

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JULY 5, 1929.

### I.—GENERAL; PLANT; MACHINERY.

**Nomography.** XIV. O. LIESCHE (Chem. Fabr., 1929, 170—171).—This chart, constructed exactly as No. 13 (B., 1929, 305), permits of immediate reading of English weight units (troy and avoirdupois) into metric units, and *vice versa*. S. I. LEVY.

**Heat transfer in recuperators.** TERRES and BESECKE.—See II. **Synthetic resins as construction materials.** KALMAN.—See XIII.

### PATENTS.

**Furnace for treatment of materials.** E. B. THORNHILL, Assr. to THORNHILL-ANDERSON Co. (U.S.P. 1,705,039, 12.3.29. Appl., 1.11.26).—A furnace has an outer roof and inner arches under the roof which support heating elements. B. M. VENABLES.

**Regulating the firing of furnaces.** SIEMENS-SCHUKERTWERKE A.-G. (B.P. 299,885, 31.10.28. Ger., 4.11.27).—Oxygen is added to the combustion air in a proportion which increases with increase of load. The rate of fuel firing is also increased, but the introduction of oxygen abolishes the time lag between the addition of fuel and consequent increase of heat. The quantity of air admitted may either remain constant at all loads or be regulated inversely to the oxygen. B. M. VENABLES.

**Recuperators for furnaces.** W. H. FITCH (B.P. 282,443, 17.12.27. U.S., 18.1.26).—The air receiving heat flows in one line through several banks of tubes, expanding between each bank into chambers the width of which is alternately larger and smaller than the length of the tubes, the wider chambers being used for withdrawal of tubes when replacement is necessary. The waste gas giving up heat flows in zig-zag fashion transversely to and outside the tubes in as many passes as there are banks of tubes, the general result being that the gases and air enter (and leave) at opposite ends of the recuperator to each other. The tubes may have metallic rods threaded through them so that the streams of air are annular. The tubes may be made of silicon carbide. B. M. VENABLES.

**Mercury boilers.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. J. NERAD (B.P. 293,756, 5.7.28. U.S., 11.7.27).—To avoid the high cost of pure mercury in a boiler in sufficient quantity to allow a good circulation, an amalgam is used, *e.g.*, of lead, which is liquid at the temperature at which the boiler operates, only mercury being vaporised. The main heating surface of the boiler preferably consists of double concentric tubes, the inner one being partly jacketed with heat-insulating medium, alternating, if desired, with heat-conducting blocks to permit enough heat to pass into

it to keep the amalgam liquid but to avoid excessive heat-transfer, which would interfere with the circulation. B. M. VENABLES.

**Heat interchanger of the plate type.** G. M. G. DE BEAUVAIS, Assr. to G. J. PRAT (U.S.P. 1,705,471, 19.3.29. Appl., 12.6.24. Belg., 13.6.23).—Channel iron is placed between the plates alternately at the horizontal and vertical edges, in each case with the flanges outwards. The plates and flanges are held together with elastic riders or U-shaped elements, no other clamping or welding being used. B. M. VENABLES.

**Heat exchangers and manufacture of tubes provided with gills.** CARRIER ENGINEERING Co., LTD., and W. H. OWEN (B.P. 309,845, 16.1.28).—Gilled tubes are constructed from thick-walled tubing by cutting the gills out of the solid, the ends of the tubes being left of slightly larger diameter than the gills so that the tubes may be withdrawn through the tube plate. If it is desired to secure the tubes in the plate by expanding them, the thickness of the ends is reduced on the inside. B. M. VENABLES.

**Effecting heat exchange.** LA MONT CORP., Assees. of W. D. LA MONT and A. F. ERNST (B.P. 278,704, 1.10.27. U.S., 7.10.26).—In a heat exchanger, a fluid stream is confined in passages (with heat-transferring surfaces) of which the cross-section is varied to maintain the fluid above the critical velocity and give it a substantial "ranging" motion. The walls confining the fluid converge in the direction of the cooler zone. To avoid excessive draught losses, spacing of the surfaces is not reduced proportionately to the reduction of volume of the gases, but is reduced sufficiently to maintain at least the critical velocity. B. M. VENABLES.

**Apparatus for carrying out exothermic gas reactions, regenerating heat, and cooling the walls of the said apparatus.** J. Y. JOHNSON, From I. G. FARBERIND, A.-G. (B.P. 306,054, 15.9.27).—Two forms of apparatus are described in which the incoming gas for the reaction keeps the outer pressure-retaining wall cool and is preheated by heat conducted from the reaction chamber or chambers. The gas is caused to follow a sinuous course while in the preheating stage. B. M. VENABLES.

**Absorption apparatus [for exothermic reactions].** G. B. TAYLOR, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,708,685, 9.4.29. Appl., 8.4.27).—A tower constructed to withstand raised pressures comprises a bottom section having a liquid outlet and a gas inlet above it, a top section having a gas outlet and a liquid inlet, and a series of alternating absorption sections fitted with bubbler-cap trays etc. and cooling sections

comprising vertical tubes in contact with a cooling medium.

L. A. COLES.

**Carrying out exothermic catalytic chemical reactions under pressure.** L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE & L'EXPLOIT. DES PROC. G. CLAUDE, ASSEES. of A. T. LARSON (B.P. 288,577, 11.4.28. U.S., 12.4.27).—The apparatus comprises a double-walled vessel having an outer pressure-resisting wall and a lighter inner one containing the catalyst. Incoming fresh gases are passed through the space between the walls, then mixed with gases that contain products of the reaction, and the whole mixture is allowed to pass through the catalyst.

B. M. VENABLES.

**Apparatus for chemical reaction on heated solid material.** T. GRISWOLD, Assr. to DOW CHEM. CO. (U.S.P. 1,705,614, 19.3.29. Appl., 27.1.27).—An apparatus in which to conduct reactions between gases or vapours and solids comprises a vertical retort below which is a chamber divided by a horizontal partition into two parts, the lower of which serves as a vaporiser and the upper as a receptacle from which ash or the treated solid material may be removed. The apparatus is particularly adapted to the manufacture of carbon disulphide.

A. R. POWELL.

**Thermostats.** R. MACLAREN (B.P. 307,135, 21.12.27).—A tube of metal which expands by heat surrounds a rod of inexpandible metal; the latter operates the short arm of a lever, the long arm of which operates electrical contacts.

B. M. VENABLES.

**Treatment of discrete materials with gaseous media, particularly applicable to drying.** R. V. FARNHAM (B.P. 308,074, 10.3.28).—Powdered material falls in a zig-zag course through a vertical casing counter-current to a drying gas. The material is guided by cellular rotary retarders (shaped like the spokes of a wheel in cross-section) which make substantially gas-tight joints with the casing alternately at either side, a free space being left for the current of gas at the other side of any retarder, which is also the discharge side for the powdered material.

B. M. VENABLES.

**Dryer.** M. D. JONES, Assr. to FULLER-LEHIGH CO. (U.S.P. 1,709,351, 16.4.29. Appl., 22.11.26).—The material under treatment passes down a casing into a hopper below fitted with a discharge spout at the lower end. The casing contains means for heating the material and a distributing device co-axial with the casing, the axis of the hopper being parallel to but at one side of that of the casing.

L. A. COLES.

**Drying processes and plant.** J. G. OLSSON and F. I. E. STENFORS (B.P. 309,331, 18.5.28).—In drying plants where the material is heated directly or indirectly from hot surfaces and the evaporated moisture condensed on cold surfaces, a substantial amount of the latent heat of the moisture is recovered by using the same medium as a heating agent for the hot, and cooling agent for the cold, surfaces, each stage being graduated. The material may be caused to travel longitudinally through the dryer by such means as trucks, and air or gas is caused to circulate transversely over the material and over longitudinal heating and cooling pipes. The medium in the pipes is heated, introduced to the hottest

zone, and travels longitudinally first through the heating pipe and becomes cooler, then (after supplementary cooling if necessary) back through the cooling pipe and becomes hotter.

B. M. VENABLES.

**Drying apparatus.** N. TESTRUP, T. GRAM, O. SÖDERLUND, and TECHNO-CHEMICAL LABS., LTD. (B.P. 306,200, 21.11.27).—In apparatus where material is dried, while borne in a moving stream of gas, by heat that is transmitted through the walls of the conduit, every part of such conduit, which includes any bends, separators, etc., as well as the actual drying zone is maintained at a temperature above that of the interior, or at any rate above the dew point of the gas stream, the object being to prevent blockages by moist material.

B. M. VENABLES.

**Vacuum drying apparatus.** A. E. JONSSON (B.P. 292,105, 11.6.28. Swed., 13.6.27).—The apparatus described in B.P. 232,962 (B., 1925, 655) is modified; e.g., the cylinder preferably oscillates only, and the inlet and outlet for heating medium and outlet to vacuum pump are made by flexible tubes which can twist.

B. M. VENABLES.

**Spray-drying plants.** J. A. REAVELL (B.P. 306,009, 14.10.27).—The disc used for spraying the liquid has its surface roughened to prevent formation of local dry spots. The roughening may be effected by sand-blasting before hardening the metal, or by turning ribs or serrations, or by building up the disc from a number of nested cups the edges of which form the rough working surface.

B. M. VENABLES.

**Drying and grading plants for granular water-containing materials.** L. HONIGMANN and F. BARTLING (U.S.P. 1,705,617, 19.3.29. Appl., 2.6.26. Ger., 3.6.25).—An apparatus for the drying of, e.g., moist coal dust comprises a revolving ring drying oven into which the material is charged in a thin layer and through which is drawn by suction air preheated by the hot, moist, waste gases from the dryer. The dust-laden air from the dryer passes through settling devices in which partial classification of the material is effected prior to passing into the preheater.

A. R. POWELL.

**Means for simultaneous drying and grinding.** E. BARTHELMESS (U.S.P. 1,702,333, 19.2.29. Appl., 15.5.26. Ger., 15.5.25).—Coal or similar material is subjected, in a cycle of operations, to the action of a hot drying fluid and sifted; the undersize constitutes finished material, and the oversize is ground, mixed with fresh material, and re-subjected to drying as above.

B. M. VENABLES.

**Pulverising apparatus.** W. R. WOOD (B.P. 292,175, 16.6.28. U.S., 17.6.27).—In a system of air-borne pulverisation working in substantially closed circuit, if the air is also used as a drying medium it is necessary to allow some of the air to go to waste. In this invention there is inserted in the return air main, between the cyclone collector and the pulveriser, a device by which the residual very fine dust is temporarily removed from the air stream, and, after removal of the necessary amount of clean waste air, the dust is returned to the main stream. One device described comprises a drum with tangential inlet at one end, tangential outlet for

the bulk of return air and dust at the other end, and an outlet for clean waste air axially.

B. M. VENABLES.

**Pulverising mill.** J. CRITES, Assr. to RAYMOND BROS. IMPACT PULVERIZER Co. (U.S.P. 1,702,248, 19.2.29. Appl., 31.12.26).—In the passage between the pulverising chamber and the air classifier above are a number of transverse baffles, of which the under surfaces are concave and which are in staggered rows; the baffles are intended to prevent large particles being flung by the pulveriser into the classifier.

B. M. VENABLES.

**Pulverising mill.** J. MEAD, JUN. (U.S.P. 1,704,757, 12.3.29. Appl., 29.9.26).—The "screens" of an impact pulveriser are supported in annular grooves in opposite ends of the casing. The "screens" comprise a complete circle of arcuate elements, all of which have pyramidal interior surfaces, and one or more have perforations through the points of the pyramids.

B. M. VENABLES.

**[Impact] pulverisers.** T. BROADBENT & SONS, LTD., and W. HALLITT (B.P. 308,095, 4.4.28).—The apparatus comprises two horizontal discs rotating co-axially in opposite directions at centrifugal speed. The material is fed through the centre of the upper disc to the space between the discs, and leaves at the periphery. The working faces of the discs are provided with intercalating annular teeth of buttress form, the steep faces being towards the axis. Fillets of the material under treatment are caught in the angles between the steep sides of the teeth and the main part of the discs, so that the impact of the material flung off from the preceding tooth is always upon a mass of the material itself, thus avoiding abrasion of the metal.

B. M. VENABLES.

**Crushing machine.** J. J. DENNY (U.S.P. 1,704,823, 12.3.29. Appl., 1.10.26).—A number of crusher plates with flat backs are linked together to form two rounds of chain-conveyor or crusher belts. The conveyors are supported at their ends by rollers (four) with sprocket wheels (eight) at the ends of the rollers. Two runs of the conveyors approach each other, forming the crushing nip, the crusher plates being supported by the above-mentioned rollers and by others with unyielding axes under and above, respectively, the straight approaching runs of the crushing belts.

B. M. VENABLES.

**Crushing machines having vibrating jaws.** F. M. VALE (B.P. 306,609, 28.11.27).—In a jaw-crusher preferably of the granulator type the corrugations on the jaws are longitudinal, of comparatively fine pitch, but formed in more than one plane; the same effect may be produced by superposing fine corrugations upon much coarser ones. Transversely, each jaw exactly mates with the other, but longitudinally one jaw may be convex to the other.

B. M. VENABLES.

**Grinding or crushing mill.** C. J. COOPER and A. M. MASON (C. J. COOPER & Co.) (B.P. 306,630, 3.12.27).—The mill, which is intended for paints and the like, is similar in construction to a multi-plate friction clutch.

B. M. VENABLES.

**Crushing and grinding machine.** A. C. HAMEY

and J. STONEHAM (U.S.P. 1,706,290, 19.3.29. Appl., 10.2.27. Austral., 19.2.26).—The material is fed through a hollow vertical shaft which rotates a muller above another fixed muller. The shaft and muller are rotated by a power-driven sleeve, and are also given a vertical reciprocating movement.

B. M. VENABLES.

**Colloid-treating apparatus.** H. W. A. DIXON (U.S.P. 1,702,380, 19.2.29. Appl., 23.3.27).—A colloid mill is contained in a casing, and the material is supplied under positive pressure and withdrawn under negative pressure by means of a pair of gear-wheel pumps operated by a common motor. Two containers are used, filled and emptied alternately by the same material until it is sufficiently dispersed, but the material always flows through the mill in the same direction, the change-over of flows being effected by a rotary valve. Spaces are provided in the body of the valve and the mill for a heating (or cooling) fluid which flows through pipes adjacent to those for material, the pairs being surrounded by insulation so that the material is always subjected to the heating (or cooling) influence.

B. M. VENABLES.

**[Rotary-drum] comminuting mills.** ALLIS-CHALMERS MANUF. Co., Assees. of [A] E. C. GREISEN, [B] R. C. NEWHOUSE (U.S.P. 1,710,659 and 1,710,666, 23.4.29. Appl., [A] 12.3.28, [B] 8.3.28).—(A) The mill is provided with scoops for feeding the material through the circumference of the drum; detachment of the scoops leaves openings for direct removal of material from the drum. (B) The mill has a grinding compartment surrounded by a cylindrical screen attached to it. The oversize travels longitudinally within the screen to a stationary pocket, whence it is picked up by scoops attached to the mill, and returned within the screen and back into the mill through the same openings, now at the top, through which it had emerged at the bottom. The undersize after passing through the screen is conveyed longitudinally in the reverse direction by worms attached to the outside of the screen, and is picked up by other scoops and delivered to another compartment of the mill for finer grinding, or disposed of as finished product.

B. M. VENABLES.

**Homogenising mill.** W. EPPENBACH (B.P. 306,502, 22.11.27).—The apparatus comprises two relatively rotatable "grinding" elements within a casing. The inlet for material is to the outside of the grinding elements, and outlet for emulsion from the space between the grinding elements through the hollow shaft of one of them. The casing may be jacketed to control the temperature and may be split in the same plane as the operating surface between the two rotating elements.

B. M. VENABLES.

**Mixer.** R. L. COOK (U.S.P. 1,702,931, 19.2.29. Appl., 17.3.28).—A rotating drum is provided with internal guides to drive material, received at one end of the drum, in one longitudinal direction when the rotation is in one direction and back again when the rotation is changed. One end of the mixer is provided with a double series of scoops—one series engaging and charging the material into the mixer and effective when rotated the first way, the other being effective for discharge on reversal.

B. M. VENABLES.

**Mixing apparatus.** P. LENART (U.S.P. 1,706,176, 19.3.29. Appl., 17.12.27. Ger., 28.12.26).—Two containers are arranged one within the other, the inner one having openings in the bottom and sides. The material is drawn through the bottom of the inner vessel by a pump below it, but within the outer vessel, and delivered up the annular space and into the inner vessel again.

B. M. VENABLES.

**Producing an intimate mixture of several media by means of centrifugal force.** P. JANES (B.P. 294,871 and 305,877, [A] 21. and [B] 23.4.28. Addns. to B.P. 283,975; B., 1929, 191).—(A) In the apparatus described in the original patent the mixture is cooled by partial evaporation by forming the deflecting or collecting shield of porous material. The shield is preferably stationary and inclined to the issuing mixture. (B) Mechanical additions and improvements are made to the original apparatus.

B. M. VENABLES.

**Apparatus for mixing or emulsifying especially viscous or adhering liquids.** O. J. BUDTZ (B.P. 307,283, 24.10.28).—A vessel with hemispherical bottom is provided with a number of concentric hoops alternately fixed and rotating. The fixed hoops are attached to a fixed bush at one side of the vessel, and the moving ones to a bush at the other side of the vessel rotated by a shaft which extends across the vessel and turns freely in the fixed bush.

B. M. VENABLES.

**Machines for kneading or mixing plastic masses, liquids, or powdery material.** R. RIEDL (B.P. 302,152, 14.11.28. Austr., 10.12.27).—A disc rotates in the bottom of a fixed bowl or container, and by means of blades on its upper surface presses the material against the wall of the bowl. Air may be drawn or blown into the material from an opening in the casing below the disc.

B. M. VENABLES.

**Screening of materials.** L. S. DEITZ, JUN. (U.S.P. 1,710,208, 23.4.29. Appl., 29.9.26).—A stream of the materials is caused to flow perpendicularly through a screen, and the screen is moved transversely to the stream of materials, but the latter is substantially prevented from following the motion of the screen.

B. M. VENABLES.

**Treatment of solids with liquids.** K. KOMERS (B.P. 288,999, 1.11.27. Czechoslov., 19.4.27).—Comminuted solid material is subjected to countercurrent leaching in an apparatus comprising a trough-like container of large diameter compared with its length. The solids are dragged round the circumference of the trough by comb-like arms attached to a drum rotating about the axis of the trough. The solids are delivered over the side of the large trough by fixed comb-like teeth alternating with the moving teeth into a smaller and longer trough provided with a permeable bottom and spiral conveyor blades, from which horizontal conveyor the material is elevated by a vertical presser worm and delivered in a nearly dry state to a final conveyor for removal to any point desired. Any leaching fluid that drains through the permeable bottom, plus a supply of fresh fluid, is forced by a pump round the trough of large diameter in the opposite direction to the solids, and overflows at a point near the entry for solid.

B. M. VENABLES.

**Apparatus for treating semi-solids and liquids.** E. E. LINDSEY (U.S.P. 1,705,822, 19.3.29. Appl., 14.11.27).—The material is circulated by means of a centrifugal pump through a conical tank, in which it meets a stream of steam or hot air, as desired, to cause the material to gelatinise or the excess moisture to evaporate. The pump withdraws the liquid from the bottom, and returns it at the top of the tank through a spray device, which causes it to run down the walls as a thin film.

A. R. POWELL.

**Separation of solids from liquids.** A. L. BLUMFIELD, L. S. HARNER, and H. S. COE, Assrs. to CYCLE Co. (U.S.P. 1,702,192—3, 12.2.29. Appl., [A] 12.5.24, [B] 2.3.26).—A pulp is thickened and clarified by admitting it above a column of filtering material, part of the clear liquid being overflowed some distance above the level of entry, and part simultaneously withdrawn as filtrate downwards through the filtering column. Settled thick pulp with thin successive layers of the filtering medium are scraped off and removed through the centre of the bottom of the tank.

B. M. VENABLES.

**Centrifugal separators.** ARTEBOLAGET SEPARATOR (B.P. 298,949, 16.10.28. Swed., 17.10.27).—In a separator for two liquids means are described for keeping the outlet for heavy liquid comparatively close to the axis without any risk of contaminating the lighter liquid.

B. M. VENABLES.

**Centrifugal separators.** E. B. MÖLBACH (B.P. 299,703, 19.10.28. Norw., 29.10.27).—In a separator for two liquids the main discs are stacked alternately with discs of smaller diameter. The smaller discs are provided with ports so that the feed passes from a common central passage above each of the smaller discs, and the lighter fluid passes under the smaller discs to a number of outlet passages parallel to, and not far removed from, the axis. The heavier fraction is passed out into a volute pump-casing, in which the loss of kinetic energy produces a certain back-pressure, which in turn helps to force out the lighter fraction. Supplementary to this, another pump, complete with rotor, may be provided for the light fraction.

B. M. VENABLES.

**Centrifugal separators and coolers.** B. C. CARTER (B.P. 310,550, 28.10.27).—The separator part comprises a bowl to which the mixed fluids are supplied under pressure through a hollow pivot, and which is rotated only by the action of a separated fluid issuing through tangential jets. In separating oil-water emulsions, the lighter fluid (oil) will issue through the jets and be cooled by impingement on a stationary outer shell provided with fins or otherwise kept cool.

B. M. VENABLES.

**Centrifuge.** NAT. ACME Co., Asses. of D. S. PATERSON and G. B. PETSCHÉ (B.P. 294,525, 9.5.28. U.S., 25.7.27).—In a centrifuge of the bowl type the bowl is supported on a door of the casing and swung out for cleaning. Other mechanical improvements are also described.

B. M. VENABLES.

**Liquid separator.** H. S. COE, Assr. to DORR Co. (U.S.P. 1,709,971, 23.4.29. Appl., 20.5.25).—For the continuous separation of liquids of different sp. gr. by settling, a number of (conical) diaphragms are

assembled in a vertical (cylindrical) casing. The feed of mixed liquids is through a common supply pipe with branches leading to the spaces between each diaphragm, at the circumference; the outlets for heavy liquid are similarly situated, but well spaced (say 180°) circumferentially from the inlets. The outlet for lighter liquids is axially upwards through the points of the cones.

B. M. VENABLES.

**Hydraulic separator.** H. H. CARL and H. E. MUEHLHOF (U.S.P. 1,710,568, 23.4.29. Appl., 26.11.27).—The separator comprises a tank having a feed at one end and discharges at the other end for heavy liquid at the bottom and for light at the top; the latter discharge is subdivided by an adjustable skimmer, the uppermost layer of liquid removed being sent to a pump which delivers it back to the tank in such a manner as to cause upward and forward currents.

B. M. VENABLES.

**Conservation of centrifuged liquids.** H. O. LINDGREN, Assr. to DE LAVAL SEPARATOR CO. (U.S.P. 1,705,934, 19.3.29. Appl., 27.7.26. Swed., 29.7.25).—The separated liquids, which are likely to be delivered in finely-divided form, are collected in vessels under slight vacuum and provided with means to separate finely-divided liquid from any gas in which it is entrained.

B. M. VENABLES.

**Means for separating air, vapour, and volatile liquids from liquids.** E. DODSON (B.P. 308,010, 11.1.28).—The liquid, *e.g.*, lubricating oil containing water and/or petrol, is passed through a Venturi tube, the drop in pressure in the throat of which causes the volatile fluids to form bubbles. After the stream of liquid has expanded to the normal bore of the conduit, but before the bubbles have had time to redissolve, the latter are allowed to rise out of the stream into a vent chamber. The action is much increased by permitting air to be drawn into the throat of the Venturi through a side passage.

B. M. VENABLES.

**Apparatus for pasteurising liquids.** A. JENSEN (U.S.P. 1,701,777, 12.2.29. Appl., 21.6.26. Cf. U.S.P. 1,693,034; B., 1929, 192).—A chamber is heated by a fluid under pressure in a jacket. The inlet for the liquid to be treated is centrally at the bottom and the outlet peripherally at the top; a rotor is provided to force the liquid centrifugally through the vessel. The shaft of the rotor is hollow and perforated within the upper part of the chamber, and is utilised to afford connexion to an exhaust pump.

B. M. VENABLES.

**Conveyance of viscous substances.** C. P. BRASINGTON, Assr. to INTERNAT. PRINTING INK CORP. (U.S.P. 1,710,144, 23.4.29. Appl., 23.3.28).—A moving endless wire dips into the fluid at the starting point and adhering fluid is removed therefrom at the delivery point.

B. M. VENABLES.

**Tubular rotary crystallisers or crystalliser-boilers.** F. LAFEUILLE (B.P. 301,453, 4.10.28. Fr., 30.11.27).—The tubes for heating or cooling agent are not expanded into tube plates, but are freely supported only by stay-plates, the junctions with the return bends and to the supply and discharge headers being all welded. The massecuite or other fluid under treatment occupies the whole space within the cylinder right up to the dished

ends, which are pressure-resisting, and one of which is provided with concentric inlet and outlet for heating or cooling fluid, the other with concentric inlet being for fluid to be crystallised and for outlet of vapour. In operation the cylinder and heating or cooling tubes rotate and with them a worm in the inlet pipe for massecuite; the actual inlet and outlet pipes remain stationary.

B. M. VENABLES.

**Separating out the mother-liquor from crystals or crystalline bodies.** RAFFINERIE TIRLEMontoise Soc. ANON. (B.P. 286,599, 24.2.28. Ger., 5.3.27).—In a centrifugal process for draining crystals, the basket is first charged, closed by an air-tight cover, and then run up to speed. This prevents circulation of air which would have an evaporating effect on the mother-liquor adhering to the crystals, and cause surface-discoloration in the case of materials like sugar with syrup.

B. M. VENABLES.

**Precipitation apparatus.** R. W. SHAFOR, A. R. NEES, and R. J. BROWN (U.S.P. 1,708,332, 9.4.29. Appl., 19.7.24).—A vessel provided with an outlet to maintain a constant liquid level inside it is divided into mixing and precipitation zones communicating with one another in the lower part of the vessel. The material under treatment is fed into the mixing zone in which means are provided to produce a current flowing downwards and into the precipitation zone, the lower part of which contains means for bringing the contents to a temperature suitable for precipitation.

L. A. COLES.

**Combined decanting and filtering apparatus.** C. PICCARDO (B.P. 305,976, 22.8.28. Ger., 13.2.28).—A sleeve of fabric is suspended within a container and spaced from the wall thereof by means of a permeable material such as spiral wire mesh, and from the annular space a draw-off for clear liquid is provided. The top of the sleeve may be sealed to an angle-iron ring within the container, and the bottom may slip into an internal channel and be sealed with sand. The mud is discharged through a hopper bottom.

B. M. VENABLES.

**Filtering or like devices.** H. A. THOMPSON (B.P. 307,267 and 308,166, [A] 31.7.28, [B] 26.10.28).—(A) The filter comprises a pack of annular laminations interleaved with scraping laminae capable of rotation or rocking by means of suitable shafts on which they are threaded. In (B) the same shafts that rotate the scrapers also carry pinions gearing with teeth formed on the edge of the main laminations, so that the latter as well as the scrapers rotate.

B. M. VENABLES.

**Distilling and like apparatus.** H. GRIFFITHS (B.P. 309,976, 19.11.28).—Condensate from a still is collected in a high-vacuum receiver which is connected to a low-vacuum receiver, some distance below it so as to create a hydrostatic head of condensate, by a pipe having a valve which is closed when it is desired to discharge the condensate from the low-vacuum receiver. The receivers may be connected to the respective stages of a single, two-stage, vacuum pump, and the low-vacuum receiver is cut off from the vacuum pump when air is admitted to discharge condensate; these operations may be effected automatically by floats in the low-vacuum receiver.

B. M. VENABLES.

**Still.** E. H. RECORDS (U.S.P. 1,710,070, 23.4.29. Appl., 24.2.25).—A removable charging bucket having a perforated bottom, perforated side walls, and a central pipe open at both ends and leading to the outside of the bucket, supported therein, forms an annular steam passage within a still-cylinder having a steam inlet and an outlet for volatile gases. J. S. G. THOMAS.

**Apparatus for separating grit, dust, etc. from smoke and gases.** A. PARKER (B.P. 307,168, 27.1.28).—The gases are admitted upwardly through an annular space between an inverted, conical, outer casing and a similarly shaped collecting surface. On reaching the top (base) of the cone the gases are caused to whirl inwards and downwards by means of fixed vanes; the solid matter separates out on the interior of the catcher cone, and the cleaned gases have their motion changed from whirling downwards to rectilinear upward motion along the axis of the cones by means of another set of fixed vanes, and are discharged through the top of the apparatus. B. M. VENABLES.

**Removal or separation from gaseous fluid of material suspended therein.** R. S. PORTHAM, and TANGENTIAL DRYERS, LTD. (B.P. 306,697, 15.2.28. Addn. to B.P. 271,545; B., 1927, 544).—The many-pointed body on which the gas impinges with change of direction is so arranged that the dust can pass away between the points in the original direction of the gas. The gas may afterwards pass through a filter bed. B. M. VENABLES.

**Rotatable air or gas filter of the plate type.** A. SCHIRP (B.P. 303,740, 5.10.28. Ger., 7.1.28).—The filter comprises a number of closely spaced plates kept moist with a liquid such as oil and secured at one end to a chain which slowly travels round two small sprockets vertically over each other. The free ends of the plates are guided only. When the plates pass over the sprockets they open out, affording opportunity for cleansing them and renewing the liquid film by means of a vessel of liquid at the bottom. Several of these chain-and-plate elements are arranged in horizontal series, and the gas passes through them all, entering through the free ends of a set of plates which is, say, rising, passing between the plates, through both runs of the chain, between a set of plates which is falling, and so on. The driving is effected at the lower sprockets by gearing outside the washing chamber. B. M. VENABLES.

**Filter for straining and clarifying such portions of the gases, from furnace flues etc., as may be desired for analysis etc.** A. and L. LUMB (B.P. 305,874, 10.4.28).—The apparatus comprises a casing with closed top and water-sealed bottom containing a filter medium (*e.g.*, coke and asbestos fibre) between two grids. B. M. VENABLES.

**Filter medium for air or gas filters.** A. JORDAHL (B.P. 306,348, 15.6.28).—The medium comprises a number of layers of knitted fabric made from a flat ribbon, preferably metallic. B. M. VENABLES.

**Separation of mixtures of gases and gases with vapours.** H. BLAU (B.P. 278,712, 4.10.27. Ger., 5.10.26).—The mixture of gases of different solubility is passed, preferably under increased pressure, up a tower where it meets a descending current of cooled

absorbing liquid, with the result that the practically pure constituent of lower solubility (A) leaves from the top of the tower. Below the absorbing tower is a rectifying tower, heated slightly at the bottom, in which the absorbing liquid is gradually heated so that all of gas A (and some of the constituent of higher solubility [B]) is expelled from solution; the evolved mixture mingles with the fresh mixture in the absorbing tower above. The liquid from the bottom of the rectifier, having only gas B dissolved in it, passes through a heat exchanger to an expelling tower, where it is more strongly heated, the vapour of pure gas B being cooled with or without condensation. The barren absorbing liquid passes through the heat interchanger and a cooler and is re-used. If there are several constituents of moderate solubility they may be further separated by the use of several expelling towers in series. The towers each have a cooled top and heated bottom, the point of entry for the liquid with dissolved constituents being intermediate. B. M. VENABLES.

**Air- and gas-washing apparatus.** PNEUMATIC CONVEYANCE & EXTRACTION, LTD., and W. A. SMITH (B.P. 306,058, 14.11.27).—In the apparatus described in B.P. 277,112 (B., 1927, 832), the roof is rigid but the baffles are loosely mounted. A hopper with suitable valves is also provided for collection of sludge.

B. M. VENABLES.

**Rectification of mixed gases.** S. G. ALLEN, Assee. of W. L. DE BAUFRE (B.P. 283,101, 285,468, and 294,994, Appl., [A—C] 11.10.27. U.S., [A, C] 3.1.27, [B] 18.2.27).—These patents refer to an elaborate system of heat exchangers, bubbling columns, and tubular rectifiers, for the purpose of separating a mixed gas (*e.g.*, air) into its pure constituents. B. M. VENABLES.

**Recovery of gases and vapours from gas mixtures.** V. PANTENBURG (U.S.P. 1,702,311, 19.2.29. Appl., 28.1.26. Ger., 7.2.25).—A number of absorption chambers are connected to a single regeneration chamber to which the absorption material is transferred in turn. B. M. VENABLES.

**Drying of gases and vapours.** HOLZVERKOHLUNGS-IND. A.-G. (F.P. 630,612, 9.3.27. Ger., 28.5.26).—The gases etc. are passed up towers containing sodium bisulphate, the aqueous solution formed of this being evaporated to dryness for re-use. L. A. COLES.

**Apparatus for preparing compressed gases.** GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 279,041, 3.9.27. Ger., 18.10.26).—A pressure vessel, in which gases are stored in their liquid condition and from which compressed gases of different controllable pressures may be drawn off, is provided (*a*) with a thin-walled inner vessel, spaced from the pressure-resisting vessel, gas only being allowed to fill the space between the two, and (*b*) with a draw-off coil leading from below the level in the liquid and round the pressure-resisting vessel in coils which gradually get further away from that vessel and in which evaporation takes place. The walls of both vessels are made of a metal that does not change its coefficient of expansion at low temperatures. A rectifying column may be placed within the inner vessel, in which an additional gas may be liquefied

(*e.g.*, oxygen-rich air) partially to take the place of that drawn off (*e.g.*, oxygen). The rectifier is used in conjunction with a double-walled inlet and outlet tube, which serves as a heat exchanger. B. M. VENABLES.

**Liquefying and supplying gases.** GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 282,813 and 307,083, 29.12.27. Ger., [A] 29.12.26, [B] 14.1.27).—(A) In transferring liquid air or other gas from an expansion machine to vessels for storage or transport, gas formed by evaporation is led back by a pipe to the expansion machine, which is operated at a higher pressure than usual (250 atm. instead of 200 in the case of air), the resulting excess cold being sufficient to re-liquefy the returned gas. (B) Since large storage vessels as described in B.P. 279,041 (p. 500) cannot produce gas at a pressure much above 40 atm., a small quantity of liquid is evaporated in a small strong vessel to give gas at a much higher pressure, say 150 atm., which is utilised to work an injector drawing medium-pressure (40 atm.) gas from one storage bottle and delivering it into other bottles at normal storage pressure, the medium-pressure bottle being thereby emptied ready to receive gas from the main liquid storage again. B. M. VENABLES.

**Accumulating, conveying, and consuming liquefied gases of low b.p. without loss.** GES. F. INDUSTRIEGASVERWERTUNG M.B.H., and C. W. P. HEYLANDT (B.P. 280,588, 12.11.27).—Means are described for reducing time and opportunities for evaporation when transferring liquefied gas from a transport vessel to measuring vessels, and thence to a storage vessel. The inner transport vessel is suspended on chains which are provided either with insulating links or with metallic links having line-contact only, and any gas (oxygen) which does escape may be used in the motor of the vehicle. B. M. VENABLES.

