

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 6 and 13, 1930.*

I.—GENERAL; PLANT, MACHINERY.

Trend in design and operation of industrial plants, with special reference to furnace volume.

H. KREISINGER (Proc. Eng. Soc. W.Pa., 1929, 45, 426—440).—A definite volume of combustion space is necessary in all furnaces to allow of almost complete combustion before the furnace gases enter the boiler. Any means to increase the rate of contact between the fuel and oxygen would permit the combustion space to be decreased, but limiting factors occur as, *e.g.*, the low heat value of blast-furnace gas and also when increased turbulence causes fuel ash to erode the combustion walls excessively. In water-cooled furnaces the limits are the accumulation of slag on the boiler tubes and the losses due to incomplete combustion. The rate of heat liberation in B.Th.U. per cub. ft. of combustion space per hr. for normal range of operation is given for various fuels: blast-furnace gas 7000—15,000, natural gas and fuel oil 10,000—30,000, powdered coal (low-fusion ash) 6000—14,000.

C. A. KING.

Pneumatic transport of granular and powdered materials. H. NEU (Bull. Soc. Ing. Civ. France, 1930, 82, 987—1041).—A pneumatic method of transporting materials requires a pressure difference of 100—450 mm. Hg and an air supply of 30—500 litres/kg. of material, according to circumstances. The calculation of an installation, which cannot be performed by theory alone, permits of two solutions: the minimum capacity and the most economical capacity. Various methods of feed and discharge are described together with types of pumps for the production of pressure or vacuum. Pneumatic transporters can deliver up to 300 tons per hr.; they are flexible and economical in labour, but they have a higher power consumption than other types. They are suitable for grain of all kinds, coal dust, cement, ammonium sulphate, etc. Portable transporters for discharging ships are described. Analysed costs of five transporters handling coal and grain are given. This method of handling has the convenience of taking up little room, and the crossing of obstacles such as railway lines presents no difficulty.

C. IRWIN.

High-pressure chemical engineering equipment of the Chemical Research Laboratory, Teddington.

H. TONGUE (Inst. Chem. Eng., Apr., 1930. Advance proof. 14 pp.).—A description of the apparatus at this laboratory, which includes low-pressure gas-storage, compression plant, a high-pressure gas-storage and distribution system, high-pressure autoclaves, and other apparatus for catalytic reactions.

C. A. KING.

Humidity chart for use with combustion gas [dryers].

S. HATTA (Chem. Met. Eng., 1930, 37,

165—166).—Humidity charts for air dryers cannot be used without error when the actual drying agent is flue gas. The author's chart is constructed on the assumption that the sp. heats of oxygen, nitrogen, and carbon monoxide are substantially equal. Humidity is expressed in lb.-mols. of steam per lb.-mol. of dry gas, and separate cooling lines are given for 0, 10, and 20% CO₂ content. In addition to the usual lines, a series of humid-volume lines are given for varying humidities. An example of the use of the chart is given.

C. IRWIN.

Use of thermal data in drying-tower design.

A. B. NEWMAN (Chem. Met. Eng., 1930, 37, 145—147).—The case is considered of a sulphuric acid drying tower without cooling coils, in which the whole of the heat of dilution of the acid is carried away in the effluent. Curves are given showing the variation of the sp. heat of sulphuric acid with concentration and the heat of dilution at various concentrations. By their aid the temperature of the effluent acid can be calculated, and from the water-vapour pressure of sulphuric acid solutions the necessary feed in any particular case can be deduced.

C. IRWIN.

Water-softening. JENKS.—See XXIII.

PATENTS.

[Open-hearth] furnace. M. C. STEESE (U.S.P. 1,737,392, 26.11.29. Appl., 13.12.20).—At each end of the furnace is a single uptake, used (at the firing end) for preheated air alone. Below an ordinary air port (leading to the furnace) is another containing a burner through which fluid fuel is injected under pressure. A preliminary combustion takes place in the port and is completed in the furnace with air from the upper port. The burner induces air proportional to the fuel supply, and the amount of air passing on to the upper port is determined by the total air allowed to enter the regenerator; the valve for this may be regulated (through electrical relays) by the pressure existing in the uptake.

B. M. VENABLES.

Pulverised-fuel furnaces. ASH CO. (LONDON), LTD. (B.P. 311,815, 26.4.29. U.S., 17.5.28).—The combustion space has its walls water-cooled to such an extent that a substantial amount of heat is extracted from the ash without causing it to solidify. The molten ash drops to a hopper-shaped zone, where it is cooled by jets of water and removed periodically through the bottom to a sluiceway.

B. M. VENABLES.

Coolers for rotary furnaces. N. YOUNG (B.P. 327,017, 8.4.29).—The product from a rotary roasting or clinkering furnace drops into a trough-like housing surrounding the kiln, and is propelled along it by inclined

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

blades attached to the outer surface of the kiln. Air for cooling may be passed through passages formed in the trough, and a water spray may be used for "hydrating" the clinker. B. M. VENABLES.

Apparatus for heat-treating articles. C. E. BUYSSE, Assr. to INDUSTRIAL HEATING EQUIPMENT CO. (U.S.P. 1,744,949, 28.1.30. Appl., 21.12.27).—The articles are placed in cylindrical containers and the rollers thus formed run down a track (hairpin-shaped in vertical section), through an ante-chamber into a heating chamber, and return from the latter chamber through the lower part of the former. While in the ante-chamber the ingoing goods are heated by waste gases above and by radiation from the outgoing goods below. B. M. VENABLES.

Fluid heaters. BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX Co. (B.P. 326,470, 11.12.28).—A method of providing joints in U-tubes such as are used in economisers is claimed. Lugs are formed on the abutting ends of the tubes, loose flanges with corresponding recessed holes are slipped over the lugs in the manner of a bayonet joint, and the flanges are bolted together. B. M. VENABLES.

Heat-exchange apparatus. R. SAMESREUTHER and G. KRÄNZLEIN (B.P. 305,172, 29.1.29. Ger., 1.2.28).—A method of attaching a number of closely spaced tubes to a header entirely by means of welding is described. The ends of the tubes are flanged out and the adjacent edges of the flanges are welded together, thus forming one wall of the header. B. M. VENABLES.

Headers for heat-exchanging apparatus. SKODA WORKS (B.P. 310,819, 30.4.29. Czechoslov., 30.4.28).—A header, which may conveniently be manufactured as a steel casting, is formed with an inwardly arched end-cover, with or without stiffening ribs, and with outwardly arched side-walls with stiffening ribs of undulatory form. Preferably the thickness of the ribs and other parts is the same throughout, but in any case no portion is to exceed $2\frac{1}{2}$ times the thickness of any other. B. M. VENABLES.

Heat-exchange apparatus. W. H. OWEN (B.P. 326,844, 21.12.28).—Methods of securing a cellular-type air heater in a conduit for flue gases are claimed. B. M. VENABLES.

Plate heat-exchange apparatus employing condensable gas or fluid. R. SELIGMAN (B.P. 327,377, 6.3.29. Holl., 7.3.28).—On each side of the heat-transferring plate passages for the respective fluids are formed by ribs in conjunction with plain or similarly ribbed closing plates. The passages may be zig-zag or give controlled parallel flow from and to cross-passages at the top and bottom; the latter arrangement is suitable for clearing condensate rapidly. B. M. VENABLES.

Device for effecting heat interchange. [Immer-sion heater.] W. S. BOWEN (U.S.P. 1,745,204, 28.1.30. Appl., 11.4.24).—In the steam generator of the type where combustion of gaseous fuel takes place in a conduit which is jacketed by the steam and water space, a sleeve and baffle are provided in the latter to promote circulation and prevent priming; also a spiral baffle of refractory material is placed in the combustion conduit,

thereby improving the heat transmission both by scouring away the film and by radiating heat through the film. B. M. VENABLES.

Power plants and apparatus therefor. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTROTRITATS-GES. (B.P. 315,274, 9.7.29. Ger., 10.7.28).—In a power station in which the feed-water heaters for the whole station form the condensers of the "house" turbines, under some circumstances there may not be enough cool feed-water to condense the exhaust from the house turbines. On the principle that it is wiser to waste heat rather than distilled water (by exhausting to atmosphere), some of the hot feed is cooled by running it back through a main condenser, which may be either that belonging to a turbine which has unexpectedly had its load reduced, or a stand-by. There is only one valve to regulate, viz., the water by-pass. B. M. VENABLES.

Crusher. W. A. BATTEY and C. A. JAMISON, Assrs. to PENNSYLVANIA CRUSHER CO. (U.S.P. 1,743,996, 14.1.30. Appl., 18.10.27).—A form of frame for a heavy-duty single-roll crusher is described. B. M. VENABLES.

Pulveriser. R. SINCLAIR, Assr. to UNITED COMBUSTION ENGINEERS, INC. (U.S.P. 1,744,927, 28.1.30. Appl., 19.6.28).—A series of pulverising zones is arranged longitudinally of the apparatus and without dividing walls. The interior of the casing is fluted except opposite the last beaters where, being smooth and volute shaped, that zone acts to a certain extent as a fan; there is, however, an additional fan drawing external air and delivering it into the outlet conduit at such an angle that it induces flow through the pulveriser. Arrangements are made at the feed end to trap, by inertia, any very heavy particles, the falling coal or other material being diverted sharply into the pulveriser by jets of high-pressure air. B. M. VENABLES.

Pulverising machine. P. A. HIRSCH (U.S.P. 1,744,895, 28.1.30. Appl., 15.11.28).—In a disintegrator suitable for powdered fuel the hammers work in annular zones spaced longitudinally of the casing and alternating with zones containing fixed vanes which tend to throw large material back into the pulverising zone from which it came. Beyond the last pulveriser is a double fan one side of which draws only just enough air through the machine to transport the fine powder, but the other side draws external air and dilutes the stream for transport to a distant point. B. M. VENABLES.

Pulverising and like mills. E. W. GREEN and G. R. UNTHANK (B.P. 326,662, 14.3. and 4.12.29).—The mill comprises a number of balls rotating between two tracks; the lower is rotated but unyielding, the upper yieldingly supported, but otherwise stationary. The ground material flows by centrifugal force over an upwardly and outwardly sloping lip on the lower race, and enters a rising annular stream of air which conveys the whole of the material to an upper separating zone from which the oversize drops back to be re-ground. The annular stream comprises practically all the air passing through the apparatus. B. M. VENABLES.

Apparatus for determining the volume and density of granular materials passing through it. A. HANIQUE (B.P. 326,583, 29.12.28).—The apparatus

comprises a measuring device, such as a rotating drum divided into four buckets by radial partitions, above which is a distributing hopper and above that a feed hopper with automatic-discharge valve. The drum and the distributing hopper are counterpoised and they drop together only when there is a certain weight of material present in the two combined; their downward motion cuts off the supply from the feed hopper and permits the drum to rotate one step and discharge itself with consequent return to the charging position. Since the drum delivers a definite volume every time, but only operates when there is a definite weight present, the excess material left in the hopper is a measure of the density of the material and can be read off on a scale down the side. A check may be provided by weighing the material into the charging hopper and dividing by the number of cycles. B. M. VENABLES.

Mixing machines. V. BOOTH (B.P. 326,969, 25.2.29).—The apparatus comprises a rotary drum with an axial conveyor which serves to charge the material, to withdraw partly mixed material and recharge it with a further quantity of original material, also to discharge the finished material. The conveyor extends beyond the drum at both ends, a hopper for original material being at one end and a final discharge at the other. The top run carries the material from the hopper in one direction, the bottom run scrapes it along a trough in the other. Buckets are provided on the interior surface of the drum which lift and drop the material into the trough. Sliding doors in the bottom of the trough determine the point of delivery.

Mixing machines. A. WALLACE and W. M. WALLACE, JUN. (B.P. 327,000, 21.3.29).—A mixer for plastic or fluid materials comprises a horizontal cylindrical chamber with filling and emptying apertures, and a stirrer with axial shaft and radial beater blades and spokes; the latter carry scroll-like scraper elements which sweep the entire internal surface as closely as possible. At least one end of the casing is removable and the stirrer is subdivisible. B. M. VENABLES.

Converting suspensions of solids into pieces [coherent masses]. L. B. GUNDERSEN (B.P. 326,999, 19.3.29).—The fluid is poured into porous moulds and the water removed by currents of air across the moulds, under pressure on one side and vacuum on the other.

B. M. VENABLES.

Magnetic separation of materials. F. KRUPP GRÜSONWERK A.-G. (B.P. 303,513, 1.1.29. Ger., 5.1.28).—A magnetic separator of the type in which a drum rotates between two poles is arranged so that the magnetic material which adheres to the drum in the first air-gap is passed through the second and is there cleaned. If additional output rather than a very clean product is desired, some additional raw material may be supplied to the second separating zone.

B. M. VENABLES.

Art of separating [discrete particles] and apparatus therefor. F. R. JOHNSON (U.S.P. 1,744,967, 28.1.30. Appl., 25.8.27).—A mixture of discrete particles having differing physical properties is fed to an intermediate point on an inclined belt which runs upwards, at such angle and speed that some particles will

roll down and others will be carried up. The actual separation may be effected by their shape or the friction of their surfaces, "by an underlying solid body obstructing the direct pull of gravity," or by electric charges. In the last case the feed chute may be electrified to one polarity and a mass underneath the belt to opposite polarity; further, by the use of another charged mass at the lower delivery point, a separation may be made there, giving three products in all. B. M. VENABLES.

Apparatus for regulating, varying, or controlling the flow of solid, gaseous, or liquid media. H. C. HEAD (B.P. 326,546, 15.12.28).—A squirrel cage is formed of overlapping longitudinal slats and a pair of end rings; the joints are left loose, the cage is inserted in a conduit, and the end rings are rotated with respect to each other. The slats then form a false wall to the conduit in the form of a Venturi throat. The rotation of a ring may be effected by a Bowden wire, and should it be desired to put the fluid in rotation the slats may be provided with radial projections. B. M. VENABLES.

Centrifugal machines. SHARPLES SPECIALTY CO., and L. D. JONES (B.P. 327,079, 11.7.29).—The apparatus is suitable for the separation of solids and liquids by straining. The rotor is constructed of a number of rings separated by circumferential drainage slits, the interior is conical, either smooth or stepped, and the solid matter is worked towards the small end by means such as helical blades rotating at a suitably different speed; this inner rotor also carries thin ploughs which extend into the slits and keep them clear. Since the solid matter moves in the opposite direction to the freely flowing pulp, which naturally moves to the large end, the product is drier. B. M. VENABLES.

Centrifugal machine. T. A. BRYSON, ASSR. to TOLHURST MACHINE WORKS, INC. (U.S.P. 1,743,753, 14.1.30. Appl., 8.7.21).—A self-balancing centrifuge is constructed of the following parts, in order downwards: a motor with centrifugal clutch; a driving spider having freedom to move radially and longitudinally; a spherical bearing permitting longitudinal and angular, but not radial, movement and a brake drum at the same height; then, some distance down, the basket; and finally a double spherical thrust-bearing permitting the shaft to gyrate. B. M. VENABLES.

Removal of vaporisable constituents from liquid, pasty, or solid matters. J. BRABAEK (B.P. 326,829, 20.11.28).—The material is spread in a thin layer on a rotating drum and subjected to currents of air or other drying medium which arrive radially, flow a short distance circumferentially under increased pressure due to the convergence of the nozzles, and then depart radially under reduced pressure. The inlets and outlets are formed from the assemblage of a number of ribbed plates and are long and narrow, a large number of alternate ones being provided to cover a considerable arc of the drum. The condition for maximum efficiency is that the width of the inlet nozzles should be greater than twice the radial distance between the nozzles and material on the drum, but less than the width of the outlet nozzles; the circumferential distance between nozzles should also be greater than twice the above-mentioned radial distance. B. M. VENABLES.

Evaporators. H. WADE. From CHEM. FABR. J. BELLAK (B.P. 326,510, 14.12.28).—In an evaporator with steam-heated tubes to which the liquid is supplied as a drizzle, the heating surface of the horizontal rows of tubes decreases in succession downwards. Preferably they are arranged in groups having equal numbers of tubes in the horizontal rows of any one group, and below each group are collectors from which the liquid drips in fewer rows on to the tubes of the next group. Splashing is not relied on to distribute the liquid and the tubes are not staggered.

B. M. VENABLES.

Combined purifying and distilling apparatus. K. E. BUETTNER (U.S.P. 1,744,747, 28.1.30. Appl., 13.3.28).—A still suitable for the purification of dry-cleaning solvents, by means of caustic soda and decolorising carbon or other reagents, is constructed with a conical lower part for settling purposes; this part is provided with a steam jacket and with a live-steam injection pipe, together with means for admitting the dirty liquid and drawing off sludge. The distilling chamber above is provided with a steam-coil and with means for sampling and drawing off clean, settled liquid and for adding reagent. There is an overflow at the top of the distilling chamber and another near the top of a continuing stand-pipe, also a vent at the extreme top of the latter. The still is used either continuously or for batches in conjunction with a heat-exchanging condenser, moisture remover, and pump.

B. M. VENABLES.

Apparatus for distilling liquids [especially crude glycerin]. W. E. SANGER, Assr. to O. H. WURSTER (U.S.P. 1,743,289, 14.1.30. Appl., 23.7.23).—A distilling system substantially as described in U.S.P. 1,452,617 (B., 1923, 536 A) may be used for the distillation of glycerin of either dynamite or pharmaceutical quality. The concentrator is provided with a superheating coil in its upper part, which gives a preliminary superheat to the mixed vapours from "sweet water" in the evaporator and from the glycerin in the concentrator prior to passing through the superheater-condenser and thence being injected into the bottom of the still. The efficiency of the vapours is increased and the amount of heat supplied to the still (closed coil and/or external) is reduced, but the amount of glycerin condensed in the superheater-condenser, hence its grade, is controlled by the amount of superheating effected in the top coils of the concentrator. The above method of operation is used for glycerin of dynamite quality; where a pharmaceutical product is desired, pure water is boiled in the evaporator and the concentrator is not simultaneously used as such, only its superheating coil being heated. Roses for injected steam are provided in the concentrator and evaporator for a preliminary removal of impurities. To allow for the difference in vacuum between the receivers and the concentrator and evaporator, the latter are at a lower level and the transfer pipes may be provided with siphons and non-return valves. For cleaning the main still a water spray is arranged above, and a steam or air spray below, the heating coil.

B. M. VENABLES.

Stuffing-box for pressure stills. F. E. WELLMAN, Assr. to KANSAS CITY GASOLINE CO. (U.S.P. 1,745,200,

28.1.30. Appl., 5.6.22. Renewed 11.7.29).—The packing is divided into two portions by a hollow spacer, the interior of the spacer being put into communication with the interior of the still through a condenser, so that any leakage that does take place is of liquid.

B. M. VENABLES.

Combination bubble cap and downflow. V. F. GRACE (U.S.P. 1,744,543, 21.1.30. Appl., 3.10.27).—The cap is H-shaped in vertical section and circular in plan, and a long downflow pipe is taken through the web; the lower flange surrounds a short rising pipe for vapour and the upper flange determines the depth of liquid in the tray. The edges of either or each flange may be serrated.

B. M. VENABLES.

Apparatus for separating dust from flowing gases. WASSMUTH, KURTH, & Co., A.-G., Assees. of A. WASSMUTH, GES.M.B.H. & Co. (B.P. 308,178, 18.3.29. Ger., 17.3.28).—A chamber is divided into two by a partition extending nearly to the bottom, and is provided with hopper-like outlets for dust. The gas is admitted downwards through a spreading device in the top of one compartment, and the clean gas exhausted through a similar device in the top of the other compartment.

