquors are caused to react with  $\text{NH}_3$  and  $\text{CO}_2$ , or  $(\text{NH}_4)_2\text{CO}_3$ , or with gas mixtures containing  $\text{NH}_3$  and/or  $\text{CO}_2$ , preferably under pressure, the solution being crystallised or evaporated to give a product consisting of  $\text{CaCO}_3$  and  $\text{NH}_4\text{Cl}$ .

W. J. WRIGHT.

**Fungicide and bactericide.** J. W. ROBERTS (U.S.P. 1,791,430, 3.2.31. Appl., 12.10.28).—The product contains  $\text{ZnSO}_4$  and  $\text{CaO}$ , together with, e.g.,  $\text{CaO}$ -casein and alum. The above in the form of a spray is effective against *Bacterium pruni*.

L. A. COLES.

**Treatment of phosphate material. Compound fertilisers.**  $\text{CaCN}_2$ . Substances containing S.—See VII. Insecticide.—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

**Determination of the sugar content of cane.** P. NEUVILLE (Internat. Sugar J., 1931, 33, 326—328).—That the use of the Java ratio may give an inexact value in calculating the sugar content of the cane was shown by crushing cane in two laboratory mills throughout an entire season, when the polarisation of the juice thus obtained (15.33) differed distinctly from that of the factory juice (14.94). It is concluded that the calculation of the Java ratio based on the juice obtained from small mills cannot be applied to the primary juice of the factory. In two factories in Egypt the sucrose of the sugar bagged, plus that left in the molasses, plus the polarisation of the bagasse, gave a sum which did not correspond to the sugar content of the cane as calc., using values of 85.2 and 85.8. An appreciably higher ratio should have been used, and it would have been raised yet higher had the sucrose content (by double polarisation) been applied in its calculation, instead of the direct polarisation.

J. P. OGILVIE.

**[Sugar juice] carbonatation studies. I. Comparison of the Pšenička and Hruđa processes.** L. DOŠTÁL (Z. Zuckerind. Czechoslov., 1931, 55, 555—563).—Using a small-scale plant, comparative experiments were made with the Pšenička and Hruđa processes, using in different series 0.75, 1.25, and 2.25% CaO. No difference was to be observed in the results given by the two methods so long as the same amount of CaO was used. In the series using 0.75% CaO there was distinctly more colour and a higher content in Ca salts than with the two larger quantities of CaO. On the whole, the 2.25% CaO series showed the best results.

J. P. OGILVIE.

**Course of the first carbonatation [of beet juices]. V. Neutralisation of sugar solutions containing lime, using oxalic acid. VI. Inactivation of calcium oxalate.** J. DĚDEK and D. IVANČENKO (Z. Zuckerind. Czechoslov., 1931, 55, 527—532, 593—596; cf. B., 1931, 560).—V. Laboratory experiments imitating factory conditions of carbonatation, but using oxalic acid for neutralisation, showed the course of the operation to proceed as when CO<sub>2</sub> is used. Entrainment of CaO by the ppt., however, took place only at high temps.

VI. On formation of a ppt. of CaC<sub>2</sub>O<sub>4</sub> in a solution of sugar at temps. below 70°, CaO is carried down, but this property is lost on heating, and in a few min. at temps. above 70°. Addition of alkalis to the suspension of the ppt. hinders this "inactivation."

J. P. OGILVIE.

**Glycerophosphoric acid in the liming and saturation [of beet juice].** B. DLOUHÝ (Z. Zuckerind. Czechoslov., 1931, 55, 431—435).—Factory scum cakes from the first carbonatation contained 1—1.5% P<sub>2</sub>O<sub>5</sub>, including 0.1—0.3% organically combined, probably as glycerophosphate and distearyl glycerophosphate, not directly precipitable by magnesia mixture or molybdate. Cakes from the second and third carbonatations contained 0.56 and 0.19% P<sub>2</sub>O<sub>5</sub>, including 0.46 and 0.11% organically combined. Small quantities of Ca glycerophosphate, added to 15% sucrose solutions, were removed to the extent of over 90% by the usual liming and carbonatation operations, but mainly by adsorption, undecomposed. Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> readily adsorbs Ca glycerophosphate.

J. H. LANE.

**Value of the addition of phosphate in difficult [sugar] juice clarification.** H. D. LANIER (Internat. Sugar J., 1931, 33, 418).—In a factory in Cuba the average phosphate content of the raw juice was 0.022 g. P<sub>2</sub>O<sub>5</sub> per 100 c.c. of juice, but on adding  $\frac{1}{4}$ — $\frac{1}{2}$  lb. of superphosphate (of 50% solubility) per ton of cane ground, and liming to  $p_H$  7.5, a considerable improvement in the brilliancy of the clarified juice was observed. The best results were obtained when the P<sub>2</sub>O<sub>5</sub> content of the juice was raised to 0.033%, which addition was estimated to have increased the capacity of the factory by 10%.

J. P. OGILVIE.

**Influence of composition of sugar juices on their darkening during evaporation.** O. REISS (Z. Zuckerind. Czechoslov., 1931, 55, 515—526).—Employing Staněk's laboratory evaporator (B., 1926, 961) and Šandera's photocolorimeter graduated in Stammer degrees (B., 1930, 1126), the author conc. 400-c.c.

portions of sugar solutions and beet thin-juices from 15° to 60° Brix in 1 hr. and determined the increase in colour at the original dilution and  $p_H$  val. With solutions of refined sugar no measurable darkening occurred at neutral or acid reactions, even after addition of 0.05% of invert sugar and 0.05% of amino-acid (aspartic acid). At initial reactions of  $p_H$  8.72—10.33, colours of 0.2—0.46° Stammer (at 15° Brix) were produced, with a fall of 0.3 in  $p_H$  val.; these effects were intensified by initial addition of 0.05% of invert sugar and much more by addition of 0.05% of aspartic acid as well. Beet thin-juices suffered a slight fall in  $p_H$  val. and an increase of 30—80% in their colour. This increase was much lessened by initial addition of 0.02% of Na<sub>2</sub>SO<sub>3</sub>, but much increased by addition of 0.001% of FeSO<sub>4</sub>. Raw beet sugars in 15% solution darkened less than thin-juices. Repeated evaporations after redilution to 15° Brix had in most cases about the same darkening effect as the first, or rather less.

J. H. LANE.

**Number of micro-organisms in diffusion [sugar] juice.** J. VONDRÁK (Z. Zuckerind. Czechoslov., 1931, 55, 601—605).—Counts of the micro-organisms present in the diffusion juices from five beet factories gave figures from 800,000 to 12,000,000 per c.c., the lowest being observed at the beginning of the campaign. *Diplococci* colonies were much in evidence, and in some cases a considerable number of *Torula* colonies developed.

J. P. OGILVIE.

**Factors affecting the ash content of [sugar] factory juices.** L. WICHERS (Arch. Suikerind. Nederl.-Indië, 1931, 39, I, 472—477).—The nature of the soil, and its content in mineral matter, particularly CaO, Cl, SO<sub>3</sub>, affect considerably the ash content of the juice, as does also the variety of cane grown. It is a general experience in Java factories that the introduction of the POJ 2878 variety has been accompanied by an increase in the ash content of the factory molasses, as compared, e.g., with cane variety EK 28.

J. P. OGILVIE.

**Chlorine as a decolorant in sugar analysis.** L. R. BLISS (Rev. Ind. Agr. Tucumán, 1930, 20, 180—186).—After treating sugar solutions with basic Pb acetate for their polarisation, dry Cl<sub>2</sub> is passed in, Al(OH)<sub>3</sub> cream added if necessary to remove traces of colloidal Pb, and the liquid finally filtered.