**Re-gasification of liquefied gases.** GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 287,909, 28.3.28. Ger., 29.3.27).—The re-gasification is accelerated by admitting a reserve supply of compressed gas to the gasifier, the pressure, number of molecules, and the rate of heat transmission from the exterior of the gasifier to the liquid being thereby increased, thus quickening (but not dangerously so) the evolution of gas from the liquid. Suitable valves are described. B. M. VENABLES.

**Continuous absorption refrigerating apparatus.** R. F. BOSSINI and G. MAIURI (B.P. 307,236, 11.4.28).—The inert gas is of approximately the same density as the vaporised refrigerant, and is either a single gas or a mixture of gases. When ammonia is the refrigerant, the inert gas may be methane or a mixture of nitrogen and hydrogen. B. M. VENABLES.

**Antifreeze composition.** J. R. PAUL (U.S.P. 1,689,153, 23.10.28. Appl., 17.2.27).—Sodium thio-sulphate and calcium chloride in approximately equal amounts and a relatively small amount of tartaric acid are added to water. R. BRIGHTMAN.

**F.p. depressant.** S. ISERMANN and W. VERNET (U.S.P. 1,687,094, 9.10.28. Appl., 9.7.25).—Formamide, acetamide, diacetone alcohol, acetylacetone, diacetoneglycerol, or mixtures of these, are used with or without glycerol to depress the f.p. of water etc. R. BRIGHTMAN.

**Disintegrating or mixing apparatus.** C. W. BOISE and W. R. DEGENHARDT (U.S.P. 1,711,259, 30.4.29. Appl., 2.8.27. U.K., 10.8.26).—See B.P. 280,276; B., 1928, 72.

**Separating and purifying apparatus for liquids.** J. SCHÄFER (U.S.P. 1,711,428, 30.4.29. Appl., 31.5.27. Fr., 15.6.26).—See B.P. 272,927; B., 1928, 216.

**[Means for excluding air to interior of] rotary furnaces and kilns.** METALS PRODUCTION, LTD. From T. J. TAPLIN (B.P. 310,780, 30.1.28).

**Heating by air and apparatus therefor.** F. LEBRE (B.P. 282,371, 1.12.27. Fr., 17.12.26).

**Refrigerating apparatus of the absorption type.** N. V. KODOWA REFRIGERATOR COMP., and W. A. SLATER (B.P. 310,901, 2.1.28).

**Absorption refrigerating apparatus.** H. D. FITZPATRICK. From N. V. KODOWA REFRIGERATOR Co. (B.P. 296,792 and 311,496, [A] 16.2.28, [B] 9.3.28).

**Refrigerating apparatus.** J. HUDSON (B.P. 311,595, 11.7.28).

**[Automatic] means for controlling the supply of materials to pulveriser and like mills.** STIRLING BOILER Co., LTD., and E. E. NOBLE (B.P. 311,606, 30.7.28).

**[Shaking table for] separating or cleaning inter-mixed divided materials.** K. DAVIS (B.P. 307,978, 13.10.27).

**[Bag] filters.** A.C. SPARK PLUG Co. (B.P. 284,982, 6.2.28. U.S., 5.2.27).

**Treating material with gases** (B.P. 305,883).—See X. **Sorting articles by light** (B.P. 292,474).—See XI. **Distillation apparatus** (B.P. 310,400 and 310,403).—See XVIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Mechanism of the carbonisation of coal.** E. AUDIBERT (Fuel, 1929, 8, 225—243; cf. B., 1927, 383).—The factors controlling the physical phenomena accompanying the carbonisation of coal and determining the strength of the coke formed are divided into two classes: (a) the initial characteristics of the network of interspaces, *i.e.*, the particle size and the density of packing, and (b) the factors determining the maximum fluidity of the coal in the plastic state, *i.e.*, the nature of the coal, its degree of oxidation, and the rate of heating. The strength of a small cylinder of coke prepared under standard carbonising conditions increases rapidly as the particle size of the coal used decreases below about  $\frac{1}{2}$  mm. diam.; above that size the strength is not greatly affected by this factor. The strengths of the cokes produced from different mixtures of coals of three different particle sizes are conveniently represented on a trilinear diagram. The mechanism of the agglomeration of the coal particles in a coke oven is discussed; neither pyrolysis of the vapours passing through the coke nor the deposition of pitch on the coal in the region within the plastic layer plays any part in this agglomeration. The maximum fluidity of a coal is measured by the "agglutination index," *i.e.*, the amount of an infusible substance that must be added

to 100 pts. of the coal in order that, at a rate of heating of  $1^{\circ}/\text{min.}$ , the mixture may be just at the limit of intumescence. This index is independent of the fineness of the particles used, the apparent density of the mass, and also, very nearly, of the nature of the infusible substance. As the rate of heating is increased the agglutination index increases until it reaches a limiting value. Consideration of the factors responsible for the fissuring of coke has led to an empirical test for distinguishing between coals giving a lump coke and those giving a "fingery" coke. The finely-divided coal is carbonised within a cast-steel cylinder under carefully-controlled conditions which give results, with respect of the type of coke, comparable with large-scale practice. A "fingery" coke can be converted into a lump coke by adding to the coal a suitable proportion of coke, semi-coke, or a very slightly fusible coal. To make a dense coke the diluent substance should contain 10–15% of volatile matter. The practical application of these principles to determine the most suitable ternary mixtures for carbonisation in the oven is described in detail. The binary mixtures of zero agglutination index are first found; the line joining the points corresponding to these on the trilinear diagram divides the latter into regions of positive and negative agglutination index respectively. It is then easy to determine by a few trials the curve in the former region which separates the zones of "fingery" coke from that of lump coke. A. B. MANNING.

**Rectangular graphs as applied to the proximate analyses of Chinese coals.** H. S. WANG (Bull. Geol. Soc. China, 1928, 7, 175; Fuel, 1929, 8, 244–248).—The smoothness of the curves obtained by plotting moisture and volatile matter against fixed carbon (on the ash-free basis) for 40 Chinese coals indicates that there is no break in the series from low-rank bituminous coal to anthracite in Palæozoic, Mesozoic, and Tertiary coals. The importance of geological age in relation to coal classification is emphasised. A. B. MANNING.

**Heat transfer in recuperators.** E. TERRES and W. BESECKE (Gas- u. Wasserfach, 1929, 72, 417–421, 447–449, 466–469, 476–477).—Experiments have been carried out on a vertical-chamber oven, heated by an internal producer, with particular reference to the efficiency of the recuperator system. Temperatures of flue gases and air were measured by rare-metal thermocouples protected by silica and by iron sheaths, and the volumes of the gases were obtained by calculation from hourly analyses, and from the coke consumption; the coke used in 24 hrs. was weighed, sampled, and analysed. Producer gas was sampled through a special pipe fitted in one of the cleaning ports, and flue gases were withdrawn from four different points in the setting; slow withdrawal of the gases was found to give unreliable results owing to change in composition, and intermittent samples were taken more rapidly. The volume of producer gas was calculated by assuming 97% of carbon in the dry, ash-free coke; the calorific value of the latter was 7950 kg.-cal./kg. The carbon content of the flue gases at different points was compared with that of the producer gas, so that the amount of secondary air drawn in through leaks could be determined. The

methods employed in interpretation of the results are explained in full, and calculations are included for gas volumes, mean temperature differences, heat balances, sp. heats, efficiency of recuperation, and coefficient of heat conductivity. The 18 tests recorded in detail gave the following mean results: coke used in 24 hrs., 2822 kg.; volume of producer gas, flue gas, and air, 470, 937, and 539 m.<sup>3</sup>/hr., respectively. The flue gases gave up 93,100 kg.-cal./hr., and the efficiency of recuperation was 33.8%; the conductivity through the bricks of the recuperator was 7.93 kg.-cal./m.<sup>2</sup>/hr.°C. for a mean temperature of 500°. The air drawn in through leaks varied widely in volume, and accounted for irregular results in the heat balance, so that the heat absorbed by the incoming air sometimes appeared to be more than that lost by the waste gases. Further calculations are included, showing the relations between gas velocity, temperatures, and the rate of heat transfer, and it is found that the coefficients of conductivity for the bricks are considerably larger than those derived from existing information on the subject. Curves are included which show the change of rate of heat transfer with temperature and with gas velocities between 0.87 and 1.22 m./sec., and the causes of the low general efficiency of recuperation are discussed and compared with those concerned in regenerative systems.

R. H. GRIFFITH.

**Distillation of peat.** V. E. RAKOVSKI (J. Chem. Ind. Moscow, 1928, 5, 915–919).—The rôle of water vapour injected into the retort during the distillation of peat, whereby the tar contains a smaller quantity of asphaltenes, is considered to be the impeding of condensation processes normally occurring with formation of water.

CHEMICAL ABSTRACTS.

**Determination of the elementary oxidisable carbon in solid fossil fuels.** J. BLUM (Bul. Chim. pura aplic. Bukarest, 1927, 30, 43–49; Chem. Zentr., 1928, ii, 2314).—From the carbon dioxide produced on combustion must be subtracted that absorbed in the coal, that arising from mineral substances, and that produced by decomposition of organic compounds.

A. A. ELDRIDGE.

**Modern illuminating gas production consequent on the Krummhübl calorific value agreement.** R. GEIPERT (Gas- u. Wasserfach, 1929, 72, 169–174, 205–210).—The Krummhübl agreement advocates the mixing of coal gas with water-gas to give a mixture of calorific value 4200 kg.-cal. This practice results in a saving of gas coal, a reduction in the number of retorts needed, and a greater flexibility in dealing with varying loads. Flue gases may be mixed with coal gas, instead of water-gas, but to less advantage. Working results are compared in the case of two plants with vertical retorts, the carbonising times being 12 and 16 hrs. respectively. In the former less water-gas is obtained and the deficiency has to be drawn from producers. The retorts carbonising in 16 hrs. are decidedly more efficient thermally, and avoid the necessity of transporting and grading the coke for the producers. The ash content of the lump coke is not increased by steaming, the superficial mineral matter rubbing off as dust; steaming in this retort does not appear to produce a greater proportion of coke fines. The quantity of



ammonia liquor is increased, but its strength is not diminished, owing to the greater yield of ammonia from vertical retorts. A portion of the hot flue gases may be by-passed to a steam boiler, the remainder passing through the recuperators. Fixed and revolving grates for water-gas producers are discussed, together with slag prevention by correct choice of coke, and prevention of explosions. Reducing the depth of the fuel bed from 1.9 m. to 1.2 m. had little effect on the working of the producer. The water-gas should be added in measured proportion to the coal gas after the latter has passed the tar extractor and scrubbers. The water-gas or mixed gas may be carburetted with crude benzol instead of with gas oil, although the high naphthalene content of the former might be a drawback. The working results of four gasworks prove the great flexibility of vertical-retort practice.

W. T. K. BRAUNHOLTZ.

**Production of blue and carburetted water-gas in continuous vertical retorts.** T. F. E. RHEAD (Gas J., Special No., Feb. 14, 1928, 59—65).—Experiments are recorded on (1) the production of blue water-gas by passing steam through graded coke in a continuous vertical retort, and (2) the cracking of light tar and gas oil under similar conditions. Effects of steam, rate of coke extraction, coke quality and size, temperature, exhaustor "pull," tightness of retort, etc. were investigated. An average hourly make of 4600 cub. ft. (290 B.Th.U. gross) was obtained from 190 lb. of steam and a coke extraction of 126 lb./hr. Thermal efficiency tests showed that 58% of the total heat in coke "consumed" in the retorts plus gross coke to producers appeared as potential heat in gas, and that waste gases could generate more than sufficient steam to be self-supporting. Scuffing of retorts was shown to be possible. The extent to which the steamed coke became deteriorated was demonstrated. Gas oil was cracked in the retort and gave gases of 375—447 B.Th.U. when using 0.97—1.86 gals. per 1000 cub. ft. of carburetted water-gas. Extensive "crude tar" cracking tests proved disappointing and impracticable. Either the seal pots, oftakes, etc. became blocked with pitch, or the retort itself with pitch-coke. Gases of 361—387 B.Th.U. were obtained using 1.73—3 gals. per 1000 cub. ft. Several suggestions of the value of the above water-gas process for emergencies are outlined.

**Grozni oil gases.** P. I. BOGARJEVSKI (Nef. Choz., 1928, 15, 636—638).—The amount of gasoline obtained from natural gas was 9.42 gals. ( $d$  0.700), 5 gals. ( $d$  0.640), and 1.69—3.93 gals. ( $d$  0.635—0.658) per 1000 cub. ft. from three sources, respectively.

CHEMICAL ABSTRACTS.

**[Production of] high-value anti-knock fuels by cracking low-temperature coal tar.** G. EGLOFF (Petroleum, 1929, 25, 573—577).—Tars from the low-temperature carbonisation of West Virginian, Ohio-Indiana, and Utah coals are cracked at 452° and 7 atm., 427° and 7 atm., and 435° and 7.7 atm., respectively, with the production of (1) 22—24% of acid-free benzine of more than 50% benzene equivalent, (2) fuel or Diesel oil, (3) tar acids of low b.p. which are suitable for making phenol condensation

products or as insecticides or wood preservatives, (4) cracked gases of calorific value 1300 B.Th.U. per 1000 cub. ft., and (5) coke of low ash content and calorific value 16,000 B.Th.U. per lb. Neutral oil distillate (freed from tar acids and pitch) from the low-temperature carbonisation of bituminous West Virginian coal, when cracked at 454° and 17 atm., gives more than 50% of petrol of more than 50% benzene equivalent and, per brl. of oil, 85 lb. of practically ash-free coke of calorific value 16,000 B.Th.U. per lb., and 840 cub. ft. of gas of calorific value nearly 1300 B.Th.U. per 1000 cub. ft.

W. S. NORRIS.

**Theory of cracking petroleum.** H. A. WILSON (Proc. Roy. Soc., 1929, A, 124, 16—45).—The cracking of hypothetical oils consisting only of paraffins and unsaturated hydrocarbons is considered. Assuming that the oil has been in the reaction chamber long enough for equilibrium to have been established between all the substances present, and that no coke is deposited in the reaction chamber, and applying the results obtained in previous investigations of the chemical equilibrium in mixtures of paraffins and unsaturated hydrocarbons (cf. A., 1927, 1139; 1928, 1190), the fractions of the oil coming out of the reaction chamber as liquid, vapour, gas, gasoline, and unsaturated hydrocarbons are calculated. The values of these fractions (in weight percentages) for four hypothetical oils having the assumed compositions  $\text{CH}_{2.1}$ ,  $\text{CH}_{2.15}$ ,  $\text{CH}_{2.2}$ , and  $\text{CH}_{2.25}$  (corresponding, roughly, to fuel oil, crude oil, gas oil, and kerosene), at temperatures ranging from 400° to 760° and pressures from 7.5 to 60 atm. are tabulated. It is found that there is good general agreement between the calculated results for the hypothetical oils and the results obtained with actual oils, and it is concluded that the hypothetical oils resemble actual oils sufficiently closely for the theory of the cracking of the former to represent the main features of actual cracking, thus confirming Berthelot's suggestion (Ann. Chim. Physique, 1866, 9, 445) that approximate equilibrium must be actually attained in actual cracking operations. As a question of practical importance in liquid-phase cracking, the amount of a given oil which can be cracked in unit time at any temperature and pressure with a reaction chamber of given volume is considered at some length. In practice, the time of passing through the reaction chamber is usually considerably longer than necessary. It is found that the production of gasoline increases with the amount of oil pumped through, at first rapidly and then more and more slowly. When the amount pumped through is small, the gasoline is nearly proportional to it. It follows as a practical rule from the theoretical results that the amount of oil which should be pumped into the reaction chamber per day is inversely proportional to the gasoline fraction. At a given temperature and pressure the gasoline production for a given reaction chamber should be about the same for any kind of oil, provided that the gasoline fraction is not less than about 10%. In any cracking process the theoretically possible maximum yield of gasoline would be obtained if the oil were converted into gasoline and carbon only, and the possibilities of obtaining such a yield by "recycling" all the products formed except the gasoline are indicated.

Further, a considerable increase in the percentage yield of gasoline should be theoretically obtainable by adding to the oil a small quantity of a mixture of hydrocarbons rich in hydrogen, but no experimental results are yet available for comparison with the theoretical calculations on the effect of adding gas to the oil. The factors affecting the quality of the gasoline produced are discussed, and the theoretical compositions of the gasoline obtained from the oil  $\text{CH}_{2.25}$  are calculated for various temperatures and pressures. L. L. BIRCUMSHAW.

**Insulating oils.** G. STADNIKOV and Z. VOSSCHINSKAJA (Petroleum, 1929, 25, 651—659).—The presence of a sulphonic acid or a salt thereof in a transformer oil may cause the results of an oxidation test to be fallacious. The action of oxygen gives rise to acids, alcohols, and carbonyl compounds. In the presence of a sulphonic acid and of resinous materials, *e.g.*, in an insufficiently refined oil, the products of oxidation condense with the resin or gum (this reaction being accelerated by a sulphonic acid or its salts) and the condensation products remain in suspension, the excess of resin acting as a protective colloid. The acid value of the oil will remain somewhat low, and the oil, according to the oxidation-acid value test, may appear satisfactory although, if it is used for some time in a transformer, sludge will be formed and the acid value will rise. If, however, the oil contains a sulphonic acid, but has been freed from resin by energetic refining, the products of oxidation will be condensed under the action of the sulphonic acid and the products of condensation, in the absence of gum, will be precipitated. Thus a well-refined oil will appear less stable according to the oxidation test than one that contains an appreciable quantity of resin. These conclusions are based on experiments in which fatty acids are heated at  $100^\circ$  in gas-oil solution, with and without the addition of a sulphonic acid, and in the presence of alcohols, aldehydes, or ketones; besides synthetic acids, volatile acids obtained by the oxidation of a transformer oil at  $160^\circ$  are employed. Since sulphonic acids or salts thereof are very difficult to wash out of a transformer oil, their presence in acid-refined oils may account for discrepancies in the results of previous workers. W. S. NORRIS.

**Treating heavy oils by acid sludge.** N. GRAMENETZKI (Nef. Choz., 1928, 15, 212—216).—Heavy oils are treated with acid sludge at  $70$ — $80^\circ$  without preliminary dilution or subsequent distillation.

## CHEMICAL ABSTRACTS.

**Paraffin wax in Apsheron crude oils.** A. N. SACHANOV and L. G. ZHERDEVA (Nef. Choz., 1928, 15, 639—642).—For the determination of paraffin wax the oil (10 g.) was mixed with light gasoline (500 c.c.) and treated at the ordinary temperature with fuller's earth (60—70 g.). The fuller's earth was extracted with gasoline in a Soxhlet extractor for 2 days, the extract evaporated to 10—20 c.c., and the wax was precipitated by Holde's method with alcohol and ether, the solid being redissolved in ether and reprecipitated. The paraffin wax contained in various crude oils was determined and examined. CHEMICAL ABSTRACTS.

**Ultra-violet spectroscopy of flames of motor fuels.** IV. Practical utilisation of a small quartz-

prism spectrograph for the determination of lead tetraethyl in gasoline. G. L. CLARK and H. A. SMITH (J. Physical Chem., 1929, 33, 659—675. Cf. B., 1926, 522; A., 1927, 810).—A small quartz-prism spectrograph provides a simple and accurate means for determining lead tetraethyl in gasoline and its effect on a detonation flame. Details are also given of a simple type of mercury arc, and a combined still and burner for liquid fuels suitable for analyses of this kind. By the method used, 5 g. of lead in  $10^6$  c.c. of gasoline have been detected, and by the use of standard spectra larger quantities can be accurately determined. Seventeen lines in the flame spectrum of lead have been identified, including a hitherto undiscovered line at  $\lambda = 2167 \text{ \AA}$ . The band positions, structures, and intensities have been compared, under similar conditions, for flames of gasoline alone and containing small amounts of ethylene bromide, lead tetraethyl, and aniline, and of benzene, alcohol, acetone, ether, and carburetted water-gas. Further evidence has been obtained that the anti-knock action of lead tetraethyl is due not to the compound *per se* but to the process of its decomposition and to its products of disintegration. L. S. THEOBALD.

**Use of the Parr calorimeter for determining the calorific value of solid fuels.** H. WINKELMANN (Z. tech. Physik, 1928, 9, 422—425; Chem. Zentr., 1928, ii, 2612—2613).

## PATENTS.

**Retort oven.** H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,707,734, 2.4.29. Appl., 31.8.21. Renewed 17.8.28. Ger., 7.12.18).—A combustion chamber containing a group of retorts is divided by partitions transverse to the retorts in such a way that vertical-flame flues are formed alongside each of the retorts and over the tops thereof. Regenerators for preheating the gas and air are arranged below the retort chamber and communicate through distributing channels with corresponding ports at the bottoms of the flues. Means are provided for periodically reversing the direction of gas flow through the flues.

A. B. MANNING.

**Apparatus for coking solid fuel.** E. RAFFLOER (U.S.P. 1,708,152, 9.4.29. Appl., 1.4.25).—A horizontal, rotary drum has longitudinal partitions forming a number of channel-shaped chambers extending along the inner side of the wall of the drum. A rotary, tubular roller is arranged longitudinally within the drum in such a position as to cover the open inner side of the lowermost chamber. As the drum rotates, the chambers are brought in succession below the rotating roller. Means are provided for passing the fuel to be coked first through the roller and then through the chambers of the outer drum, which is externally heated. A. B. MANNING.

**Destructive distillation [of solid fuels].** P. GIRARD, F. PETIT, and A. CHARBONNEAU (B.P. 290,606, 16.5.28. Fr., 16.5.27).—Solid carbonaceous fuels are distilled at low temperatures in the presence of halogens, halogen acids, or compounds, *e.g.*, bleaching powder, which yield halogens under the experimental conditions. The materials are preferably subjected at the same time to the action of a high-frequency electric discharge.

High yields of light hydrocarbon oils are thereby obtained.

A. B. MANNING.

**Apparatus for distillation [of solids].** E. PIRON and V. Z. CARACRISTI, Assr. to PIRON COAL DISTILLATION SYSTEMS, INC. (U.S.P. 1,709,370—1, 16.4.29. Appl., [A] 4.4.22, [B] 14.2.23).—(A) The solid material is conveyed over and adjacent to the surface of a bath of molten material within a distillation chamber. The molten material is heated by means of flues passing through it. (B) The solid material is conveyed over the molten bath on an endless conveyor, from which it falls on to a second endless conveyor which carries it in contact with the return run of the first conveyor to the discharge opening of the chamber (cf. B.P. 207,460; B., 1924, 122).

A. B. MANNING.

**Apparatus for distilling materials.** W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,706,421, 26.3.29. Appl., 20.1.21).—Within a cylindrical casing to which are attached a number of hollow conical, annular hearths projecting radially inwards is a revolving column from which similar hearths project outwards. The two sets of hearths fit into one another to form a confined, inclined, sinuous passageway down which the material to be distilled is passed. Hollow conical rabblers project downwards from the inclined lower surfaces of the hollow hearths almost to the upper surfaces of the next adjacent lower hearths. Hot gases are circulated in the interior of the hearths. Means are provided for charging the material into the apparatus and for discharging the solid product therefrom, as well as for collecting separately the vapours evolved from the material at progressive intervals during its passage through the retort.

A. B. MANNING.

**Distillation of solid carbonaceous materials.** W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,706,420, 26.3.29. Appl., 11.1.23).—The material is distilled in a retort provided with a number of heating mandrels round which the fuel is packed so that on removing the carbonised mass from the retort a series of openings is formed therein.

A. B. MANNING.

**Low-temperature distillation of bituminous coal.** G. E. ROHMER, Assr. to NAT. COAL DISTILLATION CORP. (U.S.P. 1,708,740, 9.4.29. Appl., 23.4.27).—A horizontal rotary retort has external and internal heating members with an annular distillation chamber between them. The outer wall of the chamber is provided with lifting baffles and the inner wall with retarding baffles, the coal being alternately advanced and retarded in its passage through the distillation chamber, and subjected at the same time to different distillation temperatures from opposite sides of the chamber.

A. B. MANNING.

**Low-temperature carbonisation of coal.** W. M. CRANSTON (B.P. 309,231, 13.8.28).—Ground or slack coal is mixed with a mineral oil and distilled, first in a retort maintained at or below 180°, then in a second retort at a higher temperature, preferably 300–500°. The pressure in the first retort is maintained slightly below atmospheric, and that in the second at about 20 lb./in.<sup>2</sup> above atmospheric. Steam may be supplied to the second retort, the contents of which are agitated by stirrers. This retort may be either horizontal or

vertical; in the latter case the temperature is varied at different levels, being highest at the bottom.

A. B. MANNING.

**Manufacture of water-soluble products from lignite and similar fossil materials.** I. G. FARBEIND. A.-G. (B.P. 284,670, 2.2.28. Ger., 3.2.27).—“Degraded” lignite is treated with chlorine in the presence of sufficient alkali to keep the solution neutral or weakly alkaline until the end of the process, when it is permitted to become acid. The products, which are precipitated from the acid solution or may be extracted therefrom by suitable organic solvents, are light-coloured water-soluble substances containing chlorine; they give the Congo-violet reaction and precipitate gelatin from solution. By “degraded” lignite is meant lignite which has been rendered soluble in water, e.g., by boiling with sodium sulphite solution.

A. B. MANNING.

**Operation of internal-combustion engines with pulverulent fuel.** J. Y. JOHNSON. From I. G. FARBEIND. A.-G. (B.P. 310,220, 31.5.28).—Coke, particularly that derived from the low-temperature carbonisation of carbonaceous materials, is conveyed directly from the coking retort through a pulverising mill to the engine in such a way as to conserve as much as possible of its sensible heat. The fuel may be further preheated, if desired, and used with or without the addition of superheated steam. Pulverised coke at the ordinary temperature may be preheated, preferably to about 600°, by admixture with highly superheated steam, and then supplied to the engine.

A. B. MANNING.

**Production of active charcoal.** SOC. ANON. DES ENGRAIS ET NOIR ANIMAL (Addn. No. 32,364, 9.9.26, to F.P. 623,455; B., 1928, 593).—Charcoal is heated gradually to 100° with constant stirring in the presence of sulphuric and phosphoric acids, and the mixture is subsequently heated at 500°.

L. A. COLES.

**Production of active charcoal.** G. WEGELIN (F.P. 631,332, 23.3.27).—Products obtained by the incomplete combustion of hydrocarbons are calcined in the presence of suitable gases or treated with activating material.

L. A. COLES.

**Preparation of highly-active charcoal.** A. SCHREINER (B.P. 292,566, 22.6.28. Ger., 22.6.27).—The carbonaceous matter (wood, peat, lignite, etc.) is impregnated with a solution of potassium thiocyanate (or ferrocyanide), dried, distilled at 300–350°, heated to bright redness in the absence of air for  $\frac{1}{2}$  hr., and leached with as little water as possible.

J. A. SUGDEN.

**Gas producers.** F. L. BROUGHTON and D. HADLINGTON (B.P. 301,208, 3.12.27).—A gas producer is provided with a combination of a supply hopper and a valve chamber, the latter containing three valves which are operated by the rotation of suitably-shaped discs about a vertical axis. One of the valves is rotated intermittently to maintain the required supply of fuel to the producer, whilst another serves to open or close communication between the hopper and the producer. The third valve, which is normally held by a spring device, is designed to move with the intermittently rotated valve in the event of any hard body becoming jammed within the valves. The fuel delivered by the

valves falls on a rotary distributor, which spreads it uniformly over the fuel bed. A. B. MANNING.

**Gas producers.** S. C. G. EKELUND (B.P. 301,241, 30.1.28).—A gas producer for use with an uncoked fuel comprises an upper distillation chamber and a lower gasification chamber, preferably of the ash-melting type. The fuel is distilled by passing part of the producer gas through the upper chamber. The mixed gas so formed, after separation of the tar, if desired, is caused to re-enter and pass through the hot zone of the producer. The gas withdrawn from the main gas outlet in the upper part of the gasification chamber consists substantially of carbon monoxide, hydrogen, and nitrogen only. Moreover, it is at a sufficiently high temperature to enable it to be used for certain metallurgical purposes without further preheating. Instead of an upper distillation chamber, a retort or rotary furnace placed outside, preferably above, the producer may be used. A. B. MANNING.

**Gas generators.** HUMPHREYS & GLASGOW, LTD., Asses. of W. I. BATTIN and C. S. CHRISMAN (B.P. 294,523, 11.4.28. U.S., 25.7.27).—A gas generator which is provided with a cooling jacket has a metallic facing interposed between the fuel bed and the jacket wall. The facing presents a smooth surface permitting the unimpeded descent of the hot fuel; at the same time it reduces the flow of heat to the jacket, and so prevents undue cooling of the periphery of the fuel bed. The facing is preferably made up of detachable blocks, of which only the lower courses need be of metal, the upper courses being of refractory material. Air spaces may be provided in the facing if desired. A. B. MANNING.

**Generation of gas.** C. W. ANDREWS and W. B. CHAPMAN, Assys. to H. A. BRASSERT & Co., and WESTERN GAS CONSTRUCTION Co. (U.S.P. 1,709,335, 16.4.29. Appl., 3.7.26).—Water-gas is made in a generator having a relatively thin fire bed, an intermediate zone of which is maintained at a sufficiently high temperature during the air blast to melt most of the ash. The molten ash passes down to a solidifying zone in which an agitating member breaks up the clinker. A. B. MANNING.

**Manufacture of water-gas.** C. W. ANDREWS (U.S.P. 1,709,107, 16.4.29. Appl., 8.1.25).—Water-gas is made by alternately air- and steam-blasting the fuel bed of a dry-bottom generator with a stationary grate, maintaining in the bed an upper fuel zone, an intermediate clinker-formation zone, and a lower ash zone. The walls of the intermediate zone are water-cooled, and any large masses of clinker which form are broken up by grinding them against one another and against the walls of the generator by imparting an up-and-down agitation to the fuel and ash beds, the latter being also agitated from the centre outwards. A. B. MANNING.

**Production of mixtures of water-gas and gases of distillation.** W. SCHWEDER (B.P. 288,336, 5.4.28. Ger., 6.4.27).—The hot coke discharged from a gas retort is transferred to a water-gas generator into which steam is then introduced until the temperature of the coke has fallen too low for any further production of water-gas. Air is then blown through the coke and the

combustion gases thereby formed are used to preheat the fuel gas supplied to the retort and/or for steam raising. Part of the combustion gases may be used, before or after their utilisation in the waste-heat boiler, for dry-quenching the coke not used in the water-gas generator. A. B. MANNING.

**Separation of gas mixtures.** A. B. RAY, Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,685,883, 2.10.28. Appl., 4.5.23).—The absorption power of activated carbon is independent of its water-content, and in the separation, *e.g.*, of gasoline from natural gas, the carbon may be cooled with running water during the absorption process, and used again for absorption immediately after expulsion of the absorbed gas. R. BRIGHTMAN.

**Gas filter.** C. C. BOARDMAN, Assr. to THERMATOMIC CARBON Co. (U.S.P. 1,710,469, 23.4.29. Appl., 24.9.27).—A number of vertical groups of filters for separating solid particles from a gas are connected to a common horizontal discharge duct, in which a liquid is maintained at a suitable level to form a seal. A pipe associated with the liquid seal controls the back-pressure which is set up in the duct. A. B. MANNING.

**Gas purifier.** F. E. LAMMERT (U.S.P. 1,709,530, 16.4.29. Appl., 24.6.24).—A central flue has lateral openings communicating with a surrounding, concentric cooling chamber. At each end of the latter are annular chambers for the cooling medium, connected by tubes passing through the cooling chamber and provided with partitions for suitably directing the flow of the cooling medium through the tubes. The flue itself is closed by an inclined partition wall, which also extends laterally into the cooling chamber. A. B. MANNING.

**Elimination of sulphur compounds from gases.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,063, 20.10.27).—The gases are mixed with a sufficient amount of air to oxidise the sulphur compounds therein, and are passed over solid active adsorbents, *e.g.*, active charcoal, at 150–250°, or over alloys or compounds of the alkaline-earth metals, aluminium, or the heavy metals with non-metals of the 5th and 6th periodic groups (with the exception of oxygen) at about 400°. Ammonia, in amount necessary for the formation of the corresponding ammonium salts, may be added to the gases either before or after the catalytic treatment and the salts separated, or the sulphur oxides may be washed out with aqueous ammonia. A. B. MANNING.

**Ionisation and hydrogenation of hydrocarbon starting material, vapour, and oil. Dissociation of hydrocarbon fluids and solids for production of gases and liquids of lighter mol. wt.** I. W. HENRY, Assr. to IONIZING CORP. OF AMERICA (U.S.P. 1,709,814—5, 16.4.29. Appl., [A] 4.8.27, [B] 20.3.28).—(A) The reaction chamber is surrounded by a coil through which a high-frequency oscillating current can be passed. The chamber is packed with stationary conducting elements which serve as short-circuited secondaries in the electric system of which the outer coil is the primary. The material to be treated and superheated steam are passed through the interspaces between the elements. (B) A chamber similar in design to that described above is constructed in the form of a still mounted on a chambered

foundation. Sludge which may be deposited on the heating elements or the walls of the still is burnt off by passing a current through the coil when the still is otherwise empty.

A. B. MANNING.

**Distillation of tar and recovery of products therefrom.** BARRETT CO., ASSEES. OF G. E. BRANDON (B.P. 282,367, 28.11.27. U.S., 14.12.26).—Tar, preferably dehydrated and preheated, is sprayed into the hot coke-oven gases in the uptake pipe from the coking chamber; it is thereby rapidly distilled and the resulting pitch is immediately withdrawn. The tar spray is placed at the upper end of the uptake pipe, whilst at the lower end is an inner pipe extending upwards from the chamber and having a baffle above its open end. The pitch is withdrawn from the annular space round this inner pipe. A pitch of any desired m.p. can be produced by suitably regulating the spraying operation or by recirculating the pitch through the still. It is necessary to modify only a few of the individual uptake pipes of a battery in order to distil the whole of the tar produced.

A. B. MANNING.

**Manufacture of asphalt emulsions.** W. H. SCHMITZ (B.P. 284,330, 24.1.28).—Bituminous material (more especially waste liquor obtained in the refining of lubricating oils etc.) is agitated with an alkaline slime containing 1% (calc. on the weight of emulsion) of fuller's earth; a stabiliser (*e.g.*, starch, gum, soap) may be added.

J. A. SUGDEN.

**Removing amorphous wax and asphaltic material from [lubricating] oil.** T. CLARKSON and H. R. HEAL (U.S.P. 1,686,437, 2.10.28. Appl., 29.10.21).—Petroleum-still residue, after removal of gasoline, kerosene, and lighter lubricating oil by distillation, and partial removal of asphaltic impurities by sulphuric acid and alkali treatment (either before or after the distillation), is treated with naphtha or other light petroleum distillate to reduce the viscosity. The mixture, *e.g.*, 60–80% of naphtha and 40–20% of heavy mineral oil, is cooled to 0° to –35°, depending on the "cold test" desired, and filtered under pressure at this temperature, using a filtering material of a close texture comparable with fine filter paper. The filtrate is passed in heat-exchange with the incoming mixture of oil and solvent, to cool the latter, and after treatment with, *e.g.*, fuller's earth, if desired, the solvent is distilled, giving a "bright stock" or cylinder oil of low cold test. The residue in the press is extracted with hot naphtha to dissolve almost the entire residue, and distillation of the naphtha gives a petroleum residue plastic at room temperature, m.p. 55–60°.