B. M. VENABLES.

Removal of dust from gases, and more particularly producer gas, a reheating device, and a steam-mixing device for the combustion air. "MATECO" SOC. POUR LA CONSTRUCTION ET L'EXPLOIT. DU MATÉRIEL COLONIAL AU GAZ PAUVRE, SOC. ANON., Assees. of M. DELVAUX (B.P. 318,965, 29.8.29. Belg., 13.9.28).—The dust-laden gases are passed through a conduit bent to circular shape, so that the dust is concentrated into the outer layer, which travels into a large expansion chamber where the dust settles, and whence the portion of gas is withdrawn and caused to rejoin the main stream. Provision for preheating the air and adding steam to it is made in a conduit surrounding the cleaner.

B. M. VENABLES.

Filtering apparatus for air and other gases. S. DUNLOP. From W. BARTEL GES.M.B.H. (B.P. 327,083, 13.8.29).—In an apparatus comprising a number of filter bands which run one behind the other, the bands are driven at different speeds but preferably by a common mechanism. They may pass over rollers or the like, which are provided with peripheral openings for the emission of steam, air, water, or other cleansing fluid.

B. M. VENABLES.

[Heat exchanger for] liquefaction apparatus. J. W. DAVIS, Assr. to S. G. ALLEN (U.S.P. 1,744,108, 21.1.30. Appl., 3.7.24).—A heat exchanger suitable for separating undesired constituents from a gas by liquefaction, and comprising a bundle of twin concentric tubes, has the outer tubes threaded through and firmly fixed to a number of metallic plates which conduct heat transversely from tube to tube and equalise the temperature. The whole unit may be placed in a casing and surrounded by insulating material.

B. M. VENABLES.

Condensation of vapours from air and gases by cooling. M. FRÄNKEL (B.P. 308,228, 19.3.29. Ger., 19.3.28. Addn. to B.P. 294,354; B., 1928, 697).—Two

reversible, regenerative, cold-storage devices and a direct-contact cooler containing grids sprayed with chilled brine or the like are operated on the cycle described in the original patent to remove moisture from air. If non-return valves are used in the conduits the change-over may be effected by only one reversing valve. B. M. VENABLES.

Separation of mixed gaseous components. E. MAZZA (B.P. 303,124, 28.12.28. It., 28.12.27).—The apparatus is intended to stratify the gaseous constituents by means of centrifugal force. B. M. VENABLES.

Gas-analysing apparatus. E. PICK, Assr. to PERMUTIT Co. (U.S.P. 1,744,415, 21.1.30. Appl., 1.8.27. Renewed 24.9.29).—To protect the analyser proper, which may have metallic parts, the sample of gas is first passed through a compact mass of steel wool preceded, if desired, by a preliminary strainer. B. M. VENABLES.

Carrying out of catalytic reactions. SELDEN Co., Assees. of A. O. JAEGER (B.P. 310,956, 6.4.29. U.S., 4.5.28).—In the catalytic air-oxidation of naphthalene to phthalic anhydride and in other exothermic vapour-phase catalytic reactions, the metal bath regulating the temperature forms a closed system in which actual boiling of the metal at the upper end of the reaction vessel (i.e., where fresh gases meet the catalyst) is prevented by the hydrostatic pressure of the metal in the higher portions of the system. The metal rises through tubes in contact with the catalyst, overflows at the top into a drum forming part of the closed system, and descends through an outer limb which is cooled so as to maintain a difference of about 100° between the bottom and top of the catalyst chamber. The drum contains an air space which may be kept at any desired increased or reduced pressure. Circulation is automatic and is accelerated, whenever incipient boiling takes place, by the "geyser" action of the bubbles of vapour. A suitable apparatus is described. C. HOLLINS.

Apparatus for carrying out endothermic catalytic reactions. J. Y. JOHNSON. From I. G. FARBEININD. A.-G. (B.P. 327,025, 15.4.29).—The catalytic tubes or chambers are heated entirely or mainly by radiation; their outer walls and the inner walls of the surrounding heating chamber may be lined with material of high emissive power. B. M. VENABLES.

Catalysing apparatus. SOC. ANON. D'OUGRÉE MARIHAYE (B.P. 327,378, 19.3.29. Belg., 7.4.28).—The catalysing agent (or its support) is shaped in the form of a surface of revolution of a catenary, the axis of revolution coinciding with the direction of admission of the gas to be treated. The containing chamber is of similar shape and the gas is led away through holes in the wall. B. M. VENABLES.

Manufacture of catalysts of high mechanical stability. J. Y. JOHNSON. From I. G. FARBEININD. A.-G. (B.P. 326,580, 24.12.28).—Finely-divided metals of groups II or III, or alloys or carbides thereof, are incorporated with the constituents of non-metallic catalysts by intensive mechanical intermixing at below 50°. *E.g.*, a catalyst for the destructive hydrogenation of hydrocarbon oils is prepared by the addition of

1–3% of aluminium or aluminium-bronze powder to a mixture of 65% MoO₃ and 35% ZnO; after thorough grinding below 50° until the metal has completely reacted with the other constituents the product dries to hard, stable granules which do not disintegrate during use. A. R. POWELL.

Manufacture of catalysts containing free phosphoric acid. J. Y. JOHNSON. From I. G. FARBEININD. A.-G. (B.P. 326,185, 27.10.28).—The catalysts, which have prolonged activity, comprise a uniform dehydrated mixture of 10 pts. of a phosphate of an alkali or alkaline-earth metal, or a mixture of the two, up to 1 pt. of free phosphoric acid (or an equivalent amount of a substance, like ammonium phosphate, which produces phosphoric acid when heated), and, if desired, a carrier (graphite, pumice), with optional addition of a pyrogenic substance (*e.g.*, oxalic acid) which decomposes into gaseous products during the dehydration or practical application. The phosphate and acid may be dissolved and crystallised out after any of the optional additions. The catalysts are adapted for use in dehydration reactions, *e.g.*, the production of butadiene from $\alpha\gamma$ -butylene glycol, for acetal splitting, for the formation of ethylene from ethyl alcohol, etc. S. K. TWEEDY.

[Carriers for] catalysts. COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (B.P. 304,654, 4.1.29. Fr., 23.1.28).—Catalyst carriers comprise material prepared by baking a mixture of refractory material, a binder, and organic material removed during the baking; *e.g.*, a mixture of dried sawdust, kaolin, water, Mexican mazout, and coal tar moulded by extrusion into hollow cylinders is baked at 1200–1400°, first in a reducing and later in an oxidising atmosphere. The product is impregnated with the desired catalyst in the usual manner. L. A. COLES.

Preparation for removing or preventing boiler scale and other like deposits. H. KÖPPLINGER (U.S.P. 1,754,171, 8.4.30. Appl., 10.2.26. Ger., 4.1.26).—See B.P. 262,823; B., 1928, 110.

[Crushing or grinding] mill. A. STEINBRÜCKNER (U.S.P. 1,752,860, 1.4.30. Appl., 30.11.27. Ger., 8.12.26).—See B.P. 301,667; B., 1929, 154.

Grinding, pulverising, or disintegrating mill. W. A. CLOUD (U.S.P. 1,752,888, 1.4.30. Appl., 14.6.26. U.K., 20.7.25).—See B.P. 260,034; B., 1927, 31.

Discharge means for tube or ball mills. P. BODENSTEIN, Assr. to F. KRUPP GRUSONWERK A.-G. (U.S.P. 1,753,685, 8.4.30. Appl., 18.6.28. Ger., 27.6.27).—See B.P. 292,941; B., 1929, 78.

Filtering and decanting apparatus. C. PICCARDO (U.S.P. 1,753,577, 8.4.30. Appl., 21.8.28. Ger., 13.2.28).—See B.P. 305,976; B., 1929, 499.

Apparatus for separating liquids of different densities. F. PINK (U.S.P. 1,754,119, 8.4.30. Appl., 29.3.28. U.K., 27.1.28).—See B.P. 298,683; B., 1929, 3.

Recovery of adsorbable substances [from gaseous mixtures]. A. B. RAY and G. T. FELBECK, Assrs. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,753,067, 1.4.30. Appl., 26.5.26).—See B.P. 291,277; B., 1928, 508.

Apparatus for separating dust from gases. A. STEVENART (U.S.P. 1,754,126, 8.4.30. Appl., 28.1.28. Belg., 4.2.27).—See B.P. 284,980; B., 1928, 432.

Gas washer. M. MALLET, ASST. to NEWTON CHAMBERS & Co., LTD. (U.S.P. 1,743,974, 14.1.30. Appl., 9.4.28. Fr., 27.9.27).—See B.P. 297,765; B., 1929, 4.

[Mechanical stoker for] furnaces. A. J. M. A. R. VAN DER DOES DE BIJE (B.P. 326,443, 11.10.28).

Closing shutters for tunnel ovens. EESTI PATENDI AKTSIASELTS (B.P. 304,238, 15.1.29. Estonia, 17.1.28).

Continuously-acting absorption refrigerating machines. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 303,504, 11.12.28. Ger., 5.1.28).

[Mounting of capillary tubes of] thermometers. H. G. A. NEWMAN, and ACCURATE RECORDING INSTRUMENT Co., LTD. (B.P. 327,205, 7.1.29).

Fractional distillation (U.S.P. 1,744,261). Mixing devices (B.P. 327,339).—See II. Fire-extinguishing and -proofing compositions (B.P. 302,172 and 326,253).—See III. Cooling brine (B.P. 327,039). Water-softening material (U.S.P. 1,744,703).—See VII. Treating solids with liquids (B.P. 327,146).—See XV.

II.—FUEL; GAS; TAR; MINERAL OILS.

Primary thermal decomposition of coal. R. HOLROYD and R. V. WHEELER (Fuel, 1930, 9, 40—51, 76—93, 104—114. Cf. B., 1928, 880).—The distillation under low pressure of some bituminous coals and coal constituents has been studied; a very slow rate of heating was adopted and the temperature, which was raised in stages, was maintained constant at each stage for 120 hrs. The heavy oils, light oils (and water), and gases evolved at each stage were separately collected. No extensive decomposition of any coal constituent occurred below 300°. Occluded gases were driven off up to 150°, and slight changes in the external groupings of the ulmin compounds occurred, particularly with weathered coals, resulting in the evolution of water and oxides of carbon. Between 220° and 300° the major part of the free hydrocarbons in the coal (γ_1 -compounds) distilled unchanged. At 300°, or just above, any plant entities which the coal contained began to decompose, giving unsaturated hydrocarbons, neutral oxygenated compounds, and water. Above 300°, marked decomposition of the ulmin compounds occurred, the products being gaseous paraffins, water, phenolic oils, and liquid aromatic and hydroaromatic compounds. Above this "active decomposition point" of the coal (cf. B., 1929, 116) the ulmin compounds, organised plant entities, and resins decomposed and distilled simultaneously, yielding a complicated mixture of liquid products. The amount of resins which distilled unchanged was usually less than that extractable from the coal by solvents (γ_2 -compounds). As the distillation was carried beyond 400°, increasingly large quantities of gases were evolved, the proportion of hydrogen they contained rapidly increasing. Compounds containing nitrogen and sulphur appeared in small quantity in both gaseous and liquid products at all temperatures of distillation.

A. B. MANNING.

Coking value of coal. T. C. LLOYD (Chem. Met.

Eng., 1930, 37, 169—171).—The plastic range of various coals was studied by measuring the rate of gas flow through a tube charged with the sample under a maximum pressure of 2.2 in. of water. Results were recorded by graphs on the basis of 1 in. pressure, it being assumed that within the slight range of pressure variation the flow is proportional to the pressure. It was found that the plastic range of coking coals extends over a considerable temperature interval (up to 150°) and the width of this interval and the absence of pronounced rise in the rate of flow of gas on the high-temperature side of the plastic range are considered to be reliable indications of the coking quality. The advantage of blending two coals of different plastic ranges is apparent on this view. C. IRWIN.

Swelling of coals during the coking process. B. HOFMEISTER (Arch. Eisenhüttenw., 1929—1930, 3, 559—569; Stahl u. Eisen, 1930, 50, 391—392).—The influence of various factors on the "swelling pressure" of a coking coal has been studied. By Damm's method (B., 1928, 802) the swelling pressure of a coal, the period during which that pressure is exerted, and the subsequent shrinkage of the coke can all be determined. The pressure exerted by both dry and moist coals increased with the bulk density, which was itself dependent on the size of the coal and the closeness of packing. Diminution in the size of the coal particles at constant bulk density, however, produced a fall in the swelling pressure. Addition of moisture up to 5% also reduced the pressure, whilst a further increase in moisture had little effect. Addition of coke breeze reduced the pressure. The swelling pressure of a blend of two coals often departed very considerably (+150 to —50%) from the mean of the values for each coal. Increased rate of heating, in general, increased the swelling pressure. The mechanism of the coking process is very briefly discussed. A. B. MANNING.

Vertical-retort practice on a small works, with special reference to coal testing under normal working conditions. S. B. JONES (Gas J., 1930, 190, 96—97).—A small bed of four vertical retorts, which is alone in operation during the summer, is used to carry out thorough tests on different coals. Methods employed in weighing, sampling, and analysis are described.

R. H. GRIFFITH.

Determination of volatile matter in coal by the standard method, with a new and an old platinum crucible and a nickel crucible. D. J. W. KREULEN (Chem. Weekblad, 1930, 27, 227—229).—A new platinum crucible was found to give a figure for volatile matter 1% higher than that found with an old crucible; a nickel crucible gave intermediate figures, closer to those given by the new platinum. The differences are ascribed to varying surface activities. The greatest swelling of the coke was observed with the nickel crucible. S. I. LEVY.

Coke grading and coal blending. T. H. MADDEN (Gas J., 1930, 190, 95).—It has been found more satisfactory to produce graded coke by cutting large pieces than by screening the coke as produced from the retort; a plant installed for this purpose is described.

R. H. GRIFFITH.

Combustibility of coke. D. J. W. KREULEN (Chem. Weekblad, 1930, 27, 229—230).—The different rates of combustion of two cokes of the same analysis supplied with air at the same rate lead to differences in the maximum temperatures attained, on account of the difference in size of the zones of combustion. The factors influencing the combustibility are considered.

S. I. LEVY.

Domestic fuel by high-temperature carbonisation. J. J. BROWN (Gas J., 1930, 190, 92—94).—Tests on graded fuels were made on 12-lb. charges, ignited by a fixed gas burner in a special grate; temperature measurements were taken at 15-min. intervals after the fire had started. Ash and unburned fuel remaining at the end of the test were weighed and the general characteristics of the fire were noted, particularly after replenishing. The effect of coke size was studied in some preliminary experiments and pieces between 2 in. and $\frac{1}{2}$ in. were finally used in the comparison of cokes prepared by the high-temperature carbonisation of blended coals. The quality of the coke was influenced by changes in the proportion of caking to non-caking coal, and by altering the temperature or time of carbonisation. Large-scale production was accomplished on lines indicated by the experimental plant results, and a dry-quenched coke has been produced which is easily ignited and gives good radiation. For domestic use it compares favourably with raw coal or with a low-temperature semi-coke. The manufacture of a coke of this kind needs very careful selection of coals, and considerable supervision during their treatment.

R. H. GRIFFITH.

Pulverised fuel. J. T. DUNN and B. MOORE (Inst. Chem. Eng., Apr., 1930. Advance proof. 19 pp.).—The type of fuel, general principles and systems of pulverised-fuel firing are discussed. The degree of fineness of the fuel should be fixed in relation to the grade of fuel and the size of combustion chamber available, though particles larger than 100-mesh should be avoided as owing to incomplete combustion a loss as high as 4% may be involved. The direct or unitary system commends itself for small-sized plant on account of its relatively low initial cost and the short length of tube transport. Burners should possess high adjustability and capacity for operating with low air pressures, at the same time imparting a turbulent motion to the combustible mixture, as, e.g., by grooving the burner nozzle. The suitable ratio of combustion rate to furnace volume depends on various factors, but in modern boiler plants a combustion rate of 5—10 lb. of coal per cub. ft. of combustion space is not considered excessive, and shows good practice with 25—35% excess air and a flue gas with 13.5% CO₂. The trend of design to reduce erosion of furnace linings is in the direction of the replacement of refractory material by screens of cooled tubes, though the former possesses the advantage of providing an incandescent surface which aids the combustion particularly of fuels of low volatile content.

C. A. KING.

Peat. II. Dehydration of peat. G. STADNIKOV (Kolloidchem. Beih., 1930, 30, 297—333; cf., B., 1930, 354).—Direct measurements of the rate of dehydration of peat are difficult because the total loss from a 10-g.

sample in several hours is too small for differences to be observed in different kinds of peat, and if 200-g. samples be used other errors arise, for, in air drying, it is not possible to ensure even layering, and in dehydration in a desiccator the absorption of water by the sulphuric acid is so great that its vapour pressure rises continuously. Measurements are best conducted with samples of intermediate weight (about 60 g.). Such measurements carried out at a vapour pressure of 3—4 mm. or above 10 mm. indicated no difference in the velocity of dehydration of natural peat and a coagulated peat suspension, but at intermediate vapour pressures it seemed that the coagulated peat was dehydrated more rapidly. It is emphasised that this result cannot be regarded as conclusive. Both forms eventually reach the same equilibrium for a given vapour pressure. In any case the velocity of dehydration is proportional to the difference between the water-vapour pressure of the peat and that of the surrounding atmosphere. Peat placed on filter paper is dehydrated far more rapidly than when placed on glazed paper, and the rate on filter paper is reduced if the pores become stopped with the matter. These experiments reproduce to some extent the conditions of the drying fields in different weather conditions. So long as peat is left on the ground the process of dehydration comes to a more or less incomplete close, depending on the conditions. Artificial methods of dehydrating peat are described; the cataphoretic method of Schwerin is inefficient. E. S. HEDGES.

Waste-wood utilisation by the Badger-Stafford process. W. G. NELSON (Ind. Eng. Chem., 1930, 22, 312—315).—The process of distillation of about 400 tons of scrap wood per day is described. Larger pieces are broken up and handled separately from sawdust and shavings. Six rotary dryers, 100 ft. long and 10 ft. in diam., reduce the moisture content to 0.5%, at which stage the wood is transferred through butterfly valves to three retorts which operate continuously by utilising the heat developed by the exothermic carbonisation of material near the centre. The direct products are charcoal, pyroligneous acid, and gases having an average heating value of 290 B.Th.U. per cub. ft. The bulk of the charcoal is pulverised and briquetted, or burnt for steam-raising purposes, and the liquors are distilled, yielding approx. 4.5% of methyl alcohol, 12% of acetic acid per ton of wood, together with other allied products.

C. A. KING.

Oxide purification [of coal gas]. S. K. HAWTHORN (Gas J., 1930, 189, 834—836).—A special iron oxide purifying material, prepared by treating spent oxide for sulphur removal and then for cyanide recovery, is found to give increased capacity to the plant. Further advantages are due to reduced handling of the ore, and improvements in working methods have greatly diminished the costs of purification.

R. H. GRIFFITH.

[Coal]-gas dehydration for small works. F. PORTEOUS (Gas J., 1930, 189, 816—817).—A brief summary of advantages resulting from dehydration of coal gas.

R. H. GRIFFITH.

Analysis of Sarmas natural gas. Z. KERTÉSZ (Petroleum, 1930, 26, 393—396).—Recent research in Turda shows that this gas is quite dry, and is the purest

natural methane gas. It contains no oxygen, carbon dioxide, carbon monoxide, or heavy hydrocarbons; the mean analytical result was CH_4 99.18%, H_2 0.43%, and N_2 0.39% by vol. It is used as a heating material in lime kilns. W. S. E. CLARKE.

Natural gas and "cracking" gas and their industrial utilisation. H. M. STANLEY (J. Inst. Petroleum Tech., 1929, 15, 516—552).—Recent advances in the subject are reviewed and a full bibliography is given.