J. P. OGILVIE.

**Conductometric determination of the affining values of raw [beet] sugars.** K. ŠANDERA and C. A. RUŽIČKA (Z. Zuckerind. Czechoslov., 1931, 55, 423—431).—A standardised affination test for raw sugars would often furnish more useful information than the conventional rendement. Comparative study of Šandera's test (B., 1928, 344) and that of Spengler and Brendel (B., 1927, 730; 1928, 30), as applied to 120 Czechoslovakian raw sugars, shows the necessity for an agreed definition of affining value, taking account of the various factors involved.

J. H. LANE.

**Comparative analysis of [beet] sugars in the usual way and by the method of single weighing with conductometric ash determination, and the times required.** A. DOLINEK, J. LINHART, and M. ŠMIDINGEROVÁ (Z. Zuckerind. Czechoslov., 1931, 55,

439—440).—With 265 raw beet sugars, not including after-products, determinations of polarisation and conductometric ash from a single weighing led to rendements differing in no case by more than 0.55% and in 208 cases by not more than 0.25% from the rendements determined in the usual way involving two weighings and an incineration. The rendement could be determined in  $\frac{1}{2}$  hr. by the former method, compared with  $1\frac{1}{2}$  hr. by the latter. J. H. LANE.

**Analysis with Wood's light and the products of sugar manufacture.** G. MEZZADROLI and E. VARETON (Atti III Cong. Naz. Chim., 1929, 580—581; Chem. Zentr., 1931, i, 2277).—The use of the fluorescence method in analysis and control is described. A. A. ELDRIDGE.

**Behaviour of cane molasses during storage.** G. L. C. LA BASTIDE (Arch. Suikerind. Nederl.-Indië, 1930, 38, II, 701—703).—Periodic analysis of a good number of samples of molasses from defecation, sulphitation, and carbonation factories showed that the polarisation gradually falls, though sometimes the sucrose (by double polarisation) remains const. The content of reducing sugars falls, fermentation apparently taking place. Glucose increases, generally in the first month, contents of from 2.22 to 5.67% being observed. J. P. OGILVIE.

**Influence of reducing sugars on the caramelisation test.** J. PUCHERNA (Z. Zuckerind. Czechoslov., 1931, 55, 663—669).—Reducing sugars are decomposed in the caramelisation test parallel with sucrose, but in the presence of the salts and amides of amino-acids, and also of alkali salts, coloured decomp. products are more readily formed from reducing sugars than from sucrose under identical conditions, and they have a more intense colour. J. P. OGILVIE.

**Devitrification of sugar products [e.g., fruit drops].** F. HALLA and E. MEHL (Z. Zuckerind. Czechoslov., 1931, 55, 551—553).—That the change undergone by fruit drops and like vitreous confectionery articles on being exposed to moist air is due to surface recrystallisation is shown by six X-ray photographs, the amorphous ring being exhibited clearly in the case of freshly made drops, and the interference rings in material which had undergone the recrystallisation change. J. P. OGILVIE.

**Determination of the acidity of potato flour.** A. P. SCHULZ (Z. Spiritusind., 1931, 54, 193).—Discordant values of the acidity of potato starch are due to the amount of NaOH required to neutralise a fixed quantity of starch decreasing to a const. as the vol. of  $H_2O$ , with which the starch paste is made, is increased to 100 c.c. The determination of the acidity is standardised by mixing 20 g. of starch with 100 c.c. of "neutralised"  $H_2O$ , adding 10 drops of 1% phenolphthalein, and titrating with 0.1N-NaOH until the red colour is permanent for 1 min. after stirring for 3 sec. New standards of quality of potato flours are chosen as 0.6, 0.8, and 1.2 c.c. of N-NaOH per 100 g. of starch for "fine," "superior," and "first" quality, respectively. C. RANKEN.

**Hydrolysis of starch by oxalic acid in the preparation of [textile] sizes.** W. SIEBER and S. GÄRT-

NER (Textilber., 1931, 12, 105—107).—Results are given showing the amount of dextrose formed by heating potato starch with various concentrations of oxalic acid at  $100^\circ$  for 3 hr. Under such conditions about 50% and 15% of the starch is hydrolysed by 5% and 0.5% solutions of the acid, respectively. The view that  $(CO_2H)_2$  is of special value in preparing sol. starch for sizing purposes because dextrose is not formed simultaneously is shown to be erroneous. A. J. HALL.

**Active carbons.**—See II. Naringin in marmalade.—See XIX.

## PATENTS.

**Treatment of saccharine fluids [with invertase].** L. WICKENDEN, Assr. to J. J. NAUGLE (U.S.P. 1,783,551, 2.12.30. Appl., 3.5.27).—Sugar syrups to be inverted, e.g., affination syrups, are treated, preferably at  $p_H$  4—5 and temp.  $55$ — $65^\circ$ , with syrupy invertase products prepared according to U.S.P. 1,689,607 (B., 1929, 70), and are afterwards clarified if necessary. J. H. LANE.

**Treatment of saccharine fluids.** (A) L. WICKENDEN and (B) J. J. NAUGLE, (A) Assr. to (B) (U.S.P. 1,787,502, 6.1.31. Appl., 28.1.26).—A fluid containing two or more types of impurities is treated by the addition of subdivided adsorbent material of varied degrees of fineness derived from leached carbonised lignin residues, in order to adsorb different types of such impurities, the mixture being stirred and heated to about  $90^\circ$ . Each fraction is separately treated for the removal of the adsorbent material. J. P. OGILVIE.

**Treatment of sugar solutions.** H. DE F. OLIVARIUS, Assr. to CALIFORNIA PACKING CORP. (U.S.P. 1,788,628, 13.1.31. Appl., 27.12.27).—In recovering sucrose from its impure solutions containing invert sugar, it is concentrated to 35—65° Brix, and the invert sugar fermented without material inversion of sucrose. Yeasts of the *Saccharomyces apiculatus* group are not used. J. P. OGILVIE.

**Treatment of sugar juices or syrups.** S. COLE. From F. CHAMIER (B.P. 350,118, 4.4.30).—Raw diffusion juices or syrups, e.g., from suitably or partly dried cane or beet slices, are acidified to  $p_H$  4.3—4.5 with  $H_2SO_4$  at  $20$ — $50^\circ$  to ppt. coagulable matters, then clarified centrifugally, adjusted to  $p_H$  7.4—7.6 with CaO and  $Na_2CO_3$ , and again centrifuged. Further known treatments, e.g., with charcoal, may precede boiling. J. H. LANE.

**Sugar process.** R. E. POSPISIL, Assr. to E. KUREK (U.S.P. 1,772,911, 12.8.30. Appl., 19.5.28).—The process of U.S.P. 1,352,084 (B., 1920, 732) is adapted to enable raw cane sugar to be worked up with beet juice for the production of white granulated sugar without the use of bone char and during an extended working season. J. H. LANE.

**Monosaccharide material [from starch].** T. B. WAGNER (U.S.P. 1,784,402, 9.12.30. Appl., 30.7.23).—Green starch or other amyloseous material is hydrolysed with acid until about 90% of dextrose has been formed and until the dextrans present have lost their gummy properties. After a crop of cryst. dextrose has been obtained by known procedure, the mother-liquor, containing about 20.5% of  $H_2O$ , 57.5% of dextrose,

19.5% of non-fermentable carbohydrates, and 2.5% of ash, constitutes a specially good material for the manufacture of bakers' yeast. J. H. LANE.

**Manufacture of sugar.** DORR Co. (B.P. 350,459, 4.12.29. U.S., 4.12.28).—See U.S.P. 1,755,165; B., 1930, 1085.

**Lixiviation of beetroot slices.**—See I. Saccharification of cellulose.—See V. Artificial wood [from cane trash].—See IX.