R. BRIGHTMAN.

**Economical treatment of mixtures containing hydrocarbon and nitrogen in the electric arc.** SOC. CHEM. IND. IN BASLE, and H. ANDRIESENS (B.P. 296,355, 29.8.28. Switz., 29.8.27).—In the preparation of acetylene from methane, and of hydrocyanic acid from hydrocarbon and nitrogen in the arc, it is necessary to dilute the gases with hydrogen in order to prevent the formation of soot. This dilution, however, reduces the yield per unit of energy consumed. By allowing both reactions to occur simultaneously the efficiency is greatly increased. The portion of acetylene

which is not used in the formation of hydrocyanic acid in the second reaction is separated in amounts sufficient to prevent the formation of soot. A mixture of methane, nitrogen, and hydrogen is circulated through a suitable arc system (B.P. 195,239; B., 1923, 498 A) until the concentration of hydrocyanic acid is about 1% and of acetylene 2–3% by vol. A fraction of the gases is withdrawn and passed first through a solution of caustic soda and then through active charcoal in order to remove the hydrocyanic acid and acetylene. The gases are then returned to the circulator.

J. A. SUGDEN.

**Production of liquid hydrocarbons.** F. J. M. HANSEN (B.P. 284,224, 24.1.28. Switz., 24.1.27).—In the process for hydrogenating hydrocarbons in the electric discharge, the dissociation of the hydrogen is inhibited by the presence of the hydrocarbon. This difficulty is overcome by allowing the dissociation to take place before admixing the hydrocarbon vapour. The dissociation of the hydrogen is maintained by periodically interrupting the admission of hydrocarbon vapour.

J. A. SUGDEN.

**Apparatus for cracking oil.** G. EGLOFF and W. R. HOWARD, ASSTS. TO UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,683,766, 11.9.28. Appl., 23.3.21. Renewed 3.7.26).—The heating tube contains a helical agitator rotated by the passage of the oil, and the agitator has a thrust bearing at the discharge end of the tube.

R. BRIGHTMAN.

**Flue-gas recirculating system for cracking-still operations.** H. A. ATWATER, ASST. TO COMBUSTION EQUIPMENT CO. (U.S.P. 1,709,764, 16.4.29. Appl., 9.5.27).—Combined with the furnace combustion chamber is an initial fuel-combustion zone and a second heat-exchange zone for the still-heating surfaces. A portion of the waste flue gases from the second zone is returned to the combustion chamber through separate discharge ducts opening at opposite sides of the initial combustion zone, which is protected from any chilling effect by partition structures above and below.

H. S. GARLICK.

**Cracking of hydrocarbons.** J. E. BELL, ASSR. TO SINCLAIR REFINING CO. (U.S.P. 1,688,325, 23.10.28. Appl., 22.1.25).—Hydrocarbon oil is circulated from a main supply drum to the lower end of vertical heating tubes, over which the heating gases pass downwards, the velocity being increased, by means of baffles, as their temperature falls. The heated oil leaves the upper end at cracking temperature, and is discharged to the main supply drum, where cracking continues. The vapours escape to a reflux tower and the oil continues in circulation.

R. BRIGHTMAN.

**Cracking of hydrocarbons.** G. EGLOFF and H. P. BENNER, ASSRS. TO UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,688,859, 23.10.28. Appl., 20.12.20. Renewed, 21.11.27).—From 10 to 60% by vol. of a solid bitumen in colloidal form is dispersed in a hydrocarbon oil, *e.g.*, fuel oil, heavy Mexican or Californian crudes, and the mixture is cracked at 400–480° under at least 50 lb./in.<sup>2</sup>

R. BRIGHTMAN.

**Cracking of hydrocarbons.** J. PERL, ASSR. TO SINCLAIR REFINING CO. (U.S.P. 1,689,362–3, 30.10.28.

Appl., 20.11.24).—To prevent or reduce the formation of carbonaceous deposits, the tubular heating coil is lined with (A) tin or (B) iron sulphide. R. BRIGHTMAN.

**Manufacture of lighter products from hydrocarbons.** F. A. HOWARD, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,686,490, 2.10.28. Appl., 16.8.22).—The still containing the hydrocarbon oil is submerged in a liquid heating medium, *e.g.*, molten lead, contact being broken immediately distillation is complete. R. BRIGHTMAN.

**Treating [cracking hydrocarbon] oils.** R. T. POLLOCK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,683,801, 11.9.28. Appl., 7.3.23. Renewed, 15.3.28).—The raw oil is fed to the heating coil after heat-exchange in the dephlegmator out of contact with the cracked vapours. R. BRIGHTMAN.

**Treatment [cracking] of hydrocarbon oils.** C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,686,654, 9.10.28. Appl., 18.8.28).—Relatively heavy hydrocarbon oil is brought to cracking temperature while circulating under pressure with relatively high velocity in a heating coil. The heated oil is passed into tanks under lower pressure, the vapours escaping to a condenser, and the unvaporised oil is recirculated to the heating coil, its relatively slow passage through the tanks allowing free carbon and other solids to deposit therein. R. BRIGHTMAN.

**Cracking of [hydrocarbon] oil. Cracking of petroleum oil.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,688,860—1, 23.10.28. Appl., [A] 31.10.21, [B] 17.10.23. Renewed, [A] 12.3.26, [B] 28.5.28).—(A) Hydrocarbon oil is raised to cracking temperature under pressure, and the unvaporised oil drawn off from the vaporiser is submitted to increased pressure and temperature in a top-fired secondary zone. The dephlegmated products from each stage are collected separately. (B) The residuum accumulating in the vaporiser or reaction vessel of a cracking apparatus is cooled and agitated with liquid sulphur dioxide. The upper layer is withdrawn, freed from sulphur dioxide by sodium hydroxide treatment, and worked up for lubricating stock; the bottom layer after distilling off liquid sulphur dioxide stock affords cylinder oil stock. R. BRIGHTMAN.

**Treating [cracking] residual [hydrocarbon] oils.** L. C. HUFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,683,826, 11.9.28. Appl., 26.5.24).—Residual oil is withdrawn from a cracking still at above 200° and the pressure released in an expansion chamber. The vapours are taken off and the residual oil is strained and circulated to burners, any excess returning under pressure to the bottom of the expansion chamber with turbulence to prevent deposition of carbon. R. BRIGHTMAN.

**Preparation of fuel oil.** W. S. HUGHES and J. HARROP, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,686,491, 2.10.28. Appl., 19.1.27).—The residual oil resulting from the distillation of hydrocarbon oil in presence of alkali is cooled, fluxed with 5–50% of gas oil or other hydrocarbon oil, and emulsified with water. The emulsion is broken, *e.g.*, with dilute sul-

phuric acid, sufficient acid being added to decompose the salts of naphthenic acids or other organic acids and to leave an acid solution, and the oil and aqueous layers are separated. The oil may be distilled to recover the fluxing oil and the naphthenic acids. R. BRIGHTMAN.

**Treating [cracked hydrocarbon] oils.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,683,767, 11.9.28. Appl., 24.3.24. Renewed, 20.2.28).—Residue,  $d\ 0.909$ — $1.06$ , from a cracking process is cooled below 95°, mixed with lighter oil, and centrifuged to give a coke-free fuel oil. R. BRIGHTMAN.

**Petroleum-cracking process and apparatus.** J. J. JAKOSKY (U.S.P. 1,689,590, 30.10.28. Appl., 15.10.25).—A series of vertical tubes allow the cracked vapours to escape readily from the horizontal cracking tube. Liquid particles are removed from these vapours by electrical precipitation methods, the liquid being returned to the cracking tube and the stripped vapours removed and condensed. The temperature of the vertical-tube walls and of the precipitation electrodes is high enough to prevent condensation of desired products, but low enough to avoid further cracking. R. BRIGHTMAN.

**Conversion [cracking] of petroleum oils.** A. D. DAVID, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,688,855, 23.10.28. Appl., 14.2.23. Renewed, 24.4.28).—Petroleum oil is raised to cracking temperature in a heating coil and passed into an expansion chamber, where a swirling motion is maintained by means of baffle plates and local circulation of oil with a pressure pump. The carbon particles resulting from the cracking process remain in suspension, and the residual oil is continuously drawn off into settling tanks, the carbon being separated before the oil comes into circulation again. The vapours escape from the expansion chamber through a dephlegmator in the usual manner, the condensate being circulated through the heating coil. R. BRIGHTMAN.

**Refining of hydrocarbon oils.** E. B. PHILLIPS and J. G. STAFFORD, Assrs. to GRAY PROCESSES CORP. (U.S.P. 1,687,992, 16.10.28. Appl., 29.5.26).—Petroleum distillates free from water are treated successively with crystalline cupric chloride and fuller's earth, or other suitable solid absorbent. R. BRIGHTMAN.

**Refining of oils etc.** W. K. LEWIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,686,493, 2.10.28. Appl., 23.8.23).—Oil is clarified with, *e.g.*, fuller's earth, and passes forward by decantation in a series of units. Refined oil is drawn off at the end of the series, and the earth in the first unit is used for a fresh charge of oil until it is no longer efficient. The soakage oil is removed, and the spent earth is extracted with solvent naphtha, dried, and discharged, for revivifying if desired, as a sandy powder. The unit is recharged with fresh earth and brought back into operation as the final unit of the series, crude oil being introduced at the second, third, fourth, etc. units in turn as the earth in each becomes inefficient. R. BRIGHTMAN.

**Production of hydrocarbons of higher b.p. from gaseous and low b.p. hydrocarbons.** A. S.

RAMAGE, Assr. to GYRO PROCESS CORP. (U.S.P. 1,687,890, 16.10.28. Appl., 13.5.25).—Hydrocarbon vapours, *e.g.*, from the lighter fractions of casing-head gasoline, are mixed with about 10% of steam and passed over ferrous oxide at 600–700°, giving a motor fuel, b.p. 20–210°, consisting essentially of cycloparaffins, cycloolefines, and polymerised olefines.

R. BRIGHTMAN.

**Imparting fluorescence to [lubricating] oil. Fluorescent product.** J. C. BLACK, W. D. RIAL, and J. R. MCCONNELL, Assrs. to PAN AMER. PETROLEUM Co. (U.S.P. 1,708,563 and 1,708,602, 9.4.29. Appl., [A] 13.8.26, [B] 6.5.27).—(A) Fluorescent substances are extracted from uncracked petroleum pitch with a non-fluorescent lubricating oil, which is then separated therefrom. (B) A concentrated extract of green fluorescent substances is obtained by mixing a mineral lubricating oil with a coal-tar product containing them and then separating the lubricating oil from the insoluble coal-tar residue.

H. S. GARLICK.

**Renovation of journal-box oil.** T. W. POTTER, L. C. MCNAMARA, C. N. LAMMERS, and J. R. MCLAVY, Assrs. to JOURNAL-BOX SERVICING CORP. (U.S.P. 1,709,230, 16.4.29. Appl., 9.5.28).—Oil is separated from lint and foreign matter by distillation in a closed tank under a controlled pressure insufficient to bring about cracking.

H. S. GARLICK.

**Preparation of valuable compounds from the refining wastes of cracked products, particularly cracked benzenes.** GALICYJSKIE TOWARZYSTWO NAF-TOWE "GALICJA" S.A., W. DE PIOTROWSKI, and J. WINKLER (B.P. 309,718, 26.3.28).—Waste sulphuric acid from the refining of cracked benzene is diluted with water or dilute alkali (*e.g.*, waste lye from the subsequent soda-washing of the benzene) and the aqueous and oily layers are treated separately. The aqueous layer is neutralised and distilled with superheated steam, and the distillate (b.p. 80–150°) is then rectified giving, *e.g.*, butyl, amyl, and hexyl alcohols. The oily layer is washed with alkali and with water and is then heated to 110° and steam is introduced. The fraction of b.p. 50–200° is refined by means of alkali hydroxide and sodium plumbite and redistilled, giving a yellow fraction, b.p. 140–200°, flash point not below 50°, which has a characteristic odour and possesses insecticidal properties; the fraction of b.p. 200–280° is boiled with a manganese or cobalt siccativ to give a varnish-like product. The residue, b.p. above 280°, is cooled to 140° and is then air-blown, yielding asphalt-like products of high elasticity, which, being soluble in benzene, can be employed as lacquers.

W. S. NORRIS.

**Heat-treatment of hydrocarbon gases.** ANGLO-PERSIAN OIL Co., LTD., A. E. DUNSTAN, and R. V. WHEELER (B.P. 309,455, 8.10.27).—For the production of aromatic hydrocarbons, a stream of gas containing gaseous paraffins is gradually heated to a temperature (*e.g.*, below 550°) substantially below that at which eventual decomposition is to be carried out. It is then heated in a determined course to a temperature (650–975°) at which the paraffins are decomposed and aromatic hydrocarbons are formed. The stream of gas is then suddenly reduced in velocity, *i.e.*, expanded, and cooled, and the free carbon formed is deposited; finally

the aromatic hydrocarbons are condensed. If necessary, the original gas is previously freed from hydrogen sulphide, *e.g.*, by limited oxidation and deposition of elementary sulphur, and from condensable hydrocarbon vapours. The yield of aromatic hydrocarbons is increased by diluting the gas with an inert gaseous material (steam), which is preferably introduced into the gas stream between the preheater and the reaction tubes. The reaction temperature is controlled according to which paraffin or mixture of paraffins is present. The temperatures are 850–975°, 800–900°, 750–850°, 700–800°, or 650–750° for methane, ethane, propane, butane, or pentane, respectively; for mixtures, a range of temperature is chosen according to the average proportions of the constituents. The velocity of the gas in the preheater tubes and in the reaction tubes is so maintained that the flow is turbulent, giving good heat-transference and maintaining the carbon particles in suspension.

W. S. NORRIS.

**Manufacture of nitrogenous bases from hydrocarbon materials.** H. K. IHRIG, Assr. to S. E. CAMPBELL and ASSOCIATED OIL Co. (U.S.P. 1,686,136, 2.10.28. Appl., 21.12.26).—Crude gasoline, after removal of sulphur compounds by treatment with sodium hydroxide, is treated with an acidic reagent, *e.g.*, 25% sulphuric acid. The acid liquor is made alkaline and steam-distilled to separate the nitrogenous bases soluble in water from those insoluble therein. The products have insecticidal properties.

R. BRIGHTMAN.

**Apparatus for distilling bituminous substances.** H. KOPPERS (U.S.P. 1,712,083, 7.5.29. Appl., 29.9.22. U.K., 28.6.22).—See B.P. 203,444; B., 1924, 548.

**Apparatus for treatment of liquid hydrocarbons.** S. J. M. AULD, A. E. DUNSTAN, and P. H. HERRING, Assrs. to ANGLO-PERSIAN OIL Co., LTD. (U.S.P. 1,711,219, 30.4.29. Appl., 11.2.24. U.K., 20.2.23).—See B.P. 220,664; B., 1924, 899.

**Apparatus for grinding coal and like material.** W. T. BELL and J. F. BENNETT (B.P. 311,505, 20.3.28).

**Conveying and quenching of coke.** W. J. JENKINS & Co., LTD., and R. M. GOODMAN (B.P. 311,487, 28.2.28).

**Preparation of charges for use in internal-combustion engines.** A. S. JOHN (B.P. 311,165, 4.9.28).

**Burner for coal dust firing.** BÜTTNER-WERKE A.-G., and H. ZIKESCH (B.P. 311,019, 13.2.28).

**Agitators or mixing devices. [Fuel burners.]** A. C. PRIOR (B.P. 308,054, 21.2.28).

**Carbide cartridge for use in acetylene gas lamps.** C. A. MONHAM and E. G. FIRTH (B.P. 311,517, 31.12.28).

**Apparatus [press] for expressing liquid from solid materials [e.g., peat].** H. SKÖLDBERG (B.P. 284,318, 13.1.28. Swed., 29.1.27).

**Drying etc. of granular materials** (U.S.P. 1,705,617). **Simultaneous drying and grinding** (U.S.P. 1,702,333). **Separating volatile fluids from liquids** (B.P. 308,010). **Separating dust, smoke, etc. from smoke** (B.P. 307,168).—See I. Lead tetraethyl (U.S.P. 1,690,075).—See VII. Road surfaces (B.P. 301,891 and 309,634).—See IX. Flotation

oil (U.S.P. 1,688,975). Anti-corrosive preparation (B.P. 310,640).—See X. Dryers for drying oils (U.S.P. 1,686,484—6).—See XIII.

### III.—ORGANIC INTERMEDIATES.

**Preparation of acetic anhydride.** E. FRITZMANN (J. angew. Chem. [Russia], 1928, 1, 1, 27—36; Chem. Zentr., 1928, ii, 2548).—Optimal conditions for the preparation of acetic anhydride by the action of chlorine on a mixture of acetate and sulphur (G.P. 372,528; B., 1923, 1200 A), and by the action of toluene-*p*-sulphonyl chloride on an acetate are described.

A. A. ELDRIDGE.

**Simple method for the determination of acetaldehyde.** Y. TOMODA (J.S.C.I., 1929, 48, 76—77 T).—The relation between the  $p_H$  and the dissociation of the sodium hydrogen sulphite compound of acetaldehyde has been investigated; at  $p_H$  6—8 dissociation is inappreciable, but above  $p_H$  8 it increases rapidly, becoming complete at about  $p_H$  12. Thus, using 0.1*N*-iodine, below  $p_H$  2 free sodium hydrogen sulphite only is titrated, and at about  $p_H$  8 the combined sodium hydrogen sulphite is also titrated. Since hydriodic acid is produced, the  $p_H$  of the medium falls, necessitating the addition of alkali; if the alkalinity of the solution rises above  $p_H$  9 oxidation of the aldehyde by the iodine occurs, so that in order to titrate both free and combined sodium hydrogen sulphite the  $p_H$  of the medium must be maintained at about  $p_H$  8; this is accomplished by addition of a large excess of sodium hydrogen carbonate. By employing these conditions, a simple and accurate method for the determination of acetaldehyde is obtained, which can be applied not only to the direct determination of acetaldehyde in the presence of sulphite, but also to the converse case. Procedure is described in detail.

C. W. SHOPPEE.

**Determination of alcohol in the presence of acetaldehyde.** Y. TOMODA (J.S.C.I., 1929, 48, 77—79 T).—When an aqueous solution of acetaldehyde is subjected to aeration, volatilisation of the aldehyde can be prevented completely by sodium hydrogen sulphite, provided that the solution has an acid reaction. On this basis a method for the determination of alcohol in the presence of acetaldehyde is described, which is accurate to 3%. By employing a large excess of sodium hydrogen sulphite the volatilisation of the alcohol is accelerated owing to its increased relative partial pressure, due to the presence of the dissolved salt. The presence of formic acid, acetic acid, glycerol, or sucrose does not interfere with the method, and with dilute solutions containing less than 1% of alcohol, increased accuracy may be obtained by using a larger volume of the solution under test and an increased amount of sodium hydrogen sulphite. Apparatus and procedure are fully described.

C. W. SHOPPEE.

**Bromometric determination of phenol and the cresols.** H. DITZ (Z. anal. Chem., 1929, 77, 186—202).—Polemical against Järvinen (B., 1927, 596) and a claim for priority together with a critical survey of recently published methods for the bromometric determination of phenols.

A. R. POWELL.

**Determination of nitrogen in dyes and their intermediate products by the Kjeldahl method.** P. SISLEY and M. DAVID (Bull. Soc. chim., 1929, [iv], 45, 312—324).—Low results obtained in the determination of nitrogen in nitrobenzene, *p*-nitrotoluene, and *o*-chloronitrobenzene, using the modified Kjeldahl method of Sisley (B., 1907, 605), are due to volatilisation; accurate results are obtained if the sample is first sulphonated, e.g., by heating with 30% oleum at 100—120°. The method is not general, the loss being reduced only with *o*-nitrotoluene, *m*-dinitrobenzene, *p*-chloronitrobenzene, picric acid, and dinitronaphthol (calcium salt), whilst in the case of dinitrophenetole and hexanitrodiphenylamine preliminary sulphonation leads to lower results. With picric acid the loss of nitrogen by Sisley's method (*loc. cit.*) is 25%, and is not reduced by the addition (to 0.5 g. of picric acid) of 1 g. of any of the following substances: benzene, toluene, benzoic acid, phthalic anhydride, oxalic, acetic, or formic acids. Phenol, furfuraldehyde, tartaric acid, acetone, and gallic acid appreciably reduce the loss, and accurate results are obtained in the presence of 1 g. of dextrose, sucrose, benzoquinone, resorcinol, anthracene, lactic acid,  $\beta$ -naphthol, quinol, pyrocatechol, and pyrogallol, the last three giving the best results; accurate results are obtained in presence of 0.3 g. of pyrogallol with many nitro-compounds and with nitroso- and azoxy-compounds, but not with all azo compounds. Since replacement of the pyrogallol by 5 g. of phosphorous acid gives accurate results, the favourable action of pyrogallol is attributed to its reducing action on the sulphuric acid. Sisley's method (*loc. cit.*) is satisfactory for azo compounds containing the azo group *para* to the hydroxyl or amino-group, but *o*-hydroxy- or *o*-amino-azo compounds yield low results. The following method gives accurate results for nitrogen in azo compounds generally, and in intermediate products with the exception of pyrazolones (cf. Ranker, B., 1927, 536). The substance (0.5—1 g.) is warmed in a 250 c.c. pyrex flask with 10 c.c. of alcohol and 5 c.c. of water, 2—4 g. of sodium thiosulphate are added, 1 g. at a time and with boiling and cooling after each addition, followed by 10 c.c. of sulphuric acid, *d* 1.84, and the flask is gently heated to expel alcohol; when the liquid becomes spongy 0.5 g. of copper sulphate, 6—8 g. of potassium sulphate (10 g. less the weight of thiosulphate used), and 12 c.c. of sulphuric acid are added. The mixture is heated until the liquid is clear blue (20—30 min.), diluted to 300 c.c., and the ammonia determined as usual, 5 c.c. of 20% sodium sulphide solution and a little granulated zinc being added with the sodium hydroxide before distillation.

R. BRIGHTMAN.

**Unsaturated compounds as germicides.** CHEESEWORTH and COOPER.—See XXIII.

### PATENTS.

**Apparatus for production of carbon disulphide.** I. G. FARBERIND. A.-G. (B.P. 291,382, 30.5.28. Ger., 31.5.27).—A subsidiary superheating retort (A) for the sulphur vapour is combined in one casting with the main retort (to contain carbon) and extends along its entire length. Retort A is filled with refractory material resistant to sulphur, or, preferably, has horizontal ribs



cast in its inner wall to support flat dishes of refractory material which are assembled so that openings therein are staggered with respect to each other, thereby lengthening the path of the sulphur vapour. The superficial area of this retort is at least one half of that of the main retort, and the lining of the latter covers the opening to retort A from the top nearly to the bottom.

W. G. CAREY.

**Manufacture of olefine alcohols.** F. W. LOMMEN, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,688,083, 16.10.28. Appl., 7.6.26).—Olefinic aldehydes are reduced by heating with a paraffin alcohol in presence of aluminium alkoxide as catalyst. Thus cinnamaldehyde, when heated at 100° with a slight excess of butyl alcohol containing aluminium ethoxide, affords a 95% yield of cinnamyl alcohol, with butyl butyrate as by-product.

R. BRIGHTMAN.

**Dehydration of alcohols.** S. R. MERLEY, Assr. to DOHERTY RES. CO. (U.S.P. 1,688,731, 23.10.28. Appl., 22.12.25).—Amyl and higher alcohols are dehydrated by adding a lower alcohol, e.g., *sec.*-butyl alcohol from the still butts obtained in its rectification, which forms an azeotropic mixture with water. Volatile odorous constituents are removed at the same time.

R. BRIGHTMAN.

**Hydrolysis of methyl chloride.** R. H. MCKEE (U.S.P. 1,688,726, 23.10.28. Appl., 7.9.22).—Methyl chloride, from the limited chlorination of natural gas or methane, is scrubbed to remove hydrochloric acid, mixed with an equal volume of steam, e.g., by bubbling through water at an appropriate temperature, and passed over an alkaline-earth hydroxide, e.g., aluminium, calcium, barium, or magnesium hydroxide, at 350–375° in an aluminium tube. The aqueous solution obtained on cooling the issuing gases affords methyl alcohol and some methyl ether, free from acetone.

R. BRIGHTMAN.

**Synthesis of [organic] nitrogen compounds, and of liquid hydrocarbons.** H. E. POTTS. From H. PLAUSON (B.P. 309,001—2, 30.12.27).—(A) Mixtures of nitrogen and water-gas or Dowson gas containing suspended, finely-divided particles of magnesia or other catalyst are subjected to the action of  $\beta$ - and X-rays, and the product is then subjected to high temperature and pressure. Carbamide, uric acid, and ammonium carbamate are stated to be among the products of the reaction. (B) Mixtures of gaseous saturated and/or unsaturated hydrocarbons or of hydrogen and carbon monoxide are subjected to a similar treatment as in (A) to obtain liquid hydrocarbons having b.p. below 150°.

A. R. POWELL.

**Catalytic manufacture of thiocarbamides.** C. N. HAND and H. P. ROBERTS, Assrs. to RUBBER SERVICE LABS. CO. (U.S.P. 1,688,707, 23.10.28. Appl., 1.10.24).—Aromatic amines are heated at 46–46.2° with about 60% of carbon disulphide in presence of an alkaline catalyst volatile below 75°, e.g., 0.1–0.25% of aqueous ammonia, *d* 0.897. The hydrogen sulphide vapours evolved are scrubbed by passing through a tank containing a second charge of amine and catalyst, this charge being admitted to the mixer when the reaction in the first charge is complete, and the excess of carbon disulphide and catalyst have been recovered by distillation at 75°.

R. BRIGHTMAN.

### Purification of benzoic acid and its derivatives.

A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,686,913, 9.10.28. Appl., 18.10.27).—Benzoic acid is separated from phthalic acid by distillation with pressure-steam below 191° (at which temperature phthalic acid is converted into its anhydride). The aqueous solution may be distilled under pressure, and the process made continuous by means of a countercurrent of steam, or the mixture may be vaporised and blown into a vessel counter to a blast of superheated steam; the vapours may be removed and cooled so that benzoic acid but very little water condenses, or the benzoic acid may be extracted from the steam above 100° with high-boiling solvents. Benzoic acid may similarly be recovered from effluent converter gases by treating the gases with steam or steam and water to reduce the temperature below 191°, when the phthalic acid separates. By treating the gases from a vapour-phase catalytic oxidation of naphthalene with a carbon dioxide-splitting catalyst and sufficient steam, the production of benzoic acid from naphthalene can be effected in one operation.

R. BRIGHTMAN.

### Production of styrene from chloroethylbenzene.

O. H. SMITH, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,687,903, 16.10.28. Appl., 4.10.27).—Halogen derivatives of alkylbenzenes, e.g., the mixture of  $\alpha$ - and  $\beta$ -chloroethylbenzenes obtained by chlorinating ethylbenzene below 30°, or the mixture of *o*-, *m*-, and *p*-chloroethylbenzenes, b.p. 90–105°/50 mm., obtained from chlorobenzene and ethylene in presence of aluminium chloride, are heated to 600–725° in a cracking tube, and the styrene or alkylstyrene is separated in known manner.

R. BRIGHTMAN.

### Production of phthalic anhydride.

F. A. CANON and C. E. ANDREWS, Assrs. to SELDEN Co. (U.S.P. 1,689,860, 30.10.28. Appl., 7.11.25).—Temperature control in the catalytic oxidation of naphthalene is effected by a cadmium-mercury alloy, containing 25% Cd, b.p. about 400°.

R. BRIGHTMAN.

**cycloHexyl alkyl phthalates.** R. ADAMS and J. F. HYDE, Assrs. to NEWPORT Co. (U.S.P. 1,689,761, 30.10.28. Appl., 11.6.26).—Phthalic anhydride is heated with 1 mol. of cyclohexanol or a substituted cyclohexanol, and the reaction product esterified with an alcohol in presence of an acid catalyst. *cycloHexyl hydrogen phthalate*, m.p. 95°, *cyclohexyl methyl phthalate*, b.p. 168°/2 mm., *cyclohexyl ethyl phthalate*, b.p. 180°/5 mm., 219–220°/21 mm., *cyclohexyl n-propyl phthalate*, b.p. 173–178°/1.5–2 mm., *cyclohexyl isopropyl phthalate*, b.p. 168–172°/1.5–2 mm., and *cyclohexyl n-butyl phthalate*, b.p. 186–188°/1.5–2 mm., are described.

R. BRIGHTMAN.

### Organic solvents. [cycloHexyl alkyl phthalates.]

R. ADAMS and J. F. HYDE (U.S.P. 1,689,762, 30.10.28. Appl., 8.12.26).—*cycloHexyl alkyl phthalates* are used as solvents for cellulose esters. The following are described: *4-Methylcyclohexyl ethyl phthalate*, b.p. 160°/3 mm.; *4-methylcyclohexyl n-butyl phthalate*, b.p. 183°/2–2.5 mm.; *3-methylcyclohexyl ethyl phthalate*, b.p. 168°/3.5 mm.; *3-methylcyclohexyl n-butyl phthalate*, b.p. 178°/2 mm.; *2-methylcyclohexyl ethyl phthalate*, b.p. 168°/3.5 mm.; *2-methylcyclohexyl n-butyl phthalate*, b.p. 185°/3 mm.

R. BRIGHTMAN.

**Reduction of aromatic nitro-compounds.** P. DIETERLE, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,689,014, 23.10.28. Appl., 4.1.26).—Aromatic nitro-compounds are reduced to azoxy-, azo, hydroxy-, amino-, and nitroamino-compounds by heating with ferrous sulphide in neutral or alkaline media in presence or absence of organic solvents. Thus 2:4-dinitrophenol is converted into 4-nitro-2-aminophenol by heating its sodium salt at 40–80° for  $\frac{1}{2}$ –2 hrs. with precipitated ferrous sulphide. Similarly, equal parts of *m*-dinitrobenzene and ferrous sulphide in water at 80–85° for 1–2 hrs. yield *m*-nitroaniline, and nitrobenzene is converted into aniline by heating it in 3% sodium hydroxide solution at 99–102° with ferrous sulphide for 2 hrs. With 6 pts. of nitrobenzene and 4.5–6 pts. of ferrous sulphide in 15 pts. of 40–48% sodium hydroxide at 80–85° for 2–3 hrs. about 20–25% of aniline and 65–75% of azobenzene are obtained. After removal of aniline and dilution to give 20–30% sodium hydroxide, the azobenzene can be converted into hydrazobenzene by heating at 70–80°.

R. BRIGHTMAN.

**Production of polymerised vinyl acetate.** W. O. HERRMANN and W. HAEBNEL, Assrs. to CONSORT. F. ELEKTROCHEM. IND. (U.S.P. 1,710,825, 30.4.29. Appl., 6.11.26. Ger., 14.11.25).—See B.P. 261,406; B., 1927, 823.

**Manufacture of *o*-aminodiaryl ethers.** A. ZITSCHER, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,703,915, 5.3.29. Appl., 16.12.27. Ger., 24.12.26).—See B.P. 307,130; B., 1929, 349.

**Preparation of homogeneous pure 2:3- and 2:5-dichloro-4-amino-1-methylbenzene [2:3- and 2:5-dichloro-*p*-toluidines].** W. SCHUMACHER and C. SEIB, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,712,173, 7.5.29. Appl., 3.2.28. Ger., 30.3.26).—See B.P. 294,078; B., 1928, 704.

**Manufacture of tetranitrodianthrone.** B. STEIN, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,686,992, 9.10.28. Appl., 28.6.26. Ger., 25.6.25).—See B.P. 289,958; B., 1928, 516.

**Preparation of monodiazo compounds of 1:4-diaminoanthraquinone- $\beta$ -sulphonic acids.** G. KRÄNZLEIN and F. ROEMER, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,690,021, 30.10.28. Appl., 18.1.27. Ger., 25.1.26).—See B.P. 264,879; B., 1928, 8.

**Apparatus for chemical reactions** (U.S.P. 1,705,614).—F.p. depressants (U.S.P. 1,687,094).—See I. Treating hydrocarbon-nitrogen mixtures (B.P. 296,355). Products from waste cracked benzines (B.P. 309,718).—See II. Flotation of ores (U.S.P. 1,686,529).—See X. Light filter (U.S.P. 1,688,259).—See XI. Stable sulpho-acids and salts (B.P. 288,126).—See XII. Vapours from alcohol (B.P. 310,400 and 310,403).—See XVIII.

#### IV.—DYESTUFFS.

**Gases emitted in the manufacture of sulphur dyes.** G. V. KOGAN (J. Chem. Ind. Moscow, 1928, 5, 799–801).—A study of the quantities of hydrogen

sulphide and ammonia which are evolved in definite time intervals in the manufacture of sulphur dyes, and of the conditions of maximum evolution.

CHEMICAL ABSTRACTS.

**Determination of nitrogen in dyes.** SISLEY and DAVID.—See III.

PATENTS.

**[Preparation of] dyes of the anthraquinone series.** K. WEINAND, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,688,256, 16.10.28. Appl., 23.12.27. Ger., 20.12.26).—*N*-Substituted aminoanthraquinone dyes giving light-fast, pure blue shades on animal fibres are obtained by the action of alicyclic amines, not of the hydrogenated benzene or naphthalene series, on a halogenated anthraquinonesulphonic acid in presence of a copper catalyst. Thus sodium 4-bromo-1-aminoanthraquinone-2-sulphonate in aqueous alcohol in presence of copper sulphate affords 1-amino-4-cyclo-pentylaminoanthraquinone-2-sulphonic acid and 1-amino-4-bornylaminoanthraquinone-2-sulphonic acid.

R. BRIGHTMAN.

**[Production of] monoazo dyes from pyrazolones and anilinepolysulphonic acids.** L. W. GELLER, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,688,044, 16.10.28. Appl., 23.12.25).—Yellow to red or brown dyes for wool are obtained by coupling a diazotised anilinedisulphonic acid with a 1-aryl-3-methyl-5-pyrazolone or 1-aryl-5-pyrazolone-3-carboxylic acid, e.g., aniline-2:4-disulphonic acid  $\rightarrow$  1-(4-sulpho-2-tolyl)pyrazolone-3-carboxylic acid. R. BRIGHTMAN.

**[Production of] monoazo dyes from disulpho-naphthylpyrazolones and *o*-aminophenols.** L. W. GELLER, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,688,045, 16.10.28. Appl., 23.12.25).—Monoazo dyes for lakes and pigments or for dyeing wool from an acid bath in yellow to red or brown shades, changed to yellowish- or bluish-red on after-chroming, are obtained by coupling a diazotised *o*-aminophenol-carboxylic or -sulphonic acid with a 1-(4:8-disulpho-2-naphthyl)-5-pyrazolone-3-carboxylic acid or the corresponding 3-methylpyrazolone.

R. BRIGHTMAN.

**Readily-soluble vat colour preparations.** W. BAUER, A. HERRE, and A. FUNKE, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,711,008–9, 30.4.29. Appl., [A] 29.11.24, [B] 17.5.26. Ger., 10.12.23).—See F.P. 589,914; B., 1926, 154.