Crystallisable phenols of brown-coal tar. F. SCHULZ and J. PRUNET (Paliva a Top., 1929, 10, 61—68; Chem. Zentr., 1929, ii, 3087).—Tar oil from coal from the Karl pit in Cukmantl contains a fraction, b.p. 254—257°, m.p. 59°, which appears to consist of a mixture of a dihydroxybenzene with a dihydroxytoluene.

A. A. ELDRIDGE.

Wood tar for making axle grease. A. DENNEMARK (Neff. Choz., 1929, 17, 236—240).—Wood tar is treated with kerosene to precipitate pitch, the acids are saponified, the soap is separated, and the residual mixture is steam-distilled. Axle grease is made from the tar (20%), lime (10%), and fuel oil (70%). Wood oil must first be eliminated. The grease should have m.p. 121°.

CHEMICAL ABSTRACTS.

Viscosities of [tar and] bituminous materials and the relationship between values obtained by different methods. H. METZGER (Z. angew. Chem., 1930, 43, 289—293).—There is a constant relationship between the temperature interval from the setting point, as determined by the Hoepfner-Metzger method, to the drop point and the interval between the first-mentioned temperature and the softening point. For the Kraemer-Sarnow softening point the factor is 0.6816, and for the ring-and-ball softening point 0.8721. The Hoepfner-Metzger system grades the setting point as 0.0 and the drop point as 1.0. If the viscosity-temperature curve of a tar or pitch on this system is depicted with right-angle co-ordinates, the softening points becoming lines parallel to the temperature axis. The Richardson penetration value has not a constant relationship to the Hoepfner-Metzger points, and is depicted by curves obtained by taking average results from a number of samples of bitumen. In a similar way curves are obtained giving the relation to the Hoepfner-Metzger values of viscosities by the Hutchinson, Engler, etc. methods, which are, of course, measured in seconds. The whole series of curves are exhibited on one diagram. The accuracy of the relationships depends largely on the accurate determination of the setting point.

C. IRWIN.

Ring-and-ball method of test for softening point of bituminous materials, resins, and similar substances. P. H. WALKER (Bur. Stand. J. Res., 1930, 4, 195—201).—Modified procedure and apparatus as used at the Bureau of Standards for carrying out this test are described.

C. J. SMITHELLS.

Magnetic double refraction of petroleum of various origin. A. COTTON and M. SCHERER (Compt. rend., 1930, 190, 700—702).—The magnetic double refraction (β) of six samples of petroleum from (i) Texaco-

West-Columbia, (ii) Pechelbronn, (iii) Mid-continent, (iv) Columbia, (v) Iraq, and (vi) Moreni (prepared by a preliminary distillation to 250°, treatment at 95° with 1% sulphuric acid, washing with alkali and water, and redistillation to 250°) has been determined at 14.8—16.7° for light of wave-lengths $\lambda = 578, 546, \text{ and } 436 \text{ m}\mu$. The values of C_m/d (the magnetic double refraction for unit field and unit mass, C_m for nitrobenzene being taken as 253×10^{-14}) have been thence deduced for $\lambda = 578$. The density (d) of the oil, values of β for $\lambda = 436$ and 578, and $(C_m/d) \times 10^{-14}$ are (in this order): (i) 0.855, 0.53°, 0.86°, 6.25; (ii) 0.779, 0.43°, 0.64°, 5.53; (iii) —, —, 0.66°, —; (iv) 0.859, 0.70°, 1.01°, 8.15; (v) 0.767, 0.63°, 0.88°, 8.21; (vi) 0.840, 2.27°, 3.34°, 27.00. These figures show differences which may be of use in distinguishing the oils. C. A. SILBERRAD.

Determination of mol. wts. of non-volatile petroleum oils. J. M. DEVINE (Univ. Oklahoma Bull., 1929, 9, 131—137).—Determinations of mol. wt. by the b.p. and f.p. methods agree for the same solvent when the values are extrapolated to zero concentration. Ethylene dichloride as solvent gives values 5% higher, and carbon tetrachloride 5% lower, than does benzene.

CHEMICAL ABSTRACTS.

Mineral oil from new sources. B. HLAVICA and F. TRČA (Paliva a Top., 1928, 10, 101—102; Chem. Zentr., 1929, ii, 3087).—Analyses of oils from Turzonka and Hodonin are recorded.

A. A. ELDRIDGE.

Composition of tar from some North Bohemian coals. B. G. SIMEK (Paliva a Top., 1928, 10, 157—160; Zentr., 1929, ii, 3086).

Low-temperature tars. J. TICHÝ (Paliva a Top., 1929, 11, 33—37, 42—50, 60—61; Chem. Zentr., 1929, ii, 3086).

Pneumatic transport. NEU. **Combustion-gas dryers.** HATTA.—See I. **Absorption of carbon monoxide.** GUMP and ERNST.—See VII. **Road asphalts.** MACZYŃSKI and SKALMOWSKI. **Road emulsions.** SHIPEROVICH and others.—See IX. **Coal-fired annealing furnaces.** STÄBLER.—See X. **[Carbon from] rice husks.** DE MONGEOT.—See XIX. **Low-viscosity nitrocellulose.** MILLIKEN.—See XXII.

PATENTS.

Coking retort ovens. KOPPERS Co., Assees. of J. VAN ACKEREN (B.P. 302,365 and 315,847, [A] 14.12.28, [B] 11.9.28. U.S., [A] 15.12.27, [B] 20.7.28. [B] Addn. to B.P. 304,743; B., 1929, 967).—(A) The heating walls of a coke-oven battery are provided with vertical flame flues divided into groups in which the gases alternately ascend and descend. The flues communicate at the top and there is no dividing wall between the different groups. Below the chambers and parallel thereto are the regenerators, which are divided into pairs by a central transverse partition. In the brickwork separating the chambers and heating walls from the regenerators are transverse horizontal flues extending the length of the battery. Some of these flues connect inflow regenerators to certain of the flame flues of each heating wall, whilst the others connect the outflow regenerators to the other flame flues of each heating wall, and *vice versa* on reversing the operating conditions. (B) The

oven described in the prior patent is modified by so connecting the regenerators, their respective flue groups, and the gas supply that the respective groups of flues of each heating system in the same heating wall simultaneously convey either burning gases or waste gases.

A. B. MANNING.

Device for the utilisation of the sensible heat of the distillation gases from ovens for production of gas and coke. N.V. SILICA EN OVENBOUW MIJ. (B.P. 310,892, 2.5.29. Ger., 2.5.28).—A removable steam generator formed by two vertical, concentric tubes is arranged centrally in each of the ascension pipes leading from the oven to the gas main. The ascension pipes are provided with fireproof linings. The inner of the two tubes serves as a supply tube for the water to be evaporated, while the outer tube serves for the production of the steam. All the inner tubes are connected, through valves, to a common water-pipe, and all the outer tubes similarly to a common steam-pipe. Tubes from the common water- and steam-pipes lead to a steam-drum.

A. B. MANNING.

[Low-temperature] distillation of fuel. C. B. THWAITE (B.P. 326,492, 13.12.28).—The finely-divided fuel is charged into pockets in a horizontal tray within a heating chamber or retort, and is carbonised therein by passing hot waste gases, *e.g.*, from coke ovens or a blast furnace, through the retort. A charging hopper is provided at the top of the retort, and the fuel, which is delivered on to the tray, is spread evenly in the pockets by means of arms operated from outside the retort. When carbonisation is complete, the tray, which is rotatable about a horizontal axis, is inverted; the low-temperature coke falls to the bottom of the retort and is withdrawn through a double-conical discharge valve.

A. B. MANNING.

Distillation and gasification of solid fuel. T. LICHTENBERGER (B.P. 309,190, 6.4.29. Ger., 7.4.28).—A gas generator has a vertical shaft the lower part of which is surrounded by an annular trough or bath through which molten salt at 800–1000° is circulated. At the base of the shaft is a rotary grate, through which steam is supplied to the generator, and a water-seal which prevents access of air to or escape of gas from the generator.

A. B. MANNING.

Distillation of coal, shale, peat, wood, and other fragmentary solid materials. S. MOORE (B.P. 326,540, 15.12.28).—The material is conveyed in a series of separate charges, *e.g.*, by means of spaced push-plates attached to an endless chain, through a horizontal retort the floor of which is heated externally by contact with a bath of molten metal. The retort is provided with gas-tight delivery and discharge orifices and means for regulating the rate of delivery and discharge of the material.

A. B. MANNING.

Carbonisation or gasification of coal and the like. K. C. APPLEYARD. From E. O'TOOLE (B.P. 326,493, 6.9.28).—The coal is charged into a vertical retort in the upper part of which are a number of vertical tubes which mould passage-ways in the coal when it becomes softened by heat and which serve to convey the heating gases and the products of distillation from the retort. The coal is distilled by passing up through it

the hot gases from a combustion chamber below the retort. The retort comprises a number of sections fitting one above the other, and preferably constructed of an outside shell of steel lined with firebrick. Each section is made up of two semi-cylindrical halves bolted together. When the coke in the bottom section is completely carbonised the section together with the coke it contains is removed to a quenching platform, the retort is lowered, and another section added at the top. Dowel pins hold the material in the upper part of the retort during this procedure. At the top of the retort is a hood to which is attached a ring of sheet iron which extends down through the first and a little into the second section; this prevents escape of gas when the upper part of the retort is lowered and before the new upper section is in place.

A. B. MANNING.

Apparatus for the carbonisation of coal and like material. ILLINGWORTH CARBONIZATION CO., LTD., S. R. ILLINGWORTH, SOUTH METROPOLITAN GAS CO., and E. V. EVANS (B.P. 326,247, 10.12.28).—The apparatus comprises a brickwork outer setting within which are retorts constructed of H-section metal conductors (cf. B.P. 245,190; B., 1926, 228); these are used for the low-temperature carbonisation of coal etc. The metal conductors are mounted on L-section seatings, detachably secured to the base of the brickwork setting, so that they may be readily withdrawn when it is desired to use the retorts for high-temperature carbonisation.

A. B. MANNING.

Plant for carbonisation of coal and like material. ILLINGWORTH CARBONIZATION CO., LTD., and S. R. ILLINGWORTH (B.P. 326,169, 29.11.28. Cf. B.P. 245,190 and 244,837; B., 1926, 228, 428).—A number of retorts in the form of vertical metal tubes are arranged in concentric rings within a brickwork setting having a central exhaust flue. The retorts are heated preferably by means of a system of flues similar to that described in B.P. 244,837 (*loc. cit.*). They are charged from chambers which are designed to deliver a measured quantity of coal into each retort, and which are mounted on a rotatable platform, one such chamber being provided for charging each ring of retorts. The measuring chambers, which are filled from a bunker overhead, are provided with inlet and outlet valve-plates. The rotatable platform forms the top of the offtake chamber for the retorts, and is provided with a peripheral flange which runs in a water-seal. Discharge of the carbonised material under the action of gravity is effected periodically by means which are automatically operated in timed relationship with the charging means.

A. B. MANNING.

Apparatus for heat treatment of the volatile products from carbonisation of coal. ILLINGWORTH CARBONIZATION CO., LTD., and S. R. ILLINGWORTH (B.P. 326,135, 29.11.28).—A retort setting of the type described in B.P. 245,190 and 244,837 (B., 1926, 228, 428) is provided with a vertical coke chamber wherein the volatile products passing from the retorts are brought into contact with coke at about 700° and are thereby converted into useful hydrocarbons. On each side of the coke chamber are vertical heating flues from which the hot gases pass to horizontal flues for heating the retorts.

A. B. MANNING.

Refining of active carbon. I. G. FARBENIND. A.-G. (B.P. 301,313, 23.11.28. Ger., 26.11.27).—Active carbons containing adsorbed acid electrolytes, *e.g.*, hydrochloric acid, chlorides, or sulphates, which would tend to corrode the containing vessels, are treated with nitric acid or a nitrate, and the nitrate ions are subsequently removed by washing, or steaming, etc. [Stat. ref.]

A. B. MANNING.

Manufacture of fuel. R. W. STREHLENERT (U.S.P. 1,743,985, 14.1.30. Appl., 16.6.27. Swed., 18.5.26).—Dry sawdust is mixed with waste sulphite liquor from cellulose manufacture and containing 25–60% of dry materials, and, after partially drying, the mixture is heated with steam and pressed into briquettes, which are dried and heated at 200°.

F. R. ENNOS.

Destructive hydrogenation of coal, tars, mineral oils, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,157, 3.9. and 19.10.28).—The yield of hydrogenated oils obtained from coal and heavy mineral oils, often low owing to the formation of sludges which cause the deposition of the catalyst, is improved by a continuous circulation or movement of the liquid. Pumps of various types may be used, such as geared, valveless, centrifugal, or reciprocating. The hydrogen is continuously introduced into the liquid through a number of jets. Light products are carried with the hydrogen in the form of vapour and are separated. From the hydrogenated, unvaporised residue lubricating oils may be separated. A portion of the circulating liquid may be continuously withdrawn and the catalyst reactivated. Fresh raw material and catalyst are continuously added.

T. A. SMITH.

Destructive hydrogenation of coal, tars, mineral oils, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,184, 3.9.28. Cf. B.P. 326,157; preceding abstract).—The materials in the form of liquids or pastes are subjected to destructive hydrogenation under pressure in the presence of colloiddally dispersed catalysts which are immune from poisoning by sulphur, *e.g.*, mixtures of molybdenum, zinc, and magnesium oxides. A portion of the reaction liquid may be branched off, either continuously or periodically, and the catalysts contained therein may be regenerated without separation from the liquid.

A. B. MANNING.

Destructive hydrogenation [of oils, tars, suspensions of coal, etc.]. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 326,586, 31.12.28).—The material in the form of a continuously falling, annular, liquid film is submitted to the simultaneous action of hydrogen under pressure and heat from a central heating element which does not come into contact with the material. Forms of apparatus suitable for carrying out the process are described.

A. B. MANNING.

Hydrogenation of coal, oils, and similar materials. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 326,238, 8.12.28).—The reaction vessel is heated by circulating hydrogen at a suitable temperature, *e.g.*, 450°, through a surrounding jacket, and all or part of this hydrogen is then admitted into the reaction vessel together with the preheated coal, oil, etc. The jacketed reaction

vessel is mounted within a high-pressure vessel. The intervening space is packed with heat-insulating material and the high-pressure vessel is also provided with a jacket, through which cooling gases are passed.

A. B. MANNING.

Water-gas generators. D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 326,293, 25.1.29).—Coke is charged into a deep vertical shaft and the lower portion is subjected to alternate blasting with air and steam. The blow gases are led off, without passing through the upper portion of the coke bed, into regenerators which are used for preheating the steam during the "run." Part of the gas resulting from the steam blast (the "run") is passed through the upper portion of the coke bed and the remainder through regenerators used for preheating the incoming air for the succeeding "blow." During the "blow" part of the preheated air is by-passed to form secondary air for the combustion of the blow gases in a chamber adjacent to the regenerators.

A. B. MANNING.

Production of gases rich in carbon monoxide. (S.I.R.I.) SOC. ITAL. RICERCHE INDUSTRIALI (B.P. 306,959, 26.2.29. It., 29.2.28).—Solid fuels are gasified by injecting oxygen and steam (or oxygen and carbon dioxide) into a producer at a pressure which is unable to cause agitation of the fuel, but is sufficient to bring about rapid combustion and to carry away the ash as a fine dust containing the alkali and alkaline-earth compounds in the form of the oxides or hydroxides. The gas is washed with water, which dissolves these alkaline compounds and fixes the greater part of the carbon dioxide and the sulphur compounds in the gas. The residual ash is removed in dust separators.

A. B. MANNING.

Purification of gases containing hydrogen sulphide. C. STILL (B.P. 297,061, 13.9.28. Ger., 13.9.27).—The gases are washed with a suspension, in an alkaline solution, of an oxide or hydrated oxide of iron containing a small proportion, *e.g.*, 5%, of an oxide or hydrated oxide of manganese. The addition of the manganese increases the rate of absorption of the hydrogen sulphide and the rate of regeneration of the spent liquor; it also considerably reduces the formation of acid by the oxidation of sulphur during regeneration.

A. B. MANNING.

Bituminous composition and its manufacture. L. KIRSCHBRAUN (U.S.P. 1,738,906, 10.12.29. Appl., 22.7.27).—A waterproofing composition is formed by agitating and thereby aerating an aqueous dispersion of asphalt or similar bituminous material, preferably containing bentonite, in the presence of an agent having a low surface tension, *e.g.*, an alkaline-earth oxide, albuminous substance, or soap. The resulting foamy mass, which has a substantially permanent cellular and sponge-like structure, contains gas, and retains its shape and characteristics after drying out, is incorporated with cementitious material.

H. S. GARLICK.

Restoring the quality of used gasoline. F. H. MCBERTY, ASSR. to DE LAVAL SEPARATOR CO. (U.S.P. 1,745,108, 28.1.30. Appl., 13.6.27).—Mechanically removable impurities are separated from dry-cleaner's solvent, which is then treated with a mineral acid, the

insoluble products are removed, the oil is treated with an alkali to effect partial neutralisation, and the remaining acidity is neutralised with ammonia.

H. S. GARLICK.

Treatment of petroleum oils. W. M. CROSS (U.S.P. 1,744,262, 21.1.30. Appl., 17.12.25. Renewed 9.8.29).—Crude petroleum is cleaned and the higher fractions are extracted by preheating the oil to above 100°, separating the liquid from the vapour-phase material, and passing the latter to a fractionating stage. The liquid products pass to a secondary heating stage where additional heat is imparted, and then on to a combined re-boiler and partial cooler in which the temperature is controlled before being admitted to the vaporisation stage, where additional fractions of vapour-phase hydrocarbons are separated and a free-flowing, residual bottom is obtained from the oil. Excess heat in the oil prior to evaporation is utilised for reboiling in the re-boiler and partial cooler, a condensate separated out during fractionation, and the vapours evolved in the vaporising stage are subjected to fractionation and final condensation.

H. S. GARLICK.

Refining of hydrocarbon oils. W. A. STREET (B.P. 326,163, 5.11.28).—Used lubricating oils are treated with a mixture of 1 pt. of sulphuric acid (*d* 1.84) and 4 pts. of new or recovered lubricating oil. This mixture (1 vol.-%) is well stirred into the used lubricating oil with the addition of 20–50% of petroleum spirit to ensure rapid settling.

T. A. SMITH.

Production of refined hydrocarbon oils and the like. I. G. FARBERIND. A.-G. (B.P. 300,900, 6.11.28. Ger., 19.11.27).—The sulphur content of impure oils is reduced by hydrogenation at temperatures of 300–420° and pressures above 20 atm. in the presence of catalysts of group VI or of cobalt and its compounds. The unsaturated content of the oils is also reduced. The process is particularly applicable to the refining of cracked distillates and rendering the oils obtained by the low-temperature carbonisation of coal suitable for use in Diesel engines. Oils low in hydrogen content may be separated from other oils by means of liquid sulphur dioxide and then refined and hydrogenated. When benzene vapour, obtained by the low-temperature carbonisation of brown coal, and containing 1% S and 25% of unsaturated hydrocarbons, is passed at 410° and 200 atm. through a tube, 2.5 cm. diam. and 150 cm. long, containing a mixture of tungstic acid and magnesia, a saturated benzene free from sulphur is obtained. The time of contact is 30 sec.

T. A. SMITH.

Treatment of hydrocarbon oil. C. W. WATSON, Assr. to TEXAS CO. (U.S.P. 1,744,599, 21.1.30. Appl., 11.11.27).—A battery of shell stills is arranged for continuous distillation in a series of groups of stills, each group being maintained under conditions of distillation and provided with a fractionating zone. Distillates suitable for forming a fraction of the boiling range of gasoline are taken off from the first group of the series, those suitable for forming a kerosene fraction are removed from the next group of the series, and the oil itself is introduced into the last still of each group at an intermediate point in the corresponding fractionating zones.