## XVIII.—FERMENTATION INDUSTRIES.

**Physical chemistry of the proteins, and some applications in brewing.** I. General. II. Swelling and protection, with special reference to isinglass and finings. R. H. HOPKINS (J. Inst. Brew., 1931, 37, 413—419, 419—424).—I. A review is given of the physical properties of proteins with special reference to the reaction of the medium and to the relationship of the isoelectric point to coagulation, turbidity, viscosity, and surface tension.

II. Although the max. amount of swelling of isinglass in acid solution varies with different acids, the point of max. swelling occurs for all acids at  $p_H$  3.3—3.5 within the swelling gel. The isoelectric point of finings made from isinglass is between  $p_H$  4.8 and 5.2, and the protective action of the finings is at a min. at that point.

C. RANKEN.

**Protein degradation during mashing.** W. WINDISCH, P. KOLBACH, and E. SCHILD (Woch. Brau., 1931, 48, 253—260, 267—273, 286—289, 298—300).—The optimum temp. for production of "permanently sol. N" falls with increasing duration of mashing from about 62° with a 15-min. mash to about 56° with a 3-hr. mash. The corresponding optima for formol-N are 56—60° and 47—51°. The latter action is considered to be in part due to the more heat-sensitive peptidase. A "congress" mash has approx. the same composition as regards total, permanently-sol., and formol N as a 2-hr. mash at 60°. The % formol-N in the permanently-sol. N produced falls from about 40 to 20% as the temp. is raised from 40° to 70°. This may be due to increasing inhibition of the peptidase or to a relatively less formation of simpler decomp. products by the proteinase at higher temps. With increasing duration of mashing the ratio of formol to permanently sol. N increases, probably owing to the continued action of the proteinase on the products first formed, since the effect is very marked at  $p_H$  5.1, at which reaction the peptidase action would be very weak. The optimal  $p_H$  zone for production of permanently-sol. N rises from 4.3—4.7 at 30° to 4.9—5.3 at 70°, that for formol-N being slightly lower at high temps. (4.6—5.0 at 70°). This optimum is referred to the proteinase, since at this reaction the peptidase is inactive, but below 50° the action of the latter is indicated by the higher proportion of formol-N produced at higher  $p_H$  values. The permanently-sol. N is increased by 6% on raising the mashing concentration from 1:5.3 to 1:2.0, in agreement with Schjerner (B., 1913, 985), but not with Oliver (B., 1929, 413). This corresponds to an increase of 12% on the permanently sol. N produced; the proportion of formol-N is slightly raised. The protein degradation is seriously inhibited by

mashing in brass beakers, and to a smaller extent by Ni. F. E. DAY.

**Principles of the malting process.** A. R. LING (J. Inst. Brew., 1931, 37, 424—433).—The structural changes which take place in barley during ripening and which lead to the production of mealiness are reviewed. The changes underlying the malting operations of steeping, flooring, and kilning of barley are also discussed. C. RANKEN.

**Determination of bitter principles of hops.** H. WILDNER (Woch. Brau., 1931, 48, 221—225, 231—235, 241—244, 260—263).—The methods for total resin proposed by Winge and Jensen (B., 1915, 43), Siller (B., 1909, 1056), and Wöllmer (B., 1929, 413), and those of Stádník (Böhm. Bierbr., 1927, No. 29 *et seq.*) and Hastings and Walker (B., 1929, 533) are applied to a varied selection of hop samples. Siller's and Wöllmer's methods give results in close agreement, the former usually slightly the higher. Stádník's method includes a small amount of wax etc., and the results are somewhat higher, whilst those of the Hastings-Walker method are 7.7—15% higher still. The Winge-Jensen method gives low results. For soft resins the Lintner-Adler (B., 1912, 1003), the same using hexane in place of light petroleum, Seibriger (B., 1913, 442), Stádník volumetric, and Siller, Wöllmer, Hastings-Walker, and Stádník gravimetric methods are compared. Seibriger's cold extraction with light petroleum gives low, and with  $CCl_4$  high, results as compared with Lintner and Adler's, in which the use of hexane is preferred to that of light petroleum except for old hops. The gravimetric methods of Siller, Wöllmer, and Stádník agree within the limits of experimental error, with a tendency to be higher in the order stated, and the Hastings-Walker method gives still higher results. The results of the first three methods are 0.1—1.7% higher than those of the Lintner-Adler, and, in general, agree with those of the hexane extraction, except that this tends to give low results with old samples. The tendency of the gravimetric results to be relatively high is attributed partly to small amounts of neutral material in the resin and partly to oxidation during drying. As regards humulone, both the Stádník iodometric and the Hastings-Walker Pb precipitation methods give higher results than the Siller and Wöllmer methods when applied to old samples, though they agree with fresh hops. The difference may be due to impurities in the humulone fraction on the one hand, or to imperfect precipitation of the Pb salt from extracts of old samples on the other, but the pre-extraction with phosphate buffer,  $p_H$  6.4, claimed by Windisch, Kolbach, and Winter (B., 1929, 412) to avoid this was not employed in the single comparison of these authors' method. F. E. DAY.

**Colorimetric method for the determination of the preservative value of hops.** J. M. FRENCH (J. Inst. Brew., 1931, 37, 436—439).—The MeOH used in the Guthrie and Philip method (B., 1930, 1086) for determining the preservative value of hops can be replaced by various grades of EtOH, provided the same alcohol is used both for the determination and for the manufacture of the standard solutions of  $\alpha$ -resins and U salt. 10 g. of minced hops are extracted with 100 c.c.

of the selected alcohol (98% EtOH or redistilled methylated spirit) for 10 min. in a corked bottle, and 0.5 c.c. of the extract after mixing with 10–15 c.c. of the alcohol and 7 c.c. of a 0.7% solution of  $\text{UO}_2(\text{NO}_3)_2$  in the same alcohol is made up to 50 c.c. with the alcohol in a Nessler tube and the colour compared with that of the standard solution similarly treated. As the colour is unstable, the determination should be made rapidly, and the solution of the U salt should be kept in the dark or in amber bottles.

C. RANKEN.

**Effect of high electric potentials on brewers' yeast.** N. FLORESCO and A. RAFALESCO-FLORESCO (Bul. Fac. Stiinte Cernauti, 1927, 1, 384–391; Chem. Zentr., 1931, i, 2352).—The capacity for resisting the action of p.d. of 2–320 volts depends on the age, which can thus be determined.

A. A. ELDRIDGE.

**Hemicelluloses of brewers' grains.** I. A. PREECE (J. Inst. Brew., 1931, 37, 409–413).—Two of the hemicelluloses extracted from the spent grains from malt are urono-xylans, whilst a third is a urono-araban. In no case does the amount of uronic anhydride exceed 9% of the hemicellulose. These hemicelluloses agree closely in composition and properties with those obtained from woods.

C. RANKEN.

**Determination of colour of wort and beer.** A. JANKE and A. SZILVINYI (Woch. Brau., 1931, 48, 281–295).—Comparison with I, dye, or mineral solutions, or with glass slides is subject to inaccuracy owing to differences in spectral composition of the light transmitted. I solutions are less objectionable than the other standards tested, but are unstable. A table and graphs of the readings given by dilutions of 0.1N-I solution containing 0.14–2.0 c.c./100 c.c. in the Zeiss-Pulfrich step-photometer are given, from which it is claimed that very accurate colour determinations may be made. The violet screen S43 is used, which was found unsuitable by Meštán (B., 1928, 725), who, however, employed a longer comparison cell.