**Preparation of dyes derived from diphthalimido-anthraquinones.** E. G. BECKETT and J. THOMAS, Assrs. to SCOTTISH DYES, LTD. (U.S.P. 1,711,873, 7.5.29. Appl., 28.7.24. U.K., 14.9.23).—See B.P. 231,206; B., 1925, 393.

**Monoazo dye.** W. DUISBERG, W. HENTRICH, and L. ZEH, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,711,930, 7.5.29. Appl., 16.1.25. Ger., 28.2.24).—See B.P. 230,055; B., 1925, 875.

**Manufacture of azo dyes for dyeing cellulose esters.** O. KNECHT, Assr. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,711,390–1, 30.4.29. Appl., [A] 23.12.25, [B] 16.3.27. Ger., [A, B] 1.6.25).—See B.P. 245,758; B., 1927, 469.

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

**Determination of wool in raw- and wool-felt pastes by chemical means.** B. SCHULZE (Papier-Fabr., 1929, 27, 299—301).—Dissolution of the wool by treating a weighed sample of the material with boiling 2% sodium hydroxide solution under standard conditions and reweighing the residue gives results for the wool content which are too high, since other materials besides the wool are thereby partially dissolved. Thus, cotton loses 4.2%, jute 16.3%, wood cellulose 4.9%, and mechanical wood pulp 4.7% by weight under these conditions. On the other hand, if the vegetable fibres are removed by sulphuric acid, leaving the wool unchanged, better results are obtained. The material is treated with 80% sulphuric acid for 3 hrs. with shaking, the mixture is poured into a large volume of water, the wool residue washed to neutrality, dried at 105—110°, and weighed. It is then found that a 0.04% increase in weight of the wool occurs, cotton is completely dissolved, whilst jute leaves 0.8%, wood cellulose 0.1%, and mechanical wood pulp 12.1% of insoluble matter. These differences are negligible except in the case of mechanical wood pulp, but use of the latter in the materials referred to is forbidden, so that its adverse effect may be disregarded. Results obtained for the analysis of different known mixtures show that the sodium hydroxide method gives values 5—9% higher than the theoretical, whereas those for the sulphuric acid method vary from 0.5% too low to 0.1% too high. Natural silk, and viscose, nitrate, cuprammonium, and acetate silks are all completely dissolved by the above acid treatment, but Tussah silk leaves a residue of about 10%. B. P. RIDGE.

## PATENTS.

**Article made of fibrous material and its production.** W. H. KOBÉ, Assr. to TEXAS GULF SULPHUR Co. (U.S.P. 1,689,394, 30.10.28. Appl., 14.3.27).—Fibrous material is treated with wet, calcined gypsum, shaped, and, after hardening and drying, impregnated with molten sulphur. R. BRIGHTMAN.

**Production of fibres from coconuts.** L. THOMAS and E. ELÖD (B.P. 310,396, 24.1.28).—Coconut husks are steeped in hot water which is maintained in motion, sufficient lime being added to prevent discoloration of the fibre. After subjection to mechanical disintegration they are given a further steeping, the fibres being finally freed and isolated in the usual way. F. R. ENNOS.

**Manufacture of cellulose acetate.** J. W. BULMER (B.P. 300,824, 2.1.28).—After steeping cellulose in glacial acetic acid for several hours in order to secure uniform absorption, the mixture is cooled until the acid crystallises, thereby causing some mechanical disruption of the cellulose, which thus becomes more receptive to the action of the acetylating mixture. F. R. ENNOS.

**Acetylation of cellulose material.** S. A. OGDEN (B.P. 310,563, 28.1.28).—Cellulose material, prepared according to B.P. 246,746 (B., 1926, 975), is treated with acetic acid and dried by heat, the partially acetylated product being then further acetylated with anhydride in the usual way. F. R. ENNOS.

**Esterification of mercerised cellulose with lower aliphatic acids.** H. T. CLARKE and C. J. MALM, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,687,059, 9.10.28. Appl., 23.4.27).—Mercerised cellulose, after washing free from alkali and drying at 30—40°, is heated with an aliphatic acid, C<sub>2</sub>—C<sub>7</sub>, for about 48 hrs. at 100—170° until the product contains at least 8% of acyl group. With heptic acid the temperature of acylation should be 145—165°. The operation may also be effected under pressure, and acetylation at 140—160° is complete in 10 hrs. Acylation may also be effected with the acid vapours under normal or reduced pressure. During the acylation water is removed by use of a fractionating column.

R. BRIGHTMAN.

**Pulping of wood.** F. G. RAWLING (U.S.P. 1,705,827, 19.3.29. Appl., 17.9.27).—Wood is digested under pressure at 190° with a solution containing sodium sulphide, sodium hydrogen carbonate, and carbon dioxide. A. R. POWELL.

**Manufacture of artificial threads, bands, films, etc. from solutions of cellulose esters and ethers.** RUTH-ALDO Co., Inc., Asses. of H. L. BARTHELEMY (B.P. 308,271—3, 28.12.27. Fr., 28.12.26).—A cellulose ester or ether is precipitated in a suitable form from its solution in a volatile solvent by means of (A) a hydrocarbon of b.p. above 80°, e.g., toluene, or an alicyclic hydrocarbon, e.g., cyclohexane, (B) a higher fatty acid ester of a monohydric alcohol, e.g., ethyl stearate, (C) a halogenated hydrocarbon, e.g., trichloroethylene, an aromatic nitrile, e.g., benzonitrile, or a nitrated aromatic hydrocarbon, e.g., nitrobenzene. F. R. ENNOS.

**Manufacture of filaments or threads of cellulose derivatives.** H. DREYFUS (U.S.P. 1,688,532, 23.10.28. Appl., 16.9.26. U.K., 29.3.26).—Addition of 0.5—15% (preferably 0.5—5%) of a high-boiling solvent or plasticiser, e.g., diacetin, triacetin, toluene- or xylene-sulphon-methyl- or -ethyl-amide, to the ripened cellulose ester solution, especially those with viscosities above 50°, gives on dry-spinning threads of considerably enhanced strength. The plasticiser may be removed from the resulting filaments by washing or extraction with liquids non-solvent for the cellulose derivative. R. BRIGHTMAN.

**Spinning of artificial silk.** I. G. FARBENIND. A.-G. (B.P. 279,888, 28.10.27. Ger., 30.10.26).—Viscose is spun into fine-denier threads of high extensibility and uniform dyeing capacity by projecting it vertically upwards from nozzles through a short length of the precipitating liquid, without the use of a second bath, in such a manner that the threads are not subjected to stretching or bending at an angle smaller than 150° during their passage to the bobbins. F. R. ENNOS.

**Manufacture of artificial threads, filaments, etc.** COURTAULDS, LTD., and E. A. MORTON (B.P. 310,172, 10.4.28).—The cake of fibre is washed while still in the rapidly rotating centrifugal box by allowing a jet of water to impinge vertically on to the middle of a circular disc placed at the centre of the bottom of the box. The centre of this disc is domed whilst the outer part has alternate ridges and hollows, so that the water

jet is broken up into a spray which reaches all parts of the cake. F. R. ENNOS.

**Manufacture of hollow artificial threads.** ERSTE BÖHMISCHE KUNSTSEIDEFABR. A.-G. (B.P. 282,687, 20.12.27. Czechoslov., 21.12.26).—An insoluble liquid or solid substance, *e.g.*, petroleum, which does not yield any bubble-forming vapours, is mixed with the solution to be spun, and is subsequently removed from the textile threads, after finishing, by means of a vacuum at ordinary or increased temperature. F. R. ENNOS.

**Paper-sizing agents.** E. C. R. MARKS. From ALOX CHEM. CORP. (B.P. 309,383, 6.10.27).—A petroleum hydrocarbon oil, mixed with 0.1% of an oxidising catalyst, is subjected to a fine spray of air and heated at 120–155° under pressure until not more than 10% of heavy organic acids insoluble in the hydrocarbon mixture are formed. After separation these acids are purified by treatment with steam above 150°, separated from sludge, and converted by treatment with alkali into neutral soaps, which are mixed with paper pulp and then converted by means of alum into insoluble aluminium soaps. F. R. ENNOS.

**Apparatus for treatment of webs or sheets of paper or textile or other material with chemicals or colouring matters or other fluids.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,135, 27.2., 23.5., and 7.8.28).—By passing the material over orifices in the cover of one or more boxes, the appropriate liquid or gaseous staining or colouring materials contained therein are drawn up by suction on to the sheet. Various effects may be produced by giving the sheets a varying absorption capacity by application of pressure, by treatment with suitable reagents, or by imparting a reciprocating movement to the boxes. F. R. ENNOS.

**Treatment of cellulosic material. Manufacture of cellulose derivatives.** H. DREYFUS (U.S.P. 1,711,110—1, 30.4.29. Appl., 31.10.24. U.K., [A] 25.9.24, [B] 6.10.25).—See B.P. 249,173 and 263,939; B., 1926, 436; 1927, 247.

**Manufacture of [compound] textile threads.** O. RASCH (B.P. 311,447, 11.2.28).

**Spinning bowls or boxes for artificial silk.** J. C. HENSING, Assee. of N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 293,871, 13.7.28. Holl., 15.7.27).

[Guider-starter for] spinning of artificial silk and the like. W. P. DREAPER (B.P. 311,454, 13.2.28).

**Centrifugal [laundry] washing and drying machines.** SIEMENS-SCHUCKERTWERKE A.-G., Assees. of SIEMENS-SCHUCKERTWERKE GES. M.B.H. (B.P. 292,512, 19.6.28. Ger., 20.6.27).

**Solvents for cellulose esters** (U.S.P. 1,689,761—2).—See III. Rubber-covered metal sheets (B.P. 301,300).—See X. Plates for electric condensers (B.P. 310,625).—See XI. Wool-washing water (B.P. 300,386).—See XXIII.

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

**Stability to light of records made with type-writer ribbon, duplicate paper, and copying ink pencils.** H. I. WATERMAN, J. GROOT and M. J. VAN

TUSSENBROEK (Chem. Weekblad, 1929, 26, 276—277).—Whilst impressions made with ribbon and papers in which carbon-black has been used do not change on keeping except for the fading of the dye, papers and pencils containing only methyl-violet do not give permanent impressions, since the dye fades completely on exposure to light. S. I. LEVY.

### PATENTS.

**Dyeing composition.** A. DAVID, Assr. to DIP-IT, INC. (U.S.P. 1,687,751, 16.10.28. Appl., 25.3.22).—1% of a pure soap is incorporated with 59–66% of water, about 30% of glycerol, and 3–10% of a suitable dye, preferably a water-soluble acid, direct, or basic dye. R. BRIGHTMAN.

**Mineral dyeing.** C. B. WHITE, Assr. to VIVATEX PROCESSES, INC. (U.S.P. 1,686,540, 9.10.28. Appl., 23.12.26).—1–5% of formic or other organic acid is added to a solution of ferric sulphate and the sulphate ions are removed by means of barium acetate; textile material is passed through the bath, dried, and treated with an alkaline solution in the usual manner. The increased solubility of the ferric acetate in presence of formic acid leads to deeper, faster, and more uniform shades. R. BRIGHTMAN.

**Dyeing and printing.** M. BATTEGAY, Assr. to CALCO CHEM. CO. (U.S.P. 1,686,224, 2.10.28. Appl., 1.11.26).—With acid dyes and their salts, the mordants obtained from sulphur and phenols (*cf.* U.S.P. 1,523,340; B., 1925, 204) can be applied simultaneously with the dyestuff. After the dyeing, padding, or printing operation, the material is passed into a fixing bath containing dilute organic or mineral acid or mineral acid salt such as aluminium sulphate, zinc sulphate, barium chloride, etc. R. BRIGHTMAN.

**Differential coloured fabric and its manufacture.** C. DREYFUS and H. PLATT, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,689,426, 30.10.28. Appl., 13.8.27).—Fabric, *e.g.*, cellulose acetate or other organic derivative of cellulose, is printed with a solution of viscose or other material of different behaviour towards dyestuffs, and after regenerating the cellulose the mixed fabric is dyed in a bath containing, *e.g.*, a cellulose acetate dye which does not affect cellulose, and a cellulose dye which does not affect cellulose acetate. R. BRIGHTMAN.

**Treatment of artificial silk and articles made thereof for rendering same water-repellent.** J. P. BEMBERG A.-G. (B.P. 286,257, 1.3.28. Ger., 1.3.27).—Artificial silk materials are impregnated with a solution of a basic aluminium soap (containing not more than twice the equivalent weight of fatty acid to three times the equivalent weight of aluminium) in an organic solvent, the latter being then removed by evaporation; other fats and waxes, *e.g.*, paraffin wax, may be added to the solution. *E.g.*, into an 8% solution of a soap containing 80% of fat is poured an excess of a concentrated solution of a basic aluminium sulphate having 1.2 hydroxyl groups to 3 atomic equivalents of aluminium; 2 kg. of the precipitated basic aluminium soap (9.6% Al<sub>2</sub>O<sub>3</sub>) are then dissolved in 100 kg. of trichloroethylene. A. J. HALL.

**Treatment [sizing] of artificial silk yarn.** O. PLOETNER (B.P. 311,137, 29.6.28).—Artificial silk yarn

is rendered smooth and pliable by sizing it with a mucilage containing 7—8 pts. of soft soap, 1—2 pts. of glycerin, and 1 pt. of potato meal or other colloidal substance such as egg albumin, gum tragacanth, or agar-agar, this process being effected by passing the yarn through the mucilage, removing the excess, and smoothing the yarn by passage between pieces of absorbent rubber sponge pressed together.

A. J. HALL.

**Mothproofing of fabrics.** H. I. JONES (U.S.P. 1,688,717, 23.10.28. Appl., 24.1.27).—Fabric is treated first with an aqueous solution of casein and borax and then with an aqueous solution of salt of copper, lead, mercury, bismuth, thallium, or a rare-earth metal. Greater waterproofing effect is obtained by adding to the casein solution an emulsion of an easily hydrolysed wax, *e.g.*, beeswax, in excess of ammonia solution.

R. BRIGHTMAN.

**Manufacture of felt and felt materials.** H. STEIN, W. E. AUSTIN, and I. LIEBOWITZ, Assrs. to STEIN FUR DYEING Co., INC. (U.S.P. 1,687,801—2, 16.10.28. Appl., 10.2.25).—Fur skins, hair, or other animal fibre, preferably without previous alkaline washing, are partly carotated using 50—75% of the amount of acid mercuric nitrate solution normally employed for complete carroting. After rinsing and hydro-extraction the fibres are immersed for 8—12 hrs. in a solution of protective agent, *e.g.*, a 0.5—5% solution of ferrous sulphate crystals, to which a stabilising agent such as ammonium chloride may be added. After further rinsing and hydro-extraction the material is bleached by treating at 15—40° with 3% hydrogen peroxide.

R. BRIGHTMAN.

**Imparting a wool finish to cotton cloth.** M. J. MACK (U.S.P. 1,689,208, 30.10.28. Appl., 28.7.27).—Cotton fabric is successively bleached, dyed, napped, mercerised, sized, and re-brushed.

R. BRIGHTMAN.

**Dyeing of artificial silk.** W. HENTRICH, R. KNOCHE, and M. HARDTMANN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,711,890, 7.5.29. Appl., 10.9.27. Ger., 22.9.26).—See B.P. 301,564; B., 1929, 127.

**Machines for treating fabrics by processes such as washing, dry-cleaning, etc.** BRIT. AMER. LAUNDRY MACHINERY Co., LTD., Asses. of G. H. WEST (B.P. 309,980, 29.6.28. U.S., 19.4.28).

[Steaming machine for] finishing of textile fabrics. G. C. HIRST (B.P. 311,355, 6.2.28).

**Treatment of paper, textiles, etc.** (B.P. 310,135).—See V. Stable sulpho-acids and salts (B.P. 288,126).—See XII. Bleaching of foodstuffs (U.S.P. 1,687,803—5).—See XIX. Multicolour screens (B.P. 309,113).—See XXI.

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

**Determination of phosphoric acid in mineral phosphates and fertilisers.** G. JORGENSEN (Ann. Falsif., 1929, 22, 199—207).—In the case of mineral phosphates a sample (5 g.) is boiled with 25 c.c. of nitric acid (*d* 1.21) and 12.5 c.c. of hydrochloric acid

(*d* 1.12) for 30 min. The solution, after cooling, is made up to 250 c.c., shaken, and filtered, the first part of the filtrate being rejected. To 50 c.c. of the solution a quantity of molybdic acid is added calculated from the given table according to the proportion of phosphate present, and the mixture is kept at 50° for 10 min. and shaken. After being kept overnight the flask is cooled, the contents are filtered, and the precipitate is washed five times with 20—25 c.c. of a 4% ammonium nitrate solution. If precipitation is thought to be incomplete, the filtrate is again heated with molybdic acid. The solution is warmed and two thirds of a standard magnesium solution (amount found from the table) are added. The flask is shaken until the precipitate becomes crystalline, the rest of the magnesium solution is added, and the liquid cooled with frequent shaking. After 15 min. the precipitate is collected, washed with ammonia solution, dried, heated, and weighed as Mg<sub>2</sub>P<sub>2</sub>O<sub>7</sub>. For superphosphates and fertilisers the total phosphoric acid is determined on 5 g. of the sample; these are heated with 20 c.c. of nitric acid only, the procedure being as above except that different quantities of reagents are added, as found from the tables. The phosphoric acid soluble in water is next determined, and then that soluble in water and in ammonium citrate, this last determination being made on a 2.5 g. sample, which is ground first dry and then with 20—25 c.c. of water, and the decanted liquid from three grindings is filtered. The residue is washed, the filtrate made up to 200 c.c., a few drops of nitric acid are added, and the whole is shaken (solution A). To the residue with the filter paper are added 100 c.c. of Petermann's ammoniacal ammonium citrate solution, the whole is shaken, and, after 15 hrs., heated to 40°, cooled, made up to 250 c.c., shaken, and again filtered (solution B). Then 50 c.c. each of A and B are mixed, and phosphoric acid is determined as for mineral phosphates. A modified procedure is given for Thomas slag. D. G. HEWER.

**Manufacture of potassium chloride from Solikamsk sylvite.** II. F. F. VOLF and V. S. YATLOV (J. Chem. Ind. Moscow, 1928, 5, 909—915; cf. B., 1929, 127).—The composition of the solutions of the sylvite, and of the precipitated crystals, is discussed. The conditions of crystallisation prevent the separation of calcium sulphate with the potassium chloride.

CHEMICAL ABSTRACTS.

[Manufacture of] bleaching powder without cooling. E. HADAMOVSKY (Chem.-Ztg., 1929, 53, 321—322).—The tower-chamber system, in which lime is rabbled over successively lower floors by means of arms on a central vertical shaft, as in the ordinary eight-hearth furnace for burning fine pyrites, the chlorine gases entering at the bottom and passing upwards, is found to operate as well without cooling as with cooling.

S. I. LEVY.

**Examination of alkali iodides.** W. MEYER (Pharm. Ztg., 1929, 74, 628).—Samples of ammonium, sodium, and potassium iodides, which on keeping darkened and separated free iodine, were found to contain thio-sulphate. The Pharmacopœia test with silver nitrate and ammonia does not show the presence of small quantities of thio-sulphate; these may be detected by

decolorisation of an acidified starch solution coloured blue with iodine. S. I. LEVY.

#### Manufacture of aluminium sulphate from clays.

I. E. ADADUROV (J. Chem. Ind. Moscow, 1928, 5, 941—942).—Clays containing alumina as kaolinite are calcined preferably at 800° before attack by sulphuric acid (20%), whilst clays containing 34% of kaolinite-alumina are preferably calcined at 700° and treated with 50% acid. Iron is best eliminated by chlorination of the crude clay, mixed with charcoal, at 300—400° or, better, of dehydrated aluminium sulphate at 400°.

CHEMICAL ABSTRACTS.

#### PATENTS.

**Nitre oven.** E. B. RUPARD, Assr. to ARMOUR FERTILIZER WORKS (U.S.P. 1,709,880, 23.4.29. Appl., 2.6.27).—The oven, for use in the manufacture of sulphuric acid, is connected by a conduit with the combustion flue and in its lower part has a refractory vessel, to contain sodium nitrate and sulphuric acid, fitted with a safety overflow through the oven wall below the conduit level. A valve normally closes the outer end of the overflow, but opens outwards automatically when necessary. W. G. CAREY.

**Manufacture of boric acid.** AMER. POTASH & CHEM. CORP., Assees. of W. E. BURKE and H. DE ROPP (B.P. 294,236, 7.6.28. U.S., 22.7.27).—A borax solution is successively treated with sulphuric acid, cooled to a temperature just above that at which sodium sulphate starts to crystallise, and filtered to remove the boric acid which has crystallised out; the mother-liquor, after dilution and dissolution in it of more borax, is successively cooled to such a temperature that sodium sulphate decahydrate crystallises out, filtered, and returned to the process. The conditions are so adjusted that the quantity of sodium sulphate crystallising out is approximately equal to that formed during the initial reaction, and the water formed by the reaction and that added to dilute the mother-liquor are removed as water of crystallisation. L. A. COLES.

**Manufacture of silica gel.** M. YABLICK (U.S.P. 1,687,919, 16.10.28. Appl., 10.3.22).—A solution of sodium silicate or other soluble silicate is treated with an ammonium salt, preferably the carbonate, and the silicic acid allowed to gel in the alkaline liquor before washing and drying. R. BRIGHTMAN.

**Synthetic production of ammonia.** NITROGEN ENGINEERING CORP. (B.P. 287,072, 12.3.28. U.S., 12.3.27).—A portion of the ammonia formed by the combination of nitrogen and hydrogen in a single converter is recovered from the gaseous products immediately they leave the converter, and the remainder after the gases have been contaminated with impurities by passage through a pump and by the addition of fresh supplies of nitrogen and hydrogen. L. A. COLES.

**Production of salts.** A. E. MITCHELL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 310,594, 16.2.28).—An acid is treated with a base or with a decomposable salt while allowing them to flow down a packed tower, using an excess of one constituent and subsequently neutralising with a volatile acid or alkali supplied to the base of

the reaction tower in a current of steam, or in a separate vessel or tower. *E.g.*, a suspension of chalk in water or in liquor from a previous operation flows down a tower, and excess of nitric acid is introduced at a lower level, the excess of acid subsequently being neutralised with gaseous ammonia. W. G. CAREY.

#### Production of clear brine or other liquors.

A. W. ALLEN (U.S.P. 1,687,703, 16.10.28. Appl., 2.9.27).—Turbid liquors, *e.g.*, potassium sodium chloride liquors, sodium nitrate liquors in the Shanks process, are clarified by the addition or the production, by evaporation with rapid agitation and cooling, of sufficient small crystals of a constituent, or other water-soluble compound, to act as a mechanical filter-aid, when the crystals and suspended matter are filtered off. R. BRIGHTMAN.

#### Manufacture of alkali nitrates.

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,687, 31.5.28).—Alkali sulphates are treated with at least 2 mols. of nitric acid per mol. of sulphate, and the alkali nitrates are separated from the mother-liquor by cooling and centrifuging. L. A. COLES.

**Production of pure ammonium sulphate.** R. E. SLADE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 310,635, 11.4.28).—Ammonium sulphate practically free from iron is obtained from the interaction of ammonium carbonate and calcium sulphate by returning a portion of the evaporated liquor, preferably from the crystal separators, to the reaction stage and removing the precipitated iron and chalk together, or to a subsequent stage where the liquor is still alkaline and removing the precipitated iron by settling. The evaporators are made of, or lined with, nickel-chrome steel or similar alloy. W. G. CAREY.

#### Apparatus for the production of ammonium sulphate.

BAYERISCHE STICKSTOFF-WERKE A.-G. (G.P. 453,941, 6.6.26. Addn. to G.P. 441,260; B., 1927, 965).—The circulating liquid is raised by compressed air from the equalising vessel to which the fresh acid is added as described in the prior patent and, after separation from vapour, is returned to the upper part of the saturation vessel where it serves to absorb liberated ammonia. L. A. COLES.

#### [Manufacture of] ammonium alum.

C. MCC. BROWN (U.S.P. 1,709,166, 16.4.29. Appl., 14.10.25).—Aluminous material containing iron is heated at a temperature above the decomposition point of ferric sulphate, ammonium hydrogen sulphate sufficient to dissolve the alumina is added, and heating is continued at the same temperature until the reaction is complete. A. R. POWELL.

**Production of ammonium salts.** R. TERN (B.P. 310,673, 16.5.28).—Volatile ammonium compounds are produced from salts capable of reacting in a dry condition, by mixing such salts, reducing the mixture to a colloidal state, heating in a revolving inclined drum to which air is supplied, withdrawing the product as vapour, and treating in an "electro-filter." W. G. CAREY.

**Production of barium hydroxide.** C. DEGUIDE (Swiss P. 122,352, 8.10.26).—Basic barium silicate

( $2\text{BaO}, \text{SiO}_2$ ) is treated with hot water whereby barium hydroxide dissolves, leaving an insoluble residue of the normal silicate which, after removal, is converted into the basic silicate by treatment with barium carbonate.

L. A. COLES.

**Recovery of oxalates.** G. H. BUCHANAN and G. BARSKY, Assrs. to AMER. CYANAMID CO. (U.S.P. 1,687,480, 9.10.28. Appl., 4.9.24).—Low-grade calcium oxalate cake, containing, e.g., 20% of calcium oxalate and 80% of calcium carbonate, is made into a slurry and heated at  $100^\circ$  with an excess (10–35%) of sodium carbonate solution, sufficient to prevent reversion on cooling when the insoluble calcium salts are filtered off. The oxalate solution is treated with milk of lime and filtered, the precipitate (about 80% of calcium oxalate and 20% of calcium carbonate) being decomposed with sulphuric acid in the usual manner and the filtrate converted into (sodium) carbonate and used again. If the crude oxalate cake contains lime, carbon dioxide may be passed into the mixture during the treatment with sodium carbonate until the hydroxide has been converted into carbonate.

R. BRIGHTMAN.

**Treatment of [ferrous] chloride solutions.** S. I. LEVY and G. W. GRAY (B.P. 309,268, 10.2.28).—The ferrous chloride solutions obtained by dissolution of the ferrous sulphide produced by heating pyrites out of contact with the air are saturated with hydrogen chloride at  $100^\circ$  and utilised in dissolving fresh quantities of ferrous sulphide until they are 90% saturated with ferrous chloride at  $100^\circ$ . On filtering and cooling to  $20^\circ$ , about half the ferrous chloride separates as  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ . The mother-liquor is saturated with hydrogen chloride and returned to the cycle. When the zinc accumulated in the liquor reaches 80–100 g./litre, the solution is removed from the cycle and evaporated to recover crude ferrous chloride crystals, and a final mother-liquor from which the zinc is readily recovered; alternatively, most of the ferrous chloride is precipitated by saturation of the solution at  $20^\circ$  with hydrogen chloride.

A. R. POWELL.

**Production of ferrous bicarbonate.** C. PEETERS (Swiss P. 122,987, 28.2.27).—The compound is precipitated by mixing aqueous solutions saturated with carbon dioxide containing, respectively, a ferrous salt and an equimolecular proportion of an alkali bicarbonate, together with 1–5% of an alkali tartrate, lactate, citrate, etc.

L. A. COLES.

**Production of alumina from clay and other aluminiferous material.** M. BUCHNER (B.P. 309,621, 13.1.28).—The clay or aluminiferous material containing but small quantities of alkali compounds is digested with nitric acid, and, after filtration, the solution is treated with nitric acid or oxides of nitrogen to precipitate crystalline aluminium nitrate. The product is converted into alumina with simultaneous recovery of oxides of nitrogen by heating it alone or mixed with alumina, or by adding it to molten alkali nitrates or calcium nitrate, the decomposition being aided by working under reduced pressure and by passing steam, hot gases, etc. through the material.

L. A. COLES.

**Production of magnesium salts or magnesia from waste material containing magnesium.**

E. SEDLER (Austr. P. 108,413, 28.8.26).—The material is dissolved in concentrated hydrochloric acid and impurities such as silica, iron, lime, alumina, etc. are precipitated by the addition of basic material containing magnesium, e.g., filter residues or calcined magnesite waste which has been rendered readily soluble by exposure to the air.

L. A. COLES.

**Production of hypochlorites.** G. ORNSTEIN (G.P. 454,406, 2.2.23. Addn. to G.P. 406,452; B., 1925, 169).—An aqueous suspension containing material capable of forming hypochlorites and free hypochlorous acid circulates continuously through a circuit comprising a container and absorption apparatus; in the latter the suspension is treated with liquid or gaseous chlorine in quantity sufficient to form some free hypochlorous acid, which in the container is converted into hypochlorites. The hypochlorite solution after clarification is withdrawn continuously from the container and replaced by equivalent quantities of water and the reacting substances.

L. A. COLES.

**Evaporation of cyanide solutions.** STOCKHOLMS SVERFOSFAT FABR. AKTIEBOLAG, Asses. of H. G. A. RAMSAY (Swed. P. 59,652, 23.2.24).—The solutions are evaporated to dryness by spraying *in vacuo* at a temperature below that at which the cyanides hydrolyse.

L. A. COLES.

**Process of alkylation. [Lead tetraethyl.]** C. A. KRAUS and C. C. CALLIS, Assrs. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,690,075, 30.10.28. Appl., 5.4.23).—Magnesium and an alkyl chloride are heated in ether with the substance to be alkylated, e.g., plumbous chloride, to above the normal b.p. of the alkyl chloride.

R. BRIGHTMAN.

**Obtaining pure oxygen from a single or double oxygen plant, with a greater output.** W. F. DALTON (B.P. 309,632, 14.1.28).—The liquid oxygen is conveyed by siphon tubes to an inner column contained in the evaporating coil of the collecting vessel, the liquid being evaporated during its passage through the tubes and the pure gas being withdrawn from the inner column. Alternatively, the siphon tubes may be closed and the liquid oxygen conveyed to the inner column for re-rectification, when necessary.

L. A. COLES.

**Liquefaction of oxides of nitrogen.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,212, 2.12.27).—Gases containing oxides of nitrogen are cooled and compressed in the presence of, e.g., 2–4% of anhydrous nitric acid, which may be introduced as a spray or may be formed *in situ* by the addition of suitable quantities of water and oxygen to the gases.

L. A. COLES.

**Oxidation of phosphorus vapour.** A./S. MALMIN-DUSTRI (Norw. P. 42,753, 20.12.24).—Roasting gases or finely-divided metal oxides are used for the oxidation.

L. A. COLES.

**Ammonia oxidation and catalyst therefor.** E. I. DU PONT DE NEMOURS & Co. (B.P. 306,382, 25.6.28. U.S., 18.2.28).—See U.S.P. 1,706,055; B., 1929, 432.

**Manufacture of hydrocyanic acid.** R. FICK, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,712,297, 7.5.29. Appl., 21.12.25. Ger., 19.12.24).—See B.P. 261,559; B., 1927, 74.

Ferric chloride (B.P. 284,991 and 288,319).—See X. Titanium compounds (B.P. 309,051).—See XIII. Halogens from sea-water (B.P. 294,655).—See XXXIII.

### VIII.—GLASS; CERAMICS.

**Kilns and kiln firing. II. The Dunnachie continuous gas-fired kiln. III. The Belgian ring kiln.** S. R. HIND (Trans. Ceram. Soc., 1929, 28, 148—164, 204—219).—II. The kiln has two parallel but unequal sets of chambers, eight on one side and ten on the other. The main producer-gas flue runs between the two sets of chambers, the exhaust flues being on the outer sides. The method of firing is that common to all chamber kilns, but two chambers are fired simultaneously in different parts of the round. The method of testing the behaviour of the kiln is described; 14.9 cwt. of fuel are consumed per 1000 bricks fired to cone 15. A heat balance is presented, and methods of improving the efficiency of the plant are indicated.

III. The kiln consists of two long parallel galleries, 12 ft. wide and 8 ft. 6 in. high internally, which communicate at the ends to form a continuous passage. Room is left between the galleries to form a smoke chamber, which leads to an outside stack. The method of operating the kiln and of setting the bricks is described. The results of an exhaustive test show that 1.83 cwt. of fuel are consumed per ton of fired ware. A summarised heat-balance is given, together with full data on the fuel, draught and temperatures, kiln atmospheres, exhaust gases, rates of firing, etc. F. SALT.

**Use of pulverised coal as a fuel for periodic kilns.** D. J. WATSON (J. Amer. Ceram. Soc., 1929, 12, 336—341).—Attempts to use pulverised fuel in periodic kilns have been unsuccessful because sufficient heat for combustion must be maintained in the fire-box. This too soon raises the whole kiln above the water-smoking temperature. A fire-box has been constructed the size of which can be varied by the movement of two baffle plates. In this way the small hot spot necessary for combustion may be maintained at low kiln temperatures.

J. A. SUGDEN.

**Glass-tank refractories and their chemical relationship with the molten glass.** K. C. LU (Ohio State Univ. Eng. Exp. Sta. Bull., 1928, No. 44, 40 pp.).—The life of a refractory depends on the rate of formation and dissolution of a porcelain-like surface at the glass interface. Digestion of this layer with hydrofluoric acid yielded mullite. The rate of dissolution was less in an aluminous than in a highly siliceous refractory. The brown, glassy layer covering the white layer contained labradorite crystals and a little mullite. In a highly siliceous refractory the brown glass was replaced by a white layer containing cristobalite.

CHEMICAL ABSTRACTS.

**Application of colloid chemistry to the study of clays. II.** A. E. J. VICKERS (Trans. Ceram. Soc., 1929, 28, 124—147; cf. B., 1929, 394).—A comprehensive review is presented of the literature dealing with clays from the colloid aspect. The relation between the general properties of clays and their origin is discussed, and the present position regarding the nature of clay substance is summarised. The colloid chemistry of

clays is outlined in some detail and its practical applications are indicated. F. SALT.

**Obtaining raw clays which are not washed away by water.** P. P. BUDNIKOV (Trans. State Exp. Inst. Silicates, Moscow, 1927, No. 21, 97—106).—The times required for clay, mixed with reagents and then air-dried, to disintegrate when immersed in water are tabulated. Calcium hydroxide (more than 5%) markedly increased the stability of the clay. Of the salts and acids, phosphoric acid (3—5%) was most effective. Addition of phosphoric or sulphuric acid decreases the burning shrinkage. Clays containing 5.5—42.5% SiO<sub>2</sub>, when treated with sulphuric acid or a mixture of this with calcium phosphate, manifested stability up to 21.7% SiO<sub>2</sub>. CHEMICAL ABSTRACTS.

**Action of sulphuric acid on clay at the ordinary temperature.** P. N. GRIGORIEV and P. I. GALKIN (Trans. State Exp. Inst. Silicates, Moscow, 1927, No. 21, 91—95).—The production of "waterproof" clay ("keramolite") by the action of sulphuric acid (cf. preceding abstract) is attributed to dehydration. The presence of impurities, especially iron, appears favourable. CHEMICAL ABSTRACTS.