H. S. GARLICK.

Cracking of mineral oil. J. H. PEW, Assr. to SUN OIL CO. (U.S.P. 1,744,574, 21.1.30. Appl., 10.2.26).—A stream of oil is flowed through a series of cracking zones in heat-exchange relation with a vaporised metallic heating medium, *e.g.*, mercury, each zone being maintained at a temperature high enough to effect cracking of the oil with formation and accumulation of asphaltene within the cracking zones, but below that required to form any substantial amount of insoluble coky residue. This is effected by maintaining the metallic vapour at progressively higher temperatures in the successive heating zones and arranging in each zone for condensation of metallic vapour and transfer of its latent heat to the oil. At definite periods the process is stopped and a solvent for the asphaltene is flowed through and out of part of the cracking zones.

H. S. GARLICK.

Carburetted or mixing devices for air and liquid fuel, applicable also to the mixing of liquids or gases together. J. W. SMITH (B.P. 327,339, 7.6.29).—The fluids are passed through an annular conduit which is provided with a number of baffles alternately and oppositely inclined from each wall. The free edges of the baffles are formed with a large number of pointed blades like multi-pointed stars; the blades may be given a twist alternately in opposite directions, and their number and angle of inclination may be different on different stars.

B. M. VENABLES.

Coking retort oven. J. VAN ACKEREN, Assr. to KOPPERS CO. (U.S.P. 1,754,131, 8.4.30. Appl., 4.5.23).—See B.P. 215,306; B., 1925, 274.

Coking retort oven. J. BECKER and J. VAN ACKEREN, Assrs. to KOPPERS CO. (U.S.P. 1,752,363, 1.4.30. Appl., 27.9.26).—See B.P. 278,012; B., 1929, 160.

Binder for fuel agglomerates. F. M. CROSSMAN (U.S.P. 1,752,838, 1.4.30. Appl., 5.12.24).—See B.P. 244,053; B., 1927, 98.

Polymerisation of oils. S. P. MILLER, Assr. to BARRETT CO. (U.S.P. 1,752,921, 1.4.30. Appl., 23.1.25).—See B.P. 246,491; B., 1927, 722.

Production of montan wax. F. ZSCHOCK and H. RODRIAN, Assrs. to A. RIEBECK'SCHE MONTANWERKE A.-G. (U.S.P. 1,753,816, 8.4.30. Appl., 13.3.28. Ger., 16.9.27).—See B.P. 297,102; B., 1929, 1007.

Pulverisers (U.S.P. 1,744,927 and 1,744,895). **Mixing of liquids** (B.P. 326,924). **Stuffing-box for stills** (U.S.P. 1,745,200). **Removing dust from gases** (B.P. 318,965). **Filtering gases** (B.P. 327,083). **Gas cleaner** (U.S.P. 1,743,344).—See I. **Hydrocarbons from methane** (B.P. 316,126).—See III. **Carbon monoxide** (B.P. 326,227).—See VII. **Catalysts** (B.P. 304,654).—See VIII. **Dispersions of bitumens** (U.S.P. 1,738,776). **Bituminous paving** (B.P. 326,863).—See IX. **Catalysts** (B.P. 326,580).—See X. **Insecticide** (U.S.P. 1,744,324).—See XVI.

III.—ORGANIC INTERMEDIATES.

Preparation of glycol by hydrolysis of ethylene [di]chloride. H. BAHR and H. ZIELER (Z. angew. Chem., 1930, 43, 286–289).—The manufacture of glycol in America owes its origin to the availability of ethylene

in cracking gases, but fractions rich in ethylene are also obtained in Europe in synthetic ammonia plants employing coke-oven gas from which the nitrogen-hydrogen gas is obtained by partial liquefaction. This may be converted into glycol either through ethylenedichlorohydrin or ethylene dichloride. The latter is not decomposed by water under ordinary conditions and alkalis produce principally vinyl chloride and acetylene. It is necessary therefore to convert the ethylene dichloride into an ester by treatment with, *e.g.*, sodium acetate and then effect hydrolysis with alkali. Experiments showed that the direct hydrolysis of ethylene dichloride (b.p. 84°) was facilitated by increased temperature and pressure. The yield per hr. rose from nil at 120° to 34% at 180°. Aldehydes begin to be formed in quantity if the hydrochloric acid concentration rises above 1%. A method was therefore adopted of autoclave treatment with addition of alkali at intervals to neutralise the hydrochloric acid. Yields of about 70% were obtained, working at 150–170°, with only small quantities of aldehyde and vinyl chloride. A loss of 20–25% was attributed to aldehyde having been volatilised or resinified. On a larger scale a yield of 1.5–2.0 kg. of glycol per hr. per 100 litres of liquid was obtained, using a reaction mixture containing 4–5% of ethylene dichloride. It is scarcely practicable to replace caustic soda by lime, and little advantage would be obtained as the sodium chloride is required for electrolysis. C. IRWIN.

Vapour pressure and [latent] heat of vaporisation of toluene. D. S. DAVIS (Ind. Eng. Chem., 1930, 22, 380–381).—The following equation, relating the vapour pressure, p , of toluene with T , the absolute temperature, $\log (41.6/p - 0.28) = 3.106(593.7/T - 0.08158)$, is suggested to correlate recent vapour-pressure measurements on toluene. The different empirical equations used to calculate the latent heat of toluene are compared. H. INGLESON.

Waste wood utilisation. NELSON.—See II. Citric acid. CATULLO.—See VII. Absolute alcohol. GUINOT.—See XVIII. Utilisation of rice husks. DE MONGEOT.—See XIX. Detection of isopropyl alcohol. BOEHM and BODENDORFF.—See XX.

PATENTS.

Production of higher hydrocarbons from methane. F. FISCHER and H. PICHLER (B.P. 316,126, 14.12.28. Ger., 23.7.28).—Methane, in the pure state or diluted with other gases, is converted into higher unsaturated and aromatic hydrocarbons by subjecting it to a temperature of at least 1000° for a period not exceeding 1 sec., in the absence of catalysts or oxygen-containing gases. The process is carried out by passing the gas at a high velocity through narrow tubes maintained at the required temperature. The free hydrogen produced at the same time may be reconverted into methane, *e.g.*, by interaction with carbon monoxide in the presence of a suitable catalyst, and this again subjected to thermal decomposition. A. B. MANNING.

Manufacture of butylene from ethylene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,322, 22.2.29).—Ethylene under a pressure of 55–60 atm. is led with a little boron fluoride over a nickel catalyst at

15° (contained, *e.g.*, in a 4-litre autoclave packed with Raschig rings); 1.5 litres of butylene per hr. are obtained. C. HOLLINS.

Preparation of higher alcohols from ethylene and its homologues. COMP. DE BÉTHUNE (F.P. 636,651, 20.10.26).—In the process of B.P. 273,263 (B., 1927, 859), one tower is charged with dilute acid, cooled to 15–20°, which after absorption is worked up for higher alcohols and ethers in the usual way.

R. BRIGHTMAN.

Concentration of aqueous acetic acid. I. G. FARBENIND. A.-G. (B.P. 302,174, 10.12.28. Ger., 8.12.27).—In the extraction method for the concentration of acetic acid a secondary liquid (light petroleum, petroleum, benzene, tetrahydronaphthalene, trichloroethylene) is used so that less water is extracted and on subsequent distillation of the extract all remaining water is carried over with the secondary liquid. The latter is preferably a liquid boiling below 119°, and is used in conjunction with a high-boiling solvent. Alternatively, a low-boiling solvent and a high-boiling secondary liquid are used. Basic solvents are excluded. C. HOLLINS.

Hydrogenation of polyhydroxy-compounds [glycerol from dextrose etc.]. I. G. FARBENIND. A.-G. (B.P. 299,373, 24.10.28. Ger., 24.10.27).—Glycerin, or its reduction product, propylene glycol, is obtained by catalytic hydrogenation of polyhydroxylated compounds in presence of metals of the iron or platinum group (nickel). Sorbitol at 200–250° with hydrogen at 70–100 atm. gives first glycerol and then propylene glycol, b.p. 186–188°; dextrose or sucrose is reduced at 150° and 40 atm. to the hexitol and then at 200–250° and 100 atm. to glycerol and the glycol. Starch and cellulose are similarly reduced at 210–260° and 70–100 atm. C. HOLLINS.

Preparation of cyclohexylbenzene and its homologues. P. SCHVING (F.P. 636,686, 23.10.26).—*cyclo*-Hexanol or one of its homologues is heated with benzene or homologues in presence of sulphuric acid. *cyclo*-Hexanol and benzene give 56% of *cyclohexylbenzene* and 12–16% of *p*-*dicyclohexylbenzene*.

R. BRIGHTMAN.

Manufacture of triarylcarbinols. IMPERIAL CHEM. INDUSTRIES, LTD., and S. COFFEY (B.P. 325,933, 1.1.29).—A diaryl ketone (except tetra-alkyldiaminobenzophenones) mixed with an aryl halide is added to sodium or potassium in hot benzene or other diluent. The preparation of diphenyl-*p*-tolylcarbinol, m.p. 72–73°, from benzophenone and *p*-chlorotoluene, phenyldi-*p*-tolylcarbinol, m.p. 76–77°, from di-*p*-tolyl ketone and bromobenzene, triphenylcarbinol from benzophenone and chlorobenzene, *p*-methoxytriphenylcarbinol from benzophenone and *p*-chloroanisole, diphenyl- α -naphthylcarbinol from phenyl α -naphthyl ketone and bromobenzene, and 4:4'-dimethoxytriphenylcarbinol from 4-methoxybenzophenone and *p*-chloroanisole is described. C. HOLLINS.

Manufacture of organic phosphorus compounds [moth-proofing agents]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,137, 30.11.28).—Phenols are heated with triarylphosphine oxides, PAR_3O , to give products of the type, $\text{PAR}_3(\text{OH})\cdot\text{OAr}$, which are

useful moth-proofing agents. Compounds of triphenylphosphine oxide with phenol (m.p. 106°), *o*-, *m*-, and *p*-cresols (m.p. 67°, 89–90°, 44–45°), *o*- and *p*-chlorophenols (m.p. 105–106°, 80–81°), thymol (m.p. 67–68°), 6-chlorocarvacrol (m.p. 65–66°), 2:6-dichlorophenol (m.p. 97–98°), 2:4:6-trichlorophenol (m.p. 70–71°), *m*-5-xylenol (m.p. 83–84°), 6-chloro-*o*-cresol (m.p. 91–92°), 3-chloro-*p*-cresol (m.p. 64–65°), ethyl *p*-hydroxybenzoate (m.p. 104–105°), *p*-nitrophenol (m.p. 107–108°), pyrocatechol monoethyl ether (m.p. 93–94°), resorcinol (m.p. 119–120°), *p*-hydroxybenzaldehyde (m.p. 66–67°), α - and β -naphthols (m.p. 106–107°, 89–90°), diethyl-*m*-aminophenol (m.p. 81–82°), *ar*-tetrahydro- β -naphthol (m.p. 120°), *p*-hydroxy-*n*-butylbenzene (m.p. 73–74°), 2:4-dichlorophenol (m.p. 54–55°), and of tri-*o*-tolylphosphine oxide with phenol (m.p. 149–150°) are described. C. HOLLINS.

Manufacture of aromatic nitriles. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,149, 4.12.28).—Aromatic amines are diazotised and treated with aqueous potassium cupridiammonium cyanide prepared by stirring a solution of copper sulphate (1 mol.) into aqueous potassium cyanide (4 mols.) and ammonia (2 mols.). A saving of half the copper salt and half the cyanide used in the ordinary Sandmeyer reaction is effected. The yield of *p*-tolunitrile from *p*-toluidine is 83%; of benzonitrile from aniline, 65%. C. HOLLINS.

Manufacture of aromatic aminosulphochlorides substituted in the amino-group [*N*-substituted arylaminesulphonyl chlorides]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,226, 30.11.28 and 27.7.29).—By the action of chlorosulphonic acid *N*-substituted arylaminesulphonic acids are converted, according to conditions, into corresponding sulphonyl chlorides or into compounds containing additional sulphonyl chloride groups. In particular the *sulphonyl chlorides* of acetyl-*J*-acid and acetyl- γ -acid are obtained for the first time. Other compounds described are: acetyl- α -naphthylamine-4-sulphonyl chloride, m.p. 170°; disulphonyl chloride of acetyl-*H*-acid, m.p. 188–189°; 1-acetamido-8-naphthol-4:6-disulphonyl chloride, m.p. 145–147°; benzoyl- α -naphthylamine-6-sulphonyl chloride, m.p. 185–186°; sulphonyl chlorides from phosgenated *o*-aminophenol-4-sulphonic acid (m.p. 210–214°), 2:1-aminonaphthol-4-sulphonic acid [m.p. 262–263° (decomp.)], and *H*-acid (m.p. 214–216°); 2-methylbenzoxazole-5-sulphonyl chloride, m.p. 189–190° (decomp.); 2-hydroxyperimidine-5:8-disulphonyl chloride, decomp. 250°; 2-methylperimidine-6-sulphonyl chloride, m.p. 200–204°; carbanilide-4:4'-disulphonyl chloride; methyl-*o*-toluidine-4-sulphonyl chloride, m.p. 99–100°; dimethylaniline-*m*-sulphonyl chloride, m.p. 38°; isomeric dimethyl- β -naphthylaminesulphonyl chlorides, m.p. 86° and 108–110°, respectively, from the 5-sulphonic acid; 4-chlorosulphonylnaphthalene-1:2-diazo oxide, m.p. 131–132°. The products are of use as dye intermediates, for therapeutical purposes, and for combating insect pests. C. HOLLINS.

(A) Fire-extinguishing and (B) fire-proofing compositions. I. G. FARBENIND. A.-G. (B.P. 302,172 and 326,253, 10.12.28. Ger., 10.12.27).—Alkylated or aral-

kylated aromatic sulphonic acids are added as wetting agents (A) to foam-producing fire-extinguishing materials and (B) to fire-proofing compositions. C. HOLLINS.

Manufacture of sulphonic acids of 1-aminomethylnaphthalene. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 326,022, 12.3.29).—Sulphonation of 1-aminomethylnaphthalene with 100% sulphuric acid below 50°, or with oleum at 60–70°, yields, respectively, the 2-sulphonic acid or the 2:4-disulphonic acid from which the corresponding aldehydes are obtainable. C. HOLLINS.

Preparation of camphor by catalytic hydrogenation of borneol or isoborneol. Soc. NOBEL FRANÇ. (F.P. 636,809, 29.6.27).—Formation of camphene and the reverse reaction to borneol and isoborneol are reduced by the use of calcium carbonate as support for the catalyst. *E.g.*, reduced nickel oxide catalyst on powdered chalk gives 90% of camphor at 200–350°.

R. BRIGHTMAN.

Manufacture of nitro-compounds [of 2:4-dihydroxyquinolines]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,398, 2.5.29).—2:4-Dihydroxyquinoline or an *N*-substituted 4-hydroxy-2-quinolone is nitrated with nitric and sulphuric acids. The nitro-derivatives retain the power of coupling with diazo-compounds (nitration in acetic acid gives non-coupling products), and may be reduced and/or sulphonated. The nitrated 4-hydroxy-1-methyl-, -1-ethyl-, and -1-phenyl-2-quinolones melt above 300°.

C. HOLLINS.

Manufacture of condensation products of polynuclear hydrocarbon compounds and olefines [wetting agents]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,500, 11.12.28).—Solid hydrocarbons (or their halogen derivatives) containing at least 3 ring-nuclei are condensed with olefines in presence of aluminium chloride etc. and preferably hydrogen chloride. Examples are: phenanthrene with propylene or cyclohexene, acenaphthene or 5-bromoacenaphthene with propylene, crude anthracene with propylene.

C. HOLLINS.

Organic intermediate compounds and [vat] dyes [of the anthraquinone series]. (SIR) G. C. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 326,487, 8.12.28).—1-Chloroanthraquinone-2-carboxylic acid is heated with alkaline polysulphide and ammonia to give anthrathiazolecarboxylic acid, the chloride of which is condensed with amino- or diamino-anthraquinones to form vat dyes, *e.g.*, with α - or β -aminoanthraquinone (greenish-yellow), 1:5-diaminoanthraquinone (yellow), 1:4-diaminoanthraquinone (yellow-brown), 1:5-aminobenzamidoanthraquinone (greenish-yellow), 6-chloro-1-aminoanthraquinone (greenish-yellow), or 2-amino-1-thiolanthraquinone.

C. HOLLINS.

Manufacture of anthanthrone derivatives. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 325,797, 25.8. and 9.10.28).—The nitro-group or -groups in nitrated anthanthrones may be replaced by chlorine or bromine by the action of halogen or of benzoyl chloride or bromide, or may be reduced and the amino-group or -groups may be replaced by halogen, hydroxyl, carboxyl, cyano-, thiol, or thiocyano-groups. The hydroxy- or

carboxy-compounds may be etherified or esterified to give vat dyes. Mono- and di-chloroanthranthrones are orange-yellow vat dyes; the di-iodo- (bluish-red), dimethoxy- (bluish-red), mono- and di-cyano-, methoxy- (red), bromo- (orange-yellow), iodo- (orange), β -chloroethoxy- (red), dibromo- (orange-red), and thiol (brown) derivatives are also described. C. HOLLINS.

Compound from indene and phenols. L. ROSENTHAL and W. KROPP, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,754,052, 8.4.30. Appl., 22.6.27. Ger., 28.6.26).—See B.P. 297,075; B., 1928, 845.

Manufacture of naphthisatin compounds. J. HALLER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,754,098, 8.4.30. Appl., 29.11.26. Ger., 3.12.25).—See B.P. 286,358; B., 1928, 327.

Distilling apparatus [for glycerin] (U.S.P. 1,743,289). **Carrying out catalytic reactions** (B.P. 310,956). **Catalysts containing free phosphoric acid** (B.P. 326,185).—See I. **Vulcanisation accelerators** (B.P. 326,256).—See XIV. **Light-sensitive surfaces** (B.P. 326,287).—See XXI.

IV.—DYESTUFFS.

PATENTS.

Preparation of *o*-nitroso-[hydr]oxy dyes and their heavy-metal compounds. J. R. GEIGY A.-G. (B.P. 305,648, 30.11.28. Ger., 9.2.28).—Pyridacetyl or other basic derivatives of aminonaphthols are nitrosated and converted into heavy-metal salts which are soluble in water. Pyridylacetyl-2:7-aminonaphthol, $C_5H_5N(Cl) \cdot CH_2 \cdot CO \cdot NH \cdot C_{10}H_6 \cdot OH$, after nitrosation gives a copper salt which dyes brown-yellow. Dimethylaminoacetyl-2:7-aminonaphthol (yellowish-green iron salt), 7-hydroxy- β -naphthyltrimethylammonium chloride (blue-green iron salt), pyridylacetyl-7-amino- α -naphthol (green iron salt), pyridylacetyl-3-amino- α -naphthol (green iron salt), and pyridylacetyl-5-amino- α -naphthol (red-brown copper salt) are also nitrosated and converted into dyes. C. HOLLINS.

Manufacture of [vat] dyes [of the benzanthrone series]. I. G. FARBENIND. A.-G. (B.P. 308,651, 25.3.29. Ger., 23.3.28).—Benzanthrone-3:4-dicarboxylic anhydride (or a derivative) is condensed with an *o*-diamine to yield iminazole compounds. With *o*-phenylenediamine, 4-chloro-*o*-phenylenediamine, and 3:4-diaminophenetole orange vat dyes are obtained; with 1:2-naphthylenediamine a deep brown. C. HOLLINS.