F. E. DAY.

**Fermentation in open and closed vessels.** G. VIERECK (Woch. Brau., 1931, 48, 303–306).—In agreement with the results of Windisch (B., 1928, 685; 1929, 69), fermentation in closed vessels improves the appearance and fermentative power of the yeast. The closed fermentations generally proceeded rather more rapidly than those in open vessels, but no differences could be detected in the finished beers.

F. E. DAY.

**The cider industry.** G. WARCOLLIER (Chim. et Ind., 1931, 26, 15–32).—The natural sweetness of cider is due to a slow, incomplete fermentation of a thoroughly defecated juice with a low content of N, and to the action of tannin on the yeast. Juice from apples grown in flinty soils gives good defecation, whilst lime and argillaceous soils yield a rapidly fermenting juice and less stable ciders. Sweet cider can be prepared by arresting the fermentation by centrifuging the fermenting juice under pressure of  $\text{CO}_2$ , and thus decreasing the content of N and withdrawing the yeast. Similar results follow by exclusion of the air from the fermenters and chilling to 2–3°. For addition to cider, juice is conc. in vac. at 45–50° until the content of sugar is 700 g. per litre. Chilling will not raise the concen-

tration of sugar above 450 g. per litre. The juice remains stable provided the content of free  $\text{SO}_2$  is 200 mg. per litre, but 150 mg. per litre are sufficient if the juice is previously centrifuged. Unripe apples should not be used, as the enzyme which coagulates the pectic matters is inhibited by the high acidity of the juice, and the greater amount of starch left in the pulp lowers the content of EtOH in the cider. Cider brandies made by distilling naturally sweet ciders contain an abnormally high content of ethers and have a low volatile acidity.

C. RANKEN.

**Analysis of mixed alcohols.**—See III.

## PATENTS.

**Brewing of beer.** A. E. BERRY (B.P. 353,214, 28.6.30).—As a substitute for the use of patent or crystal malt in the mash tun, pale wort is boiled under pressure after the addition of 0.05–0.1% of  $\text{NH}_3$  or of  $\text{NH}_4$  salts.

C. RANKEN.

**Manufacture of vinegar.** H. FRINGS (B.P. 352,740, 14.7.30. Ger., 6.5.30).—By means of valves in the cooling-water supply and in the mash-outflow pipe, the rate and temp. of infusion are automatically co-ordinated, so that the temp. in individual zones of the filling material in the generator remains const. at an optimum temp.

C. RANKEN.

**Roasting malt etc.**—See I. **Treatment of sugar solutions.** Monosaccharide material.—See XVII.

## XIX.—FOODS.

**Vitamins. IV. Vitamin[-C] contents of common German vegetables.** F. V. VON HAHN (Z. Unters. Lebensm., 1931, 61, 545–610; cf. B., 1931, 740).—Full data and growth curves are given, and the samples are classified as follows: (1) Extremely rich in vitamin: raw white, red, and green cabbages, kohlrabi, and cauliflower; raw and cooked Brussels sprouts. (2) Rich in vitamin: raw swedes, turnips, peas, asparagus, leeks, horseradish, and green beans; cooked green and red cabbages and kohlrabi. (3) Containing vitamin: raw carrots; fresh pickled cabbage, spinach, head-lettuce, yellow beans, and comfrey; cooked turnips, peas, and yellow beans. (4) Poor in vitamin: cooked carrots, green beans, horseradish, and asparagus; raw endive, parsley, radish, pumpkin, and cucumber. (5) Almost free from vitamin: raw and cooked mushrooms and red beetroots; raw chives; old cucumber and radish; cooked pickled cucumber, spinach, comfrey, celery, cucumber, and pumpkin.

J. GRANT.

**Corrosion of metals by milk and its relation to the oxidised flavours of milk.** E. S. GUTHRIE, C. L. ROADHOUSE, and G. A. RICHARDSON (Hilgardia, 1931, 5, 425–453).—Cr-Ni alloys, pure Al, glass enamel and carefully Sn-plated metals showed little corrosion by sweet milk and imparted no flavour to the milk. Pure Ni was very unsatisfactory, and plated Cu or Cu alloys were rendered unsuitable through the rapid mechanical wearing of the plating. Aeration of milk during pasteurisation increased the tendency for the development of oxidised flavours.

A. G. POLLARD.

**Swelling of canned prunes.** E. M. MRAK and P. H. RICHERT (California Agric. Exp. Sta. Bull., 1931, No. 508, 24 pp.).—The effects of various types of

containers and of methods of procedure are examined. Sun-dried and dehydrated prunes after canning swelled at similar rates. The rate of swelling was increased by increasing the time or temp. of blanching and by pressure-cooking, and decreased by raising the concentration of the syrup (> 30% syrup causes shrivelling). Decreasing the  $p_H$  of the syrup decreased swelling but not corrosion. A. G. POLLARD.

**Occurrence of naringin in marmalade made from S. African grape fruit.** B. SEGAL and T. DE KIEWIET (J. S. Afr. Chem. Inst., 1931, 14, 43—46).—During the storage of S. African grape-fruit marmalade white spots developed; these consisted of clusters of white radiating needles of naringenin, which disappeared when the jars were immersed in boiling  $H_2O$  and did not reappear on cooling. Naringin is shown to be a glucoside which on hydrolysis yields naringenin, which appears to be 2:4:6-trihydroxyphenyl 4-hydroxystyryl ketone. A. R. POWELL.

**Use of ethylene oxide (T-gas) in the destruction of pests in the presence of foods.** T. SUDENDORF and E. KRÖGER (Chem.-Ztg., 1931, 55, 549—550, 570—572).—From tests on numerous foodstuffs it is established that the destruction of pests (in rooms or containers used for the storage of foodstuffs) by means of ethylene oxide in concentrations up to 320 g. per cu. m. has no adverse effect on the quality or flavour of the foods provided that the space treated is thoroughly aerated after treatment. A. R. POWELL.

**Grain diseases.**—See XVI.

#### PATENTS.

**Manufacture of products having a saline taste suitable for food purposes.** DEUTS. GOLD- u. SILBERSCHNEIDANSTALT, VORM. ROESSLER (B.P. 352,673, 4.6.30. Ger., 11.6.29).—To obviate stale after-taste, Na glutamate is treated with an org. acid (formic or adipic) to form a stable double salt of pleasant taste.

E. B. HUGHES.

**Drying of tea. Roasting grain etc.**—See I. **Porcelain for cooking.**—See VIII.

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

**Influence of variations of the concentration of alcohol in tinctures.** I. M. PERINO (Boll. Chim. Farm., 1931, 70, 489—492).—When extracted with EtOH of 35, 62.5, and 90% concentrations, *Digitalis purpurea* yields tinctures of approx. the same digitoxin contents. The use of 35—40% EtOH solution is, however, to be preferred, as the tincture obtained (1) withstands dilution without becoming turbid better than that given by the more conc. EtOH and is thus more suitable for pharmaceutical preparations, and (2) contains less dry residue which is inert or disturbing. T. H. POPE.

**Investigation and evaluation of chlorophyll and chlorophyll preparations.** (A) ZICKGRAF, (B) U. DREHMANN (Arch. Pharm., 1931, 269, 427—428, 428—429).—(A) Polemical against Drehmann (B., 1931, 223). The luminescence test should not be used to evaluate the preps. owing their therapeutic activity to chloro-

phyllin. (B) A reply. The therapeutic activity of chlorophyllin in man is unproved. R. K. CALLOW.