**Rate of drying of a plastic porcelain mass due to reduced pressure and heat.** L. NAVIAS (J. Amer. Ceram. Soc., 1929, 12, 305—318).—A body consisting of china clay 20%, ball clay 25%, flint 25%, and felspar 30% was subjected to pressures of 0.7, 5, 15, and 35 cm. and temperatures of 23°, 50°, and 65°. The relationship between the logarithms of the amount of evaporation and of the time was found to be linear. At the lowest pressure the relationship has a single slope, but at the higher pressures two distinct slopes indicate two stages in the drying. The connexion between rate of drying and lowering of temperature of the drying mass is indicated, and the effect of the humidity of the air and the shape of the specimen is discussed.

J. A. SUGDEN.

**Functions of regenerators in relation to the refractory materials of construction.** A. T. GREEN (Trans. Ceram. Soc., 1929, 28, 165—191).—The principles of regeneration, with particular reference to the open-hearth furnace, are explained, and the theory of regenerator action is discussed. Essential properties of refractories for use in regenerators are: suitable thermal characteristics to ensure maximum absorption and transmission of heat; resistance to the action of slags and dust; and mechanical strength and resistance to spalling. Data are presented on the heat capacities and other relevant properties of firebrick, silica, magnesite, zirconia, and carborundum refractories. For economic reasons, the choice of regenerator fillings is restricted to silica, siliceous, and fireclay bricks. The available data indicate that silica is less efficient from the point of view of operation and thermal capacity than are firebrick and siliceous products. To guard against warping and deformation of the bricks, the material should be tested for refractoriness under a load of 20—25 lb./in.<sup>2</sup> at about 1200°, and for after-contraction, which should not exceed 0.5—0.7%. F. SALT.

**Drying cracks in firebricks.** C. E. MOORE (Trans. Ceram. Soc., 1929, 28, 193—200).—Drying cracks in



firebricks are classified as due to faulty drying treatment and to methods of preparation of the material, *i.e.*, mixing, extruding, moulding, and pressing. A "danger zone" is reached in drying when movement of moisture through the mass is rapidly becoming more difficult, when the rate of contraction with loss of water is decreasing, and when the strength is slowly increasing. The effects of additions of grog are described. The various causes of drying cracks are enumerated, and the advantages of humidity drying over the hot-floor method are pointed out.

F. SALT.

**Sand-blast abrasion test for glazes.** E. SCHRAMM (J. Amer. Ceram. Soc., 1929, 12, 356—359).—The essentials of a good test are (1) the use of a stream of sand which may easily be reproduced, (2) the rotation of the test plate so that a large area can be covered and a weighable amount removed without cutting deeply into the glaze. A suitable apparatus for the purpose is described in which a  $\frac{1}{8}$ -in. jet is inclined at an angle of 60° to the plane of the test plate. Tests showed that the weight of sand delivered and extent of abrasion are independent of the humidity of the atmosphere (*cf.* Harrison, B., 1927, 749). The comparative hardness of a number of typical glazes is given; it is found that glaze fire within normal maturing range has little effect on hardness. Glaze composition is the chief factor governing hardness.

J. A. SUGDEN.

**Prediction of service value [of ceramic materials] from laboratory test data.** A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1929, 12, 319—329).—A statistical treatment shows that such prediction depends not only on the applicability of the particular test but also on certain precision conditions which become very important in work with materials such as refractories the properties of which are variable. Charts have been constructed which show the effect of errors of measurement on accuracy of prediction for cases arising in ceramic work.

J. A. SUGDEN.

**Monolithic cements.** CLARK.—See IX.

## PATENTS.

**Tunnel kilns.** W. L. HANLEY, JUN. (U.S.P. 1,705,475 and 1,705,477, 19.3.29. Appl., [A, B] 30.8.26. Renewed [A, B] 8.8.28).—Tunnel kilns are provided with central firing zones into which the products of combustion enter, and with preheating and cooling zones in opposite directions therefrom. A single line of goods passes through the three zones in succession. In (A), the products of combustion are removed from the firing zone without permitting much longitudinal flow and are conducted by outside passages into the preheating zone. In (B), air is admitted to the cooling zone, heated by contact with the goods therein, withdrawn, and conducted outside the kiln to the preheating zone.

B. M. VENABLES.

**Pottery kiln.** A. C. WARD (U.S.P. 1,710,558, 23.4.29. Appl., 20.12.27).—A special sub-floor flue system is described.

J. A. SUGDEN.

**Laboratory muffle [for metal recovery].** E. B. FORCE, Assr. to CARBORUNDUM Co. (U.S.P. 1,710,870, 30.4.29. Appl., 2.9.27).—The walls of a heating chamber comprise at least two sections, that more directly

exposed to the action of oxidising or easily reducible materials in the chamber being made of a refractory metallic oxide material, whilst that through which heat is principally transmitted to the chamber is made of silicon carbide.

J. S. G. THOMAS.

**Production of opacifying media from zirconium silicate.** DEUTS. GASGLÜHLICHT-AUER GES. M. B. H. (B.P. 306,019, 9.7.28. Ger., 14.2.28).—Zirconium silicate is fused with about an equimolecular quantity of oxide of calcium, strontium, barium, magnesium, or zinc in presence of a small quantity of a flux.

W. G. CAREY.

**Apparatus for drawing sheet glass.** P. HADDAN. From VIRGINIA PLATE GLASS CORP. (B.P. 311,171, 24.9.28).

**Manufacture of non-splintering glass sheets.** G. E. HEYL (B.P. 310,995, and Addn. B.P. 311,098 and 311,151, [A] 4. and 17.2.28, [B] 10.5.28, [C] 8.8.28).

**Manufacture of compound sheets of glass and cellulose derivative compositions.** G. E. HEYL (B.P. 311,128, 15.6.28. Addn. to B.P. 310,995).

**Protecting wood or metal** (B.P. 310,554).—See IX.  
**Furnace for glass** (Swiss P. 122,176). **Marking of lamps** (Dutch P. 17,399).—See XI.

## IX.—BUILDING MATERIALS.

**Factors governing the durability of clay building materials.** I. W. A. MCINTYRE (Trans. Ceram. Soc., 1929, 28, 101—123).—The destructive agencies involved in the "weathering" of building materials are classified as biological, physical, and chemical. Bacterial action is not an important factor in this connexion. The various physical and chemical factors, such as moisture expansion, thermal expansion, erosion, rupture by frost and by crystallisation of salts, the presence of soluble salts, etc., are described, and an attempt is made to estimate their relative importance. Texture has an important bearing on the durability, but the crystallisation of soluble salts, mainly sulphates, is the chief disintegrating influence. The salts may be derived from the soil, the atmosphere, the mortar, or from the material itself, but the various actions governing the formation, distribution, and crystallisation of salts in brickwork are not yet elucidated. Frequent washing prevents or greatly reduces the action; it is noted that the effects of these salts are absent in roofing tiles, which are subjected to more constant washing by rain.

F. SALT.

**Comparative tests on monolithic refractory cements.** H. N. CLARK (J. Amer. Ceram. Soc., 1929, 12, 330—335).—Nine leading cements on the market (plastic and dry) were tested for composition, fineness, drying and firing shrinkages, dried and fired strengths, refractoriness, and fire behaviour. The "dry" cements had much lower shrinkages and higher strengths. Two cements had low refractoriness. A small cylindrical furnace 18 in. long, 6 in. internal diam., with 3 in. walls was rammed or poured from each of the cements. These small furnaces were used to test fire behaviour and spalling tendency by firing them to 1500° and rapidly reheating three times after cooling. As a result of

the small-scale tests the three best cements were chosen and submitted to a large-scale test on the walls of an oil-fired boiler.

J. A. SUGDEN.

**Litharge-glycerin cements.** H. STÄGER (*Z. angew. Chem.*, 1929, 42, 370—379).—Seven different samples of lead oxide to be used for the cements were examined microscopically; samples 1—4 were prepared by precipitation and ignition under different conditions, sample 5 was commercial litharge, samples 6 and 7 were obtained by burning lead in vapour form. Samples 1—4 showed irregular particle size, doubtless due to agglomeration; sample 3 (red) appeared most uniform (cf. Glasstone, *B.*, 1921, 846A), whilst samples 6 and 7 showed very fine regular structure. Sedimentation tests confirmed that the red oxide (3) had average particle size 2—6  $\mu$ , whilst the particle size in the yellow samples varied up to 30  $\mu$ . The oil numbers (c.c. of linseed oil required to make a uniform paste from 100 g. of oxide) and readiness of reaction to form soaps and glycerides were also recorded, and conclusions drawn as to the surface activity of the different samples. It was observed that the agglomerated particles of some of the yellow samples were readily broken down by rubbing. The highest surface activity was shown by the high-dispersion sample 7. The time of setting and the breaking strains of the cements were observed for mixtures of all the oxides with 85% glycerin in varying molecular proportions. The best results were obtained with different proportions for the varying samples, the ratio  $\text{PbO} : \text{C}_3\text{H}_5(\text{OH})_3$  for suitable cements being 2:1 for samples 1—4, 3:1 for sample 5, and 1:1 for samples 6 and 7. Varying the dilution of the glycerin employed also gave results varying with the sample; for the less active (1, 2, 4, and 5), 97% glycerin gave as good cements as 85%, but with 75% they were definitely weaker, whilst the more active oxides (samples 3, 6, and 7) gave better cements with 75% and worse with 97% glycerin than with 85%. Greater dilutions gave worse results in every case. Atmospheric moisture has no appreciable effect on the setting of the cement. Tests on absorption of carbon dioxide by the cements showed that the finer oxides, with the higher oil numbers, give more open and porous cements, which are more readily acted on by the gas; this was confirmed by hardness tests carried out by the die-pressure method (cf. Meyer, *Elektrotechn. Z.*, 1922, 42, 1285). The times of setting for maximum hardness and strength depend on the nature of the oxide and proportion of glycerin; where these are correctly chosen, maximum values were reached in every case in 20 hrs.

S. I. LEVY.

**Relation between the temperature curve and the expansion curve in the setting of plaster.** F. J. WILLIAMS and F. C. WESTENDICK (*J. Amer. Ceram. Soc.*, 1929, 12, 377—381).—A form of volumeter was used to determine the time-temperature and time-expansion curves. The relationship of the two curves suggests that the calcium hemihydrate first goes into solution, causing the decrease in volume and evolution of heat which are observed. When the solution becomes saturated with respect to the hemihydrate the crystallisation of the dihydrate begins. This causes an expansion to occur, whilst the temperature reaches a maximum

owing to the adsorption of heat by the crystallisation of the dihydrate.

J. A. SUGDEN.

**Refractory construction materials.** GREEN.—See VIII.

#### PATENTS.

**Kiln for calcining finely-divided material, e.g., lime.** O. DEMISCH (G.P. 454,402, 13.3.25).—A powdered mixture of fuel and the material to be calcined is forced through a jet leading upwards into a kiln of approximately semi-ellipsoidal vertical cross-section. The mouth of the jet and the outlets for the waste gases lie on the horizontal, diametrical plane, and the receiver for the calcined material lies below this plane.

L. A. COLES.

**Burning of argillaceous material.** S. J. HAYDE, Assr. to AMER. AGGREGATE Co. (U.S.P. 1,707,395, 2.4.29. Appl., 10.2.20).—The material is suddenly subjected to a temperature above 820° to cause rapid expulsion of combined water and the production of a cellular aggregate, the burning of which is completed at a lower temperature.

A. R. POWELL.

**Production from magnesia cement of articles resistant to chemical attack.** S. J. BENNETTER (Norw. P. 42,716, 5.5.25).—Aqueous emulsions of cellulose derivatives are added to the material prior to moulding.

L. A. COLES.

**Production of building stones.** A. TETZNER (B.P. 292,925, 25.6.28. Ger., 24.6.27).—Clay and sand (6 pts. each) are ground to a meal-like mass, and cement (1 pt.) and water are added to form a moist mass.

H. ROYAL-DAWSON.

**Production [by a cold process] of articles with a glaze-like covering.** K. and C. FRIEDRICH (B.P. 309,755, 7.5.28).—Articles constructed of porous material, e.g., mixtures of cement with ground pumice-stone, slag, etc., are coated with a paste of finely ground cement containing small quantities of metallic (preferably zinc) soaps, and, while the coating is still plastic, a thin layer of cellulose varnish is applied.

L. A. COLES.

**Protection of wood and metal from corrosive influences.** H. KRELL (B.P. 310,554, 25.1.28).—The material is coated with glass, fused or sprayed, molten on to the surface by electrical means. Wood may be soaked in a mixture of water-glass and asbestos before treatment. [Stat. ref.]

L. A. COLES.

**Manufacture of road surfaces.** W. H. SCHMITZ (B.P. 301,891, 3.10.28. Ger., 8.12.27).—Mineral aggregates are coated uniformly with a mineral, vegetable, or animal oil, e.g., tar oil or turpentine oil, before admixture with bitumen emulsions.

L. A. COLES.

**Manufacture of road-surfacing materials or compounds.** J. Y. JOHNSON. From AMIESITE Co. OF AMERICA (B.P. 309,634, 14.10.27).—Stone aggregate is treated with a partly neutralised solution of oleic acid in hydrocarbon oils, and lime is added after admixture of the aggregate with bitumen to form calcium oleate in the product.

L. A. COLES.

**[Concrete] mixing machines.** KOEHRING Co., Assees. of G. E. WEBB (B.P. 307,869, 7.6.28. U.S., 15.3.28).

**Manufacture [laying] of stone asphalt roadways.** J. Y. JOHNSON. From AMIESITE ASPHALT CO. OF AMERICA (B.P. 310,294, 14.10.27).

**Asphalt emulsions (B.P. 284,330).**—See II. **Rubber surfacing (U.S.P. 1,705,273).**—See XIV.

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

**Corrosion of iron.** J. F. G. HICKS (J. Physical Chem., 1929, 33, 780—790).—The corrosion of iron has been investigated by measuring the increase in weight of a spiral of pure iron wire suspended in various atmospheres of dry or moist air, oxygen, carbon dioxide, oxygen-carbon dioxide mixtures, and of water vapour. It is concluded that the primary cause of corrosion is the actual dissolution of iron in water which takes place before any other chemical reaction sets in, and that a film of liquid water adhering to the metallic surface is essential for corrosion. The mechanism given is that iron dissolves as ferrous hydroxide until  $p_H=9.4$ , the equilibria being  $Fe(\text{solid}) \rightleftharpoons Fe(\text{dissolved}) + 2HOH \rightleftharpoons Fe(OH)_2 + H_2$ . These are shifted to the right, and corrosion is maintained by oxygen and carbon dioxide in the following ways. Oxygen removes ferrous hydroxide by oxidation to the less soluble ferric hydroxide, and also hydrogen (depolarisation). Oxygen also produces the maximum *E.M.F.* with respect to dissolving iron, and hence acts as an accelerator; its oxidising action is a maintenance factor. Carbon dioxide removes ferrous hydroxide by conversion into the less soluble carbonate (maintenance factor), and forms carbonic acid, the hydrogen ions of which dissolve metallic iron and favour corrosion (accelerating effect).

L. S. THEOBALD.

**Influence of copper on mild steel.** S. KODAMA (J. Study Ferrous Met., 1928, 8, 1—23).—Experimental results accord with Ruer's equilibrium diagram, according to which 0.3% Cu can be completely dissolved in iron as a solid solution. The diffusibility of copper in solid iron is small at 1100°, but great at 1200°; copper tends to destroy the ferrite grain. In the surface oxidation of steel copper remains unoxidised and enters the boundary of ferrite at the surface, causing fissures in hot-rolling.

CHEMICAL ABSTRACTS.

**Cause of the thermal brittleness of cupriferous steel.** I. KOTAIRA (J. Study Ferrous Met., 1928, 8, 25—39).—Steels containing more than 1% Cu have the dendritic structure of a solid solution rich in copper; this cannot be removed by simple annealing. When cupriferous steel is heated at 1000° for 22 hrs. the surface oxide contains only 10—20% of the original copper, the copper content of the unoxidised steel increasing by 5—10%.

CHEMICAL ABSTRACTS.

**Analysis of zinc dust.** S. S. FROLOV and K. O. SVETLYAKOV (J. Chem. Ind. Moscow, 1928, 5, 942—943).—The zinc dust, moistened with alcohol, is warmed with a known quantity of copper sulphate in aqueous solution: after 30 min., a few drops of sulphuric or acetic acid are added, the liquid is filtered, the filtrate and washings are treated with ammonia, and the copper is determined colorimetrically. CHEMICAL ABSTRACTS.

**Protection from corrosion of aluminium armatures by anodic oxidation.** W. E. VON GRONOW (Gas- u. Wasserfach, 1929, 72, 429, 436).—When aluminium is coated with a hard film of oxide, it will often resist further attack of oxidising agents; this coating is best obtained by anodic oxidation of the perfectly clean metal, using a graphite cathode. The process is carried out at 40° in a 2% solution of chromic acid; the current necessary is 0.3—0.4 amp./dm.<sup>2</sup> of metal surface, and it is applied at 0—40 volts during the first 15 min., then at 40 volts for 35 min., and at 50 volts for 5 min. The precautions necessary to obtain completely satisfactory results are described, and the extent to which the treatment can be applied to alloys is discussed.

R. H. GRIFFITH.

**Properties of carbonised tungsten.** B. T. BARNES (J. Physical Chem., 1929, 33, 688—691).—The spectral emissivity for  $\lambda=0.660 \mu$  and  $\lambda=0.472 \mu$  and the total emissivity of tungsten carbonised to the carbide  $W_2C$  have been investigated. Photomicrographs of filaments carbonised at 1950—2150° Abs. showed the presence of only the carbides  $W_2C$  and WC. The m.p. of the carbide  $W_2C$ , or more probably the eutectic point for  $W_2C$  and tungsten, is estimated to be 3000° Abs.  $\pm 15^\circ$ .

L. S. THEOBALD.

**Effect of certain cations on the flotation of galena.** N. W. TAYLOR and H. B. BULL (J. Physical Chem., 1929, 33, 641—655).—The flotation recovery of galena by means of eucalyptus oil in the presence of the nitrates of thorium, cerium, chromium, aluminium, cadmium, lead, nickel, cobalt, barium, magnesium, and silver has been measured. In every case the cation has a toxic effect on flotation and reduces the yield, the reduction being greatest with chromium, thorium, aluminium, and cerium. The shape of the curve obtained by plotting the loss in yield against concentration of added electrolyte suggests that of an adsorption isotherm and leads to the conclusion that adsorption of the cation by the galena is responsible for the toxic effect. This appears to be due to the electrostatic repulsion of similarly charged mineral particles which, in turn, reduces the adherence to air bubbles necessary for flotation. (Cf. following abstract.)

L. S. THEOBALD.

**Electrostatics of flotation.** H. B. BULL (J. Physical Chem., 1929, 33, 656—658).—The *E.M.F.* produced by the fall of crystallised galena through solutions of thorium, chromium, silver, lead, and cobalt nitrates of various concentrations have been measured. The results indicate that the toxicity of lead, silver, and cobalt, but not of thorium and chromium, on the flotation of galena (cf. preceding abstract) and the electrostatic charge carried by galena in solutions of their nitrates are closely connected. The large effect of the silver ion compared with those of the lead and cobalt ions is attributed to greater adsorption.

L. S. THEOBALD.

**Determination of sulphur in galena and metallic lead.** H. LEYSAHT (Z. anal. Chem., 1929, 77, 209—213).—Sulphur is determined in lead by heating 5 g. of fine filings with 50 c.c. of hydrobromic acid (*d* 1.49) and a few drops of stannous chloride solution, and collecting

the hydrogen sulphide evolved in 50 c.c. of cadmium acetate solution (25 g. of the salt in 1 litre of 20% acetic acid). The precipitated cadmium sulphide is determined iodometrically. Sulphur may be determined in galena in a similar manner using 0.15–0.2 g. of the ore and 2 g. of pure zinc instead of stannous chloride.

A. R. POWELL.

**Theory of chromium plating.** E. A. OLLARD (Korrosion u. Metallschutz, 1928, 4, 208–209; Chem. Zentr., 1928, ii, 2507).—The electrolyte must contain chromic hydrogen chromate, chromic dichromate, sulphuric acid or a sulphate, and a colloidal compound of the type  $\text{Cr}_2\text{O}_3 \cdot x\text{CrO}_3$ . The chief cathodic processes are:  $\text{Cr}^{6+} \rightarrow \text{Cr}^{3+}$ ,  $\text{H}^+ \rightarrow \text{H}$ , and  $\text{Cr}^{3+} \rightarrow \text{Cr}$ .

A. A. ELDRIDGE.

**Comminution of nickel catalyst.** E. MASCHKILLEISSON (Oil Fat Ind. [Russia], 1928, No. 1, 24–27; Chem. Zentr., 1928, ii, 2417).—After an optimal degree of fineness of division of a nickel catalyst has been attained, further dispersion is accompanied by reduction and, finally, cessation of activity. Nickel, which is thus removed from the catalytic surface, should therefore be deposited on the carrier after it has been reduced to the desired state of division.

A. A. ELDRIDGE.

**Apparatus for reduction of nickel catalyst.** E. WOLFSON (Oil Fat Ind. [Russia], 1928, No. 2, 8–11; Chem. Zentr., 1928, ii, 2417).

#### PATENTS.

**Sintering apparatus.** A. HOLMBERG (B.P. 310,089, 21.1.28).—Portable sintering containers are conveyed for discharge to a mechanical tipper which can be rotated on a horizontal axis.

C. A. KING.

**Metallurgical furnaces.** B. TALBOT (B.P. 293,010, 2.12.27. Belg., 30.6.27).—A tilting, reversible, regenerative furnace is provided with auxiliary baths at both ends of the main hearth which serve as reservoirs for molten metal or slag or may be used for melting material to be charged into the main hearth. The auxiliary hearths are movable longitudinally to the main hearth, joined thereto by means of unitary removable throats, and are provided with two gas ports oppositely directed across the bath, one of these ports having an air port associated with it.

A. R. POWELL.

**Apparatus for subjecting a mass of powdered or granular material to the action of gases, particularly applicable to the calcining and roasting of ores and similar materials.** H. M. RIDGE (B.P. 305,883, 1.5.28).—In a roaster the ore or material to be roasted lies upon a bed of inert granular material, which, in turn, lies on grate bars bridging gas-supply chambers. The granular material may be either of uniform size or in strata of several sizes, with the object of providing a good distribution of the reacting gas or air which passes upward through the ore. Rabbles, if used, extend into the ore but not into the inert material. The grate bars may be so formed that hood-like projections cover the spaces between the bars.

B. M. VENABLES.

**Mechanical roasting furnaces.** METALLGES. A.-G. (B.P. 297,053, 7.9.28. Ger., 13.9.27).—In mechanically-rabbed multiple-hearth furnaces in which the charge

travels alternately towards the centre and towards the periphery on succeeding hearths, the middle hearths are provided with further openings located between the centre and periphery at various distances apart so as to accelerate the passage of the charge through the furnace.

A. R. POWELL.

**Mechanical [ore-roasting] kilns.** ERZRÖSTUNG GES.M.B.H., and G. BALZ (B.P. 309,830, 14.4.28. Addn. to B.P. 288,823; B., 1928, 451).—The lowest roasting chamber of a kiln discharges the material into a hopper which is an intrinsic part of the kiln structure, and which is fitted with a perforated pipe to supply an air blast to the material. The hopper may be situated centrally with a bottom discharge opening, or laterally with a discharge chute outside the kiln.

C. A. KING.

**Furnaces for extracting a metal from an ore.** S. C. G. EKELUND (B.P. 309,651, 26.1.28).—Ore is caused to move on a travelling base through a horizontal or inclined tunnel furnace in opposite direction to a stream of gas generated in a slagging gas producer. By a heat interchange with the cooling charge, the gas is preheated and a portion may be diverted and burned at the hot zone of the furnace.

C. A. KING.

**Rotary furnace for melting metals.** K. SCHMIDT, Assce. of K. SCHMIDT GES.M.B.H. (B.P. 297,432, 20.9.28. Ger., 21.9.27).—The axis of a metal-melting furnace is disposed obliquely to increase the capacity. The charging aperture, burner, and outlet for waste gases are combined at one end, the burner being fitted through the furnace cover, which is made adjustable to allow of the directional heating of the furnace.

C. A. KING.

**Reduction of ores of metals capable of volatilisation.** F. L. DUFFIELD (B.P. 310,252, 4.8.28).—A charge of zinc, lead, or similar volatile metal, carbon, and lime is introduced at the bottom of a vertical furnace and raised through progressively hotter zones of the furnace. The metal vapours evolved are condensed, and the gases are burned in a combustion chamber surrounding the reduction furnace.

C. A. KING.

**Treatment of moulds for producing soft castings.** H. A. MYERS, Assr. to METAL CASTINGS HOLDING CO. (U.S.P. 1,706,858, 26.3.29. Appl., 30.8.26).—The mould is faced with a mixture of graphite, magnesia, and potassium cyanide, then heated to such a temperature that the surface is case-hardened by the cyanide.

A. R. POWELL.

**Cooled rim on cyanide pots.** C. H. CARPENTER and A. D. KEENE, Assrs. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,705,714, 19.3.29. Appl., 4.6.27).—Cyanide case-hardening pots are provided with a wide curved flange dipping into a shallow annular trough, supported by the surrounding refractory brickwork. The pot is heated electrically, and the flange kept cool by air circulation.

A. R. POWELL.

**Recuperative annealing furnace.** J. SCHNEPF, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,705,686, 19.3.29. Appl., 20.10.27. Ger., 14.8.26).—The furnace comprises an open-end furnace chamber in which the main heating takes place, and two laterally adjacent chambers communicating with the main chamber and open at their outer ends for charging and

discharging the material undergoing heat treatment. One of the lateral chambers receives the material from the main chamber to allow it to cool slowly, and the heat from this chamber is used to preheat a new charge in the other lateral chamber. A. R. POWELL.

**Recovery of iron from iron-containing ores with formation of ferric chloride.** VEREIN. STAHLWERKE A.-G. (B.P. 284,991 and Addn. B.P. 288,319, [A] 25.1.28, [B] 5.4.28. Ger., [A] 7.2.27, [B] 7.4.27).—(A) The ore is heated below 600° in an atmosphere of hydrogen chloride or chlorine, and the volatilised ferric chloride is reduced by hydrogen to obtain sponge iron and hydrogen chloride for use again in the process. A small quantity of sponge iron is added as catalyst in the reduction stage. If the ore contains arsenic or antimony it is prechlorinated at about 100°. (B) Iron ore is mixed with magnesium chloride and hydrochloric acid, and the mixture is heated at 150–250° to produce ferric chloride and magnesia. The product is then heated more strongly to volatilise the ferric chloride, which is subsequently reduced to iron by hydrogen. Alternatively, the chloridised product is heated at 150–250° in hydrogen, and the iron is separated from the magnesia by screening and magnetic separation. The hydrochloric acid and magnesia are allowed to interact for the production of magnesium chloride for further use in the process. A. R. POWELL.

**Manufacture of cast iron.** E. PIVOVARSKY, ASSR. to EDELGUSSVERBAND G.M.B.H. (U.S.P. 1,705,995, 19.3.29. Appl., 11.2.26. Ger., 21.2.25).—Cast iron is heated in the cupola to a temperature above 1400° at which the carbide present begins to decompose with the separation of finely-divided graphite. On subsequently casting, the whole of the uncombined carbon separates in a finely-divided form, so that a particularly even-grained, tough casting is obtained. A. R. POWELL.

**Metallurgical process and product [cast iron].** L. H. MARSHALL, ASSR. to OHIO BRASS CO. (U.S.P. 1,707,529, 2.4.29. Appl., 19.1.25).—Cast iron is rendered malleable by annealing above 750° until graphitisation is complete, cooling slowly to about 670°, and quenching in water or in air. A. R. POWELL.

**Welding of cast iron and filler rod therefor.** H. V. WILLE (U.S.P. 1,708,815, 9.4.29. Appl., 14.4.21).—A welding rod for producing a soft weld in cast iron comprises an iron alloy containing 4–20% C. A. R. POWELL.

**Production of pure iron. Manufacture of steel.** H. J. VAN ROYEN (B.P. 282,365–6, 26.11.27. Ger., [A] 14.12.26, [B] 15.12.26).—(A) The charge of pig iron is blown in the converter until it contains less than 0.01% P and 0.02% (Si+Mn), and its ferrous oxide content is then reduced to zero by blowing through it a mixture of carbon dioxide and monoxide in the ratio 1:2.5–7 together with an excess of nitrogen. (B) The charge is converted into steel of any desired carbon content by introducing aliphatic hydrocarbons into the gas mixture when the iron is completely deoxidised. A. R. POWELL.

**Manufacture of iron and steel insensitive to cold-shortness, blue-shortness, and ageing.** H. J.

VAN ROYEN (B.P. 295,315, 27.3.28. Ger., 9.8.27).—Iron or steel obtained by the methods described in B.P. 282,365–6 (preceding) is used as the raw material for the process described in B.P. 285,814 (B., 1929, 360). A. R. POWELL.

**Sherardising [iron and steel].** S. O. COWPER-COLES, L. TAYLOR, A. A. GOULD, and P. G. LUCAS (B.P. 308,180, 19.9., 29.11., and 2.12.27).—To obtain a bright lustrous coating of zinc in the sherardising process, 0.5% of arsenic or phosphorus is added to the zinc dust mixture with or without powdered mica or talc. A highly corrosion-resisting coating on iron and steel is obtained by sherardising with electrolytic chromium powder. The sherardising operation may be made continuous by enclosing the articles to be treated in a rotating perforated drum through which the sherardising mixture falls, and which can be replaced by a similar drum in the treatment vessel without introducing air or cooling the apparatus. A. R. POWELL.

**Pickling of metals.** IMPERIAL CHEM. INDUSTRIES, LTD., E. CHAPMAN, and A. HILL (B.P. 309,314, 14.4.28).—In pickling iron and steel in dilute sulphuric acid the addition of a thiazole derivative, e.g., dehydrothio-*p*-toluidine, prevents the action of the acid on the metal. The inhibitor may be prepared by heating 7 pts. of *o*-toluidine and 2 pts. of sulphur at 185° for 6 hrs., then at 210–215° for 18 hrs., and dissolving the product in 9 times its wt. of 70% sulphuric acid. A. R. POWELL.

**Bessemer process of making steel.** F. W. DAVIS, ASSR. to S. G. ALLEN (U.S.P. 1,709,389, 16.4.29. Appl., 2.6.24).—Non-Bessemer pig iron is blown with a blast enriched in oxygen sufficiently to supply the necessary heat for the reaction. A. R. POWELL.

**Treatment of freshly-cast steel ingots.** GEBR. BÖHLER & CO. A.-G., WIEN (Austr. P. 108,125, 4.5.26).—The ingots are cast in double-walled moulds, the space between the walls being evacuated or packed with heat-insulating material. The thickness of the inner wall is so adjusted that considerable supercooling of the steel takes place before solidification. Subsequent annealing is thus rendered unnecessary. A. R. POWELL.

**Surface-hardening of metal bodies or articles by heat-treatment.** CAMELL, LAIRD & CO., LTD., J. McN. ALLAN, and A. P. HAGUE (B.P. 310,347, 23.1.28).—Steel articles are heated in a furnace maintained at 1100–1500° by rotating either the article in the furnace or the furnace around the stationary article. The article on cooling is withdrawn when it has attained a suitable hardening temperature (about 800°) and is then chilled. C. A. KING.

**Preparation of low-carbon [iron-chromium] alloys.** V. B. BROWNE (U.S.P. 1,710,446, 23.4.29. Appl., 21.10.25).—Rustless chromium-iron alloys are obtained by melting in a carbon-arc furnace a charge comprising a low-carbon iron-chromium alloy, lime, and manganese oxide so proportioned as to prevent access of carbon to the bath. A. R. POWELL.

**Malleable iron alloy.** A. L. BOEGEHOLD, ASSR. to GEN. MOTORS RES. CORP. (U.S.P. 1,707,753, 2.4.29. Appl., 14.3.27).—The alloy contains 1.3–2.5% Si,

2—2.75% C, and about 0.5% Mo, the last ingredient serving to obstruct the formation of flake graphite during solidification so that a white iron is obtained on casting.  
A. R. POWELL.

**Annealing of silicon-steel sheets.** W. E. CAUGHERTY and C. J. S. STROBLE, ASSTS. to ALLEGHENY STEEL CO. (U.S.P. 1,706,438, 26.3.29. Appl., 30.6.27).—Thin silicon-steel sheets are laid one, two, or three at a time on a flat base in an annealing furnace, and as soon as the first charge has reached the annealing temperature a second equal charge is placed on top of it, heated to the same temperature, and the operation repeated until the furnace is full; it is then closed and maintained at the annealing temperature (830°) for 1—3 hrs.

A. R. POWELL.

**Manufacture of iron or alloys thereof.** A. A. FREY (B.P. 284,976, 6.2.28. U.S., 5.2.27).—Iron or steel scrap is melted under a lime-carbon charge until the sulphur is completely eliminated, the slag is removed, and the metal tapped into a basic converter in which it is blown until all the carbon, phosphorus, manganese, and silicon are removed and the metal contains 1% or more of ferrous oxide. At this stage the blast is turned off and the charge allowed to settle before running off the slag. The converter is again turned on but, instead of air, a reducing gas, preferably hydrogen, is blown through the metal until the latter is thoroughly de-oxidised. Silicon, arsenic, or aluminium may be added at the end of the reduction to obtain alloys of these metals with iron.

A. R. POWELL.

**Manufacture of materials [steel] containing tungsten.** H. G. BATCHELLER and J. O. KELLEY, ASSTS. to LUDLUM STEEL CO. (U.S.P. 1,709,817, 16.4.29. Appl., 6.10.25).—Tungsten is introduced into the steel bath in the form of a friable conglomerate comprising partially reduced tungsten trioxide and a slag composed of sodium carbonate and the manganese, arsenic, and tin compounds present as impurities in the oxide.

A. R. POWELL.

**Metallic alloy.** BARBER ASPHALT CO., ASSEES. of A. B. DAVIS (B.P. 300,249, 16.4.28. U.S., 10.11.27).—An alloy of iron containing 15—25% (20) Ni, 10—20% (14.5) Cr, 3—8% (5) Co, 2—8% (4.5) Cu, the quantity of the last two elements being more than 7%. Tungsten, molybdenum, and silicon may also be included as modifiers.

C. A. KING.

**Refractory iron alloys.** E. O. VOGEL and C. F. C. WEICHELT (MEIER & WEICHELT) (B.P. 287,876, 27.3.28. Ger., 28.3.27).—The alloys contain 12—20% Ni, 16—20% Cr, 0.3—3% Si, and up to 1% C, the silicon content being at least equal to 2.5 times the carbon content and the sum of the silicon and chromium exceeding 19%. [Stat. ref.]

A. R. POWELL.

**[Iron-nickel-chromium] alloys.** W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 308,643, 23.12.27. Addn. to B.P. 284,789; B., 1928, 271).—Alloys having a differential permeability greater than iron and a high electrical resistance comprise 40—50% Fe, 1—5% Si or Al, 1—5% Cr or similar element, and the remainder (not less than 42%) nickel. In addition the alloys may contain 0.2—0.3% Mn and up to 5% Cu. The

alloys are substantially free from carbon; they are heat-treated for 15 min. at 900—1100°, then slowly cooled to the ordinary temperature. A. R. POWELL.