Manufacture of vat dyes of the benzanthrone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,148, 4.12.28 and 3.9.29).—A mono- or di-halogenated benzanthrone, or 3:3'- or 4:4'-dibenzanthronyl, or 3:3'- or 4:4'-dibenzanthronyl sulphide is condensed with 1-amino-2-aldehydoanthraquinone (or an anil etc. thereof); or the corresponding mono- or di-amino-derivative is condensed with 1-chloro-2-aldehydoanthraquinone. Preferably a diluent (nitrobenzene), and an acid-binding agent (sodium acetate), and a catalyst (copper oxide) are used. The resulting acridones or acridones are brown vat dyes. 1-Amino-2-aldehydoanthraquinone is thus condensed with 3-bromo-, 11-chloro-, 9-chloro-, 4:9-dichloro-, 3:9-dibromo-, 3-bromo-

9-nitro-, and 3-bromo-9-benzenesulphonamido-benzanthrones, with dibrominated 3:3'- and 4:4'-dibenzanthronyls, and with dibromo-3:3'-dibenzanthronyl sulphide; 1-chloro-2-aldehydoanthraquinone is condensed with 9-amino- and 4:9-diamino-benzanthrones.

C. HOLLINS.

Manufacture of nitrogenous vat dyes [of the dibenzanthrone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,217, 7.9.28. Addn. to B.P. 314,593; B., 1929, 710).—Grey vat dyes are obtained by heating nitrodibenzanthrones or nitroisodibenzanthrones alone or in nitrobenzene in the absence of alkaline condensing agents. C. HOLLINS.

Manufacture of vat dyes containing nitrogen [from pyrazolanthrone/benzanthrones]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,268, 19.12.28).—The condensation products from pyrazolanthrone and 4-chlorobenzanthrones carrying a 3-nitro- or 3-chloro-substituent are converted by alkaline fusion (*e.g.*, alcoholic alkali at 110°) into violet vat dyes. Examples are the condensation products from pyrazolanthrone and 4-chloro-3-nitrobenzanthrone, m.p. 268°, or 3:4:9-trichlorobenzanthrone, and from 5-benzamidopyrazolanthrone and 4-chloro-3-nitrobenzanthrone. The products may be halogenated.

C. HOLLINS.

Manufacture of vat dyes [of the pyranthrone series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,263, 15.12.28).—Reddish-brown vat dyes are obtained by oxidising di- or tetra-bromopyranthrone, *e.g.*, with manganese dioxide or potassium iodate, in sulphuric acid. C. HOLLINS.

Manufacture of acid wool dyes [of the anthraquinone series]. I. G. FARBENIND. A.-G. (B.P. 302,928, 21.12.28. Ger., 23.12.27. Addn. to B.P. 282,409; B., 1928, 442).—A 4-halogeno-1-aminoanthraquinone-2-sulphonic acid is condensed with a *m*-aminoacetalkylanilide to give blue acid dyes.

C. HOLLINS.

Manufacture of monoazo [chrome wool] dyes. I. G. FARBENIND. A.-G. (B.P. 301,423, 26.11.28. Ger., 29.11.27).—Clear yellowish chrome-greens for wool are obtained by coupling 2:3-aminonaphthoic acid with a diazotised *o*-aminophenolsulphonic acid which may contain halogen but no nitro-group, *e.g.*, 4-chloro-*o*-aminophenol-6-sulphonic acid. C. HOLLINS.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 302,601, 17.12.28. Ger., 17.12.27).—An arylamide of 2:3-hydroxynaphthoic acid is coupled in substance or on the fibre with a diazotised 3:6- or 5:6-dihalogenated *o*-toluidine. C. HOLLINS.

Manufacture of dyes [gallocyanine derivatives]. DURAND & HUGUENIN A.-G. (B.P. 301,329, 26.11.28. Ger., 26.11.27).—Readily dischargeable gallocyanines are prepared from *m*-substituted alkylanilines. Examples are dyes from: gallamide and nitrosodiethyl-*m*-toluidine (converted into anilide and sulphonated for chrome-printing greenish-blue); gallamide and *m*-chloronitrosodiethyl- or -dimethyl-aniline (anilide, sulphonated, redder blue); gallamide and nitrosoethyl-*m*-toluidine

(anilide, sulphonated); gallic acid and nitrosomethyl-*m*-toluidine (decarboxylated and reduced, violet).

C. HOLLINS.

Black sulphur dyes. R. VIDAL (F.P. 633,395, 14.8.26).—Nitroso-*o*- or -*m*-cresol is added to a solution of sodium sulphide and after cessation of the reaction 2 : 4-dinitrophenol and sodium sulphide are introduced. Sulphur is then added at 85°, the mixture being thereafter maintained at boiling temperature for 16–24 hrs. and the dye precipitated with the aid of a current of air. Liquid dyes of high concentration are obtained by mixing the moist precipitate with sodium sulphide and concentrating to a 50% content of dye. D. F. TWISS.

[Manufacture of] azo dyes. H. WAGNER, E. HOFFA, E. RUNNE, E. THOMA, and H. HEYNA, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,754,070, 8.4.30. Appl., 5.7.27. Ger., 9.7.26).—See B.P. 274,128; B., 1929, 351.

Manufacture of azo dyes. E. HOFFA and E. THOMA, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,745,676, 4.2.30. Appl., 24.2.28. Ger., 2.3.27).—See B.P. 313,865; B., 1929, 711.

[Manufacture of azo] dyes containing chromium. H. KAMMERER, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,753,120, 1.4.30. Appl., 13.4.28. Ger., 14.5.27).—See B.P. 302,709; B., 1929, 165.

Anthraquinone dyes (B.P. 326,487). **Anthanthrone derivatives** (B.P. 325,797).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Commercial possibilities of the common milkweed. F. GERHARDT (Ind. Eng. Chem., 1930, 22, 160–163).—In an experimental crop from milkweed (*Asclepias syriaca*), 30 bushels of seed, 280 lb. of seed fibre, and 1 ton of air-dry stems were obtained per acre. The seed fibre is too brittle for use in the textile industry, but might be a substitute for kapok. The seed contains 21% of semi-drying oil and 47% of the remaining meal is crude protein. The dry stem contains 10% of bast fibre, which might be used in the textile industry. In view of the α -cellulose content (36.5%), the stems might be used for pulps for making paper or board.

E. B. HUGHES.

Silk—a field for research. E. M. SHELTON and T. B. JOHNSON (Ind. Eng. Chem., 1930, 22, 387–390).—The present state of knowledge of the chemistry of silk is discussed, and some valuable lines of investigation are indicated.

F. R. ENNOS.

Calcium chloride and flue gas for waste disposal of kier liquors from textile plants. R. L. CORSON and H. A. CURTIS (Chem. Met. Eng., 1930, 37, 167–169).—The waste liquor resulting from the boiling of unbleached cotton with dilute caustic soda being highly charged with organic matter is an undesirable effluent to run into streams. The most feasible method of treatment is as follows. The liquor is run down a carbonating tower against a current of flue gases. The bicarbonate content is then neutralised with the requisite amount of untreated liquor, and a slight excess of calcium chloride is added. The solids are settled and the clarified liquor is run away. About 75% of the colour and organic matter are so removed as insoluble

calcium salts of organic acids. Variation in temperature of the liquor has little influence on the results.

C. IRWIN.

Evaluation of unbleached chemical wood pulps. G. HALL (Proc. Tech. Sect. Papermakers' Assoc., 1929, 10, 85–138).—The unbeaten strength test is not considered suitable for unbleached pulps, particularly sulphate pulps, owing to the difference in response to beating. The strongest pulp on unbeaten test is not necessarily the strongest after beating. Unbeaten tests depend more on felting capacity than on the ultimate strength of the fibres. For beating under standard conditions, the Abbé pebble-mill and the Lampén ball-mill are compared. The newer type of Lampén mill is recommended. After beating the pulps are made into circular sheets, 10 in. in diam., couched on to ferro-type plates, pressed at 214 lb./in.², dried at 50–60°, and tested for strength. The effect of varying the pressure on the waterleaf is discussed. Increasing pressure gives rise to increased tensile, folding, and bursting strengths, and lower tearing strength. Difference in strength of machine-made papers and laboratory-made sheets is attributed to the high pressures exerted by the press-rolls of the paper machine, the presence of fragmentary fibres in the back-water, and the more even distribution of the fibres in the laboratory method. Conditions may be chosen so that the different factors cancel out to give comparable results. Pulps beaten in a Lampén mill need only be pressed at 50 lb./in.² to give sheets of equal strength to machine-made paper from the same pulp. The lignin content of pulps is a measure of the degree of cooking, and determines the bleachability. The amount of rosin present is independent of the degree of cooking, but varies according to the process used. The cooking degree is determined rapidly by permanganate absorption tests. α -Cellulose tests are of little value for unbleached pulps. For a given bleachability, sulphite pulps hydrate more readily than do sulphate pulps; similarly, sulphite pulps cooked with indirect steam have the higher bursting and tensile strengths, but the lower folding and tearing strengths. Strength increases with lignin content, tearing strength showing a maximum at 5% of lignin. Porosity, or air-permeability, is a better indication of the condition of the fibres in a sheet than "beating degree." The same amount of beating produces a more porous sulphate sheet than sulphite sheet. The porosity of sheets made from sulphate pulps is proportional to the cooking degree, the harder the pulp the more porous is the sheet. Absorbency is proportional to cooking degree for both kinds of pulp.

T. T. PORTS.

Wood. II. Water-soluble polysaccharide of western larch wood. L. E. WISE and F. C. PETERSON (Ind. Eng. Chem., 1930, 22, 362–365; cf. Harlow and Wise, B., 1928, 636).—The presence of water-soluble ε -galactan in western larch wood (*Larix occidentalis*) described by Schorger and Smith (B., 1916, 750) is confirmed. On complete hydrolysis it yields 11.95% of arabinose and 84.6% of galactose, no other hexoses being detected, and it appears to be an arabogalactan containing arabinose and galactose molecules in the ratio 1:6; the arabinose obtained on hydrolysis

accounts quantitatively for the furfuraldehyde yield on distilling with acid. F. R. ENNOS.

Rayon as a paper-making material. M. B. SHAW and G. W. BICKING (Bur. Stand. J. Res., 1930, 4, 203—211).—It is shown that rayon is valueless in rag stock used for making high-grade paper, and may actually be detrimental, since the rayon fibres break into short lengths when wetted, without the fibrillation and fraying necessary for good felting. C. J. SMITHELLS.

Rice husks. DE MONGEOT.—See XIX.

PATENTS.

Lubricating, conditioning, and laying the fly of raw cotton. G. J. EDWARDS. From E. F. HOUGHTON & Co. (B.P. 325,938, 5.1.29).—The raw stock is treated with a mixture containing at least 60% of mineral oil, 5% of soap, 2% of glycerin, 2% of sulphonated vegetable or animal oil, and the balance non-mineral oil, the whole being emulsified with not less than an equal quantity of water. F. R. ENNOS.

Impregnation of textile filaments with rubber. C. H. GRAY (B.P. 325,916, 19.12.28).—The filaments are passed through a dispersion or solution of rubber and thence between a pair of rotating rubber-covered rollers. F. R. ENNOS.

Means for testing the dryness of fibrous material. C. Q. IVES, Assr. to BROWN CO. (U.S.P. 1,744,120, 21.1.30. Appl., 12.12.24).—The relative dryness of similar materials is determined by comparing the electric resistance between a pair of pointed metallic contacts, connected to a source of electric current, when inserted in the materials to a fixed depth and at a suitable distance apart. F. R. ENNOS.

Manufacture of cellulose esters. H. DREYFUS (B.P. 325,822, 21.11.28).—Cellulosic material is pre-treated with an organic base, *e.g.*, methylpiperidine, above 100° and afterwards esterified with a carboxylic acid anhydride in the presence of the organic base above 100°. F. R. ENNOS.

Manufacture of cellulose esters. I. G. FARBERIND. A.-G. (B.P. 303,006, 6.12.28. Ger., 24.12.27).—Cellulose is treated for 1 hr. below 60° with air saturated with nitric acid vapour, so that its fibrous structure is preserved; the product is subsequently esterified in any desired manner. F. R. ENNOS.

Manufacture of esters of cellulose or other carbohydrates. I. G. FARBERIND. A.-G. (B.P. 301,036, 22.11.28. Ger., 23.11.27).—Esterification of cellulose with a carboxylic acid anhydride and/or acid halide is carried out in presence of liquid sulphur dioxide, with or without the usual catalysts. F. R. ENNOS.

Manufacture of acetylcellulose. I. G. FARBERIND. A.-G. (B.P. 301,755, 3.12.28. Ger., 2.12.27).—By incorporating a catalyst comprising zinc chloride, sulphuric acid, and hydrochloric acid or a substance yielding hydrochloric acid (acetyl chloride or tetrachloroethane), an ester soluble in acetone is produced. F. R. ENNOS.

Manufacture of chlorinated organic acid esters of cellulose. I. G. FARBERIND. A.-G. (B.P. 306,132, 7.2.29. Ger., 17.2.28).—A cellulose ester of a saturated

organic acid is chlorinated, *e.g.*, with phosphorus pentachloride, under suitable conditions according to the chlorine content desired, while suspended in an indifferent medium (tetrachloroethane); the product is soluble in organic solvents and stable towards acids. F. R. ENNOS.

Manufacture of rayon of increased whiteness from viscose. DU PONT RAYON CO., Assees. of C. HENNINGSEN (B.P. 301,307, 30.10.28. U.S., 26.11.27).—Sufficient bluish colouring matter or dye is added to the viscose at any stage of its manufacture prior to coagulation and spinning. F. R. ENNOS.

Manufacture of artificial silk. P. VANDEMAELE (B.P. 327,220, 22.1.29).—Artificial filaments produced from viscose etc. are prevented from stretching or slipping while passing over rotary spinning pots by an arrangement of three godet wheels. F. R. ENNOS.

Production of artificial [silk] filaments. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 326,461, 28.9.28).—A fluid stream is suitably directed on to a number of artificial filaments, extruded from spinning orifices into a medium rich in the solvent employed, so that they converge together while in a tacky condition and unite to form one filament of large cross-section. F. R. ENNOS.

Manufacture of artificial threads and webs of the same. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 325,857, 27.8.28).—The solution of cellulose ether or ester in a volatile solvent is passed through nozzles, the straight bore of which is at least 10 times the internal diameter, and thence directly through a liquid containing an organic solvent and a coagulant for the cellulose derivative, *e.g.*, aqueous acetone, so that the threads are only slightly coagulated superficially. The threads, which may be stretched during spinning if desired, are then solidified in the usual air-spinning space. F. R. ENNOS.

Manufacture of artificial products from wood. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 326,502, 12.12.28).—Wood, straw, hay, rushes, etc. are treated with an organic acid halide in the presence of a basic reagent such as a tertiary amine, whereby a mouldable product softening below its decomposition point is obtained. F. R. ENNOS.

Cellulose solutions and their applications. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 326,579, 22.12.28).—Cellulose is dissolved in anhydrous or aqueous methanesulphonic acid at 10–20°, and precipitated in any desired form by means of water. F. R. ENNOS.

[Cellulose binding agent for] joining wood, metal, glass, etc. COMP. FRANÇ. D'EXPLOIT. DES PROC. PLINATUS, Assees. of W. PLINATUS (B.P. 302,324, 14.12.28. Ger., 14.12.27).—A cellulose ester is stirred with an aqueous emulsion of a high-boiling solvent, *e.g.*, an ester such as glyceryl benzoate, and the mixture is heated until the water evaporates; the powdery material obtained is pressed and rolled out to a thin adhesive layer, which is applied between the surfaces to be joined, the whole being subjected to gentle

pressure and increased temperature and allowed to cool under pressure. F. R. ENNOS.

Fireproofing of cellulose. F. S. VIVAS, Assr. to INTERNAT. FIREPROOF PRODUCTS CORP. (U.S.P. 1,738,976, 10.12.29. Appl., 11.3.27).—Cellulosic materials are impregnated with a warm solution of boric acid, borax, ammonium sulphate, and sodium tungstate, and, after partially drying, are immersed in a solution of calcium chloride and dried. F. R. ENNOS.

Production of paper. D. K. PATILLO and J. H. MACMAHON, Assrs. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,737,330, 26.11.29. Appl., 23.12.27).—The stock is sterilised with chlorine while at a point of high dilution, subjected to a coagulating treatment, and formed into paper. F. R. ENNOS.

Smoothing of paper. I. G. FARBENIND. A.-G. (B.P. 310,340, 23.4.29. Ger., 23.4.28).—After spreading with a melted hydrocarbon (paraffin wax), wax, resin, or a mixture of these, the paper is passed over a support heated above the m.p. of the smoothing agent, while removing the excess by means of a scraper. F. R. ENNOS.

Production of [moiré-patterned fabrics] from organic derivatives of cellulose. C. DREYFUS (U.S.P. 1,754,164, 8.4.30. Appl., 4.11.26).—See B.P. 280,195; B., 1929, 279.

Coating of paper-pulp vessels for protection thereof against the action of moisture, fat, soap, etc. F. FRANK (U.S.P. 1,753,565, 8.4.30. Appl., 26.10.25. Ger., 27.10.24).—See B.P. 241,876; B., 1926, 783.

Combined acid channel and thread guide for artificial silk threads. O. (FRH.) VON KOHORN, and A. PERL (B.P. 316,253, 27.5.29. Austr., 26.7.28).

Spinning of artificial silk threads [on to a "ring" or "traveller" spindle]. C. C. TYRER (B.P. 326,452, 11.12.28).

Machine for production of wood pulp for manufacture of paper. MAGNET-WERK G.M.B.H. EISENACH SPEZIALFABR. F. ELEKTROMAGNET-APPARATE (B.P. 304,233 and Addn. B.P. 311,386 and 326,708, [A—C] 9.1.29. Ger., [A] 17.1.28, [B] 6.2.28).

Attrition mills (U.S.P. 1,744,226 and 1,744,235). **Pressure regulator for pumped liquids** (B.P. 309,086).—See I. **Fuel** (U.S.P. 1,743,985).—See II. **Hydrogenation of polyhydroxy-compounds** (B.P. 299,373).—See III. **Laminated glass** (B.P. 326,450).—See VIII. **Lead-coated paper** (B.P. 326,792).—See X. **Wool fat** (B.P. 303,890 and F.P. 569,624).—See XII. **Rubber-fibre articles** (U.S.P. 1,737,133). **Rubber goods** (B.P. 326,782). **Rubbering of fabrics** (G.P. 461,134).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Effect of exposure of wool before dyeing. W. VON BERGEN (The Melliand, 1929, 1, 1084—1093).—Wool containing alkali is more rapidly affected by sunlight than wool which is slightly acid. The behaviour on dyeing of the exposed wool depends on the colour used. CHEMICAL ABSTRACTS.

Action of light on cotton dyed with certain vat dyes. F. SCHOLEFELD and E. H. GOODYEAR (The Melliand, 1929, 1, 1389—1393).—Cotton dyed with indanthrene-yellow G or certain other yellow or orange vat dyes is more susceptible to tendering by light than that dyed with blue or violet vat dyes. The effect, which is attributed to the formation of hydrogen peroxide, is increased by alkali.

CHEMICAL ABSTRACTS.

PATENTS.

Increasing the wetting and penetrating capacity of liquors employed in the textile industry. H. T. BÖHME A.-G. (B.P. 290,256, 10.5.28. Ger., 11.5.27).—Sulphonated fats and oils prepared as described in B.P. 284,280 (B., 1928, 943) are more suitable than is Turkey-red oil as wetting-out agents for addition to liquids which are acid or alkaline or which may contain soluble salts, since they are stable under such conditions and produce a greater corresponding degree of penetration. [Stat. ref.] A. J. HALL.