**Ipecacuanha preparations and their stability.** E. H. MADSEN (Pharm. Ztg., 1931, 76, 901—902).—Preparations of ipecacuanha ( $p_H$  3.6) made by double infusion in presence of a suitable amount of HCl (25 c.c. N-HCl per 50 g. drug) contain 90% of the total alkaloids, are quite stable, and retained their total activity after 3 years (cf. B., 1931, 780). E. H. SHARPLES.

**Analysis, preparation, and composition of hydrargyrum salicylicum.** F. VIEBÖCK and C. BRECHER (Arch. Pharm., 1931, 269, 398—416).—Na or K hydroxymercurisaliculates are formed by treating hydrargyrum salicylicum (*o*- and *p*-anhydrohydroxymercurisalicic acids) with NaOH or KOH, but the isolated cryst. products are anhydrides of uncertain composition. The method of determining Hg in hydrargyrum salicylicum which depends on the replacement of Hg by I on treatment with I solution is interfered with by side-reactions (hydrolytic demercuration:  $R \cdot HgI + H_2O = RH + IHgOH$ , and nuclear iodination) which may be avoided by methods in which excess of KI is used. Total Hg or organically combined Hg alone may be determined. Na or K hydroxymercurisaliculates are not alkaline to thymolphthalein, and hydrargyrum salicylicum dissolved in excess of alkali hydroxide may be titrated with  $H_2SO_4$  either to the thymolphthalein end-point or, better, until precipitation begins. In a second method,  $Na_2S_2O_3$  is added and the alkali liberated by the reaction  $R \cdot HgOH + Na_2S_2O_3 \rightarrow R \cdot HgS \cdot SO_3Na + NaOH$  is titrated. Thirdly, salicylsalicic acid can be determined by dissolving the substance in 0.01N-NaOH, adding KI, and titrating with acid, using phenolphthalein; excess of acid is then added, the solution refluxed until hydrolytic demercuration is complete, and the excess of acid titrated. The presence of Hg salts is detected by precipitation of Hg, when an alkaline solution of  $N_2H_4$  or of  $CH_2O$  and KBr is added to the solid substance. Analyses have been made of hydrargyrum salicylicum prepared by nine methods, and the amounts of salicylsalicic acid and dimercursalicisalicic acid present as impurities, as shown by the differences in titration figures under different conditions, have been estimated. The presence of Hg salicylate and phenylmercurisaliculate is also indicated in some cases. R. K. CALLOW.

**Determination of the alkaloid content of alkaloid salts. Rapid method for the determination of alkaloids in medicinal preparations.** E. SCHULEK and F. SZEGHÖ (Pharm. Zentr., 1931, 72, 497—506).—To an amount of the substance containing 0.03—0.04 g. of the alkaloid base are added 2 c.c. of  $H_2O$ , 1—2 drops of 10% HCl, and 70 c.c. of  $CHCl_3$ . After shaking well, 3—4 drops of 10% aq.  $NH_3$  are added, the mixture is vigorously shaken, and about 4 g. of  $Na_2SO_4$  are then added in small quantities at a time. The  $CHCl_3$  is filtered off, the residue washed 3 times with 8 c.c. of  $CHCl_3$  each time, and about 90% of the combined  $CHCl_3$  extract is distilled off, 10 c.c. of 0.02N- $H_2SO_4$  are added to the residue, and the remainder of the  $CHCl_3$  is removed on the water-bath. The excess of acid is then titrated with 0.02N-NaOH. Values identical with those given



by the usual extraction methods are obtained and the method is also successfully adapted to the determination of alkaloids in presence of other substances.

E. H. SHARPLES.

**Determination of morphine in opium.** N. RUSTING (Pharm. Weekblad, 1931, 68, 767—774).—The brown coloration observed during the extraction, which persists in the separated morphine and renders titration difficult, is attributed to atm. oxidation of an unstable alkaloid generally present. Addition of  $MnCl_2$  during extraction simplifies the manipulation. The ethereal solution is titrated with  $NaOH$ , using phenolphthalein; if methyl-red be used, results are uniformly higher, probably by reason of Ca meconate present.

S. I. LEVY.

**Microtitrimetric determination of nicotine in tobacco smoke.** L. BARTA and E. TOOLE (Z. angew. Chem., 1931, 44, 682—683).—The smoke from 0.5—1 g. of tobacco is drawn over glass-wool impregnated with dil.  $H_2SO_4$ . The wool is washed successively with hot  $EtOH$  and  $H_2O$  and the washings are made alkaline and distilled with steam. Nicotine in the distillate is precipitated with picric acid. The dipicrate is distilled with steam from alkaline solution and nicotine is determined in the distillate by titration with 0.01N-HCl in presence of methyl-red.

H. WREN.

**Influence of comminution of drugs in determination of essential oils.** L. KOFLER and F. KRÄMER (Arch. Pharm., 1931, 269, 416—422).—Higher values for the content of essential oil are sometimes obtained when drugs are not powdered before steam-distillation. This occurs particularly with labiate and composite plants in which the oil is in glandular hairs or on the surface. The degree of comminution should be specified in each case. Dafert's method is the best, but  $NaCl$  should always be added to the distillate before measuring the oil layer.

R. K. CALLOW.

**Essential oil from the fruits of *Phellodendron Japonicum*, Maxim.** I. K. BRAND and G. WESTERBURG (Arch. Pharm., 1931, 269, 369—383).—The imperfectly ripe fruit yields on steam-distillation 1.1% of an essential oil, from which, by fractional distillation, myrcene may be separated. It has been characterised by conversion into *cis*-4-*isohexenyl*- $\Delta^4$ -tetrahydrophthalic anhydride by treatment with maleic anhydride (Diels and Alder, A., 1929, 819). The amount of condensation product formed directly from the crude oil corresponds to the presence of 78.4% of myrcene.

R. K. CALLOW.

**Use of the analytical quartz lamp for the examination of perfumes.** E. EKMAN and A. SANAYSCHLAYEVA (Riechstoffind., 1930 5, 196—197; Chem. Zentr., 1931, i, 2403).—The fluorescence in ultra-violet light of the following is described: geranyl acetate, benzyl acetate, Et benzoate, *isoamyl* acetate, and benzyl salicylate.

A. A. ELDRIDGE.

**Fe in pharmaceutical preps.**—See XXIII.

#### PATENTS.

**Preparations for immunising against infectious diseases.** E. LOEWENSTEIN (B.P. 352,210, 17.6.30. Austr., 19.6.29. Addn. to B.P. 314,854; B., 1930, 640).

—Salves, protective against diseases other than diphtheria, are prepared by adaptations of the method described previously; absorption of such salves is increased either by increasing the dispersive power or adding substances which irritate the skin.

E. H. SHARPLES.

**Antiseptic and disinfectant solutions.** PEPSODENT Co. (B.P. 352,397, 2.4.30. U.S., 3.4.29).—Dil. chlorothymol solution (0.01—0.5%) is germicidal if acidified to  $p_H$  4.5 or less with an org. acid. The solvents used are  $H_2O$ -miscible monohydric alcohols, the harsh flavour of which is reduced by polyhydric alcohols or sugar products in the case of mouth-wash solutions.

E. B. HUGHES.

**Manufacture of  $\beta$ -hydroxy-derivatives of alkylpyridines.** I. G. FARBENIND. A.-G. (B.P. 348,988, 18.9.30. Ger., 21.10.29. Addn. to B.P. 335,818; B., 1931, 13).—The process of the prior patent is extended to other homologous pyridines. 2-Hydroxy-4-methylpyridine, m.p. 118—120°, b.p. 285—290° (slight decomp.), and 2-hydroxy-6-ethylpyridine, m.p. 170—173°, are obtained from the corresponding sulphonic acids.