**[Nickel-molybdenum-iron] alloy.** F. M. BECKET, ASST. to ELECTRO METALLURGICAL CO. (U.S.P. 1,710,445, 23.4.29. Appl., 10.1.28).—The alloy contains Ni 40%, Mo 15—40%, and Fe not less than 10% nor more than five times the amount by which the molybdenum exceeds 10%.

A. R. POWELL.

**Alloy steels [for permanent magnets].** C. K. EVERITT, and E. ALLEN & Co., LTD. (B.P. 308,549, 20.9.28).—The steel contains 0.5—2% C, 6—12% Cr, 15—40% Co, 0.3—1.5% Si, 1—6% W, and 0.5—4% Mo. The cast alloy is heated at 750°, cooled, reheated at 950—1000°, and quenched in oil or air. Magnets made from this steel are characterised by a high remanence and coercive force.

A. R. POWELL.

**Treatment of slags containing metals obtained in the production of ferrochromium from ores.** D. W. BERLIN (Swed. P. 59,975, 4.10.22).—The slags are fused in an electric furnace with aluminium or silicon, and the resulting metal is fused with ferric oxide to remove excess of the reducing agent.

A. R. POWELL.

**Coating of metallic surfaces for prevention of corrosion.** J. DAVIES (B.P. 310,696, 7.6.28).—For the protection of iron and steel, and particularly water tanks, the metal is coated with a mixture of 65% of Portland cement, 10% of casein, 15% of calcined magnesia, and 10% of asbestos powder to which an equal volume of water is added. Magnesia and asbestos may be omitted from the composition. After drying, the coating is washed with formaldehyde and sprayed with cellulose lacquer.

C. A. KING.

**Coating band iron with a rust-protection material.** C. RÖTZEL (B.P. 284,191, 23.1.28. Ger., 22.1.27).—Band iron after passing through a hot-rolling mill is immediately immersed, while still hot, in oil or grease.

J. S. G. THOMAS.

**Froth-flotation concentration of ores.** R. B. MARTIN, ASST. to MINERALS SEPARATION N. AMER. CORP. (U.S.P. 1,686,529, 9.10.28. Appl., 11.12.26).—In the concentration of zinc ores the copper sulphate used in the froth-flotation process may be replaced with advantage by an organic copper compound, e.g., aniline cupric chloride, benzidine cupric chloride.

R. BRIGHTMAN.

**[Flotation] concentration of oxidised ores.** J. T. TERRY, ASST. to C. E. TERRY (U.S.P. 1,709,329, 16.4.29. Appl., 23.11.25).—Oxidised ores of lead, zinc, silver, mercury, or bismuth are subjected in the form of a finely-ground pulp to the action of a soluble chromate which converts the surfaces of the mineral particles into insoluble chromates; the pulp is then subjected to froth-flotation.

A. R. POWELL.

**Flotation oil.** R. LUCKENBACH (U.S.P. 1,688,975, 23.10.28. Appl., 23.5.25).—Flotation oil suitable for the recovery of, e.g., oxidised portions of copper ore, consists of 50—70% of heavy residuum, 10—15% of petroleum diluent, and 10—30% of a frothing agent containing 20—50% of soft soap, together with, e.g., cotton-seed foots, pine creosote, and Turkey-red oil.

R. BRIGHTMAN.

**Concentration of manganese ore.** H. and A. J. TRUMBO, Assrs. to MANGANESE PATENTS CORP. (U.S.P. 1,710,522, 23.4.29. Appl., 20.9.27).—The ore is ground, classified, cleaned with a solution of sodium carbonate, and concentrated on jigs, tables, and magnetic separators. The tailings from the concentration processes are digested with sulphur dioxide solution to recover their manganese content.

A. R. POWELL.

**Recovery of copper from copper-rich material.** S. I. LEVY and G. W. GRAY (B.P. 309,269, 10.2.28).—The insoluble residue containing copper, obtained by digestion with hydrochloric acid of the product resulting from heating pyrites at 600° in a neutral atmosphere, is dried at 100° and again digested with hot hydrochloric acid to remove the greater part of the zinc and iron. The residue from this treatment is roasted, with or without the addition of ferrous chloride, and leached to recover copper and zinc sulphate or chloride.

A. R. POWELL.

**Melting and refining of copper.** H. H. ALEXANDER (B.P. 310,356, 16.1.28. Cf. B.P. 309,848; B., 1929, 479).—Dissolved oxide is removed from molten copper by blowing beneath the surface of the metal fine streams of steam or other gas containing powdered coal, coke, or charcoal in suspension.

A. R. POWELL.

**Recovery of copper and nickel.** CHEM. FABR. JOHANNISTHAL G.M.B.H., and F. TROSTLER (B.P. 283,132, 19.12.27. Ger., 4.1.27).—Copper-nickel alloys, mattes, or speisses are oxidised anodically in slightly alkaline 10–20% sodium chloride solution to produce an anode slime containing copper and nickel hydroxides. The slime is collected, washed, and digested with copper sulphate solution, whereby the nickel hydroxide dissolves with the precipitation of an equivalent quantity of copper hydroxide. This is collected, washed, and converted into metal in the usual way.

A. R. POWELL.

**Manufacture of moulding bodies of metals.** GEBR. SIEMENS & Co., Asses. of E. BIRNBRÄUER (Austr. P. 108,103, 4.11.22. Ger., 5.11.21 and 5.8.22).—Solutions of copper salts or of salts of the metals of the iron group are treated in such a way as to obtain the metal or its hydroxide in a finely-divided form. The precipitate is collected, washed, and reduced above 600° with hydrogen, carbon monoxide, or methyl alcohol. The metal is thus obtained in a plastic, difficultly oxidisable form suitable for use as a solder or metallic cement.

A. R. POWELL.

**Metallurgy of ores etc. containing tin.** E. A. ASHCROFT (B.P. 309,307, 5.4.28).—The material is heated with ammonium chloride and, if necessary, with finely-divided iron to convert the tin into stannous chloride, which is recovered by volatilisation or by lixiviation. The ammonia evolved in the first stage is utilised in recovering the metal subsequently obtained in solution.

A. R. POWELL.

**Recovery of tin from ores etc.** H. L. SULMAN and H. F. K. PICARD (B.P. 310,639, 14.4., 5.7., and 1.8.28).—A mixture of low-grade tin concentrates and pyrites is bonded with coal dust and tar, and the briquettes are heated to effect sulphidisation of the tin without volatilisation. The porous masses so formed are heated

on a grate in a current of air, whereby the tin sulphide volatilises and is immediately converted into tin oxide fume, which is recovered in a bag plant or other collector.

A. R. POWELL.

**Production of metallic tin.** CAYZER TIN SMELTING Co. (PROPRIETARY), LTD., Asses. of W. J. CAYZER (B.P. 306,445, 29.9.28. S. Africa, 20.2.28).—A mixture of tin ore or concentrate and an excess of carbonaceous material is charged on to a bed of spongy metal forming the hearth of a furnace which is maintained at 900–1000°, the furnace atmosphere being not less than 60% CO. Molten tin, as produced, percolates through the bed of sponge metal (iron), which may be formed or maintained by the introduction of hæmatite with the charge.

C. A. KING.

**Recovery of zinc [from sulphate leach liquors].** L. F. W. LEESE (B.P. 309,288, 19.3. and 13.9.28).—Zinc sulphate solutions obtained in the hydrometallurgical treatment of ores containing zinc are passed over scrap iron to remove copper, treated with zinc hydroxide to precipitate ferrous hydroxide, and passed through a granular mass of magnesia to precipitate zinc hydroxide.

A. R. POWELL.

**Manufacture of electrolytic zinc.** I. G. FARBENIND. A.-G. (B.P. 285,373, 6.2.28. Ger., 14.2.27).—A solution of zinc sulphate containing 15–25% of free sulphuric acid is electrolysed at a current density of 200–500 amp./m.<sup>2</sup>

J. S. G. THOMAS.

**Electrodeposition of pure chromium in thick layers.** G. GRUBE (G.P. 454,168, 20.7.21).—The electrolyte contains chromic acid, chromic oxide, and a mineral acid in such proportions that the concentration of the chromic acid is more than double that of the total other constituents and that of the chromic oxide is more than sufficient to neutralise the mineral acid.

A. R. POWELL.

**Smelting of tungsten ores.** F. M. BECKET and W. C. READ, Assrs. to ELECTRO METALLURGICAL Co. (U.S.P. 1,705,655, 19.3.29. Appl., 26.3.28).—Tungsten ores containing sulphur, arsenic, tin, and/or copper are ground finely together with silicon, bleaching powder, and sulphur chloride, with or without a reaction promoter such as sodium chloride. The mixture is heated to such a temperature that an exothermic reaction takes place and the tungsten ore is reduced to ferrotungsten without fusion; complete volatilisation of the impurities as chlorides takes place simultaneously.

A. R. POWELL.

**Aluminium-base alloy.** S. DANIELS (U.S.P. 1,710,148, 23.4.29. Appl., 27.3.25).—The alloy contains 2.25–3% Cu, 0.75–1.5% Fe, and a small percentage of magnesium and silicon, the ratio Mg : Si being about 1.7 : 1.

H. ROYAL-DAWSON.

**Apparatus for centrifugal purification of metals and the like.** E. R. HEWITT (U.S.P. 1,709,939, 23.4.29. Appl., 7.4.23).—The apparatus is similar to a centrifuge, the basket being replaced by a container lined with refractory material. After centrifuging, the container is brought steadily to a standstill and the purified metal is run off from the bottom.

C. A. KING.

**Purification of light metals.** H. E. BAKKEN, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,710,398,

23.4.29. Appl., 21.7.26).—Entrained impurities are removed from a readily oxidisable light metal by passing the liquid metal through a porous mass of a neutral metal.  
C. A. KING.

**Production of metallic caesium.** R. E. MIESSE, Assr. to NEW PROCESS METALS CORP. (U.S.P. 1,707,637, 2.4.29. Appl., 3.1.27).—A mixture of caesium chloride and a rare-earth metal alloy is heated at 270° in an exhausted glass vessel until all traces of gas are removed, and is then more strongly heated by radiation from an electric heater until reaction takes place with the production of caesium metal and rare-earth chlorides.  
A. R. POWELL.

**Manufacture of thin metallic foil.** C. MÜLLER, Assr. to K. MEY (U.S.P. 1,709,801, 16.4.29. Appl., 9.3.25. Ger., 4.3.24).—A coherent foil-forming layer of desired thickness produced by deposition is sandwiched between two other layers, which are then removed by molecular decomposition, the outer layers being of sufficient thinness to avoid detrimental stress being exerted on the inner layer during their removal.  
J. S. G. THOMAS.

**Production of porous metal articles from metal powder.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,141, 9.7.28).—Iron or nickel powder produced by the thermal decomposition of the corresponding carbonyl is charged into a flat, rectangular mould until the mould is loosely filled, and the whole is heated at 500–600° in a current of hydrogen with or without a previous application of pressure. The product is a porous sintered plate suitable for use as an electrode in alkaline storage batteries.  
A. R. POWELL.

**Coating of perforated metal sheets and articles with rubber.** ANODE RUBBER Co., LTD., Asses. of ANODE RUBBER Co. (ENGLAND), LTD. (B.P. 301,300, 5.4.28. Ger., 26.11.27).—Perforated metal sheets or articles, e.g., reels or spools for the rayon industry, are covered with a protective seamless layer of rubber, which also rounds off any sharp edges, by dipping or by electrophoretic deposition, using aqueous dispersions containing rubber. According to the composition of the deposit produced, the layer, after drying and vulcanisation, may be either of soft rubber or vulcanite.  
D. T. TWISS.

**Treatment of metal articles for prevention of tarnish and rust.** H. A. SCRIVEN and B. F. G. GUISE (B.P. 309,339, 7.6.28).—The articles are plated with a coating of nickel and silver in equal proportions, then dipped in a hot solution of celluloid and soap in amyl acetate, and finally dried at 32° for several hrs.  
A. R. POWELL.

**Anti-rust or anti-corrosive preparation.** E. W. TERRY (B.P. 310,640, 16.4.28).—Bitumen and coal tar are mixed with petroleum to the consistency of treacle.  
C. A. KING.

**Steel alloy.** E. HOUDREMONT and V. EHMCKE, Assrs. to F. KRUPP A.-G. (U.S.P. 1,711,733, 7.5.29. Appl., 11.1.28. Ger., 28.1.27).—See B.P. 284,314; B., 1928, 756.

**Refinement of nickel alloys.** W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (U.S.P. 1,710,846, 30.4.29.

Appl., 3.5.28. U.K., 25.4.27).—See B.P. 296,112; B., 1928, 820.

**Solder for aluminium or its alloys.** L. R. PRESTON (U.S.P. 1,711,807, 7.5.29. Appl., 8.12.26. U.K., 14.12.25).—See B.P. 265,733; B., 1927, 303.

**[Magnetic alloys for] loaded conductor.** W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (U.S.P. 1,710,805, 30.4.29. Appl., 3.5.28. U.K., 30.4.27).—See B.P. 296,137; B., 1928, 790.

**Mercury boilers (B.P. 293,756).**—See I. **Treating ferrous chloride solutions (B.P. 309,268).**—See VII. **Muffle (U.S.P. 1,710,870).**—See VIII. **Protection of metal (B.P. 310,554).**—See IX. **Heating strip metal (B.P. 297,311).**—See XI. **Bonding rubber to metal (U.S.P. 1,689,628).**—See XIV. **Precious metals from sea-water (B.P. 294,655).**—See XXIII.

## XI.—ELECTROTECHNICS.

**Insulating oils.** STADNIKOV and VOSSCHINSKAJA.—See II. **Aluminium armatures.** VON GRONOW. **Theory of chromium plating.** OLLARD.—See X.

### PATENTS.

**Electric furnace.** P. K. DEVERS, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,708,833, 9.4.29. Appl., 13.7.25).—A heater is arranged between a vacuum-tight outer envelope and an enclosed, charge-receiving graphite vessel which can be evacuated.  
J. S. G. THOMAS.

**Electric furnace.** O. A. COLBY, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,709,658, 16.4.29. Appl., 14.7.27).—Gas is forced through oxidisable material to be heated in the furnace chamber, and then passes into and through the furnace chamber so that a non-oxidising atmosphere is maintained therein.  
J. S. G. THOMAS.

**Electric furnace.** H. ELDRIDGE, Assr. to M. K. ELDRIDGE (U.S.P. 1,711,088, 30.4.29. Appl., 19.5.25).—An electrode extends into a stationary side-wall structure of a furnace having a portable bottom formed with a central cavity the side walls of which can be raised into intimate contact with the stationary side wall. A discharge port communicates with the cavity and can be closed by a second electrode arranged therein.  
J. S. G. THOMAS.

**Electrical [resistance] furnace for production or fusion of silicates, e.g., glass.** C. E. CORNELIUS (Swiss P. 122,176, 4.5.26).—The material under treatment serves as the resistance, and the electrodes are constructed of metals having the property of storing heat, e.g., iron, are of such a shape that they are in maximum contact with the charge, and are provided with openings through which the molten material is withdrawn.  
L. A. COLES.

**Electric furnace for heating strip metal by induction currents.** HIRSCH KUPFER- u. MESSING-WERKE A.-G., and M. TAMA (B.P. 297,311, 15.3.28. Ger., 17.9.27).—Metal strip wound into the form of a coil is placed in an ironless induction furnace and forms a secondary circuit in which currents are induced by the field generated by a current of 50–60 cycles in the primary.  
J. S. G. THOMAS.



**Copper oxide electrode and its manufacture.** E. W. WESCOTT, Assr. to COMSTOCK & WESCOTT, INC. (U.S.P. 1,711,462, 30.4.29. Appl., 10.11.25).—Granular copper oxide is bonded by fine soft copper flowed under high pressure. J. S. G. THOMAS.

**Copper oxide electrodes for galvanic purposes.** W. ADLER and F. SCHIEBALY (Austr. P. 108,153, 6.10.25).—A plastic mass composed of copper oxide, a plastic binder, *e.g.*, clay, chamotte, and water is moulded, if desired about wire gauze etc., coated with strengthening material, and burned. J. S. G. THOMAS.

**[Oxide electrode for] galvanic batteries.** E. WOLFF (B.P. 305,009, 30.4.28. Ger., 28.1.28).—Copper oxide is carried between two undulated sheet-metal plates having numerous narrow perforations and placed so that the apices of the respective sets of undulations cross each other at right angles. J. S. G. THOMAS.

**Manufacture of electrodes for Edison accumulators.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,383, 25.11.27).—Extremely fine, pure, non-pyrophoric iron and nickel powders suitable for use, respectively, in the cathodes and anodes of Edison accumulators are produced by thermal decomposition of the corresponding metallic carbonyls. Thus, iron carbonyl is decomposed in the free space of a heated vessel at about 250°, whilst nickel carbonyl vapour is decomposed at 200°. The powders may subsequently be treated with hydrogen at about 500°, and/or may be subjected to pressure. J. S. G. THOMAS.

**Electrodes for use in electric arc-welding.** QUASI-ARC Co., LTD., and A. P. STROHMENGER (B.P. 308,475, 27.3.28).—A core metal containing 2–4% Mn is covered with a flux-forming material, *e.g.*, blue asbestos with a strip of aluminium, the percentage of manganese being proportioned relatively to the gauge of the core metal so that the fused metal deposited contains about 0.45% Mn. J. S. G. THOMAS.

**Electrode for use in apparatus for the decomposition of gases.** NORDISKE FABRIKER DE NO FA, A./S., and C. F. HOLMBOE (Austr. P. 108,289, 28.11.25).—Fluted ribs separated by conducting distance pieces are held together by screw bolts. J. S. G. THOMAS.

**Manufacture of electron-emitting bodies [cathodes].** E. Y. ROBINSON, and METROPOLITAN-VICKERS ELECTRICAL Co., LTD. (B.P. 307,099, 1.12.27).—A core of metal, *e.g.*, nickel or platinum, or of carbon, immersed in alkaline-earth metal powder or amalgam, *e.g.*, of barium, is heated *in vacuo* or in an inert or reducing atmosphere at about 1000° so that metal vapour penetrates the core, which is afterwards assembled in a valve. J. S. G. THOMAS.

**Production of oxide incandescence cathodes.** V. H. WOHL (Austr. P. 107,954, 26.5.26).—Electrically heated metallic filaments are drawn a number of times through solutions or suspensions of compounds of the alkaline-earth metals from which the alkaline-earth metal is deposited on heating, or through liquid amalgams of the alkaline-earth metals, and the deposited metal is oxidised by heating in air. J. S. G. THOMAS.

**[Gas-filling for] electron tubes, more especially for incandescence cathode Röntgen tubes.** C. H. F. MÜLLER (Swiss P. 122,456, 18.6.26).—Attack of metal parts of the tubes is prevented by filling the tubes with neon containing a small proportion, *e.g.*, 10%, of hydrogen or helium, at a pressure exceeding 0.0006 mm. of mercury. J. S. G. THOMAS.

**Marking of incandescence lamps and the like.** N. V. PHILIPS' GLOEILAMPENFABR. (Dutch P. 17,399, 10.3.26).—The internal surfaces of the glass bulbs etc. are marked by heating a stamped mixture of silica, alumina, and oxides of lead (litharge), boron, calcium, magnesium, cobalt, and an alkali oxide. J. S. G. THOMAS.

**Luminescent tube.** P. F. J. LEBRUN (U.S.P. 1,709,858, 23.4.29. Appl., 26.8.27).—The tube is filled with argon, neon, and mercury vapour. H. ROYAL-DAWSON.

**Electro-endosmosis method and apparatus.** V. A. LAPENTA (U.S.P. 1,711,416, 30.4.29. Appl., 10.8.25).—A membranous bag containing material to be treated, and a pair of porous cells containing respectively a positive and a negative electrode, are arranged in a vessel containing electrolyte, and current is passed between the electrodes. J. S. G. THOMAS.

**Magnetic material. [Nickel-iron-cobalt alloy.]** P. P. CIOFFI, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,708,936, 16.4.29. Appl., 23.12.26).—The heated alloy is subjected, during cooling, to a field which substantially neutralises the effect of extraneous fields, in order to obtain a material of constant magnetic permeability over a wide range of field intensities. J. S. G. THOMAS.

**Manufacture of bodies [dynamo brushes] from metal powder.** C. F. SHERWOOD (U.S.P. 1,708,192, 9.4.29. Appl., 7.5.27).—Powdered metal in the form of a brush moulded integrally with a part of a preformed conductor is sintered in a fluxing bath of molten salt, m.p. 650–815°. J. S. G. THOMAS.

**Production and application of plates for electric condensers etc.** J. Y. JOHNSON. From I. G. FARBENIND, A.-G. (B.P. 310,625, 22.3.28).—Finely-divided, electrically conducting materials, *e.g.*, metals, graphite, charcoal, etc., incorporated with paper pulp are worked up into foil of suitable thickness as, *e.g.*, in the process of paper manufacture. J. S. G. THOMAS.

**Impregnation material for use in electrical condensers.** TELEPHON-APPARAT-FABR. E. ZWIETUSCH & Co., G.M.B.H. (B.P. 299,697, 27.7.28. Ger., 29.10.27).—A mixture of ozokerite and colophony (9 : 1) is used. For use in an atmosphere permeated with steam or varying materially in temperature 0.5 pt. of carnauba wax is used in place of 0.5 pt. of colophony. J. S. G. THOMAS.

**Electrical [electrolytic] condenser.** S. RUBEN (U.S.P. 1,710,073, 23.4.29. Appl., 21.3.27).—A highly viscous paste composed of glycerin and a film-forming, supersaturated, plastic composition of boric acid and sodium borate is introduced between film-forming electrodes separated by a spacer medium, impregnated

to prevent electrolytic action between its fibres, the electrolyte, and the surface of the electrodes.

J. S. G. THOMAS.

**Apparatus for sorting or classifying articles by means of light.** WESTINGHOUSE ELECTRIC & MANUF. CO., ASSEES. OF V. K. ZWORYKIN (B.P. 292,474, 18.6.28. U.S., 16.6.27).—The surface of such materials as tinned or galvanised sheets, cardboard, paper, etc. is inspected for imperfections by a number of closely-spaced photo-electric cells illuminated (preferably from a single linear source of light) by reflection from the article to be tested, which is moved beneath the cells by means of rollers or other means. If no sheet is present under the cells, light is reflected from a special reflector just below the plane of travel of the sheets to prevent unnecessary operation of the rejecting mechanism. Any diminution of the light reaching any cell operates, through a thermionic valve and amplifiers, a solenoid-deflecting mechanism causing rejection of defective plates. A balanced circuit is used in which the cells are connected in series in two groups (like a Wheatstone bridge) so that one thermionic valve only need be provided for all the cells.

B. M. VENABLES.

**[Colourless] light filter [for ultra-violet rays].** F. ZERNIK (U.S.P. 1,688,259, 16.10.28. Appl., 27.4.25. Ger., 8.5.24).—Hydroxyquinolinesulphonic acids and their alkyl or aryl others, e.g., 8-methoxyquinoline-5-sulphonic acid, m.p. 302° (decomp.), 8-ethoxyquinoline-5-sulphonic acid, m.p. 283° (decomp.), and sodium 6-methoxyquinoline-5-sulphonate [free acid, m.p. 345° (decomp.)], are used.

R. BRIGHTMAN.

**Electrolytes for electrolytic rectifiers or the like.** FANSTEEL PRODUCTS CO., INC., ASSEES. OF E. W. ENGLE (B.P. 283,208, 30.12.27. U.S., 7.1.27).—See U.S.P. 1,680,210; B., 1928, 760.

**Electric discharge tubes.** S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 310,829, 17.7.28).

**Ionisation etc. of hydrocarbon materials (U.S.P. 1,709,814—5). Treating hydrocarbon-nitrogen mixtures (B.P. 296,355).—Liquid hydrocarbons (B.P. 284,224).—See II. Synthesis of nitrogen compounds etc. (B.P. 309,001—2).—See III. Iron-nickel-chromium alloys (B.P. 308,643). Steel for magnets (B.P. 308,549). Recovery of copper and nickel (B.P. 283,132). Porous metal articles (B.P. 311,141). Zinc (B.P. 285,373). Chromium (G.P. 454,168).—See X. Plastic insulating materials (U.S.P. 1,688,500).—See XIV. Beet juice (G.P. 452,436).—See XVII.**

## XII.—FATS; OILS; WAXES.

**Relation between titre and refractive index of fat [during hardening].** A. MARKMAN and M. SERGEJEV (Oil Fat Ind. [Russia], 1928, No. 7, 27—29; Chem. Zentr., 1928, ii, 2418).—No definite and constant relation was observed.

A. A. ELDRIDGE.

**Hardening of linseed oil.** V. VASSILIEV (Oil Fat Ind. [Russia], 1928, No. 7, 9—11; Chem. Zentr., 1928, ii, 2417—2418).—Chemical constants of linseed oil before and during saturation with hydrogen were determined; 11.7% of the hydrogen was required for the

reduction of hydroxyl groups, the original acid value being high (37.1).

A. A. ELDRIDGE.

**Determination of moisture and oil in soya beans.** L. BURSCH (Oil Fat Ind. [Russia], 1928, No. 2, 13—15; Chem. Zentr., 1928, ii, 2418).—The moisture content of cleaned and ground soya beans is about 1.94% smaller than corresponds with the analysis of the whole bean, the value for the oil content being correspondingly (0.46) high. It is necessary to determine (by distillation with xylene) the moisture content and the oil content of the ground bean, and the moisture in the whole bean, and to calculate the oil content of the latter.

A. A. ELDRIDGE.

**Detection of adulterants in cacao butter by oxidation with perbenzoic acid.** K. BODENDORF (Pharm. Ztg., 1929, 74, 384—385).—The degree of unsaturation and the rates of reaction determined with perbenzoic acid are highly characteristic for fats commonly used as adulterants. These fall into two classes, respectively much more and much less unsaturated than cacao butter itself. The fat dissolved in chloroform is treated with the acid in the same solvent, and samples withdrawn at intervals of 20 min. are titrated iodometrically; the results are plotted. The method will not detect simultaneous presence of a highly unsaturated and a nearly saturated adulterant.

S. I. LEVY.

**Solubility tests of castor oil.** H. P. TREVITHICK and M. F. LAURO (Oil & Fat Ind., 1929, 36, [3], 27—29).—The sp. gr., free fatty acids, and viscosity of castor oil increase slightly on ageing; the most important change, however, is the decreased solubility in alcohol. It is recommended that failure to pass solubility tests specifying alcohol below 95% in strength should not be considered proof of adulteration of castor oil since samples of oil, possessing otherwise the characteristics of pure castor oil and completely soluble in 95% alcohol, yielded turbid mixtures in 90% alcohol (1:2) and by the Finkener test after keeping for 10 months. The insoluble portion was isolated and had acetyl value 136, iodine value 79.4, showing that this portion was hydroxylated and not an adulterant. Concordant results could not be obtained by the filtration method of determining the acetic acid liberated from the acetylated oil; the distillation method is recommended, and details are given of a modified procedure wherein phosphoric acid is substituted for sulphuric acid.

E. LEWKOWITSCH.

**Alkali-refining of castor oil.** M. BAUMAN (Oil Fat Ind. [Russia], 1928, No. 7, 12—14; Chem. Zentr., 1928, ii, 2520).—Before treatment with alkali the oil is mixed at 20° with an equal volume of petroleum. The refined oil had  $d_{20}^{22}$  0.9682, iodine value 82.4, ash less than 0.01%, ignition temperature (Brenken) about 300°.

A. A. ELDRIDGE.

**Extraction of castor oil with benzene.** A. SLASCHTSHEV (Oil Fat Ind. [Russia], 1928, No. 3, 9—11; Chem. Zentr., 1928, ii, 2520).—The yield of castor oil on extraction with benzene is 1.5—2% less than when alcohol is used.

A. A. ELDRIDGE.

**Petroleum and alcohol as solvents for castor oil.** A. SLASCHTSHEV (Oil Fat Ind. [Russia], 1928, No. 7,

15—20; Chem. Zentr., 1928, ii, 2520).—Benzine, near the b.p., is a satisfactory solvent for castor oil; benzene, carbon tetrachloride, and carbon disulphide are effective, but the resulting oil has a dark colour. Alcohol gives inaccurate values, since other substances are also extracted.  
A. A. ELDRIDGE.

**Synthetic crude oil from cholesterol and from *Lycopodium clavatum*.** N. A. ORLOV (J. Appl. Chem. Russia, 1928, 1, 117—118).—Cholesterol (50 g.) was heated with a mixture of alumina and ferric oxide (3 g.) in hydrogen at 400—450° and 175 atm. for 14 hrs. The hydrogen then contained 7% of saturated compounds (calc. as CH<sub>4</sub>), and 43 g. of a transparent, yellow oil,  $[\alpha]_D^{25} +1^\circ 54'$ , remained. The oil was fractionated and its behaviour with sulphuric acid and with potassium permanganate was examined. The oil from lycopodium heated under pressure with hydrogen was likewise examined.  
CHEMICAL ABSTRACTS.

**Alkaline-earth stearate [emulsions].** A. P. LEE and J. E. RUTZLER (Oil & Fat Ind., 1929, 6, [3], 15—18).—The relative properties of emulsions of purified cottonseed oil stabilised by the stearates of magnesium, calcium, strontium, and barium have been investigated. All the emulsions were of the water-in-oil type. The magnesium and strontium stearates were insoluble in the oil at 25°, although traces of the metals could be found spectroscopically. The stability of emulsions stabilised with magnesium stearate (as measured by the degree of separation after 15 days) was the greater as the amount of peptiser was increased (0.001—0.006 mol./50 g. of oil), the curve showing a tendency to flatten. The stability and viscosity of emulsions containing equimolar (0.004/50 g.) quantities of the alkaline-earth stearates increased with the weight of the metal: barium stearate produced the only truly stable emulsion, which further showed a disproportionally great viscosity, perhaps attributable to a specific action of this peptising agent. Preliminary experiments showed that the viscosity of emulsions stabilised by strontium stearate increased continuously for a short time after production.  
E. LEWKOWITSCH.

**Electrical apparatus for the extraction in the laboratory of fats and oils.** F. GOGOLEV (Oil Fat Ind. [Russia], 1928, No. 3, 18—19; Chem. Zentr., 1928, ii, 2417).

**Litharge-glycerin cements.** STÄGER.—See IX.  
**Nickel catalyst,** MASCHKILLEISSON; WOLFSON.—See X.  
**Butter fat in dairy products.** CHAPMAN.—See XIX.

#### PATENTS.

**[Manufacture of soft] soap.** O. H. CARLSON (U.S.P. 1,705,852, 19.3.29. Appl., 16.9.27).—Soft soap is obtained by adding to a boiling mixture of Castile soap, sodium bicarbonate, borax, and glycerol a boiled mixture of stearic acid and sodium carbonate, boiling the product, cooling, and adding ether until the mass gels.

A. R. POWELL.

**Extraction of unsaponifiable and difficultly-saponifiable matter from fatty material.** J. K. MARCUS (U.S.P. 1,690,091, 30.10.28. Appl., 30.4.27).—Fatty material, e.g., cod-liver oil, is saponified with alcoholic potassium hydroxide, and the viscous-solid

soap mass, adjusted to 30% aqueous alcohol, is extracted with ethylene dichloride, the heavier extract layer being run off free from dissolved soap.  
R. BRIGHTMAN.

**Production and use of stable sulpho-acids of high mol. wt. and their salts.** ORANIENBURGER CHEM. FABR. A.-G., Assees. of CHEM. FABR. MILOH A.-G. (B.P. 288,126, 20.12.27. Ger., 1.4.27).—When neutral fats, fatty acids, resins, etc. are mixed with aliphatic or aromatic carboxylic acids, hydroxy-acids, keto-acids, or their anhydrides or chlorides, and treated with strong condensing-sulphonating agents such as halogenated sulphonic acids or mixtures of sulphuric acid and phosphoric pentoxide, water-soluble products of enhanced utility in the pasting of dyes, stabilising of dye solutions, splitting of fats, and for cleaning, wetting, and oiling purposes, are obtained.  
E. HOLMES.

**Manufacture of compressed cakes [from soap powder].** F. KILIAN (B.P. 311,070, 14.4.28).

**Composition for bleaching (U.S.P. 1,687,803—5).** See XIX.

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

**Chemical composition of Russian turpentine oil from *Pinus sylvestris*.** B. ARBOUSOV (J. Russ. Phys. Chem. Soc., 1929, 61, 255—268).—A résumé of previous researches on the chemical composition of turpentine from various conifers. The presence of *d*- $\alpha$ -pinene in the turpentine from *Pinus sylvestris* had been definitely established, but data concerning the two other main constituents (nopinene and *d*- $\Delta^3$ -carene) has been indefinite. In the present investigation the turpentine was thoroughly dried and fractionated, and the sp. gr. b.p., sp. rotation, and coefficient of dispersion were determined. The turpentine contained 80—85% of *d*- $\alpha$ -pinene, b.p. 49.6°/13 mm.,  $d^{15}_D$  0.8625,  $[\alpha]_D^{15}/[\alpha]_C = 1.97—1.98$ . Examination of the physical constants of the higher fractions showed the presence of about 14% of *d*- $\Delta^3$ -carene, b.p. 165—168°,  $d^{17.2}_D$  0.8593,  $[\alpha]_D^{17.2} +13.34^\circ$ ,  $[\alpha]_D^{17.2}/[\alpha]_C = 2.12$ ; there were no indications of the presence of nopinene. That *d*- $\Delta^3$ -carene was present in the turpentine fraction was confirmed by Aschan's method—a purple coloration with acetic anhydride and concentrated sulphuric acid, and by the formation of a crystalline nitroso-compound, m.p. 145.5°, on treatment with amyl nitrite, glacial acetic acid, and sulphuric acid.  
M. ZVEGINTZOV.

**Synthetic resins as construction materials in chemical industry.** E. KALMAN (Chem. Fabr., 1929, 169—170).—On account of their indifference to acids and alkalis, chlorine, and organic and inorganic solvents and reagents, the phenol-formaldehyde resins are employed for the construction of vessels and plant of all kinds; a container weighing 1.8 tons, constructed in one piece without strengthening by iron or otherwise in any way, is claimed to be the biggest piece of apparatus of any kind ever constructed without joints from a single piece of material.  
S. I. LEVY.

**Litharge-glycerin cements.** STÄGER.—See IX.

#### PATENTS.

**Manufacture of titanium-containing compounds.** F. G. C. STEPHENS, L. J. ANDERSON, and W. A. CASH

(B.P. 309,051, 1.10.27).—A titanium salt solution is hydrolysed by adding it to warm or boiling water to which has previously been added silicic acid or other colloid (excluding colloidal titanium dioxide) in order to produce a hydrated titanium dioxide that is filterable, although sufficiently fine for use as a pigment. The pigment may be precipitated or subsequently associated with an insoluble alkaline-earth sulphate. S. S. WOOLF.

**Production of stable [colour] pastes and lakes.** I. G. FARBENIND. A.-G. (F.P. 629,764, 24.2.27. Belg., 2.3.26).—The products comprise zinc lakes and solvents which yield or contain free fatty acids. L. A. COLES.