Immunisation of [cellulose] threads [in wound packages]. CHEM. WORKS, FORMERLY SANDOZ (B.P. 325,961, 26.1.29. U.S., 22.10.28).—Cotton or viscose wound yarn is treated with alkali and esterified in the usual manner without unwinding. Preferably the alkalisated cops are esterified under pressure. C. HOLLINS.

Bleaching of vegetable fibres. R. VIDAL (F.P. 634,962, 24.9.26).—A hypochlorite solution mixed with a soap obtained by the process of F.P. 584,738 (cf. B.P. 250,108; B., 1926, 553) is used. R. BRIGHTMAN.

Bleaching of materials for the manufacture of hats. H. GOLDBERGER (B.P. 293,828, 27.6.28. Austr., 13.7.27. Addn. to B.P. 291,743; B., 1930, 54).—The process described in the prior patent is suitable for the bleaching of hats, and particularly of hare's fur, which have been previously mordanted with the salts of heavy metals such as mercury, zinc, and tin. A. J. HALL.

Treatment [washing, bleaching, etc.] of skeins of artificial silk. J. P. BEMBERG A.-G. (B.P. 312,034 and Addn. B.P. 313,945, [A], 17.9.28, [B] 22.9.28. Ger., [A] 19.5.28, [B] 20.6.28).—(A) Cross-lacing of skeins of artificial silk previous to their treatment with liquids is avoided and entangling prevented by laying the cross-reed skeins in separate layers closely over one another and then exposing them to the wet treatment. The silk yarn may or may not be in a twisted condition, and complete permeation of the skeins by the liquid is obtained. (B) Cross-lacing may also be avoided if the skeins are treated in a stationary condition. A. J. HALL.

Reserving of wool in dyeing with substantive dyes. I. G. FARBENIND. A.-G. (B.P. 304,742, 25.1.29. Ger., 25.1.28).—In the dyeing of mixed fabrics containing wool with direct cotton dyes the wool is reserved by addition to the bath of a small amount (about 5%) of a water-soluble salt of a sulphonic acid obtained (cf. B.P. 314,588; B., 1929, 728) by condensing a natural resin or mixture of resins with an aralkyl halide or substitution product and then sulphonating the product. A. J. HALL.

Production of fast tints on chrome-mordanted fibres. SOC. CHEM. IND. IN BASLE (B.P. 303,384, 31.12.28. Switz., 30.12.27).—Improved fastness to rubbing, light, fulling, and potting results when fully prechromed azo dyes are dyed on chrome-mordanted wool or cotton. Examples are: 5-nitro-*o*-aminophenol \rightarrow Brönner or phenyl-J-acid; 4-nitro-*o*-aminophenol-6-sulphonic acid \rightarrow acetoacetanilide. C. HOLLINS.

Dyeing of fabric. SPECTRUM DYES PROPRIETARY LTD. (B.P. 305,922, 21.8.28. Austral., 11.2.28).—Coloured textile materials are obtained by saturating a dyed fabric with a solution obtained by treating sodium bisulphite with zinc, and then further treating it until of the desired shade with a similar solution after the addition of the product obtained by treating coal-tar naphtha with sulphuric acid. A. J. HALL.

Treatment [coloration] of fabrics. BRIT. CELAN-ESSE, LTD. (B.P. 316,983, 11.8.28. U.S., 13.8.27. Cf. B.P. 295,582; B., 1930, 54).—The process of spraying cellulose acetate materials with dyes in organic solvents described in B.P. 314,208 (B., 1929, 716) is suitable for use with textile materials containing other derivatives of cellulose. A. J. HALL.

Production of dyeings [ice colours from 4-hydroxy- α -naphthyl ketones.] SOC. CHEM. IND. IN BASLE (B.P. 303,179, 29.12.28. Switz., 29.12.27).—Fabrics padded with 4-acylated α -naphthols may be steamed before development; the process is thus suitable for production of variegated effects on mixed cotton-acetate silk goods, and for prints. Phenyl 4-hydroxy- α -naphthyl ketone and aminoazobenzene are padded with alkali on cotton-acetate silk fabric, which is then steamed to fix the aminoazobenzene and developed with diazotised 4-chloro-*o*-aminophenyl phenyl ether to give red (acetate silk) and yellow (cotton) effects. C. HOLLINS.

Dyeing of [cellulose] acetate silk. I. G. FARBENIND. A.-G. (B.P. 320,422, 10.7.28. Addn. to B.P. 293,766; B., 1930, 185).—Processes additional to those described in the prior patent include dyeing with Benzo Brown G, Rhoduline Orange NO, New Methylene Blue F, Bismarck Brown FR extra, Brilliant Rhoduline Violet R, Rhoduline Heliotrope 3B, Methylene Blue BB, and Safranin FF extra in the presence of 3% of hexahydroaniline. A. J. HALL.

Dyeing of esters and ethers of cellulose or of its transformation products. I. G. FARBENIND. A.-G. (B.P. 304,739, 25.1.29. Ger., 25.1.28).—Greenish-yellow shades on acetate silk etc. are obtained by the application of 4-amino-1:8-naphthalimide and its *N*-alkyl, -aryl, or -aralkyl derivatives, *e.g.*, the *N*-ethyl or *N*-*o*-tolyl compound. C. HOLLINS.

Dyeing and printing. SOC. ALSACIENNE DE PROD. CHIM. (F.P. 636,665, 21.10.26).—Aqueous solutions of the sulphurisation product of phenols are mixed with the alkali salts of acid dyes and fixed with acid or acid salts. *E.g.*, a solution of alkali-blue in aqueous soda is mixed with a phenol sulphurisation product obtained according to F.P. 577,653 (cf. B.P. 215,012; B., 1925, 541), and thickened with neutral starch and tragacanth. The fabric is printed, dried, and the dye is then fixed with

dilute acetic, hydrochloric, or sulphuric acid, or with zinc sulphate or aluminium chloride solution. Sulphonated basic dyes can be used. R. BRIGHTMAN.

Printing of textile fabrics [resists against synthetic mordants]. IMPERIAL CHEM. INDUSTRIES, LTD., and L. SMITH (B.P. 325,935, 3.1.29).—Zinc chloride or other metal halides (except alkali halides) are applied as resists against sulphurised phenols. C. HOLLINS.

Preparations for finishing textile fabrics. H. T. BÖHME A.-G. (B.P. 291,094, 12.5.28. Ger., 27.5.27).—Epsom salt finishes on textile fabrics are produced by using the products obtained by sulphonating fats and oils in the presence of organic acid anhydrides or chlorides or of strong anhydrous organic acids (cf. B.P. 274,104; B., 1928, 613). Such sulphonated products are preferred to Turkey-red oil, since they hydrolyse less readily and acid-tendering of the fabric during calendering or similar treatment is thus avoided (cf. also B.P. 293,746; B., 1929, 640). A. J. HALL.

Stabilisation of bleaching liquors. F. DRAISBACH, Assr. to J. A. BENCKISER CHEM. FABR. (U.S.P. 1,754,163, 8.4.30. Appl., 16.8.26. Ger., 14.1.26).—See B.P. 265,417; B., 1927, 252.

Washing, dyeing, mixing, or other machines in which the materials treated are subject to kneading or squeezing action. E. FREUND (B.P. 302,316, 13.12.28. Austr., 14.12.27).

Making pattern collections of textile print and dye works. SOC. FINANCIÈRE & COMMERCIALE SOC. ANON. (B.P. 326,528 and Addn. B.P. 318,600, [A] 11.10.28, [B] 29.10.28. Ger., [A] 9.7.28, [B] 7.9.28).

Purifying etc. apparatus [for dry-cleaners' solvent] (U.S.P. 1,744,747).—See I. Restoring used gasoline (U.S.P. 1,745,108).—See II. Wetting agents (B.P. 326,500).—See III. Increasing the whiteness of rayon (B.P. 301,307).—See V. Bleaching of fatty acids (U.S.P. 1,740,012). Sulphonation product (U.S.P. 1,745,221). Bleaching of beeswax (U.S.P. 1,739,796).—See XII.

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

Preparation of citric acid. M. CATULLO (Boll. Chim. Farm., 1930, 69, 234–238).—The manufacture of citric acid from crude calcium citrate in Italy is described. T. H. POPE.

Ammonium sulphate from gypsum. L. WASILEWSKI, A. KACZOROWSKI, and S. ZABICKI (Przemysl Chem., 1930, 14, 150–158).—The following apparatus for the continuous preparation on a semi-technical scale of ammonium sulphate from gypsum is described. A suspension of gypsum in ammonia is passed through a series of horizontal tubes containing stirrers fixed to a central axis. A temperature gradient is maintained from 14° at the point where the substrates enter to 120° at the point where the products leave the apparatus. The pressure maintained within the system amounts to 2–3 atm. Solutions containing 40% of ammonium sulphate are obtained, using 12 pts. of 18% ammonia to 50 pts. of gypsum, with a throughput

of about 800 kg./24 hrs. Of the sulphuric acid present as gypsum 96.5–98.5% is thus recovered, with a loss of ammonia not exceeding 0.2%.

R. TRUSZKOWSKI.

Preparation of pure monoammonium and monocalcium phosphate from bone phosphoric acid. H. J. BRAUN (Metallbörse, 1929, 19, 1912–1913; Chem. Zentr., 1929, ii, 2927).—Treatment with ammonia removes the impurities; evaporation affords ammonium dihydrogen phosphate. Monocalcium phosphate, however, does not crystallise from solution unless a large excess of acid is present. A. A. ELDRIDGE.

Removal of ions from solutions of calcium dihydrogen phosphate by treatment with hydrous gels of alumina, silica, and their mixtures. J. C. GHOSH and P. B. BHATTACHARYYA (Soil Sci., 1930, 29, 311–322).—Silica gel removes the ions Ca^{++} and $\text{H}_2\text{PO}_4^{--}$ from calcium dihydrogen phosphate solutions in equivalent proportions. The Ca^{++} ions are assumed to be absorbed by valency forces and the resulting electropositive gel removes the $\text{H}_2\text{PO}_4^{--}$ ions by electrical attraction. The absorption of these two ions by alumina gel is much greater than in the case of silica, and the p_{H} value of the gel is increased considerably. This is explained by chemical interaction. Results of the treatment of calcium phosphate solutions with mixtures of alumina and silica gels are recorded and discussed. A. G. POLLARD.

Treatment of sylvite above 100°. W. FROELICH and E. RITTER (Mitt. Kali-Forsch.-Anstalt, 1929, 67–89; Chem. Zentr., 1929, ii, 2485).—A study of the separation of salts on cooling saturated solutions of sodium and potassium chlorides and its application to the working-up of sylvite. The maximum solubility of sodium chloride in saturated potassium chloride solutions is at 80°. At solution temperatures below 150° potassium chloride can be obtained free from sodium chloride.

A. A. ELDRIDGE.

Rotatory furnace for extraction of aluminium salts from clay. L. WASILEWSKI and J. Z. ZALESKI (Przemysl Chem., 1930, 14, 181–188).—A rotatory furnace for the continuous preparation by a modification of Rinman's method is described. A mixture consisting of 4 pts. of ammonium sulphate to 1 pt. of alumina present in the clay is passed through a furnace the distal three fourths of which is maintained at 400°; where the time of passage of the reacting mass is 30 hrs. 88% yields of alum are obtained, with only very small losses of ammonia. Higher temperatures cause decomposition of ammonium sulphate, but it is probable that prolongation of the duration of reaction would give practically theoretical yields. The emerging product (at about 95°) is at once placed in water, in which the alum dissolves, leaving a residue of silica, suitable for the preparation of water-glass. The extract, on keeping, deposits fairly pure alum, the iron content of which is, after one recrystallisation, 0.002% FeO; this degree of purity suffices for the preparation of pure aluminium.

R. TRUSZKOWSKI.

Extraction of copper from residues of copper sulphate manufacture. C. FICAI (Rass. Min. Met. Ital., 1929, 69, 4–7; Chem. Zentr., 1929, ii, 2931).—A

method depending on the roasting of the residues and extraction of the cupric oxide with dilute sulphuric acid is described. Iron and arsenic are removed from the solution before crystallisation; the noble metals remain in fairly pure form in the residue. A. A. ELDRIDGE.

Determination of small amounts of silver in pyrolusite. A. KUNDERT (Chemist-Analyst, 1930, 19, No. 1, 11).—The silver is extracted by repeated treatment of the ore with concentrated hydrochloric acid; the solution is neutralised with ammonia, treated with hydrogen sulphide, the precipitate washed with cold 10% sulphuric acid, ignited, and the ash treated with nitric acid. The silver is precipitated with hydrochloric acid, freed from lead by means of ammonia solution, and dissolved in potassium cyanide solution, this solution then being electrolysed. CHEMICAL ABSTRACTS.

Solubility of salts in water and in lyes at temperatures below and above 100°. W. FROELICH (Mitt. Kali-Forsch.-Anstalt, 1929, 37–66; Chem. Zentr., 1929, ii, 2485).—The solubilities of potassium chloride, sodium chloride, magnesium sulphate, and of sodium and potassium chlorides in water, of magnesium sulphate in aqueous magnesium chloride solutions, of sodium and potassium chlorides in technical sylvite extraction liquor, and of that mixture and carnallite in van't Hoff's liquor Q20 have been determined.

A. A. ELDRIDGE.

Absorption of carbon monoxide by cuprous ammonium salts. W. GUMP and I. ERNST (Ind. Eng. Chem., 1930, 22, 382–384).—A large number of absorbents for the extraction of carbon monoxide from water-gas were examined from the point of view of the commercial preparation of hydrogen or carbon monoxide. Various mixtures containing cuprous ammonium salts were studied and found to be unsatisfactory, and then a large number of organic acids and phenols as substitutes for the carbonate in cuprous oxide-ammonium carbonate-ammonia mixtures were examined. Cuprous ammonium lactate was found to be the most satisfactory absorbent, and the mixture with ammonia did not attack iron and was stable. The absorptive power remained the same after 20 regenerations, in which the solution was heated to 80° to expel carbon monoxide. A solution containing 13 g. of cuprous oxide, 25 g. of lactic acid, 85 c.c. of ammonia solution (d 0.910), and 100 c.c. of water absorbed 15 vols. of the gas at 0° or 0.99 mol./mol. Cu_2O . Experiments on a semi-works' scale showed that carbon monoxide of 97–99% purity could be produced. No trouble was experienced in an iron plant when run for several weeks, and no deposition of copper took place. The same method was then used successfully in a large-scale plant. H. INGLESON.

Reliability of the Reich-Raschig method of determining sulphur dioxide. H. E. WOISIN (Z. angew. Chem., 1930, 43, 293–294).—The reliability of Raschig's modification of Reich's method of determining sulphur dioxide, in which the error caused by the presence of oxides of nitrogen, as in sulphuric acid chamber gases, is claimed to be suppressed by the use of sodium acetate, was examined. It was found that if oxides of nitrogen were absent, identical results were obtained with or without sodium acetate, and whether

the gas was drawn through the iodine slowly or quickly. With concentrations of 0.7% N_2O_3 , however, results by Raschig's method were seriously below the truth, although not so low as in absence of sodium acetate, and the slower the gas current the higher was the sulphur dioxide figure obtained. For Gay Lussac exit gases the values were particularly faulty and this test should only be used with great caution in calculating the output of units in a sulphuric acid plant. C. IRWIN.

Volatilisation of phosphorus from phosphate rock. II. Volatilisation of phosphorus and potash in a blast furnace. III. Calculations of performance of a blast furnace for volatilisation of phosphorus and potash. R. D. PIKE (Ind. Eng. Chem., 1930, 22, 344—354).—II. Two experimental runs of a blast furnace indicated that most of the reduction of phosphate was effected in the bosh of the furnace, any reduction by carbon monoxide in the shaft being of minor importance. Cold oxygenated blast containing 40—45% O proved most suitable, though a hot blast sufficiently oxygenated to give the same theoretical temperature of combustion would be preferable. A free-running slag closely resembling that of an iron blast-furnace should be tapped at 1450—1500° and from smaller-scale trials extractions of 97% P_2O_5 and 92% K_2O can be reasonably expected with continuous smooth operation. As the only function of the shaft is to preheat the charge, a squat type of furnace, probably not taller than 20—25 ft., is indicated.

III. The design of a suitable furnace would be similar to but lower than a standard, iron, blast furnace except that the blast would contain 30% O. The gases would then pass through a waste-heat boiler to burn phosphorus to oxide and then to a Cottrell precipitator in which the mixture of phosphorus pentoxide and potassium phosphate would be collected in liquid form. The slag must be fluid and basic to volatilise potash, which is apparently eliminated by the direct replacement by lime without the intervention of carbon. When supplies of coke and potash-bearing flux are favourable, the blast-furnace process appears to offer distinct economic advantages over the present sulphuric acid method for making phosphoric acid. C. A. KING.

Pneumatic transport. NEU. **Thermal data in drying-tower design.** NEWMAN.—See I. **Rare elements in clays.** FIOLETOV.—See VIII. **Electro-reduction of silver chloride.** YAGI.—See X. **Preparation of basic copper sulphate.** HOLLAND and others.—See XVI.

PATENTS.

Separation of mixtures of sulphuric acid and nitric acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,977, 28.2.29).—Alkali sulphate or nitrate is added in quantity sufficient to form alkali hydrogen sulphate with the sulphuric acid. The separation of the alkali hydrogen sulphate is preferably assisted by cooling, but in order to prevent separation of alkali nitrate as well it is frequently necessary to remove part of the water from the mixture by evaporation.

S. K. TWEEDY.

Production of hydrocyanic acid. T. S. WHEELER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 325,860, 3.9.28).—Hydrocarbons, such as methane or coal gas,

are heated to at least 800° before or after admixture with (preferably excess of) nitrogen or ammonia, and the hot mixture is subjected to an arc or spark discharge. The gas mixture is preferably free from oxygen.

S. K. TWEEDY.

Production of cooling brine. H. STRATMANN and F. G. WERNER (STRATMANN & WERNER) (B.P. 327,039, 1.5.29).—Brine is rendered permanently neutral by adding "buffers," e.g., an acid component such as sodium acetate, and an alkaline component such as a primary phosphate. Alternatively, an amphoteric electrolyte, such as an amino-acid, may be added.

S. K. TWEEDY.

Production of sodium metaborate. A. KELLY (B.P. 326,378, 9.4.29).—Boron mineral (e.g., crude borax) is heated for 5 hrs. at 80° with sodium carbonate, lime, and water. The sodium metaborate solution formed is filtered off, bleached with hypochlorite, if necessary, and crystallised.

S. K. TWEEDY.

Manufacture of potassium nitrate. KALI-FORSCHUNGS-ANSTALT GES.M.B.H. (B.P. 327,047, 10.5.29. Ger., 11.2.29).—A solution containing nitric acid is treated with potassium chloride at ordinary or raised temperatures in quantities such that at ordinary temperatures merely potassium nitrate separates out, and approximate saturation exists with respect to potassium chloride. The solution, if desired after separation of the potassium nitrate, is treated with nitrous gas (e.g., produced by the combustion of ammonia) and air until the initial nitric acid concentration is regained, nitrosyl chloride being formed also. The latter, separated from inert gases by absorption in silica gel, ferric chloride, etc., is decomposed with formation of nitric oxide which is employed to enrich the nitrous gases. The process may be conducted continuously.

S. K. TWEEDY.

Production of zinc salt solutions. METALLGES. A.-G. (B.P. 302,924, 20.12.28. Ger., 23.12.27).—The zinc oxide material is treated with a quantity of acid which is less than the equivalent of the zinc present, in such a manner that excess of acid with respect to zinc is always avoided. Thus the acid may be added continuously or intermittently to the oxide material, e.g., in the form of a paste, with continuous stirring, the temperature preferably never exceeding 40°. Further zinc oxide and acid may also be added to the mass under conditions adapted to prevent local excess of acid. The well-stirred mixture is then immediately filtered or centrifuged.