C. HOLLINS.

**Manufacture of derivatives of 1-benzyl-3-methylisoquinoline.** G. B. ELLIS. From E. MERCK (B.P. 348,956, 15.7.30).—The Bischler-Napieralski method is applied to the synthesis of papaverine-like compounds containing a 3-Me group. The amide, m.p. 146°, from homopiperonylic acid and  $\beta$ -(3:4-methylenedioxyphenyl)isopropylamine is heated in PhMe with  $POCl_3$  to give a dihydroisoquinoline, m.p. 130° [hydrochloride, m.p. 237° (decomp.)], which is dehydrogenated with Pd-black to give 6:7-methylenedioxy-1-(3':4'-methylenedioxybenzyl)-3-methylisoquinoline, m.p. 141° [hydrochloride, m.p. 254° (decomp.)]. The corresponding homoveratric amide, m.p. 123°, and  $\alpha$ -phenyl-*n*-butyramide, m.p. 138°, give isoquinolines, m.p. 125° [hydrochloride, m.p. 220° (decomp.)] and 143° (hydrochloride, m.p. 80°), respectively. 3-Methylpapaverine, m.p. 136° [hydrochloride, m.p. 234° (decomp.)], is similarly obtained from the  $\beta$ -3:4-dimethoxyphenylisopropylamide of homoveratric acid, and analogous isoquinolines, m.p. 109° and 168—169°, respectively, from the corresponding amides of phenylacetic and homopiperonylic acids.

C. HOLLINS.

**Manufacture of *o*-nitroarylsarmonic acids.** I. G. FARBENIND. A.-G. (B.P. 349,037, 19.2.30. Ger., 19.2.29).—An *o*-nitroarylamine (e.g., 2:4-dinitroaniline, 7-nitro-6-amino- and 6-nitro-7-amino-benzomorpholones, *o*-nitroaniline, 3-nitro-*p*-phenetidine, 3-nitro-*p*-tolyl  $\beta$ -hydroxyethyl ether, 3-nitro-*p*-aminoacetanilide, 5-chloro-2:4-dinitroaniline, 2:5-dinitro-4-aminophenylacetic acid, 5:6- and 6:5-nitroaminobenzoxazolones) is diazotised in presence of  $As_2O_3$  or an arsenite in absence of a reducing agent. Preferably an accelerating agent (Cu, kieselguhr, animal charcoal) and/or  $NaOAc$  or other sol. org. salt are/is added.

C. HOLLINS.

**Manufacture of easily soluble [ $\beta$ -hydroxyethylamine] salts of 3-acetamido-4-hydroxybenzene-arsinic acid.** I. G. FARBENIND. A.-G. (B.P. 348,921, 13.6.30. Ger., 14.6.29).—The arsenic acid is pasted with aq. aminoethyl alcohol, dissolved in  $H_2O$ , and

EtOH is added; the salt which crystallises out is more sol. and better tolerated than are other salts. C. HOLLINS.

Cigarette paper.—See V. Substances containing S.—See VII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Wave-length and colour balance in screen plates.** T. T. BAKER (Brit. J. Phot., 1931, 78, Col. Suppl., 25, 29—30).—Gamma infinity is reached in min. development time when the gelatin: Ag ratio in an emulsion is as low as is practicable. Change in gradation due to wave-length is then obviated. J. LEWKOWITSCH.

**Antifogging agents in [photographic] developers.** II. A. P. H. TRIVELLI and E. C. JENSEN (J. Franklin Inst., 1931, 212, 155—169; cf. B., 1930, 1005).—Of many compounds tried, only cysteine hydrochloride and thiobarbituric acid showed antifogging properties. Their action is less than that of 6-nitrobenzimidazole, and produces greater density depression. Photomicrographs of the action of these compounds, KBr, and thioacetanilide are given. J. LEWKOWITSCH.

**Relative masses of photo-silver and sensitivity specks in the photographic latent image.** S. E. SHEPPARD (Phot. J., 1931, 71, 313—317).—In emulsions digested with gelatin containing labile S, the average amount of Ag<sub>2</sub>S, per g. AgBr, is  $5 \times 10^{-4}$  g. The no. of photo-Ag atoms per g. AgBr is too small to agree with "crit. size" theories of latent image nuclei. The theory that the essential process is the orientation of Ag atoms to initiate a Ag space lattice is supported. J. LEWKOWITSCH.

### PATENTS.

**Protective paper strips for photographic roll films.** I. G. FARBENIND. A.-G. (B.P. 353,324, 29.9.30. Ger., 16.12.29).—Paper sized with montan wax is quite pliable and has no tendency to uncoil quickly after winding. J. LEWKOWITSCH.

**Manufacture of transparent non-inflammable film.** I. KAWAI (B.P. 343,702, 21.1.30).—An aq. solution of agar-agar is treated with CH<sub>2</sub>O (40%), boiled, diluted with H<sub>2</sub>O, and set to a hard jelly. This is shredded, washed free from excess CH<sub>2</sub>O, remelted, filtered, and used to prepare the film. J. W. GLASSETT.

**Photographic emulsion for electronic discharge.** A. G. COOLEY (U.S.P. 1,791,053, 3.2.31. Appl., 7.6.28).—Finely-divided metallic particles (*e.g.*, Mg) are incorporated in the emulsion used for recording corona discharge in the process of U.S.P. 1,702,595, thereby increasing the potential gradient through the emulsion and diffusing the discharge. J. LEWKOWITSCH.

**Imparting printing capability to photographic images in emulsions which are excessively poor in silver.** F. LIERG (B.P. 345,311, 18.9.29).—The Ag image is converted into a mordant compound, *e.g.*, AgI or Cu<sub>2</sub>I<sub>2</sub>, and treated with a solution of a phenosafranin alone or admixed with a yellow dye such as thioflavine-yellow. The dye can be fixed by a lake-forming substance, and the mordant compound blackened with Na<sub>2</sub>S. J. W. GLASSETT.

**Colour photography. [Multicolour screens on film.]** J. N. GOLDSMITH, T. T. BAKER, C. BONAMICO, and SPICERS, LTD. (B.P. 352,949, 14.4.30).—The film is impregnated with one component of a dye, *e.g.*, β-naphthol or Naphthol AS in alcoholic NaOH or KOH. Red lines are produced by ruling with a solution of a diazo compound, *e.g.*, diazotised α-naphthylamine or Fast Scarlet Salt GG, and blue lines, crossing these, with, *e.g.*, diazotised dianisidine or Variamine Blue Salt B. A third (yellow) dye, *e.g.*, Auramine, may be applied. No overlap colours are formed, as the impregnated solution is exhausted where the previous diazo compound is ruled on. Pretreatment of the film for dyeing is unnecessary. J. LEWKOWITSCH.

**Colour photography.** J. N. GOLDSMITH, T. T. BAKER, C. BONAMICO, and SPICERS, LTD. (B.P. 339,238, 1. and 14.8.29).—In the preparation of multicolour screens the dyes are confined to the surface layers of the film by using (a) a non-penetrative solvent, (b) a viscous solution of the dye, *e.g.*, in castor oil or gum arabic, (c) a suspension of the dye, or (d) by rubbing or spreading a powdered dye on the surface of the film or forcing a finely-divided dyed substance in contact with the film. J. W. GLASSETT.

**Colour photography.** I. G. FARBENIND. A.-G. (B.P. 345,575, 4.6.30. Ger., 4.6.29).—The alkali salts of the sulphuric leuco-esters of indigosol-golden-yellow ICK, indigosol-blue OCB, and indigosol-pink IR extra are recommended for the preparation of yellow, blue, and red images, respectively, by direct exposure. A suitable support is sensitised with the leuco-ester, exposed under the appropriate component negative, and the unchanged ester removed by a suitable solvent. The process is repeated on the same or on separate supports for the other colours. A substance having an oxidising action when exposed to light, *e.g.*, inorg. nitro-compounds, is added as a sensitiser. J. W. GLASSETT.