**[Nitrocellulose] lacquers.** CARBIDE & CARBON CHEMICALS CORP. (B.P. 289,373, 12.3.28. U.S., 25.4.27).—Nitrocellulose lacquers and thinners for the same containing ethylene glycol monoethyl ether and its acetate in suitable proportions are claimed.

S. S. WOOLF.

**Nitrocellulose composition.** H. BRADSHAW, E. H. NOLLAU, and R. G. WOODBRIDGE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,703,415, 26.2.29. Appl., 21.12.21. Renewed, 21.3.24).—Nitrocellulose containing more than 12.4% N is dehydrated by displacement with denatured alcohol, the excess of alcohol being expressed under high pressure, and the block is colloided with ether in presence of 0.5% of diphenylamine, comminuted, and seasoned first for 15–25 days at 60° and finally for 4 months or more until the viscosity is less than 200 sec. at 28° by the steel-ball method. The product is mixed with a softener, *e.g.*, castor oil, blown rapeseed oil, butyl phthalate, and a pigment and dissolved in a volatile solvent.

R. BRIGHTMAN.

**Driers and their combination with drying oils.** H. E. BUC, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,686,484—6, 2.10.28. Appl., [A] 16.9.27, [B, C] 26.9.27).—The oil-soluble sulphonic acids formed by sulphonating petroleum distillates, particularly lubricating distillates, are converted into the corresponding oil-soluble sodium sulphonates. These are extracted by a selective solvent, *e.g.*, 35–65% aqueous alcohol, and freed from oil by treatment with 10–20% of gasoline by vol. The sodium sulphonate, when converted into the corresponding oil-soluble lead (A), cobalt (B), or manganese (C) sulphonates and freed from insoluble inorganic matter by dissolution in an organic solvent, *e.g.*, a mixture of benzene and 75% of ethyl or isopropyl alcohol, affords driers which may be used alone or in combination with other driers.

R. BRIGHTMAN.

**Artificial resin and its manufacture.** F. SEEBACH, Assr. to BAKELITE GES.M.B.H. (U.S.P. 1,683,701, 11.9.28. Appl., 21.3.27. Ger., 29.3.26).—Fusible resins containing about 4% N and of mol. wt. 370–380, which are soluble in organic solvents, are obtained by the action of formaldehyde on a mixture of 1 mol. of an aromatic amine or a salt or derivative thereof (including, *e.g.*, anthranilic acid, 1-naphthylamine-5-sulphonic acid) with more than 0.5 mol. of phenols. The condensation product is heated alone or in presence of organic acid anhydrides.

R. BRIGHTMAN.

**[Artificial] resin and its manufacture.** A. E. MAZE (U.S.P. 1,683,835, 11.9.28. Appl., 14.9.26).—

Diacetone alcohol is condensed with 3 mols. of formaldehyde in presence of an alkali or alkaline-earth hydroxide or soluble carbonate or phosphate. R. BRIGHTMAN.

**Manufacture of phenol resins.** BAKELITE CORP. (B.P. 304,659, 27.10.27. U.S., 11.11.26).—A phenol (2 mols.) is condensed with formaldehyde (more than 2 mols.) in the presence of aniline (1 mol.) and a basic catalyst, *e.g.*, hexamethylenetetramine. The aniline is subsequently expelled (and recovered) under conditions avoiding complete transference of the reactive resin to the "resinoid" state.

S. S. WOOLF.

**Manufacture of resin-impregnated sheets.** G. E. WIGHTMAN, Assr. to BAKELITE CORP. (U.S.P. 1,703,414, 26.2.29. Appl., 22.6.26).—Fibrous sheets impregnated with reactive and plastic phenolic resin are dried by continuous movement below 40° through a zone of reduced pressure.

R. BRIGHTMAN.

**Producing a viscous phenol condensation product.** R. GÜNTZEL, Assr. to SCHIEFERWERKE AUSDAUER A.-G. (U.S.P. 1,711,411, 30.4.29. Appl., 8.3.28. Ger., 10.3.27).—See B.P. 286,731; B., 1929, 294.

**Impregnation of articles with varnishes of synthetic resins.** MANUF. DE MACHINES AUXILIAIRES POUR L'ELECTRICITÉ & L'INDUSTRIE (B.P. 286,305, 28.2.28. Belg., 3.3.27).

**Grinding mill** (B.P. 306,630).—See I. **Products from waste cracked benzines** (B.P. 309,718).—See II. **Antifouling coatings** (U.S.P. 1,689,008).—See XXIII.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

### PATENTS.

**Reclamation of rubber.** J. J. MORIARTY, Assr. to J. H. S. KERR (U.S.P. 1,710,100, 23.4.29. Appl., 8.12.27).—Fibrised, vulcanised rubber is disintegrated, mixed with a plasticiser, and milled; during the last operation a reclaiming agent is introduced. The mixture is then subjected to the action of steam under pressure.

D. F. TWISS.

**Manufacture of reinforced rubber.** H. H. KELLER (U.S.P. 1,710,481, 23.4.29. Appl., 31.3.27).—Rubber (100 pts.) is mixed with sulphur (3), zinc oxide (26½), extremely fine aluminium oxide (26), and an appropriate quantity of a vulcanisation accelerator.

D. F. TWISS.

**Manufacture of reinforced hard rubber.** W. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,689,570, 30.10.28. Appl., 23.12.26).—Comminuted hard rubber, disintegrated partly-cured rubber, sulphur, cotton, or other fibre, and rubber latex are mixed, dried, moulded, and vulcanised.

R. BRIGHTMAN.

**[Accelerators for] manufacture of vulcanised rubber.** W. SCOTT, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,688,755—8, 23.10.28. Appl., [A] 17.2.25, [B—D] 8.4.27).—(A) The condensation products of aryl- or diaryl-guanidines and polyphenols, *e.g.*, quinol and its derivatives, give improved ageing properties. Further, 4-dimethylamino-4'-ethoxy-s-diphenylguanidine (B) from *p*-phenethylthiocarbimide and *p*-aminodimethylaniline with subsequent desulphurisation, 4-dimethyl-

*amino-s-diphenylguanidine* (c), and *4-dimethylamino-2'-methyl-s-diphenylguanidine* (d) are used as accelerators.

R. BRIGHTMAN.

**Manufacture of rubber surfacing on a rigid base.** M. C. TEAGUE, ASSR. to NAUGATUCK CHEM. CO. (U.S.P. 1,705,273, 12.3.29. Appl., 22.4.25).—A concrete base is scored while still moist, allowed to set, and surfaced with a rubber mixture comprising latex, fillers, sulphur, a vulcanising agent, and sulphonated castor oil.

A. R. POWELL.

**Fixing a thin layer of sponge rubber to the surface of india-rubber goods.** S. MORIMOTO (U.S.P. 1,685,954, 2.10.28. Appl., 14.4.27. Japan, 24.6.26).—A sponge-rubber solution, obtained by mixing, *e.g.*, 600 pts. of unvulcanised india-rubber, 100 pts. of zinc white, 8 pts. of lithopone, 8 pts. of sulphur, 5 pts. of ammonium carbonate, 7 pts. of white substitute, 2 pts. of colouring matter, and 140 pts. of spindle oil, dissolving the mixture in carbon disulphide, and diluting with naphtha, is applied to the surface of unvulcanised rubber goods and the whole is hot-cured.

R. BRIGHTMAN.

**Manufacture of plastic and mouldable composition.** C. KULAS (U.S.P. 1,688,500, 23.10.28. Appl., 2.1.26).—Plastic material for electrical insulation purposes is obtained, *e.g.*, by adding to the viscous solution of 50 pts. of resol in 50 pts. of alcohol or acetone, 100 pts. of pulverised scrap or waste rubber, or 50 pts. of rubber and 50 pts. of filler or colouring material. The mixture is stirred and gently boiled, the solvent recovered, and the residual mass rapidly calendered to expel volatile constituents, seasoned at about 30—40° for, *e.g.*, 14 days, moulded, and dried at 60—160° at a gradually increasing temperature.

R. BRIGHTMAN.

**Bonding of rubber to metal.** H. GRAY, ASSR. to B. F. GOODRICH CO. (U.S.P. 1,689,628, 30.10.28. Appl., 27.4.27).—The metal surface is coated with sulphur chloride or a solution thereof and placed under pressure in contact with a rubber composition, which is then vulcanised.

R. BRIGHTMAN.

**Stabilised latex and its production.** M. R. DAY, ASSR. to RUBBER LATEX RES. CORP. (U.S.P. 1,689,581, 30.10.28. Appl., 3.1.27. Renewed 27.3.28).—See B.P. 279,336; B., 1928, 62.

**Rubber-covered metal sheets** (B.P. 301,300).—See X.

## XV.—LEATHER; GLUE.

**Water penetration tests for sole leather.** F. O. SPRAGUE (J. Amer. Leather Chem. Assoc., 1929, 24, 87—88).—A copper cylinder, 2 ft. high and 2 in. in diam., is filled with water after clamping to a disc of the leather to be tested. At the first drop of water penetrating through the leather, electrical contact is made which stops a clock. The time of penetration varies from a few minutes up to more than one week for samples from different parts of a waterproofed hide. (Cf. Gayley, *ibid.*, 1916, 11, 37.)

D. WOODROFFE.

### PATENTS.

**Chrome tanning material.** C. D. MARLATT, ASSR. to INDUSTRIAL WASTE PRODUCTS CORP., and W. H. DICKERSON (U.S.P. 1,698,505, 8.1.29. Appl., 15.4.26).—A dry powder is produced by heating chrome tanning

liquor to 113° and spraying it into a hot gaseous drying medium.

H. S. GARLICK.

**Water-soluble product from lignite** (B.P. 284,670).—See II.

## XVI.—AGRICULTURE.

**Significance of water movements in a sandy soil in its management.** K. WIESENTHAL (Z. Pflanz. Düng., 1929, 8B, 128—136).—Records of moisture contents at varying depths, and depth of permanent water level in a sandy soil for considerable periods are presented. There is a saturated layer of soil just above the permanent water level, and above this an "intermediate layer" with the lowest mean water content rising to the top soil. The higher mean water content of the top soil is due to its organic matter. The "intermediate layer" is characterised by the widest variations in moisture content, and in periods of drought this loses more water than the top soil. In two sandy soils examined the water supply on the surface layer was mainly dependent on the rainfall, the capillary rise from the subsoil not being sufficiently great to reach the plants during considerable periods. On these soils hoeing resulted neither in increased crops nor in increased water content of the surface soil.

A. G. POLLARD.

**Nutrient requirement of soils as indicated by the Mitscherlich and Neubauer methods.** H. WIESSMANN and E. SCHRAMM (Z. Pflanz. Düng., 1929, 8B, 105—128).—In the Mitscherlich method comparison of nutrient contents based on the total, straw, and grain yields shows the grain : straw ratio to depend on the potash content of the soil, and crop yields in soils of varying potash deficiency are not strictly comparable. Again, soils deficient in potash suffer by the dilution with sand in these experiments. Differences in nutrient values obtained on a straw basis and on a grain basis increase with potash deficiency, and only in rich soils do these values approximate. In practice nutrient values based on the total crop yields are preferable. Differences in nutrient values for phosphate based on grain and straw yields are less than in the case of potash, and these differences decrease with increased phosphate deficiency. Grain yield values are 16—21% higher than those calculated on straw yields, and show a greater divergency from the corresponding Neubauer values. In the numerous soils examined agreement between Mitscherlich and Neubauer values was very imperfect, although there was a general tendency for relative sufficiency and deficiency to be similarly indicated by both. Mitscherlich values for potash content averaged about one half those by Neubauer's method, and for the phosphate content 47—80% greater. Probably Mitscherlich's growth factor is too high and the potash values correspondingly low. The relatively lower phosphate values shown by Neubauer's method in rich soils is ascribed to the less complete assimilation in this case compared with poor soils. Preliminary heating of the soil (Dirks) leads to poorer results for potash in both methods, and to an improved but by no means good agreement in phosphate values. On the average the poor soils among those examined showed lower  $p_H$  values than the richer ones. A. G. POLLARD.

**Nitrogen content of Red River Valley soils.** J. H. ELLIS and W. SHAFER (Sci. Agric., 1928, 9, 231—248).—The high nitrogen content of these Manitoba soils is established. Dark intrusions of surface material extending into the underlying horizon have a higher nitrogen content than the normal profiles.

## CHEMICAL ABSTRACTS.

**Soil moisture at permanent wilting of plants.** F. J. VEIHMEYER and A. H. HENDRICKSON (Plant Physiol., 1928, 3, 355—357).—The quantity of water available for plant growth cannot be determined by the "moisture equivalent" alone. It is necessary to know the residual moisture at permanent wilting for a particular soil.

## CHEMICAL ABSTRACTS.

**Huminit.** DENSCH (Z. Pflanz. Düng., 1929, 8B, 142—147).—Huminit is a finely-powdered preparation from peat or lignite in which the humus acids are neutralised. Its objects appear to be to act as a soil ameliorant, to improve the physical condition, to act as a nitrogen fertiliser, and to provide material inducing greater bacterial activity, notably the fixation of nitrogen. Pot and field trials showed no proof of any effects other than the improved soil texture commonly associated with added organic matter. A. G. POLLARD.

**Effects of nitrogen fertilisers.** HASELHOFF (Z. Pflanz. Düng., 1929, 8B, 136—142).—General comparisons are made of the common fertilisers. The iodine content of Chile saltpetre has no favourable influence on crop yields, on the grain : straw ratio, or on the weight per 1000 grains. In general, calcium nitrate and sodium nitrate gave similar results, and there was no confirmation of the opinion that the sodium content of the latter improved the yield of root crops. As regards efficiency calcium cyanamide was more dependent on soil characteristics than were ammonium salts or nitrates, but, in general, was rather less efficient. Injury to seedlings often associated with the use of cyanamide was not influenced by the conjoint application of iron or manganese salts. A. G. POLLARD.

**Calcium, potassium, and iron balance in certain crop plants in relation to their metabolism.** W. F. LOEWING (Plant Physiol., 1928, 3, 261—275).—Two acid muck soils, when treated with calcium carbonate, showed depressed yields of maize and wheat; this result is attributed to potash insufficiency and to reduction of sap acidity. Potash additions were injurious with 3 of 4 acid muck soils. With such soils high in iron content, toxic accumulations of iron may occur in tissues owing to increased sap acidity. In soils low in calcium and magnesium the addition of potassium chloride may depress the lime and magnesia to the starvation point. High yields in young grain plants were associated with high carbohydrate and organic nitrogen content, and low yields with low protein, low carbohydrate, and high nitrate content.

## CHEMICAL ABSTRACTS.

**Lime penetration resulting from surface application to pasture land.** P. R. NELSON (Soil Sci., 1929, 27, 143—146).—The greater part of the lime applied to pastures remains at the surface, but there is a gradual and slight increase in  $p_H$  value of the soil to a depth of

9 in. Among mineral fertilisers, gypsum alone showed a slight tendency to neutralise soil acidity.

A. G. POLLARD.

**Effects of crude petroleum on nitrate production, seed germination, and growth.** H. F. MURPHY (Soil Sci., 1929, 27, 117—120).—Application of crude petroleum to the soil surface considerably reduced nitrate production. The effect was intensified when the oil was mixed with the soil. 1% of oil mixed with soil practically inhibited nitrification. Germination of seed was seriously reduced by treatment of the soil with petroleum.

A. G. POLLARD.

**Influence of potash manuring on the hectolitre-weight of cereals.** A. JACOB (Z. Pflanz. Düng., 1929, 8B, 61—76).—Numerous results of field trials show that potash manuring results in a definitely higher volume-weight of cereal grains.

A. G. POLLARD.

**Nitrogen contents of weeds and their relation to soil fertility.** H. J. HARPER and H. F. MURPHY (Proc. Oklahoma Acad. Sci., Univ. Oklahoma Bull., 1928, [ii], No. 410; Studies Series 30, 73—77).—Weeds transfer to the soil large amounts of organic and nitrogenous substances. The nitrogen content (usually 1.0—2.5%) of the following plants was determined: sunflower, ragweed, rosin weed, coreopsis, wild lettuce, cocklebur, crab grass, black-eyed Susan, water-hemp, Russian thistle, Bermuda grass, oat and wheat straw (lowest, 0.519%), soya beans, peas (2.36—3.24%), hairy vetch (highest, 2.67—4.1%), sweet clover, cotton burrs, sesbanis, and rye.

CHEMICAL ABSTRACTS.

**Analysis of insecticides. Liquid insecticides immiscible with water. Hydrocarbon mixtures, carbon tetrachloride, nitrobenzene, naphthalene, methyl salicylate.** M. FRANÇOIS and L. SEGUIN (Ann. Falsif., 1929, 22, 226—232).—See B., 1929, 222.

**Rhode Island soils.** B. L. HARTWELL and J. B. SMITH (Rhode Island Agric. Exp. Sta. Bull., 1928, No. 214, 24 pp.).

**Potential fertility of Oklahoma soils.** H. J. HARPER and H. F. MURPHY (Proc. Oklahoma Acad. Sci., Univ. Oklahoma Bull., [ii], No. 409, 1928, 7, 138—141).

**Soils of the Punjab.** P. E. LANDER, R. NARAIN, and M. M. LAL (Mem. Dept. Agric. India [Chem.], 1929, 10, 25—142).

**Determination of phosphoric acid in fertilisers.** JORGENSEN.—See VII.

## PATENTS.

**Destruction of cacti.** H. STOLTZENBERG (U.S.P. 1,686,582, 9.10.28. Appl., 20.10.25).—Cacti are subjected to the action of aromatic arsenic compounds, e.g., phenyl- and diphenyl-arsine oxide, chloride, or cyanide, phenarsazine chloride, etc., either as vapours or sprays or as injections of 5% solutions in cresol or sulphuric acid.

R. BRIGHTMAN.

**Products from waste cracked benzines (B.P. 309,718). Nitrogenous bases (U.S.P. 1,686,136).—See II. Germicidal coatings (U.S.P. 1,689,008).—See XXIII.**

## XVII.—SUGARS; STARCHES; GUMS.

## Coagulation of colloids from beet sugar liquors.

E. GUNDERMANN (Chem.-Ztg., 1929, 53, 305—307, 322—323).—A résumé of the literature is given. The velocity of coagulation by addition of acids reaches a maximum at  $p_H$  3.2, and then diminishes with increasing acidity; it increases with rising temperature. The coagulated sediment goes into solution again on shaking and again separates on keeping; it is only partially retained by filter paper. The proportion of sediment is also at a maximum at  $p_H$  3.2; relatively more is obtained from more dilute solutions than from stronger solutions. The colour of the solution improves with coagulation, the improvement being greatest at  $p_H$  3.2; further addition of acid results in a darker colour.

S. I. LEVY.

**Solid molasses.** STAIGER and GLAUBITZ (Z. Spiritusind., 1929, 52, 163—164).—A sample of raw molasses in the solid form prepared by the aid of the heat of the sun contained 8.22% of moisture and 0.32% N, and for the neutralisation of 20 g. required 0.6 c.c. of *N*-sodium hydroxide. There were also present substances which reduced Fehling's solution but were not fermentable. The yield of alcohol varied from 31.75 to 34.25 c.c. per 100 g. of molasses according to the race of yeast used for the fermentation. Sterilised wort with added molasses showed a strong development of *Bacillus subtilis*, butyric and lactic acid bacteria, and streptococci, whilst a gelatin plate culture yielded over 1000 colonies of red yeast, fungi, torulæ, etc.

C. RANKEN.

**Determination of starch syrup and dextrose in presence of sucrose and invert sugar.** C. I. KRUISHEER (Chem. Weekblad, 1929, 26, 254—263. Cf. B., 1926, 963; also Schoorl, B., 1929, 336).—A number of formulæ are given, from which the composition of mixtures can be calculated after the determination of six values, viz., original reducing power and lævulose content,  $R_1$  and  $F_1$ , the same after partial inversion with warm dilute hydrochloric acid,  $R_2$  and  $F_2$ , and again after complete inversion at 100° with hydrochloric acid,  $R_3$  and  $F_3$ . A large number of control analyses on mixtures specially prepared, and of analyses of jams, sweets, and chocolates is given.

S. I. LEVY.

## PATENTS.

**Improvement of crude beet juice.** A. GRÄNTZ-DÖRFFER (G.P. 452,436, 22.1.25).—The juice after treatment with lime is subjected to the action of an electric current, using non-reacting electrodes brought into direct contact with the juice, which may simultaneously be treated with air, with alkalis, e.g., lime, or with carbon dioxide, sulphur dioxide, etc.

L. A. COLES.

**Rotary crystallisers. Separating mother-liquor from crystals** (B.P. 286,599).—See I.

## XVIII.—FERMENTATION INDUSTRIES.

**Drying of hops.** Institute of Brewing Research Scheme. Report of the seventh season's work at the experimental oast, 1927. A. H. BURGESS (J. Inst. Brew., 1929, 35, 235—246).—The depth of loading, air speed, and time of drying are related in the

following manner:  $T = 87.3L/a^{1.047} + M$ , where  $T$  is the time of drying in min.,  $L$  the loss of water per sq. ft. of kiln floor in oz.,  $a$  the air speed in ft. per min., and  $M$  is the minimum time depending mainly on the temperature employed and slightly on the air speed. Hops dried at 40° have a superior preservative value to those dried at higher temperature, the amount of  $\alpha$ -acid being reduced at the higher temperatures. Hops dried in nitrogen have a higher preservative value than those dried in air, whilst the use of sulphur improves the aroma and colour. The amount of sulphur retained by the hops is greater if the hops are wet when the sulphur is burned. The moisture content of hops varies at different depths of the load during drying in the intermittent type of kiln, and to avoid overdrying the lowest hops and underdrying the top hops a continuous type of kiln should be used.

C. RANKEN.

**Preservative principles of hops. X. Modification of Ford and Tait's gravimetric process for the evaluation of hops.** J. J. H. HASTINGS and T. K. WALKER (J. Inst. Brew., 1929, 35, 229—233; cf. B., 1928, 229).—10 g. of freshly-minced hops are either shaken vigorously or mechanically stirred for 10 min. with 100 c.c. of methyl alcohol in a wide-necked bottle of 250 c.c. capacity. The extracted hops are allowed to settle and the supernatant liquid is decanted on to a fluted filter paper. The percentage of  $\alpha$ -soft resin in the alcoholic extract of the hops is determined by precipitation with a 1% solution of lead acetate in methyl alcohol, and the  $\beta$ -soft resin is determined by extraction of the filtrate from the  $\alpha$ -lead salt with light petroleum, subsequent to dilution with twice its volume of water. Alternatively, the total soft resin can be determined by extracting a further portion of the alcoholic extract of the hops, and the amount of  $\beta$ -resin calculated by subtracting from the total soft resins the value found for the  $\alpha$ -resin. It is emphasised that the precipitation of the  $\alpha$ -lead salt should be carried out at 60°.

C. RANKEN.

**Interstitial liquid and [yeast] cell moisture.** N. C. BEEBLESTONE (J. Inst. Brew., 1929, 35, 260—263; cf. B., 1928, 170).—The author reviews the criticisms of his proofs that the yeast cell contains approx. 54% of moisture, and points out that the theory that the moisture content of the cell is 70% depends experimentally on results obtained from cells which are not normal, but which have been distended by the entry of interstitial liquid during pressing. The results should only be taken for yeast cake formed by drainage, where the cake consists of spheres grouped in the hexagonal form of packing. Since the amount of space between such spheres is approx. 27% of the total volume, it follows that the interstitial liquid is 25% of the weight of the yeast cake, and that the remaining 75% of the weight of the cake consists of the cells. From this latter value, and taking the calculated minimum moisture of a cake as 69%, it is calculated that the maximum cell moisture is 59% approx.

C. RANKEN.

**Reproduction of yeast in open, loosely covered, and closed fermentation vessels.** E. LÜHDER and W. KILP (Z. Spiritusind., 1929, 52, 160—162).—With equal seeding rates, the reproduction of yeast in wort

varied according to the type of fermentation vessel. The reproduction, which was greatest in open vessels, diminished if the vessel was loosely covered, and was lowest when the vessel was completely shut off from the air by a water-seal. With the seeding rate of 5 g. of yeast per litre of wort, the reproduction at the end of 72 hrs. with the open vessel was 6.68-fold, with the loosely closed type 6.10-fold, and with the completely closed vessel 5.97-fold. With a seeding rate of 0.5 g. per litre, the corresponding values were 45.90, 29.88, and 23.12. The loss by evaporation during fermentation in the open vessels amounted to  $\frac{1}{3}$  of the original volume of the liquid. With the partly-closed vessel  $\frac{1}{5}$ — $\frac{1}{10}$  of the original volume was lost, whilst with the closed type the volume remained constant. The evaporation was accompanied by a loss in alcohol, and to this deficiency with its favouring effect on yeast growth was partly attributed the greater yeast reproduction in open vessels. If the volumes of the fermenting liquids were retained constant by the addition of water during fermentation, the relative reproduction rates in the three types of vessel were again as those given above. C. RANKEN.

**Heating of the vintage.** J. DUBAQUIÉ (Ann. Falsif., 1929, 22, 211—213).—The whole grapes ( $\frac{1}{10}$ — $\frac{1}{8}$  of the total vintage) are placed in a hermetically sealed container kept at 40—50° for several hours. Conditions favourable for intercellular fermentation are set up, and the temperature favours the action of the acids on the pectoses. An intense and very desirable odour is developed and the must appears "fatter" and sweeter than usual. The heated product, which is sterile, is added to the vats after the first fermentation. The simplicity and beneficial results are lost if crushed grapes are heated. D. G. HEWER.

**Determination of corrected volatile acidity of wines.** R. MARCILLE (Ann. Falsif., 1929, 22, 224—225; cf. B., 1922, 911 A).—Mathieu's modification of Sadler's method (Compt. rend. du 6<sup>e</sup> Congrès de Chimie industrielle, p. 670) for determination of volatile acidity in sulphited wines needs modification when a high proportion of aldehydes is present, since the aldehyde may combine with the total sulphuric anhydride, thus causing the two determinations to take place under similar conditions. It is necessary to determine free and total anhydride, the difference giving the combined acidity. The total volatile acidity and that of the free sulphuric anhydride, and 70% of the combined anhydride after being expressed in terms of sulphuric acid, are deducted from the total to give the corrected volatile acidity. To accomplish this the free anhydride (g./litre) is multiplied by 1.55 and the combined anhydride by 1.08 and the results are added.

D. G. HEWER.

**"Bacteriophage," a new antiferment with formic acid base.** R. MARCILLE (Ann. Falsif., 1929, 21, 19—22).—A sample of the Belgian product had total acidity (as sulphuric acid) 190 g./litre, fixed acidity (Roos' method) 41 g./litre, and yielded a residue (per 100 c.c.) of 10.4 g. (syrupy liquid) at 105°, of 6.6 g. at 120°, and of 3.73 g. (black, viscous mass) at 130°. Fractional distillation, which could not be completed, gave 60% of a liquid of acidity 92 g. per litre; the mol. wt. of the

acid derived from the sodium salt approximated to that of formic acid. As an antiseptic for wort the product was very active, but should be used in doses 4 or 5 times that recommended by the makers. About 140 g. of formic acid per litre are present. Formic acid added in efficacious doses to wines would cause deterioration, augment their volatile acidity, and might be injurious to the health of the consumer. D. G. HEWER.

**Solid molasses.** STAIGER and GLAUBITZ.—See XVII. **Essential oil of hops.** CHAPMAN.—See XX.

#### PATENTS.

**Test reagents for determining alcohol and colour [added caramel] in alcoholic liquids.** J. F. WILLIAMS, Assr. to C. J. ROBERTS and L. C. JACKSON (U.S.P. 1,689,901—2, 30.10.28. Appl., [A] 30.7.26, [B] 2.8.26).—(B) 70 pts. of amyl alcohol or refined fusel oil ( $d$  0.8103—0.8119, b.p. 110—130°), 28 pts. of toluene (or xylene), and 2 pts. of tartaric acid, acetic acid, or other soluble acid are mixed to give a reagent of  $d_{20}^{20}$  0.8333. (A) This reagent when added to whiskey etc. extracts alcohol and natural colouring matter. The decrease in volume of the whiskey shows by reference to a curve (given) the percentage of alcohol originally present; added colouring matter (caramel) is shown by the colour of the extracted samples. R. BRIGHTMAN.

**Distillation of liquids. Apparatus for recovering alcohol from vapours.** A. FREYMAN (B.P. [A] 310,400 and [B] 310,403, 25.1.28).—(A) Vapour generated by heating in a still or oven is conducted to a dephlegmator, and is thence passed into a cooler. The vapour on its way to the condenser is used to heat vessels or containers charged with condensate from the condenser for the distillation of volatile matters from such condensate. (B) Vapour containing alcohol, e.g., the vapour issuing from a baker's oven, prior to passing to a cooler where the more volatile constituents collect, is conducted to a condenser which is kept at a predetermined temperature by water or liquid the b.p. of which corresponds to the maximum temperature at which the required condensation takes place. C. RANKEN.

**Apparatus or mills for treating or grinding malt.** G. PORTEUS (B.P. 311,074, 21.4.28).

#### XIX.—FOODS.

**Electrolytic conductivity of aqueous extracts of flour.** K. SCHMORL (Z. ges. Mühlenwesen, 1928, 5, 62—65, 88—90, 126—127; Chem. Zentr., 1928, ii, 2299).—Of the water-soluble organic constituents, chiefly the nitrogenous substances affect the conductivity. With the degree of grinding the mineral matter and nitrogen increase regularly. The use of conductivity determinations in control is discussed.

A. A. ELDRIDGE.

**Effect of lecithin in dairy products on butter fat determinations.** O. W. CHAPMAN (J. Dairy Sci., 1928, 11, 429—435).—Lecithin contents (average) are reported as follows: milk 0.0447, cream 0.1981, skim milk 0.0165, buttermilk 0.1302%. The fat found in buttermilk contains 13% of lecithin.

CHEMICAL ABSTRACTS.



**Lactometer as used to determine solids-not-fat in milk.** C. F. HOYT, N. C. SMITH, L. M. LAMPERT, and L. G. SAYWELL (Calif. Dep. Agric. Bull., 1928, 17, 594—603).—The calibrated lactometer reading should be made at the top of the meniscus. Unless the prior treatment of a sample is known, the values are subject to uncertainty. Average values for samples of maximum sp. gr. (using the Babcock formula) agree fairly well, and those for samples in which the original sp. gr. has been restored by heating (using the formula  $S - F = (L/4) + (L/5) + 0.2$ , where  $S - F =$  solids-not-fat,  $L =$  lactometer reading, and  $F =$  fat %) agree well with gravimetric results. CHEMICAL ABSTRACTS.

**Connective tissue content of beef muscle.** H. H. MITCHELL, T. S. HAMILTON, and W. T. HAINES (J. Nutrition, 1928, 1, 165—178).—Methods for the determination of elastin and collagen are described. The distribution of elastin and collagen in various "cuts" of meat has been examined. Age probably does not greatly influence the content of connective tissue in muscle. CHEMICAL ABSTRACTS.

**Proximate composition of fresh fruits.** C. CHATFIELD and L. McLAUGHLIN (U.S. Dep. Agric. Circ., 1928, No. 50, 1—19).—Data for 65 fresh fruits or their products are tabulated. CHEMICAL ABSTRACTS.

**Hydrogen swelling of canned ready-to-serve prunes. Effect of blanching.** E. M. MRAK and P. H. RICHERT (Fruit Prod. J., 1929, 8, 11—13, 14—15, 15).—The rate of spoilage under various conditions of canning has been studied. Least corrosion occurred when the amount of oxygen originally present was greatest. Blanching increases the rate of formation of hydrogen. CHEMICAL ABSTRACTS.

**Flour quality: its nature and control.** E. A. FISHER (Nat. Ind. Council for Flour-Milling Ind., Pamphlet No. 3, 56 pp.).

**Separation of the components of frozen milk by fractional melting.** V. V. WINTER (Chem. News, 1929, 138, 321—324).—See B., 1928, 108.

**Adulterants of cacao butter.** BODENDORF.—See XII. **Determination of mixed sugars.** KRUISHEER.—See XVII.

#### PATENTS.

**Composition for bleaching. Bleaching of food-stuffs.** W. B. STODDARD and V. R. KOKATNUR, Assrs. to PILOT LABORATORY, INC. (U.S.P. 1,687,803—5, 16.10.28. Appl., [A] 8.3.27, [B] 7.6.27, [C] 2.9.27. Renewed [A] 29.12.27, [B] 19.12.27).—(A) Acids obtained from natural fats and oils are converted into their mixed chlorides, and the latter, by treatment with hydrogen peroxides and alkali, into acid peroxides, which are used for bleaching foodstuffs, e.g., by incorporation with the material and keeping them at ordinary temperatures. (B) Inactive or relatively inactive organic peroxides are activated by addition of small amounts (1—10%) of active organic peroxides. Suitable bleaching agents are obtained by the action of alkaline hydrogen peroxide at  $-5^{\circ}$  to  $0^{\circ}$  on a mixture of 2 mols. of benzoyl chloride, 1 mol. of fumaryl chloride, and 12.5% of a fatty acid chloride. By incorporation of 5—10% of the "active"

peroxide with the "inactive" peroxide a product may be obtained which is 2—6 times as effective in bleaching power as the "activating" peroxide alone. (C) The material to be bleached is treated with a peroxidised compound, particularly peroxidised food material, which is constitutionally similar to the vehicle of the colouring matter present and physically similar to the colouring matter. When decomposed the peroxidised compound leaves a residue possessing food value.

R. BRIGHTMAN.

**Manufacture of wheaten flour.** R. W. DUNHAM (B.P. 311,034, 5.3.28).—The flour is matured by repeatedly subjecting it to alternate periods of light and comparative heat, and darkness and comparative cold, so as to continue the natural ripening occurring in the field. The material may be passed along under electric lamps heated to a red glow so that its temperature does not exceed  $60^{\circ}$  and preferably is slightly below  $38^{\circ}$ . It is then passed into a dark chamber at  $10-16^{\circ}$ .

W. J. BOYD.

**Treatment of flour and the like.** J. A. HALL and W. F. C. GEORGE (B.P. 310,125, 18.2. and 12.7.28).—In a series of three interconnected tubes or ducts, an improver, to be added to untreated flour, is prepared by subjecting flour, while it is being agitated, to hot steam-laden air whereby it is maintained at  $77-93^{\circ}$  for 5—20 min., then allowing it to rest until the heat is distributed uniformly through it, and finally agitating it in the presence of cool air.

W. J. BOYD.

**Preservation of grain, flour, and bran of cereals.** R. A. LEGENDRE (B.P. 310,980, 5.11.27).—The material is treated in such a manner that the  $pH$  value of its diastase cells is increased to above 7. Preferably at least one salt of a weak acid with a strong base is used, or at least one free base. The alkaline material may be applied (a) in solution by spraying, (b) as a solid by mixing it with the cereal, or (c) as a gas, e.g., ammonia.