S. K. TWEEDY.

Manufacture of anhydrous zinc chloride by reacting zinc oxide with chlorine. C. VON GIRSEWALD and H. NEUMARK (U.S.P. 1,743,740, 14.1.30. Appl., 27.3.28. Ger., 9.4.27).—The reaction is effected in the presence of hydrogen, or a gas containing hydrogen, e.g., water-gas, the zinc chloride being distilled off, or drawn off from the reaction vessel in the molten state. The zinc oxide, in a finely-divided condition, may be carried into the reaction vessel in the stream of hydrogen.

F. G. CLARKE.

Purification of natural heavy spar. SACHTLEBEN A.-G. F. BERGBAU U. CHEM. IND. (B.P. 316,542, 22.4.29.

Ger., 30.7.28).—The spar is calcined under reducing conditions at 1300–1350° and quenched. The barium sulphide formed in small quantity during the calcination converts the metal oxide impurities during quenching into metal sulphides, which are readily removed by treatment with acid. Any quartz present is also removed. If the spar is non-bituminous it is mixed with bituminous spar or carbonaceous material before calcination. However, the impure spar, bituminous or non-bituminous, may also be calcined at 1300–1350° in an oxidising or reducing atmosphere and quenched in dilute sulphide solutions, *e.g.*, barium sulphide solutions. S. K. TWEEDY.

Manufacture of chromium oxide. I. G. FARBENIND. A.-G. (B.P. 302,178, 10.12.28. Ger., 10.12.27).—Alkali chromate or dichromate is mixed with red phosphorus in quantity insufficient for the appreciable formation of chromium phosphates and the whole is ignited, with or without preheating at 300°. Reducing agents (carbon or sulphur) may be added to combine with the liberated oxygen. Preferably a mixture of phosphorus and potassium dichromate is made into a paste with at most 20% of water and the whole caused to react. The oxide produced is eminently suitable as pigment. S. K. TWEEDY.

Impregnation of porous gels with (A) metals or other insoluble material, or (B) solid material. H. N. HOLMES (U.S.P. 1,739,306—7, 10.12.29. Appl., 13.8.27).—(A) The gel is impregnated, cold, with a solution of a catalyst-yielding compound containing also a reagent, which at an elevated temperature reacts to give the required insoluble deposit upon the gel. *E.g.*, silica gel is soaked in a solution of ammoniacal silver nitrate containing formaldehyde. The temperature is then raised to 100° to precipitate the metal. The pores in the interior of the gel thus become impregnated, since the surface is not choked by deposited metal. (B) The gel is impregnated with a solution of the metal salt etc., *e.g.*, ferric chloride, and then dried. A gas, *e.g.*, ammonia, which will react with the metal salt when in solution to give the catalyst, is introduced into the gel, and the latter then immersed in water, washed, and dried. F. G. CLARKE.

Manufacture of water-softening material of the exchange-silicate type. F. K. LINDSAY, Assr. to ARIZONA MINERALS CORP. (U.S.P. 1,744,703, 21.1.30. Appl., 19.2.25).—Finely-divided non-reacting material, *e.g.*, sand or base-exchange silicates which have fallen to powder, is added to a well-stirred mixture of solutions containing, respectively, sodium silicate and a sodium salt containing another metal, *e.g.*, sodium aluminate, so that gel formation takes place around the added particles; the gel is drained, dried for about 12 hrs. at the usual temperature, and then at about 60° in a current of warm air, preferably in a revolving drum, so that the product is obtained in a granular, non-powdery form. L. A. COLES.

Manufacture of complex antimony salts. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,176 and 326,231, [A] 5.12.28, [B] 6.12.28).—(A) Antimonic acid, freshly precipitated from the penta-

chloride, reacts with aliphatic hydroxy-acids (tartaric, gluconic, mucic, lactic, citric, etc.) to give relatively non-toxic complex salts of quinquivalent antimony. An organic base (hydroxytriethylamine) may be present. (B) Complex metal salts of aliphatic hydroxy-acids are treated with oxidants, *e.g.*, hydrogen peroxide, magnesium peroxide, to give less toxic compounds. An organic base, *e.g.*, diethylamine, may be present.

C. HOLLINS.

[Removal of carbon monoxide and dioxide from gas mixtures for use in] synthetic ammonia process. J. G. DELY, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,739,217, 10.12.29. Appl., 19.9.23).—Gas mixtures produced, *e.g.*, as described in the De Jahn process (Can.P. 201,059; cf. B.P. 120,546; B., 1920, 231 A) are brought, without pretreatment with hot alkali solutions, in contact with ammoniacal cuprous (carbonate) solutions at high pressure and at 0° to remove carbon monoxide and dioxide, final traces of the latter gas being removed, if necessary, by subsequent scrubbing with cold sodium hydroxide solution.

L. A. COLES.

Continuous manufacture of carbon monoxide. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,227, 1.12.28).—Oxygen is blown into coke packed in a generator at a velocity (*e.g.*, 50 m./sec.) such that the primary reaction zone is rendered conical in form and is blown away from the nozzle and kept within the coke away from the generator walls. The ash of the coke is carried off as a vapour, containing combined sulphur (for the combination of which basic substances, like quicklime, may be added), and is separated from the carbon monoxide by washing. S. K. TWEEDY.

Production of [volatile inorganic] acids. H. FRISCHER (U.S.P. 1,754,156, 8.4.30. Appl., 16.5.27. Ger., 26.5.26).—See B.P. 271,881; B., 1928, 90.

Catalysts containing phosphoric acid (B.P. 326,185).—See I. **Acid-resistant lead** (B.P. 326,818 and 327,106).—See X.

VIII.—GLASS; CERAMICS.

Dependence of transparency of copper ruby glass on annealing time and temperature and the concentration of copper. L. RIEDEL and E. ZSCHIMMER (Keram. Rundsch., 1929, 37, 197–200, 237–239, 270–272, 545–547, 581–582, 634–636; Chem. Zentr., 1929, ii, 2488).—The temperature function of transparency for λ 5500 Å. was determined; the minimum is at 685°. Debye diagrams showed that the colouring matter of the glasses is metallic copper. It is technically desirable that the transparency to green light should be small; this is attained by increasing the copper content and annealing time and maintaining the most favourable temperature as exactly as possible.

A. A. ELDRIDGE.

Occurrence of rare elements in clays. A. FIOLETOV (Keram. Rundsch., 1927, 35, 270–272; Chem. Zentr., 1929, ii, 2489).—Primary Russian clays contain 0.22–1.35% TiO₂, and secondary clays 0.94–2.72%. The clays and kaolins examined contained 0.004–0.075% V₂O₅. A method for the determination of vanadium in clays is described. A. A. ELDRIDGE.

Glass distillation apparatus. OTHMER.—See I. [Ash from] rice husks. DE MONGEOT.—See XIX.

PATENTS.

Treatment of glassware [to prevent "bumping"]. J. A. JOBLING & Co., LTD., and G. L. HOCKENYOS (B.P. 307,793, 22.2.29. U.S., 13.3.28).—The whole or a part of the inner surface of beakers, coffee urns, etc. is roughened by sandblasting, frosting, etching, etc.

L. A. COLES.

Manufacture of safety glass. T. O. HENZE (B.P. 326,926, 9.5.29).—Two sheets of glass, the inner surfaces of which have been treated with an aqueous solution of isinglass, and an intermediate sheet of cellulose which has been treated with a solution of celluloid, urea, and formaldehyde in amyl acetate, are united, after the solvents have evaporated, in a bath of alcohol by the application of a pressure of 2–3 lb./in.² at about 15°; the edges are subsequently sealed.

L. A. COLES.

Cellulose method of laminating glass. L. BARTELSTONE (B.P. 326,450, 10.12.28).—Sheets of glass with their inner surfaces conditioned by spraying with a solution of a cellulose derivative (cellulose nitrate) free from camphor, and having an intermediate sheet of celluloid conditioned superficially by spraying with a celluloid solution, are cemented together under heat and pressure, after the solvents have evaporated, by an expressed vegetable oil, *e.g.*, castor oil.

L. A. COLES.

Manufacture of white enamels [for sheet or cast metal]. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 310,528, 18.4.29. Ger., 28.4.28).—The highly basic complex compounds of oxides of antimony, zirconium, tin, or titanium, etc., containing the radical of thermally decomposable acid or acids (*e.g.*, nitric or formic), with or without salts of such acids, may be employed in quantities of at most 6% for rendering white and opaque enamels which are free from, or deficient in, fluorine. The opaquing action of the media rises to a definite limit with increase in the content (up to 0.5–3%) of the acid or acids unstable at incandescence.

S. K. TWEEDY.

Removal of sulphates from clays or like argillaceous materials. A. L. MOND. From KALI-CHEMIE A.-G. (B.P. 326,236, 8.12.28).—Barium salts (*e.g.*, carbonate, or more particularly fluosilicate) are added to the argillaceous material and the precipitation of the soluble sulphates is accelerated by an addition of soluble chlorides (*e.g.*, sodium chloride).

S. K. TWEEDY.

Ceramic mass. J. M. LAMBIE and D. W. ROSS (U.S.P. 1,745,102, 28.1.30. Appl., 16.10.24. Renewed 14.11.28).—A ceramic casting slip of suitable composition is prepared with the use of a deflocculating mixture which contains ammonia solution, a basic compound of a metal, *e.g.*, litharge, and an organic polyhydroxy-compound of an acid character having at least two adjacent hydroxyl groups. Suitable reagents include oxalic acid, citric acid, gallic acid, mucic acid, pyrocatechol, quinol, and pyrogallol.

C. A. KING.

Drying of ceramic articles in drying chambers and apparatus therefor. K. LÜHMANN (U.S.P. 1,744,398, 21.1.30. Appl., 30.9.27. Ger., 2.10.26).—A transverse flue passes beneath a battery of parallel

drying chambers, by which means hot air may be introduced into any one chamber and successively into others according to the hygroscopic and temperature needs of the material, *e.g.*, clayware, to be dried.

C. A. KING.

Refractory materials. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 326,279, 1.1.29).—High spalling-resistance is conferred to articles of refractory material (*e.g.*, fused alumina) by bonding the latter with an interlocking crystalline material other than the said refractory material. The crystalline material may be a titanium compound (titania alone or combined with magnesia) or an alkaline-earth oxide mixed with titania, or with a silicate, or with calcium fluoride.

S. K. TWEEDY.

Manufacture of fire-resisting material. W. T. WINCKLER, ASSR. to VON WINKLER CHEM. Co. (U.S.P. 1,743,176, 14.1.30. Appl., 2.2.27).—A mixture of silica 10 pts., lead oxide 8 pts., borax 8 pts., and calcium carbonate 1 pt. is fused to a glass, pulverised, and mixed with a fibrous material, *e.g.*, asbestos. The mixture is then heated until the flux fuses and is then pressed.

C. A. KING.

Refractory articles. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 326,560, 20.11.28).—The articles consist of, *e.g.*, silicon carbide and a composite binder, both stable under reducing conditions at 1300–1600°, the constituents of the binder being mutually soluble in varying proportions so that the binder matures continuously over a range of at least 300° between 1150° and 1900°, giving a m.p. curve approximating to a straight line over the same temperature range, and yielding a binder having a porosity at 1300° differing by less than 1% from that at 1500°. Suitable binders comprise mixtures of a (calcium) fluoride and a metal oxide (alumina or magnesia), and of diopside and forsterite.

L. A. COLES.

Refractory structure and article and method of forming the same. F. L. ARENSBURG, A. J. JACKMAN, and C. L. JONES, ASSRS. to VESUVIUS CRUCIBLE Co. (U.S.P. 1,743,803, 14.1.30. Appl., 21.8.28).—Crucibles particularly adapted for melting steel in induction furnaces are made from chrome ore, magnesite, etc., with a porosity of at least 20%, the voids being lamellar in character. The pores are formed by introducing flat or flaky organic material, *e.g.*, bran, flat seeds, or fish scales, into the raw mixture, orientating the flat plates in a direction substantially parallel to the surface of the crucible wall.

C. A. KING.

Furnaces for annealing glass and other articles. T. TEISEN (B.P. 326,636, 19.2.29).

Joining glass to wood etc. (B.P. 302,324).—See V.

IX.—BUILDING MATERIALS.

Asphalt road emulsions. V. SHIPEROVICH, G. SHAVIRO, and O. PIPIK (Azerbaij. Neft. Choz., 1929, No. 10, 36–46).—The production of stable asphalt-water emulsions with "kontakt," naphthenic acids, or acid fuel is described. To the bitumen at 105–120° is added the emulsifying agent, and the mixture is poured into aqueous sodium hydroxide at 50–60°.

CHEMICAL ABSTRACTS.

Standards for and methods of testing of Polish road asphalts. M. MACZYŃSKI and W. SKALMOWSKI (Przemysł Chem., 1930, 14, 121—131).—The following official standards are given for Polish road asphalts respectively for the surface and basis of the road: d_{25}^{25} 1.220, 1.235 (± 0.002); viscosity (Hutchinson) 3—15, 20—100; free carbon 5—16, 5—18%; water 0.05, 0.5% (± 0.05); distillates boiling below 170° 1, 1%; at 170—270° 12—17, 8—18%; at 270—300° 4—12, 6—12%; at 300—350° 14—27, 6—16%. In both cases the products had: residue 55—65%, sintering point 60—75°, phenols 4% \pm 1, naphthalene 4% \pm 1, and ash 0.5%. Standard methods recommended for the testing of such asphalts are given in detail. R. TRUSZKOWSKI.

Cement from pyrites cinder. J. A. ALEKSANDROV (Nef. Choz., 1929, 17, 193—197).—A cement made from limestone (13), clay (1.3), and cinder (1 pt.) was resistant to sulphur compounds and gave the following values: volatile matter trace, SiO_2 24.22, Fe_2O_3 6.15, Al_2O_3 3.05, CaO 66.18, MgO trace, SO_3 0.35, R_2O 0.05%; d 3.26, hydraulic modulus 1.98%, silicate modulus 2.63%, setting time (beginning) 4 hrs. 55 min., (final) 6 hrs. 20 min. CHEMICAL ABSTRACTS.

Constitution of hydrated Portland cement G. ASSARSSON and N. SUNDIUS (Sver. geol. Unders., 1929, 23, 7 pp.; Chem. Zentr., 1929, II, 2490).—In setting and hardening, the compound $3\text{CaO} \cdot \text{SiO}_2$ liberates 1 mol. of calcium oxide to the water.

A. A. ELDRIDGE.

Pine wood. P. M. SOUM (Bull. inst. Pin, 1929, 221—230, 253—264, 313—316).—Chiefly an account of work already published. CHEMICAL ABSTRACTS.

Pneumatic transport. NEU.—See I. **Waste-wood utilisation.** NELSON. **Natural gas [for lime kilns].** KERTÉSZ.—See II.

PATENTS.

Production of cement. G. IMAI (B.P. 319,342, 15.2.29. Jap., 21.9.28).—A mixture of calcined igneous rock containing quartz and felspar with a substance containing large quantities of soluble acidic components, e.g., naturally or artificially weathered tuff, basalt, or andesite, is ground and mixed with Portland cement clinker, and the mixture is pulverised. L. A. COLES.

Manufacture of plaster for building and like purposes. E. JOSZ (B.P. 326,625, 5.2.29).—A mixture of plaster of Paris, 0.6% by wt. of powdered erythro-dextrin, and 0.5—1.2% of sodium borate is tempered by the addition of water, or, alternatively, the dextrin and sodium borate are dissolved in the water used for tempering the plaster. L. A. COLES.

Production of moulded articles. BELL'S UNITED ASBESTOS CO., LTD., J. A. CANN, and E. R. HARRAP (B.P. 326,825, 16.11.28).—Compositions for moulding under heat and pressure comprise finely-divided mixtures of alkaline-earth or magnesium compounds which set by combination with water (e.g., plaster of Paris, Portland cement, hydraulic or fat limes, magnesia) together with salts containing water of crystallisation, especially heavy-metal sulphates, chlorides, phosphates, and nitrates; compounds which react under heat and pressure with the heavy-metal salts (e.g., zinc oxide or

magnesia when zinc sulphate is used), as well as fillers, colouring material, etc. may also be added.

L. A. COLES.

Manufacture of [waterproofing] aqueous dispersions [of bitumens etc.]. L. KIRSCHBRAUN (U.S.P. 1,738,776, 10.12.29. Appl., 6.10.27).—Aqueous dispersions containing, e.g., asphalt with clay as the dispersing agent are stabilised by the presence of a silica gel formed *in situ* by the addition of sodium silicate solution and hydrochloric acid to the dispersing agent or to the dispersion, the quantity of acid being such that the product has p_H 3.0—3.5 when normal brick-making clays are used, or somewhat higher (e.g., 6.0—6.5) with clay having poor suspensive properties. L. A. COLES.

[Cold] bituminous paving. (SIR) G. C. MARKS. From ROL LISTER & CO. (B.P. 326,863, 23.11.28).—Preheated and dried mineral aggregate, which may contain 5—20% of fine dust or "filler," is coated with bitumen previously softened by heating with sufficient refined tar or bituminous or asphaltic base oil to yield a matrix of m.p. 15—25°.

L. A. COLES.

Preparation of cellular building material. G. M. THOMSON, ASSR. to CANADA GYPSUM & ALABASTINE, LTD. (U.S.P. 1,753,255, 8.4.30. Appl., 20.12.26).—See B.P. 305,806; B., 1929, 325.

Bituminous compositions (U.S.P. 1,738,906).—See II. **Joining wood to metal etc.** (B.P. 302,324).—See V. **Insecticide [for wood]** (U.S.P. 1,744,324).—See XVI.

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

Electric-furnace iron. G. L. SIMPSON (Amer. Electrochem. Soc., May, 1930. Advance copy. 6 pp.).—The present status of the electric furnace in the iron foundry is discussed with special reference to continuous processes which have become important in recent years. Examples are given of the duplex process in which the metal is first melted in the cupola and of the process in which the charge is melted in the electric furnace itself.

H. J. T. ELLINGHAM.

Influence of phosphatisation and a finish on the electrical insulation of steel plates. J. COURNOT (Compt. rend., 1930, 190, 934—936).—As ferrous alloys phosphatised by the author's method (cf. B., 1928, 18) showed poor superficial conductivity for electrodeposition, the electrical effect of phosphatisation has been investigated. Phosphatised plates covered with a thin layer (0.15—0.3 mm.) of an insulating varnish or of bakelite, and an unphosphatised plate for comparison, were placed (i) dry, and (ii) after being 24 hrs. in a saturated atmosphere, between electrodes by which was applied (a) an alternating current (50 cycles) of voltage increasing until rupture occurred, and (b) a direct current of 300 volts, the current passing being measured. The voltage for rupture was lower (1/2 to 1/9), whilst insulation was much greater (10—100 times for dry, and more for moist, plates) in the phosphatised plates.

C. A. SILBERRAD.

Properties of special steels. K. SASAKAWA (J. Iron Steel Inst. Japan, 1929, 15, 201—224).—A study of

the change of structure and hardness on quenching specimens of nickel, chromium, nickel-chromium, and nickel-chromium-molybdenum steels.

CHEMICAL ABSTRACTS.

Permeability of electric steel. M. NAKAMURA (J. Iron Steel Inst. Japan, 1929, 15, 95—123).—In cast steel the permeability (determined with Baro's permeameter) increases with decrease (up to a point) of the carbon content; below 0.12% C the permeability is generally low. Silicon somewhat increases the permeability, the limit depending on the manganese and carbon contents; its effect is that of deoxidation.

CHEMICAL ABSTRACTS.