**Light-sensitive paper.** J. TACKELS (B.P. 348,281, 13.3.30).—The paper support is treated with dil. H<sub>2</sub>SO<sub>4</sub> and then coated with a Ag halide emulsion containing 0.2—6% of fatty or oily substances. Examples are: poly-alcohols (*e.g.*, glycerin), lanoline, vaseline, or hydrocarbon derivatives (liquid paraffin). J. W. GLASSETT.

**Manufacture of photographically sensitive materials.** GEN. ELECTRIC Co., LTD., and C. J. SMITHELLS (B.P. 344,354, 29.11.29).—Plates of Cu or its alloy are sensitised by bathing in a 10% aq. solution of CuCl<sub>2</sub> followed by rinsing in H<sub>2</sub>O and in EtOH. The image is fixed by immersion in a dil. solution of a neutral salt, *e.g.*, NaCl or KBr. J. W. GLASSETT.

**Manufacture of [light]-sensitive diazotype layers.** N. V. CHEM. FABRIEK L. VAN DER GRINTEN, Assees. of F. VAN DER GRINTEN (B.P. 344,721, 14.5.30. Holl., 15.5.29).—Difficultly sol. diazo compounds such as the diazonium salts of chlorostannic acid are used in the sensitive layer, which also contains (a) acid-reacting substances and hydroxylamine to lower the coupling activity, (b) neutral salts which reduce the spreading of the image, and (c) NH<sub>4</sub> salts which buffer the alkali of the developer. The support is also treated with acid and salt solutions, and, after coating, the paper is subjected to intense drying. J. W. GLASSETT.

**Manufacture of photographic diazotype prints.** M., G., H., and A. RENKER (DÜRENER FABR. PRÄPARIERTER PAPIERE RENKER & Co.) (B.P. 353,486, 27.2.30. Ger., 22.3.29).—Ureas (*e.g.*, thiourea) or  $\text{NH}_4$  salts of org. acids are added to the diazo compound in the paper, to prevent discoloration. Shrinkage of the paper is avoided if excess developer is rapidly applied and immediately removed by squeegee rollers.  $\text{Na}_2\text{S}_2\text{O}_3$  may be added to the developer. J. LEWKOWITSCH.

**Fixing of light-sensitive layers containing basic dyes.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 348,102, 13.8.29).—After exposure, the sensitive layer is treated with a 12% aq. solution of Na fluoroborate followed by a bath of 1% concentration, whereby the sensitiser is removed but the dye is converted into the insol. fluoroborate. J. W. GLASSETT.

**Preparation of photo-sensitive layers.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 353,511, 25.4.30).—Photographic emulsions made with vegetable colloids, particularly polyglycuronic  $\text{NH}_4$  salt, are softened by incorporation of soaps or sulphonic derivatives of fatty compounds. The tendency to cloudiness is reduced by addition of aromatic sulphonic salts, especially the Na salt of mono- or di-butyl-naphthalene-sulphonic acid. J. LEWKOWITSCH.

**Production of photomechanical printing surfaces.** H. T. P. GEE. From MEISENBACH RIFFARTH & Co., A.-G. (B.P. 348,162, 7.11.29).—After development, the traces of unexposed chrome colloid remaining on the plate are removed by applying a mixture of a dehydrating agent and  $\text{HNO}_3$ . J. W. GLASSETT.

**Photomechanical printing surfaces.** PINCHOT-NEUMANN HOLDING CORP. (B.P. 349,933, 27.2.30. U.S., 27.7.29).—Gelatin printing surfaces are applied to metal plates by means of a transfer paper consisting of a paper support coated with a  $\text{H}_2\text{O}$ -sol. layer of sugar and starch, and a superimposed layer of gelatin, which may be sensitised. J. W. GLASSETT.

**Production of photomechanical printing plates for relief printing.** N. LUDVIGSEN (B.P. 345,255, 13.12.29. Fr., 19.12.28).—A negative resist screen is applied direct to the metal plate and a layer of sensitised colloid superimposed upon it. J. W. GLASSETT.

**Photogravure processes.** S. SAWARAGI (B.P. 346,483, 22.1.30).—The Cu plate is coated (as a substratum) with a mixture containing beer 100 c.c.,  $\text{Na}_2\text{SiO}_3$  (1:1) 120 c.c., egg albumin 100 g., water 100 c.c., and while wet the sensitised gelatin solution is poured on as the plate is heated and rotated. J. W. GLASSETT.

**Device for ascertaining the gradation of graduated series of blackenings in photographic layers.** I. G. FARBENIND. A.-G. (B.P. 353,641, 19.6.30. Ger., 20.12.29).

**Production [with copying apparatus] of prints from photographic negatives.** DÜRKOPFERWERKE A.-G. (B.P. 353,964, 12.5.30. Ger., 21.2.30).

## XXII.—EXPLOSIVES; MATCHES.

**Detonating ability and explosive power of chlorate mixtures.** HAID, GOETZE, SELLE, KOENEN, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930,

8, 115—118; Chem. Zentr., 1931, i, 2426).—With addition of liquid hydrocarbons detonation proceeds more readily than when solid hydrocarbons are used. Detonating ability rapidly diminishes with increase in hydrocarbon content, but markedly increases on addition of nitrobenzene or nitrotoluene. Mixtures with carbohydrates, soot, or metals are less shattering. Mixtures of  $\text{KClO}_3$  with up to 3% of sawdust are not explosive; with at least 10% shattering explosives are produced.

A. A. ELDRIDGE.

**Pentaerythrityl tetranitrate and glyceryl nitrate.** HAID, GOETZE, SELLE, KOENEN, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 121; Chem. Zentr., 1931, i, 2426).—A mixture (80:20) is pulverulent, but cakes on slight pressure; with more than 20% of glyceryl nitrate, or at a higher temp., the oil separates in droplets. The explosive properties are recorded.

A. A. ELDRIDGE.

**Sources of error in the determination of the chemical stability of cellulose nitrate and smokeless powders by Bergmann and Junk's method.** LENZE, METZ, and RUBENS (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 28—31; Chem. Zentr., 1931, i, 2427).—Deviations are attributed to separation of  $\text{H}_2\text{O}$  which hydrolyses the cellulose nitrate. The small separation of NO from powders containing  $\text{NHPh}_2$  at high temp. is ascribed to the fact that at low temp.  $\text{NHPh}_2$  is deposited on the cooler particles as a fused sublimate, and that at high temp. this remains gaseous and combines with liberated NO.

A. A. ELDRIDGE.

**Determination of chemical stability of smokeless powder and explosives by determination of hydrogen-ion concentration.** LENZE, METZ, and RUBENS (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 26—28; Chem. Zentr., 1931, i, 2426—2427).—The test is of value only when the  $\text{CaCO}_3$  content is not too high (0.5—2%). Addition of amyl alcohol,  $\text{NH}_2\text{Ph}$ , and inorg. salts of strong acids is without effect, but the presence of dichromate leads to incorrect results. The method may be applied to celluloid, the presence of  $\text{ZnO}$  and  $\text{PbCrO}_4$  not affecting the results. The specimens are heated to decomp., and extracted with dil. aq.  $\text{HNO}_3$  ( $p_{\text{H}}$  5.5). With cellulose nitrate or glyceryl nitrate powders heated at moderately high const. temp. the  $p_{\text{H}}$  val. of the aq. extract at the beginning of heating usually falls considerably. The decomp. velocity diminishes with stable powders after about 2 hr., after which the decomp. usually proceeds continuously. After a  $p_{\text{H}}$  val. of about 2.5 has been reached, the evolution of acid vapours again increases until  $p_{\text{H}}$  1.3 is reached, when the quantity of  $\text{HNO}_3$  liberated again diminishes with simultaneous formation of slightly dissociated acids, *e.g.*, oxalic and mucic.