W. J. BOYD.

**Production of metal glutins.** K. KÜTTEL, Assrs. to E. J. WATZL and H. J. TRENKAMP (U.S.P. 1,686,281, 2.10.28. Appl., 7.8.25).—A gluten in aqueous solution (20%) is treated at  $28^{\circ}$  with the equivalent amount of alkali hydroxide to produce a saturated alkali gluten, and an excess of an insoluble glutinate-forming salt, e.g., aluminium or zinc sulphate, is then added, the product thus containing the anion of the precipitating salt.

R. BRIGHTMAN.

**Sterilisation of double cream.** S. GRASSE (B.P. 309,932, 18.1.28).—The cream, preheated to about  $37^{\circ}$ , is maintained in a state of gentle agitation and passed under pressure through a continuous and closed conduit in which it is first heated to about  $118^{\circ}$  and then cooled to ordinary temperature. A suitable apparatus is described.

W. J. BOYD.

**Cheeses and their preparation.** PHENIX CHEESE CORP. (B.P. 300,113, 27.2.28. U.S., 6.11.27).—A mixture of cheese and dairy products is melted at  $74^{\circ}$  and emulsified by stirring, with or without addition of an emulsifier such as sodium citrate. The mass is then "viscolised" to impart a smooth texture and fluffy appearance, to blend the ingredients, and to stabilise the emulsion.

W. J. BOYD.

**Manufacture of cheese products.** SKINNER MANUF. Co., Assees. of L. M. SKINNER and J. L. HEID (B.P. 297,025, 24.1.28. U.S., 12.9.27).—To a mixture of flour and powdered cheese water is added to form a dough which is kneaded, pressed into the form of macaroni, and dried in air first at 32° and then at normal temperature.  
H. ROYAL-DAWSON.

**Preparation of vegetable food.** E. H. MILES and G. REILLY (B.P. 310,788, 31.1.28. Addn. to B.P. 256,765. Cf. B.P. 274,051; B., 1928, 425).—The juices of preservative fruits (grapes, apples) are mixed with the juices of fruits less rich in preservative factors (lemons, grape fruit), the mixture is heated at not above 80° until hydrolysis of the sucrose is well advanced, and the whole is concentrated rapidly *in vacuo* at low temperature to a thick syrup. The press-cakes of both classes of fruit may be treated with an acid, preferably an acid fruit juice, to extract further calcium and other salts, and the liquid then expressed and added to the other extracts before heating and concentration of the latter. The insoluble outer part of the orange peel may be added, and the essential oil of lemon rind may be expressed and used as a flavouring agent.  
W. J. BOYD.

**Removal of residual poisons from and the preservation of fruits.** R. H. ROBINSON and H. HARTMAN, Assrs. to U.S.A. (U.S.P. 1,708,330, 9.4.29. Appl., 19.5.27).—The fruit is treated with an aqueous solution of hydrogen chloride and formaldehyde.  
L. A. COLES.

**Removal of substances producing turbidity from pectin solutions [e.g., fruit juice].** POMOSIN-WERKE GES.M.B.H. (B.P. 284,273, 26.1.28. Ger., 26.1.27).—The solutions are cooled until freezing commences, and the precipitate and ice formed are removed by filtration or other means.  
W. J. BOYD.

**Treatment of cannery waste.** J. T. TRAVERS (U.S.P. 1,711,105, 30.4.29. Appl., 15.11.27).—To the waste, rendered alkaline, are added a soluble calcium salt and a coagulating agent containing a primary phosphate of calcium.  
W. J. BOYD.

**Treating and drying fish.** W. H. HASLAM (B.P. 310,765, 26.1.28).—Fish or other animal or vegetable material is dried in an uninterrupted current of air, the temperature rising periodically during the process according to the temperature required to dry the material. The velocity of the air is approx. 1000 ft./min. at 27°, being increased 1000 ft./min. for every rise of 5½°. The temperature may vary from 21° to about 77°. A dry, uncooked, sterilised fish powder can thus be obtained in 12 hrs.  
W. J. BOYD.

**Kneading machines** (B.P. 302,152).—See I. Alcohol from vapours (B.P. 310,400 and 310,403).—See XVIII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Decomposition of acetylsalicylic acid in alkaline solution.** D. B. DOTT (Pharm. J., 1929, 122, 302).—Contrary to the U.S. and British pharmacopœias, no decomposition into acetic and salicylic acids occurs when the drug is dissolved in sodium carbonate or bicarbonate solutions.  
S. I. LEVY.

**Bacterial content of certain medicaments.** J. P. TODD and (MISS) H. M. SMITH (Pharm. J., 1929, 122, 301—302).—A number of chemicals commonly employed in pharmaceutical preparations were examined; all were found to be sterile except glucose and gum arabic.  
S. I. LEVY.

**Evaluation of materials in the production of essential oils.** V. VARENZOV (Trans. Sci. Chem.-pharm. Inst., Moscow, 1928, 203—208; Chem. Zentr., 1928, ii, 2516).—Evaluation of the total surface of fruits in various size-groups gives results in accord with the relative yields of products.  
A. A. ELDRIDGE.

**Separation of citronellal from essential oils.** J. ZIMMERMANN (Pharm. Tijdschr. Ned.-Indie, 1928, 5, 293—295; Chem. Zentr., 1928, ii, 2601—2602).—Separation by sodium hydrogen sulphite is described in detail.  
A. A. ELDRIDGE.

**West Australian sandalwood oil.** I. A. R. PENFOLD (J. Proc. Roy. Soc. N. S. Wales, 1928, 62, 60—71).—An examination of commercial samples of West Australian and East Indian sandalwood oils shows that a considerable difference exists between the alcoholic constituents of each class of oil. Each contains about 70% of alcohols. The West Australian oil contains 40—45% of santalol (allopphanate, m.p. 162—163°) and a mixture of isomeric sesquiterpene alcohols ("fusanols") having b.p. 160—161°/4—5 mm., *d* 0.942—0.943,  $\alpha_D \pm 5^\circ$ , and  $n_D^{20}$  1.5030, together with a small quantity (less than 10%) of a dextrorotatory alcohol. The characteristics of 9 oils are tabulated. Oil from the wood of *Santalum lanceolatum* gave 70% of an alcohol having b.p. 163—165°/5 mm., *d* 0.9474,  $\alpha_D -66.7^\circ$ ,  $n_D^{20}$  1.5074 (allopphanate, m.p. 114°), and a formula approximating to C<sub>15</sub>H<sub>24</sub>O. Australian oils may be distinguished from East Indian oils by the violet-red colour produced when an acetic acid solution of the oil is treated with bromine vapour. A modified potassium permanganate oxidation process which gives increased yields of santalenic acid and freedom from tarry products is described (cf. May, B., 1928, 426).  
E. H. SHARPLES.

**Hungarian essential oils.** M. FÖLSCH (Riechstoffind., 1928, 3, 123—125, 141—142, 162—163, 180—181; Chem. Zentr., 1928, ii, 2295).—The production of the following oils is discussed: acacia blossom, dill, fennel, camomile, coriander, spearmint, and lavender.  
A. A. ELDRIDGE.

**Norwegian juniper oil.** I. A. JERMSTAD (Riechstoffind., 1928, 3, 159—160, 178—179; Chem. Zentr., 1928, ii, 2296).—The juniper oil had *d* 0.8684—0.8775,  $n_D$  1.4729—1.4752,  $\alpha_D$  (200 mm.) +34.05—41.0°, acid value 0.33—0.60, ester value 2.50—8.56, saponif. value 3.0—8.88, acetyl value 20.43—30.40,  $n_D$  of acetylated oil 1.4724—1.4747. Juniper-berry oil had  $d^{15}$  0.860—0.882,  $n_D$  1.479—1.484,  $\alpha_D^{20}$  (100 mm.)  $-1^\circ$  to  $-15^\circ$ , acid value 0—3, ester value 1—8. The juniper oil contained 11% of  $\Delta^1$ -menthen-4-ol.  
A. A. ELDRIDGE.

**Composition of pine-needle oil from Crimean *Pinus halepensis*, Mill.** B. RUTOVSKI and I. VINOGRADOVA (Trans. Sci. Chem.-pharm. Inst., Moscow, 1928, 109—114; Chem. Zentr., 1928, ii, 2516).—The

oil had  $d_{20}^{20}$  0.8960,  $\alpha_D$   $-49.44^\circ$ ,  $n_D^{20}$  1.4940, acid value 1.13, ester value 18.8 (after acetylation, 54.83); it contained  $\alpha$ -pinene (*l*- and *r*-) 10%, camphene 6%, tertiary alcohols 15%, and sesquiterpenes (apparently a mixture of isomeric caryophyllenes) 8%, and a less volatile compound (50%) which had a high optical activity. Borneol and phenylethyl alcohol were absent.

A. A. ELDRIDGE.

**Caucasian Thuja oils.** B. RUTOVSKI and K. GUSSEVA (Trans. Sci. Chem.-pharm. Inst., Moscow, 1928, 123—143; Chem. Zentr., 1928, ii, 2413—2414).—Oil of *Thuja occidentalis* had  $d_{20}^{20}$  0.9203,  $\alpha_D$   $-7.58^\circ$ ,  $n_D^{20}$  1.4605, acid value 1.05, ester value 25.46 (after acetylation 39.43);  $\alpha$ -pinene,  $\alpha$ -thujone, fenchone, and probably borneol and its esters were present. Oil of *Thuja varreana* had  $d_{20}^{20}$  0.9078,  $\alpha_D$   $-1.23^\circ$ ,  $n_D^{20}$  1.4550, acid value 1.5, ester value 16.36 (after acetylation 30.36); sabinene, thujone, and thujyl alcohol were present. Oil of *Thuja gigantea*, var. *semperavrea*, had  $d_{20}^{20}$  0.9145,  $\alpha_D$   $-1.21^\circ$ ,  $n_D^{20}$  1.4552, acid value 2.34, ester value 26 (after acetylation 47.15);  $\alpha$ -pinene, thujone, and thujyl alcohol were detected, the presence of sabinene being assumed.

A. A. ELDRIDGE.

**Essential oils of wild plants of Voronezh Government.** A. TSCHERNUCHIN (Trans. Sci. Chem.-pharm. Inst., Moscow, 1928, 196—202; Chem. Zentr., 1928, ii, 2413).—The following values refer, respectively, to the oils of *Thymus odoratissimus*, *Achillea millefolium*, *Mentha aquatica*, *Acorus calamus*, and *Hyssopus cretaceus*:  $d_{15}^{15}$  0.8682, 0.904, 0.9625, 0.960, 0.9122;  $\alpha_D$   $+15.22^\circ$ ,  $-$ ,  $+28.2^\circ$ ,  $+12.0^\circ$ ,  $-24.29^\circ$ ;  $n$  1.4755,  $-$ , 1.4865, 1.5033, 1.4743; acid value 0, 2.2, 6.8,  $-$ , 4.1; saponif. value 23.0, 26.1, 54.2,  $-$ , 48.2; ester value 23.0, 23.9, 47.4,  $-$ , 44.1; ester value after acetylation 50.2, 81.0, 91.5,  $-$ , 88.0.

A. A. ELDRIDGE.

**Preparation of menthol from Ukrainian peppermint oil.** M. EIDERMAN (Pharm. J. Russia, 1928, 35—37; Chem. Zentr., 1928, ii, 2355).—The fraction of b.p. 88—90°/8—10 mm. contains 80% of menthol, which is purified by cooling, separation from oil, and recrystallisation from petroleum.

A. A. ELDRIDGE.

**Essential oil from the leaves of *Rhus cotinus*.** B. RUTOVSKI and N. PROKOPTSCHUK (Riechstoffind., 1928, 3, 172—173; Chem. Zentr., 1928, ii, 2295—2296).—The leaves of *Rhus cotinus* yielded an oil (fresh 0.07—0.19%, dried leaves 0.134—0.207%) having  $d_{20}^{20}$  0.843,  $\alpha_D$   $+5.25^\circ$ ,  $n_D^{20}$  1.4825, acid value 0.66, ester value 20.1, ester value after acetylation 27.2. Aldehydes and other compounds containing oxygen were not present. The fraction of b.p. 155—184°/13—15 mm. contained  $\alpha$ -pinene (10—11%), camphene (8—9%), and a hydrocarbon (50%),  $C_{10}H_{16}$ ,  $d$  0.8209—0.824, probably myrcene, having three double linkings and polymerising readily.

A. A. ELDRIDGE.

**Essential oil from a *Boronia* in the *pinnata* section, from Frazer Island, Queensland.** A. R. PENFOLD (J. Proc. Roy. Soc. N.S. Wales, 1929, 62, 225—234).—Essential oils obtained by steam-distillation of leaves and terminal branchlets of the following plants are described. *Boronia thujona*, var. "A," from Frazer Island: yield 0.53—0.62%,  $d_{15}^{15}$  1.0563—1.0565,  $\alpha_D^{20}$

$-8.6^\circ$  to  $-11.2^\circ$ ,  $n_D^{20}$  1.5255—1.5260, ester value 6.3—10.8, ester value after acetylation 20.5—22.1, and solubility in 80% alcohol (by wt.) 1 in 5½—9 vols. The principal constituents are safrole (75—80%) and *l*-limonene, with small quantities of phenolic substances, sesquiterpene, and a paraffin, m.p. 65—66°. *B. thujona* (Penfold and Welch): yield 0.5—0.8%,  $d_{15}^{15}$  0.9121—0.9152,  $\alpha_D^{20}$   $+12^\circ$  to  $-56.54^\circ$ ,  $n_D^{20}$  1.4526—1.4543. The oil contains  $\alpha$ - and  $\beta$ -thujone (80—90%), sesquiterpene, and paraffin. *B. Muellieri* (Cheel) yields 0.38—0.57% of oil having  $d_{15}^{15}$  1.0197—1.0265,  $\alpha_D^{20}$   $+1.5$ — $3.8^\circ$ ,  $n_D^{20}$  1.5125—1.5150, and containing elemicin (70—90%), *d*- $\alpha$ -pinene, geraniol, and geranyl acetate. *B. pinnata* (Smith) yields 0.02—0.1% of oil having  $d_{15}^{15}$  0.8784—0.8917,  $\alpha_D^{20}$   $-4.7^\circ$  to  $-15.25^\circ$ ,  $n_D^{20}$  1.4762—1.4825, and containing limonene, *d*- $\alpha$ -pinene, sesquiterpene, and a paraffin.

E. H. SHARPLES.

**Essential oil of a new species of anemone leaf *Boronia* rich in ocimene.** A. R. PENFOLD (J. Proc. Roy. Soc. N.S. Wales, 1929, 62, 263—272).—Steam-distillation of the leaves and terminal branchlets of *Boronia dentigeroides* yielded 1.3—2% of pale, mobile oil with a pleasant characteristic odour. The oil has  $d_{15}^{15}$  0.8421—0.8466,  $\alpha_D^{20}$   $+10.2^\circ$  to  $+16.22^\circ$ ,  $n_D^{20}$  1.4778—1.4793, ester value 14.9—34.2, ester value after acetylation 33.2—88.7, solubility in 80% alcohol (by wt.) 1 in 7.5—10 vols., and contains ocimene (75—80%), *d*- $\alpha$ -pinene (less than 30%), *d*-limonene (total terpenes 90%), darwinol, and the corresponding deoate, isovalerate, and acetate, ethyl formate (?) and isovalerate, together with small quantities of sesquiterpenes, phenolic substances, and a paraffin, m.p. 64—66°. The following values for the oil from *B. anemonifolia* are also given: yield 0.6—1%, ester value 54—128, pinene 75%, and ocimene (trace only).

E. H. SHARPLES.

**Concrete otto of *Boronia metastigma* (Nees).** A. R. PENFOLD (J. Roy. Soc. W. Australia, 1927, 14, 1—5).—Four commercial samples of concrete otto of *B. metastigma* had the following characteristics:  $d_{15}^{15}$  0.8989,  $d_{15}^{15}$  0.9091—0.9189,  $n_D$  1.4752—1.4852, acid value 22.8—30.5, acid value plus ester value 95.9—109.3, ester value after acetylation 136.5—147.8, and m.p. from 35—36° to 40—41°. A large amount of triacontane, m.p. 64°, was present together with a glyceride of palmitic and stearic acids, phytosterols, [ $\alpha_D^{160}$   $+60^\circ$ , m.p. 162°, free octoic and palmitic acids, ethyl alcohol, and ethyl formate, unidentified phenolic substances, and the odoriferous constituents consisting of an unidentified alcohol and probably  $\beta$ -ionone.

E. H. SHARPLES.

**Occurrence of a number of varieties of *Eucalyptus dives* as determined by chemical analyses of the essential oils.** II. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. N.S. Wales, 1928, 62, 72—78; cf. B., 1927, 858).—The following varieties of *E. dives* from Tumberumba, N.S.W., are described: Type, var. "B" (Mannus Hill), var. "B" (School Hill), and var. "C." Essential oils from them (yield 1.62%, 3.1%, 2.32%, 4.2%) have the following respective constants:  $d_{15}^{15}$  0.9099, 0.9077, 0.8904, 0.9214;  $\alpha_D^{20}$   $-42.6^\circ$ ,  $-21.4^\circ$ ,  $-36.8^\circ$ ,  $+4^\circ$ ;  $n_D^{20}$  1.4817, 1.4686, 1.4721, 1.4627; solubility in 70% alcohol

1.4 vols., 1.4 vols., insol. in 10 vols., 1.1 vols.; piperitone content 52%, 8%, 5%, —; cineole —, 17%, 11%, 58% (all by *o*-cresol method); phellandrene about 40%, abundance, abundance, absent; ester value —, 52.2, 12.7, 12.1; ester value after acetylation —, 100.1, 77.1, 58.8. In using the *o*-cresol method, abnormal percentages of cineole were found with oils of the *E. Australiana* type due to the presence of  $\alpha$ -terpineol, and it is suggested that the determination be made on the portion distilling below 190° when applied to oils of this type.

E. H. SHARPLES.

**Determination of cineole in eucalyptus oil, with special reference to the crude oil obtained from *Eucalyptus cneorifolia*.** P. A. BERRY (Australasian J. Pharm., 1929, 203—206).—The *o*-cresol method gives satisfactory results in the determination of cineole in *E. cneorifolia*, whilst the phosphoric acid method gives very inaccurate results due to the solvent action of the aldehydes on the cineole phosphate. A detailed description of the *o*-cresol method, together with f.p. curve and a review of other analytical methods, are given.

E. H. SHARPLES.

**Exudation from the wood of *Pentaspodon Motleyi*.** A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. N.S. Wales, 1929, 62, 218—224).—The dark brown, viscous exudation from *P. Motleyi* occurring in New Guinea has an odour like that of boiled linseed oil and gives the constants:  $d$  1.01—1.011,  $n^{20}$  1.5280—1.5295, acid value 138.24—139.08, saponif. value 142.09—146.64, acid value after acetylation 102.07—102.53, iodine value (Wijs) 192.1, solubility in 70% alcohol (by wt.) 1 in 2.5—2.6 vols. It consists of 90—95% of acid substances and gives a deep violet colour with alcoholic ferric chloride solution. The principal constituent is a dihydroxymonocarboxylic acid,  $C_{24}H_{36}O_4$ , having  $d$  1.0132,  $n^{20}$  1.5270, acid value 145.52, acid value after acetylation 106.5, saponif. value 152.90, saponif. value after acetylation 203.24, iodine value (Wijs) 188.3, and solubility in 70% alcohol 1 in 3.3 vols. The alkaline salts have good emulsifying properties.

E. H. SHARPLES.

**Essential oil of hops.** A. C. CHAPMAN (J. Inst. Brew., 1929, 35, 247—253; cf. B., 1928, 501).—Myrcene, linalool, geraniol, linalyl isononoate, humulene, luparone, luparenol, and luparol were isolated from hop oil by repeated fractional distillation. Of these constituents the last three were isolated from the higher-boiling fractions of the oil, details of which have already been published (*loc. cit.*)

C. RANKEN.

## PATENTS.

**Production of codeine from opium.** J. BYLINKIN (Russ. P. 3381, 15.9.24).—The solution obtained by adding ammoniacal alcohol or a mixture of acetone and alcohol to an aqueous opium extract and removing the precipitated morphine is distilled to remove the alcohol, treated successively with ammonia to precipitate tarry impurities, with calcium hydroxide to remove meconic acid, and with acidified alcohol to remove final traces of tar, after which the alcohol is removed by distillation and the codeine salt recovered by crystallisation.

L. A. COLES.

**Ester of salicylic acid and pyruvic acid [for elimination of uric acid].** S. L. SUMMERS (U.S.P. 1,689,696, 30.10.28. Appl., 30.6.25).—Salicylic acid and 2 mols. of pyruvic acid heated at 160° yield *pyruvylsalicylic acid*,  $C_6H_4(O \cdot CO \cdot COMe) \cdot CO_2H$ .

R. BRIGHTMAN.

**Production of esters of di-iodobenhenolic acid.** A. WINGLER, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,688,100 and 1,688,169, 16.10.28. Appl., [A, B] 11.5.26. Ger., [A, B] 12.5.25).—(A) *isoAmyl di-iodobenhenolate*, m.p. 5—6°, and (B) *isobutyl di-iodobenhenolate*, m.p. 14°, are obtained by heating di-iodobenhenolic acid with excess of the alcohol and 4% of sulphuric acid at 100°.

R. BRIGHTMAN.

**Basic ether of resorcinol.** H. HAHL, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,711,020, 30.4.29. Appl., 13.8.27. Ger., 20.9.26).—See B.P. 300,695; B., 1929, 149.

**Preparation of arylazodiaminopyridines.** A. K. CROAD. From PYRIDIUM CORP. (B.P. 311,349, 10.11.27).—See U.S.P. 1,680,108—1,680,111; B., 1928, 837.

**Metálmercapto-acid esters and their manufacture.** W. SCHOELLER and H. G. ALLARDT, Assrs. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,689,366, 30.10.28. Appl., 16.12.27. Ger., 16.12.26).—See B.P. 282,427; B., 1929, 302.

**Production of unsymmetrical arseno-compounds.** J. PFLEGER and A. ALBERT (U.S.P. 1,688,351, 23.10.28. Appl., 29.11.24. Austr., 12.4.23).—B.P. 249,584; B., 1926, 462.

**Complex antimony compound.** H. HAHL, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,688,964, 23.10.28. Appl., 8.10.26. Ger., 8.6.25).—See B.P. 271,940; B., 1927, 573.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Photographic sensitivity. III. Sensitising action of previous exposure to a dim light. IV. Desensitising action of previous fogging by X-rays.** O. MASAKI (Mem. Coll. Sci., Kyoto, 1929, 12, 107—116, 117—126).—III. Faint spectral lines can be successfully developed if the plate is previously exposed to a dim light. It is now shown that with certain plates the density of the image is greater than would result from the sum of the two exposures, and that the fogging therefore increases the sensitivity. Fogging is more effective when made before the main exposure than when made after it. The sensitising action diminishes with increase in the time between the two exposures. The sensitising action is greatest when fogging is produced by red light, and least by violet light. The effect is more marked in panchromatic than in ordinary plates.

IV. The effect of fogging by X-rays on the sensitivity of commercial photographic plates is examined. A fog impression produced by X-rays is not reversed by light, but the inertia of the plate is increased. The decrease in sensitivity is greater when the fogging precedes the main exposure than when it follows it, and is equal for all wave-lengths. The effect is not modified by lapse

of time between the exposures. The change in sensitivity is greatest for very rapid plates. The decrease in sensitivity produced by X-rays is restored by a second fogging by a dim light, and the increase in sensitivity produced by light-fogging (cf. *supra*) is destroyed by subsequent fogging by X-rays. C. J. SMITHELLS.

## PATENTS.

**Manufacture of photographic silver-salt emulsions.** I. G. FARBENIND. A.-G. (B.P. 283,222, 7.1.28. Ger., 7.1.27).—The active sulphur content of gelatins or other proteins to be used for photographic purposes is increased by treatment during manufacture with traces (1 pt. to 500,000 pts. of protein solution) of colloidal sulphur, inorganic sulphides, particularly alkali and alkaline-earth sulphides, or carbon disulphide.

J. W. GLASSETT.

**Multicolour screens for colour photography.** J. H. CHRISTENSEN (B.P. 309,113, 5.1.28).—Acid dyes are added to a mixture of tannic acid and a basic dye and are thereby strongly held by the insoluble compound formed on drying. Tungstic acid, molybdic acid, and casein may be used to replace tannic acid as the mordant. Suitable acid dyes are brilliant-croceine, sea-blue, patent-blue, erioglaucine, and naphthalene-green.

J. W. GLASSETT.

**Production of photo-prints and photo-copies.** I. G. FARBENIND. A.-G. (B.P. 289,895, 4.5.28. Ger., 6.5.27. Addn. to B.P. 286,233; B., 1928, 466).—In the process of the prior patent (cf. also B.P. 286,736 and 289,386; B., 1928, 549, 944), a salt of a sulphonic acid derivative of 1:2-naphthaquinone or its halogen, hydroxy-, or other substitution product may be used as the light-sensitive material, but 1:2-naphthaquinone-4-sulphonic acid or its substitution products are excepted.

J. W. GLASSETT.

**Production of relief images upon sensitised, continuous film-strips which have been exposed or printed.** J. E. THORNTON (B.P. 303,262, 6.12.27).—The exposed films are passed continuously through a series of tanks or sprays in which the unhardened colloid is first removed by hot water or other suitable solvent, leaving a relief image which is then hardened by a solution of chrome alum, formalin, etc., and finally rendered flexible by immersion in dilute solutions of glycerol or sugar. The last two operations may be carried out simultaneously by combining the solutions.

J. W. GLASSETT.

**Printing or reproducing photographic films bearing colour records.** SOC. CIVILE POUR L'ETUDE DE LA PHOTOGRAPHIE ET DE LA CINÉMATOGRAPHIE EN COULEURS (B.P. 295,313, 26.9.27. Fr., 9.8.27).

## XXII.—EXPLOSIVES; MATCHES.

**Vapour pressure of nitroglycerin and nitroglycol.** A. MARSHALL (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 177). P. NAOUM and K. P. MEYER (*Ibid.*, 177).—The higher values obtained by Naoum and Meyer (B., 1929, 379) as compared with those of Marshall and Peace are explainable by the differences in technique. The former workers determined the amount of nitroglycerin that was evaporated by weighing the condensed nitro-

glycerin, whilst the latter determined it by finding the loss of weight of the mixture of nitroglycerin and guhr when air was drawn over it. The loss of weight would include that due to moisture and volatile matter present in the nitroglycerin. Contrary to Naoum and Meyer's statement, different velocities of air gave the same values for the vapour pressure of nitroglycerin.

Marshall's belief that the higher values obtained by Naoum and Meyer were due to moisture and volatile matter in the materials is unfounded as the nitroglycerin was pure and the guhr was ignited before the determinations were made. His statement that the values for the vapour pressure are independent of the velocity of the air is contrary to the experience of other workers on the determination of vapour pressures by the aspiration method. S. BINNING.

**Determination of moisture in smokeless powder.** W. TIBELL and G. AHLFELDT (Chem.-Ztg., 1929, 53, 356).—Benesch's method (B., 1927, 716) cannot be employed for the determination of powder made with ether-alcohol instead of acetone-alcohol as solvent; in this case the indirect method yields equations for the three unknowns in which the denominator is zero, and no solution is therefore possible. A. R. POWELL.

## PATENTS.

**Preparation of explosives.** H. C. BUGBIRD (U.S.P. 1,706,871, 26.3.29. Appl., 2.5.25. Renewed 7.8.28).—Granular carbonised lignin residues are leached to extract water-soluble mineral substances and then ground to such a degree that the apparent sp. gr. is at least 0.2 and so that the amount of liquid oxygen that can be absorbed is at least 2.6 times the weight of the carbon.

A. R. POWELL.

**Blasting explosive.** L. N. BENT, Assr. to HERCULES POWDER CO. (U.S.P. 1,706,517, 26.3.29. Appl., 26.7.27).—A mixture of 25–80% of ammonium nitrate, 0–25% of sodium nitrate, 10% of nitroglycerol, and 1–18% of wood which has been disintegrated by saturation with high-pressure steam at a high temperature followed by sudden release of the pressure is claimed.

A. R. POWELL.

**Explosive composition.** E. SORENSON (U.S.P. 1,709,498, 16.4.29. Appl., 17.9.27).—A combination of ammonium and potassium nitrates, sulphur, manganese dioxide, paraffin wax, petroleum naphtha, rosin, potato flour, aluminium, and nitroglycerin is claimed.

H. ROYAL-DAWSON.

**Tracer compositions.** S. ST. P. MEEK (U.S.P. 1,708,186—7, 9.4.29. Appl., [A] 21.5.24, [B] 18.1.26).—(A) The composition includes an oxygen-containing compound of an alkaline-earth metal, salts of a rare-earth metal, a fuel, and a binder. (B) The mixture contains magnesium, a metal resinate, a zinc compound, and a suitable oxygen carrier. H. ROYAL-DAWSON.

**[Pyrotechnic] tracer compositions.** H. C. PRITTHAM (U.S.P. 1,708,151, 9.4.29. Appl., 23.5.24).—A non-explosive composition contains salts of thorium and cerium together with a binder.

H. ROYAL-DAWSON.

**Igniting mixture for [pyrotechnic] tracer compositions.** L. HENDLER, Assr. to U.S. SECRETARY OF

WAR (U.S.P. 1,708,174, 9.4.29. Appl., 22.10.23).—The mixture includes an alkaline-earth peroxide, red lead, and a metallic fuel.  
H. ROYAL-DAWSON.

**Manufacture of propellant powders.** A. S. O'NEIL, Assr. to WESTERN CARTRIDGE Co. (U.S.P. 1,709,868—1,709,870, 23.4.29. Appl., [A] 20.6.24, [B, C] 12.10.25).—(A) The characteristics, (B) densities, and (C) compositions of blended grains are chosen so as to give a progressive-burning powder.  
S. BINNING.

**Smokeless powder and its treatment.** W. T. INGRAHAM (U.S.P. 1,710,024, 23.4.29. Appl., 24.7.25).—The powder is gelatinised and grained, the grains after drying being dipped in a solvent for nitrocellulose.  
S. BINNING.

**Diethylene glycol dinitrate and its preparation.** W. H. RINKENBACH, Assr. to W. O. SNEILING (U.S.P. 1,686,344, 2.10.28. Appl., 31.5.27).—Diethylene glycol dinitrate, m.p.  $-11.3^{\circ}$ , is obtained in 60–70% yield by nitrating pure diethylene glycol below  $15^{\circ}$  with mixed acid, sufficient of the ether being added to exhaust completely the nitric acid taken (cf. Rinckenbach, B., 1927, 763).  
R. BRIGHTMAN.

### XXIII.—SANITATION; WATER PURIFICATION.

**Disinfectant action. III. Unsaturated compounds as germicides.** H. D. CHEESEWORTH and E. A. COOPER (J. Physical Chem., 1929, 33, 720–728; cf. Cooper and Mason, A., 1928, 702).—The bactericidal and protozoicidal action of various aliphatic unsaturated compounds and their corresponding saturated derivatives, and of phenols and cyclohexanols have been compared. In general, unsaturated compounds are the more effective germicides; thus, in respect to various bacteria, allyl alcohol, and crotonic, fumaric, and maleic acids are more effective than the corresponding saturated compounds, and the phenols are more effective than the cyclohexanols. Exceptions to this rule, however, are indicated. The same general relation holds for unsaturated and saturated compounds with respect to paramecium. Benzyl alcohol is a much weaker germicide than are the isomeric cresols and even than the methylcyclohexanols, indicating that the presence of a hydroxyl group in a cyclic structure is more favourable to germicidal power than its presence in a side-chain. Fumaric acid shows a stronger action than maleic acid, an indication that configuration is a more important factor than ionisation. The precipitating power of hydroxy-compounds runs parallel with germicidal action, the unsaturated compounds having a greater effect on egg-albumin and lecitho-protein than the corresponding saturated compounds; it is suggested that greater germicidal power is connected with an increased precipitating or denaturing power of colloidal suspensions.  
L. S. THEOBALD.

#### PATENTS.

**Extraction of halogens and precious metals from sea-water.** H. BARDT (B.P. 294,655, 26.7.28. Ger., 29.7.27).—Iodine, bromine, and precious metals may be recovered from the precipitate obtained by treating sea-water simultaneously or successively with a

reducing agent, e.g., sulphur dioxide, and a mixture of finely-divided, activated carbon and a powdered metal, e.g., copper.  
C. JEPSON.

**Apparatus for purifying waste water with quickened sludge.** M. KUSCH (B.P. 310,749, 2.11.28).—In this modification of the activated-sludge process the floor of the aeration tank consists of a series of inverted pyramids, and the circulation and aeration of the contents are effected by means of air-lifts reaching from the bottom of each funnel to a point just below the surface and having an outlet intended to impart an undulating motion to the discharge. The air is applied through a series of small holes from an annular chamber encircling the pipe.  
C. JEPSON.

**Sterilisation of water and other liquids.** G. A. KRAUSE (B.P. 279,085, 12.10.27. Ger., 14.10.26).—A vessel, lined with or made of any "oligo-dynamic" metal, e.g., silver, copper, bismuth, antimony, manganese, nickel, aluminium, or their alloys, is so filled with pieces of such metal that the liquid to be sterilised is brought into intimate contact with the maximum amount of metallic surface, thus causing an increased rate of sterilisation. The spaces between the pieces of metal used for filling are made too large to retain micro-organisms by mechanical filtration.  
C. JEPSON.

**Purification of industrial [wool] washing water and the like.** C. VAN OVERSTRAETEN (B.P. 300,386, 5.11.27).—To mud and suspended fatty matter, separated or decanted from the water from wool washing etc., is added the precipitate formed by treating the separated water with acid, bleaching powder, or aluminium sulphate. The magma is washed with the least possible amount of water, with or without alkali, and is passed through a mud separator. The mud is discarded after washing again, if necessary, and all the wash waters from the muds are boiled, with or without alkali, and treated to separate the neutral grease. The water from the hydro-extractor etc. is used again as washing agent for the magma. (Cf. B.P. 275,627; B., 1928, 636.)  
W. G. CAREY.

**Antiseptic, antifouling, germicidal, waterproof coatings.** A. BIDDLE, Assr. to UNITED PRODUCTS CORP. OF AMERICA (U.S.P. 1,689,008, 23.10.28. Appl., 3.7.26).—A water-repelling material, e.g., rubber latex, is incorporated with poisons and/or antiseptic or growth-preventive materials. Thus a sanitary paint is obtained by mixing 10 pts. of calcium rosolate and/or hydroxide with 10 pts. of water and 2 pts. of latex. Methods are given for preparing a plant spray and an antifouling composition. Vulcanising and accelerating agents may also be incorporated.  
R. BRIGHTMAN.

**Vermin-killer.** F. FLURY, Assr. to DEUTS. GES. F. SCHÄDLINGSBEKÄMPFUNG M.B.H. (U.S.P. 1,712,917, 14.5.29. Appl., 29.8.21. Ger., 8.4.20).—See B.P. 196,524; B., 1923, 796 A.

**[Pendant] apparatus for filtering and treating air [of rooms].** W. JENKS (B.P. 310,351, 24.1.28).

**Protective gas-masks, respirators, etc.** E. DRÄGER (B.P. 292,531, 4.5.28. Ger., 21.6.27).