A. A. ELDRIDGE.

**Explosive properties of trinitronaphthalene.** HAID, GOETZE, SELLE, KOENEN, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 119—121; Chem. Zentr., 1931, i, 2426).—The behaviour of technical material, m.p. 145—155° (N 14—14.2%), granulated or compressed, is recorded.

A. A. ELDRIDGE.

**Guncottons.** LENZE, METZ, and RUBENS (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 22—24; Chem.

Zentr., 1931, i, 2412).—The degree of degradation of the denitrated cellulose of 12 samples of cellulose nitrate was determined by means of the Cu no., Ba(OH)<sub>2</sub> resistance, and solubility in 5% NaOH. A. A. ELDRIDGE.

**Significance of composition of nitroglycerin waste acids in the rational manufacture of nitroglycerin.** G. WALLERIUS (Proc. World Eng. Cong., 1929, 31, 75—76).—The yield of nitroglycerin is a function of the wt. of waste acid and the solubility of nitroglycerin therein. Hence for any given nitrating acid mixture there is an optimum waste-acid composition. The solubility of nitroglycerin in waste acids containing 10—16% HNO<sub>3</sub> with 12—20% H<sub>2</sub>O content was determined, it being known that 13% H<sub>2</sub>O is the low limit for H<sub>2</sub>O content and 17% HNO<sub>3</sub> the high limit for HNO<sub>3</sub> content. On the basis of these results the ratio of glycerin to mixed acid and the corresponding concentration of HNO<sub>3</sub> in the latter for varying H<sub>2</sub>O content are calc. C. IRWIN.

**Explosive fertilisers.**—See XVI.

PATENTS.

**Nitrocellulose. Pyroxylin sheets.**—See V.

XXIII.—SANITATION; WATER PURIFICATION.

**Rapid determination of the active content of insecticides. VI. Titrimetric determination of arsenic.** A. TERÉNYI and J. PÁSKUJ (Z. anal. Chem., 1931, 84, 416—441).—As may be quantitatively distilled from a conc. HCl solution; KBr does not aid the distillation of As<sup>+++</sup> or As<sup>++++</sup>, but it takes part in the reduction of the latter by FeSO<sub>4</sub>. In 19—28% HCl solution KBr and, particularly, H<sub>2</sub>SO<sub>4</sub> aid the distillation, and the H<sub>2</sub>SO<sub>4</sub>, in addition, accelerates the reduction of As<sup>++++</sup>. Details are given of the determination of As in both easily and difficultly decomposable insecticides; for the latter, 4—5 hr. are required, and a micro-method, requiring only about 1.5 hr., is recommended. H. F. GILLBE.

**Manipulation of p<sub>H</sub> [of water] at Springfield, III.** C. H. SPAULDING (J. Amer. Water Works' Assoc., 1931, 23, 1190—1201).—By adding Ca(OH)<sub>2</sub> at a varying instead of a uniform rate an increased reduction of 10 p.p.m. of total hardness is obtained, probably due to precipitation of Mg during periods of high p<sub>H</sub>. Scrubbed and filtered flue gas is used for recarbonation, but deposits on the filter sand have been observed (attributed by Fleming in the discussion on the paper to an insufficient settling period between carbonation and filtration). Sterilisation produced by lime treatment is thought to be due more to mechanical removal by the bulky floc obtained than to any direct toxic action. C. JEPSON.

**Silicic acid in mineral water analyses.** F. HUNDESHAGEN and F. W. SIEBER (Z. angew. Chem., 1931, 44, 683—685).—Sources of error are discussed. H. WREN.

**Colorimetric determination of iron in drinking water and [pharmaceutical] iron preparations.** K. SCHERINGA (Pharm. Weekblad, 1931, 68, 735—738).—The precautions to be taken when the thiocyanate method is employed in presence of org. and other materials are described. S. I. LEVY.

**Destruction of pests.**—See XIX.

PATENTS.

**Sterilisation of liquids.** G. A. KRAUSE (B.P. 353,409 and Addn. B.P. 353,686, [A] 21.2.30, [B] 19.7.30. Ger., [A] 1.3.29).—(A) Very low concentrations of salts of oligodynamically active metals (not exceeding 2 mg./litre) can be utilised for the sterilisation of H<sub>2</sub>O on the large scale if the time of exposure is sufficiently long and contact with baser metals avoided. (B) Very sparingly sol. salts, e.g., AgBr, are substituted for freely sol. ones, thus avoiding possibility of errors, e.g., overdosing, and manipulative troubles without sacrificing sterilising efficiency. The valuable metals may be recovered after use by deposition on a collector of a suitable base metal, e.g., Fe or Al. C. JEPSON.

**Vermin-destroying compositions.** DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 353,354, 14.11.30. Ger., 14.11.29).—Low-boiling, gaseous or liquid vermin-destroying substances, e.g., ethylene oxide, CNCl, or HCN, are mixed with solid CO<sub>2</sub>. Alternatively, the substances may be cooled to the solid state before mixing, or they may be solidified after mixing with liquid CO<sub>2</sub>. W. J. WRIGHT.

**Disinfection, and destruction of insect pests.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 349,004, 12.11.29, 10.12.29, and 24.6.30).—Dihydroxydiaryl sulphides or polysulphides or their alkali salts are used in bactericidal, fungicidal, and insecticidal preparations. Examples are: 5 : 5'-dichloro-2 : 2'-dihydroxydiphenyl sulphide with soft soap for use against animal pests, or in water against mildew or moths in fabrics, or with talcum etc. for face powder or against *B. pyocyaneus*; the 5 : 5'-dibromo-compound with talcum against mildew on roses, or with talcum and a wetting agent for disinfecting seed grain, or in aq. NaOH for treating anthrax-infected skins or dead wood, or in aq. NH<sub>3</sub> for addition to albumin solutions; 4 : 4'-dihydroxydi-*o*-tolyl sulphide with soft soap; 4 : 4'-dihydroxydiphenyl sulphide with glycerin, wool fat, and petroleum jelly as a cosmetic against hyperidrosis, or the 2 : 2'-diarsinic acid with talcum against mildew on roses; 4 : 4'-dihydroxy-2 : 2'-dimethyl-5 : 5'-diisopropylidiphenyl sulphide in H<sub>2</sub>O against staphylococci; 3 : 3'-dibromo-4 : 4'-dihydroxydi-*m*-tolyl sulphide with yellow wax in disinfectant polishes. C. HOLLINS.

**Treatment of [coke-oven] effluents.** W. W. GROVES. From KLÄR & ENTPHENOLUNGSGES. M.B.H. (B.P. 353,625, 10.6.30).—The effluents after dephenolating by solvent extraction are aerated by compressed air, cascades, etc. at 50—60°, whereby thiosulphates, pyridines, etc. are oxidised or volatilised. The waste air is discharged through the chimney stack. C. JEPSON.

**Incinerator structures.** H. KLEIN (B.P. 353,623, 6.6.30).

**Water-softening systems.** FLORIDA WATER SOFTENER, INC., Assees. of W. E. DUNBAR (B.P. 353,543, 1.5.30. U.S., 8.5.29).

**Depuration of waste water containing cyanide.**—See VII. **Electrolysis of aq. solutions.**—See XI. **Disinfectant.**—See XX.