Dilatometric analysis of certain nickel, vanadium, and nickel-vanadium steels. J. CHALLANSONNET (Compt. rend., 1930, 190, 939—940).—The steels examined all contained approx. 3.75% C, (a) had no further addition; (b) and (c) contained, respectively, 1 and 2% Ni; (d) and (e) 0.25 and 0.5% V; and (f) 1 or 2% Ni each with 0.25 or 0.5% V. They were all poured at 1500° into cylinders 8 or 25 mm. in diam. and 95 or 90 mm. high. Photomicrographs showed the carbon in all cases to be combined, and (a—c) showed eutectic cementite and primary pearlite in dendrites. All those containing vanadium exhibited lamellæ of pre-eutectic cementite and a network of eutectic cementite. On examination with Chevenard's dilatometer (Pyros standard), it was found that nickel had no effect on the Curie point (200°), but 0.25 and 0.5% V lowered it, respectively, to 160° and 130°. Nickel lowers the Ac and Ar points; vanadium alone has no effect, but when present with nickel it cancels the effect thereof. Nickel lowers the temperature of graphitisation, vanadium in amount equal to one quarter that of the nickel cancels its effect; alone it prevents graphitisation, at least up to 1100°.

C. A. SILBERRAD.

Change in density and electrical conductivity of copper during polishing. P. SIEBE (Metall u. Erz, 1929, 26, 397—400; Chem. Zentr., 1929, ii, 2931).—The density is maximal at 0.2% O, falling on overpolishing owing to absorption of hydrogen and sulphur dioxide. The electrical conductivity increases with falling oxygen content, further rising and then falling on overpolishing. In general, the danger of overpolishing is the greater the purer is the copper.

A. A. ELDRIDGE.

Determination of copper in commercial aluminium. J. C. GHOSH (J. Indian Chem. Soc., 1930, 7, 125—126).—The aluminium is dissolved in dilute (1:5) sulphuric acid, when the copper remains, and is subsequently determined iodometrically, after washing and dissolution in nitric acid. Results are quoted showing that the method compares favourably with existing methods.

J. R. I. HEPBURN.

Influence of silicon on the mechanical properties of aluminium bronze. S. SHIOZAWA (J. Min. Met., Japan, 1929, 45, 217—220).—With 2% Al, the effect of silicon is small; with 4% Al, over 1.5% Si rapidly increases the tensile strength. Experiments with aluminium bronzes containing 0—3% Si and 2—12% Al are recorded.

CHEMICAL ABSTRACTS.

Analysis of aluminium alloys. F. L. HAHN (Z. anal. Chem., 1930, 80, 192—200).—The alloy (0.5 g.) is

placed in a boat in a silica tube and heated in a current of chlorine charged with the vapour of carbon tetrachloride, a receiver containing isopropyl alcohol being connected with the tube. The solid sublimate condensing in the cooler front portion of the tube contains the whole of the iron, nearly all the aluminium, and a little titanium, whilst the liquid in the receiver contains all the silicon and the remainder of the titanium; a small residue of alumina [? also calcium and magnesium] remains in the boat. The constituents present in the above three fractions are then determined by the usual methods. Calcium, sodium, and magnesium, if present, are determined in a separate portion of the alloy after dissolution in hydrochloric acid. Attempts to obtain a direct determination of the oxygen present were not successful, but it is concluded that the alumina remaining in the boat represents that originally present in the alloy as corundum. A detailed description is given of the method and of the apparatus employed.

H. F. HARWOOD.

Magnesium-manganese alloys. G. W. PEARSON (Ind. Eng. Chem., 1930, 22, 367—370).—As a method of preparing alloys, rather large pieces of manganese were immersed in molten magnesium and the temperature was maintained at 750° for some hours. The solid-solution area extends beyond 2.7% Mn, and in the higher ranges manganese may appear as angular masses. When the alloy contains 35% Mn or more the manganese shows as irregular masses in a background of magnesium.

C. A. KING.

The Betts electrolytic lead-refining process in practice. J. J. FINGLAND (Amer. Electrochem. Soc., May, 1930. Advance copy. 28 pp.).—Details of modern practice in the operation of the Betts process are described with special reference to the methods employed by the Consolidated Mining & Smelting Co. of Canada at Tadenac, B.C. Concrete cells lined with asphalt have now replaced wood, which is eliminated as far as possible from the cell room. The cells are arranged in cascade units through which the electrolyte is circulated by means of copper pumps with monel metal wearing parts. At Tadenac the anodes are arranged with their centres 10.5 cm. apart and usually have a life of 8 days, although 6-day anodes are sometimes adopted in obtaining specially pure lead. Two crops of cathodes are drawn from each set of anodes. The electrolyte in circulation contains an average of 12% of total hydrofluosilicic acid and 8% of lead. The anodic current density is normally 1.8—1.9 amp./dm.² To meet the stringent requirements of the white lead industry the silver content of the refined metal has to be kept extremely low. For this reason the percentage of silver in the anodes must not be too high and the anodes cannot be run below about 20% of scrap. On the other hand, the amount of impurities in the anodes should not be too low since this leads to a slime which does not settle well and may therefore be included in the cathode deposit. It is found that the best conditions are obtained when the anodes contain about 0.85% Sb, for this forms on the anode surface an adherent sponge, which includes most of the other impurities. Hence the anodes are made to contain this proportion of antimony, if necessary by adding antimonial lead or

antimony recovered from the slimes. The total impurities in the anodes are usually less than 1.25%. Under these conditions the initial potential drop between anodes and cathodes is 0.35–0.45 volt, but as the thickness of the layer of slime increases, the resistance rises and the potential-drop reaches 0.65–0.70 volt at the end of the life of the anode. This increase in resistance is partly due to the concentration of lead fluosilicate in the pores being greater than that in the bulk of the electrolyte while the concentration of free hydrofluosilicic acid is lower. The origin of these concentration differences is discussed. The electrolyte must therefore be regarded as consisting of two parts: a portion which circulates and a portion which is stationary in the slime sponge. At Tadenac the average proportion of total hydrofluosilicic acid in the former to that in the latter is 100:15. For economic success all the hydrofluosilicic acid entrained in the sponge must be returned to the electrolyte. The proportion of lead fluosilicate to free hydrofluosilicic acid in the filtrate from the slimes washing process is adjusted by addition of sulphuric acid. The slimes are dried and melted in a reducing atmosphere, whereby some lead antimonite is slagged off. Then by the action of a jet of compressed air the arsenic and most of the antimony are driven off as oxides, which are recovered by means of a Cottrell precipitator. The rest of the antimony is removed as a lead antimonite slag, after which bismuth and copper undergo oxidation yielding a slag which is worked up for bismuth. The bismuth is recovered electrolytically. The residue of doré metal is parted by the sulphuric acid process to obtain silver. The gold sludge is cast into anodes and refined by the Wohlwill process, whereby some palladium is recovered. The cathode lead obtained in the main process is melted and then treated with compressed air at 540°. Antimony is thereby reduced to 0.001% and small amounts of arsenic and tin are completely eliminated. The hydrofluosilicic acid is best prepared by passing hydrofluoric acid vapours, produced by the action of sulphuric acid on pure fluor-spar, into water containing finely-crushed quartz or silica in suspension. Best French glue is used as addition agent. The composition of the electrolyte must be carefully controlled and rapid volumetric methods which have been developed for determining the concentrations of the various constituents are described.

H. J. T. ELLINGHAM.

Electrolysis of gold, electrolytic reduction of silver chloride, bronze plating, cadmium plating, and plating with copper-cadmium alloy. H. YAGI (J. Mint, Japan, 1929, 2, 56–77).—The composition and method of stirring the gold electrolyte were studied. Electrolytic reduction of silver chloride, using a silver cathode and a lead anode in sulphuric acid in a clay crucible (diaphragm), is convenient, but more expensive than reduction with zinc and sulphuric acid. Conditions suitable for bronze, cadmium, and copper-cadmium alloy plating are recorded. CHEMICAL ABSTRACTS.

Use of steel in the oil industry, with special reference to heat-resisting steels. (SIR) R. HADFIELD and S. A. MAIN (J. Inst. Petroleum Tech., 1929, 15, 611–633).

Copper from copper sulphate manufacture. FICAI.—See VII. **Cement from pyrites cinder.** ALEKSANDROV.—See IX.

PATENTS.

Device for separating and concentrating ores. A. W. McTAGGART (U.S.P. 1,744,785, 28.1.30. Appl., 21.7.27).—Ore pulp is treated in a tank in which upper and lower baffles are set longitudinally and inclined so as to form almost an inner compartment. Paddles agitate the pulp, causing it to circulate upwardly in the centre and through perforations in the upper baffles, and a comminuted jet of air discharges close to the agitator.

C. A. KING.

Treatment of ores, metallurgical products, etc. METALGES. A.-G. (B.P. 317,001, 3.7.29. Appl., 8.8.28).—The ore mixed with fuel, if necessary, is passed successively in a Dwight-Lloyd roasting furnace under a series of ignition furnaces of gradually increasing intensity. The process is especially applicable to the roasting of zinc blende, which is thereby prevented from slagging.

A. R. POWELL.

Roasting of [sulphide] ores and minerals. M. F. COOLBAUGH and J. B. READ, ASSRS. to COMPLEX ORES RECOVERIES Co. (U.S.P. 1,744,867, 28.1.30. Appl., 13.6.24).—The sulphide is passed in direct current with air through a roasting furnace to obtain a mixture of oxides and sulphates, and this product is mixed with a raw sulphide ore or with crushed matte and the mixture sintered in Dwight-Lloyd or Huntington-Heberlein sintering plant preparatory to smelting in a reverberatory or blast furnace.

A. R. POWELL.

Production of metals from their oxide ores. TRENT PROCESS CORP., ASSEES. OF W. E. TRENT (B.P. 306,949, 15.2.29. U.S., 28.2.28).—A mixture of oxide ore, powdered fuel, and flux is heated indirectly, by means of a number of tubes passing through the chamber, until substantial reduction is effected without sintering. The reduced charge is then smelted directly by combustion of the carbon in the charge, and the gaseous products are utilised in heating the tubes in the reduction chamber and for preheating air for combustion, which may be regulated by mixing the products of combustion with inert gases.

C. A. KING.

Melting and deoxidising metals and alloys. W. REITMEISTER (B.P. 327,570, 5.4.29).—The deoxidising agent comprises a mixture of a metal oxide and sufficient finely-divided carbonaceous material to reduce it with the formation of carbon monoxide. *E.g.*, copper alloys may be deoxidised with a mixture of zinc oxide and coal dust, and iron or steel with a mixture of hæmatite and coal dust. An excess of oxide may be used to prevent carburisation of the metal.

A. R. POWELL.

Transformation of pig iron into desulphurised and dephosphorised steel or into pure iron. CARBONISATION SOC. GÉN. D'EXPLOIT. DES CARBONES (B.P. 303,891, 3.1.29. Fr., 12.1.28).—The oval bottom of an acid-lined converter mounted on hollow trunnions is raised towards the middle to impart a gyratory motion to the molten charge when rocked. The trunnions end in the upper part of the converter as tuyères for the

admission of fuel and air. Pig iron is introduced into an atmosphere of carbon dioxide and the metal is freed, in turn, from silicon, carbon, sulphur, and phosphorus, finally being purified in an atmosphere of hydrogen.

C. A. KING.

Annealing furnace. J. R. GARNER and J. H. ROYSTON (B.P. 326,562, 20.12.28).—A furnace particularly adapted for annealing metal tubes or rods is of tunnel form with a longitudinally grooved floor. Straight tubes or rods are connected together temporarily before entering the furnace and a number of such continuous lengths are pushed along the channels in the furnace floor.

C. A. KING.

Manufacture of compact metal articles from metal [iron] powders. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,536, 13.12.28. Addn. to B.P. 311,141; B., 1929, 526).—Iron powder produced by thermal decomposition of the carbonyl is pressed into an ingot heated at 1200° preferably in hydrogen, and subjected at this temperature to a pressure of about 2000 kg./cm.² Alloys of uniform composition may be obtained in a similar manner by adding the powdered alloying element to the iron powder prior to subjecting it to the above treatment.

A. R. POWELL.

Welding of metals and alloys. I. G. FARBENIND. A.-G. (B.P. 305,999, 13.2.29. Ger., 13.2.28).—Thick metal sheets or parts may be welded by applying an oxy-hydrogen or oxy-acetylene burner to the place to be welded and at the same time generating a luminous electric arc in the flame between the work and the welding rod.

A. R. POWELL.

Welding method and composition. P. L. & M. Co., Assees. of H. J. MORGAN (B.P. 317,361, 8.2.29. U.S., 14.8.28).—A mixture consisting of 90% of tungsten, 10% of carbon, and a binder is fused on to the surface of a metal to form a hard layer of tungsten carbide. Other elements may be incorporated in the mixture to form hard alloys, and such a process may be applied in the manufacture or repair of tools, *e.g.*, earth-boring drills.

C. A. KING.

Working of normally brittle sheet metal. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of W. E. RUDER (B.P. 311,290, 8.5.29. U.S., 8.5.28).—Metals which are normally brittle, *e.g.*, iron containing chromium and aluminium, are heated to below 200° and bent round a die heated to the same temperature. Thus flat, metallic ribbon may be wound edgewise on to a heated die, or sharp bends produced on sheet metal.

C. A. KING.

[Copper]-aluminium alloys. O. REULEAUX (B.P. 309,586, 27.3.29. Ger., 13.4.28).—Copper-aluminium alloys with 3–5.5% Cu are treated with up to 0.6% Ti, Zr, or B in the presence of a molten alkali fluoride flux, whereby the solid solubility of copper in aluminium at high temperatures is increased and a greater hardness and tensile strength are obtained on subsequent ageing. The titanium may be introduced by aluminothermic reduction of an alkali fluotitanate on the surface of the molten alloy or by addition of a copper-titanium alloy to the bath.

A. R. POWELL.

Manufacture of aluminium-silicon alloys. Soc. D'ELECTROCHIM., D'ELECTROMÉTALL., ET DES ACIÉRIES

ELECTR. D'UGINE (B.P. 302,692, 19.12.28. Fr., 20.12.27).—Briquettes of kaolin, alumina, and petroleum coke containing an excess of 4% over the theoretical amount of carbon are heated in a refractory crucible by means of an arc of 6000–8000 amp. at 35–50 volts under an arch of semi-fused material of the same type. Alloys containing 65–70% Al, 30–35% Si, and small quantities of iron, titanium, and carbon are thus readily obtained with a loss of less than 5% of the aluminium and silicon by volatilisation.

A. R. POWELL.

Rosin-core solder. P. C. RIPLEY (B.P. 327,337, 7.6.29).—A self-fluxing solder comprises a tube of soft solder filled with a plastic flux made by melting rosin with 2–50% of its weight of a solvent, *e.g.*, turpentine.

A. R. POWELL.

Obtaining lead from its ores or other compounds. G. L. OLDRIGHT (U.S.P. 1,744,174, 21.1.30. Appl., 22.11.26).—The ore, if necessary, after a complete or partial roasting, is heated in a reducing atmosphere or with a reducing agent to obtain the lead in the form of prills disseminated throughout the partially sintered gangue. A considerable proportion of the lead may be removed by liquation and pressing, and the remainder by smelting in the blast furnace the granular residue from the presses.

A. R. POWELL.

Improving [the resistance of] lead [to sulphuric acid]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 326,818 and Addn. B.P. 327,106, 18.12.28).—Soft lead is alloyed with (A) 0.01–0.05% Ni and 0–0.1% Se, or (B) with up to 0.1% Se alone.

A. R. POWELL.

Metal-coated articles [lead-coated paper]. L. W. CUTLER, C. A. KLEIN, and ASSOCIATED LEAD MANUFACTURERS, LTD. (B.P. 326,792, 14.12.28).—The paper is first impregnated with a naphtha solution of bitumen and oil-soluble synthetic resin ("Albertol") and dried at 30–60°, then sprayed with finely-divided lead (200-mesh), and finally burnished with steel shot or by passing the paper through burnishing rollers. The surface may subsequently be coated with a second metal, *e.g.*, copper, by electrodeposition or by simple replacement.

A. R. POWELL.

Flotation process [for lead-zinc ores]. J. H. LANE (U.S.P. 1,743,463, 14.1.30. Appl., 3.8.27).—The ore is ground with 1–4 lb./ton of sawdust (preferably from pinewood) and the usual chemicals to prevent flotation of zinc blende, *e.g.*, cyanide, xanthate, bicarbonate, and zinc sulphate. On subsequent agitation in the flotation machine the wood pulp carries the galena into a froth which is readily floated off. The zinc mineral is removed from the tailings by addition of copper sulphate and xanthate and re-treatment in the flotation machine.

A. R. POWELL.

Mercury-extracting apparatus. L. DAAMS (U.S.P. 1,744,827, 28.1.30. Appl., 25.5.27).—A combustion chamber communicates with the lower and upper end of a retort chamber containing one or more vertical retorts, with damper control of the heat entering the chamber. The upper end of the retort is surrounded by a superheating coil, and an entrainment gas may be passed through the retort.

C. A. KING.

Manufacture of articles from alloys. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 326,518, 15.9.28).—An article of the desired shape is made of one of the constituents of the alloy and the second constituent is applied to the surface of the article by electroplating or by coating with the powdered metal. The composite metal is then heated at such a temperature and for such a time that complete diffusion of the constituents into a homogeneous alloy takes place. *E.g.*, a nickel tube plated internally and externally with 25% of its weight of chromium is readily converted into a tube of nickel-chromium alloy at 1350° in a vacuum. The process is also applicable to the manufacture of thin-walled articles of cobalt-iron, silicon-iron, chromium-iron, and nickel-chromium-iron alloys.

A. R. POWELL.

Filtration of cyanide slimes and apparatus therefor. C. LINTECUM (U.S.P. 1,744,480, 21.1.30. Appl., 3.9.24).—In order to reduce the number of tanks and also the amount of pumping required, a number of filters or vats of equal size on the same level are operated in definite cyclic relation with one another, the pulp and wash solutions being transferred from one vat to another instead of from the filter to excess tanks and back again.

C. A. KING.

Galvanising bath. J. SUMMERS & SONS, LTD., and H. B. FREEMAN (B.P. 327,181, 3.8.29).—A galvanising bath containing spelter etc. is enclosed with a closed jacket containing a fusible metal, *e.g.*, lead.

J. S. G. THOMAS.

Production of steel in an open-hearth furnace. R. DURRER, F. C. SIEMENS, and A. SPRENGER, ASSTS. to F. SIEMENS A.-G. (U.S.P. 1,752,374, 1.4.30. Appl., 28.1.27. Ger., 26.9.25).—See B.P. 293,326; B., 1928, 676.

Manufacture of ductile iron-chromium-aluminium alloys. V. B. BROWNE (U.S.P. 1,754,008, 8.4.30. Appl., 19.12.27).—See B.P. 302,640; B., 1929, 781.

Manufacture of macrocrystalline [tungsten] ingot. H. ALTERTHUM, ASSR. to GEN. ELECTRIC CO. (U.S.P. 1,752,877, 1.4.30. Appl., 6.7.22. Ger., 13.7.21).—See B.P. 183,119; B., 1923, 175 A.

Non-ferrous welding rod. A. R. LITTLE, ASSR. to UNION CARBIDE & CARBON RES. LABS., INC. (Re-issue 17,631, 1.4.30, of U.S.P. 1,716,590, 11.6.29).—See B., 1929, 648.

Electrolytic recovery of metals [nickel]. S. C. SMITH (U.S.P. 1,754,125, 8.4.30. Appl., 19.4.29. U.K., 30.3.28).—See B.P. 314,579; B., 1929, 725.

Magnetic separator (B.P. 303,513). **Filtering medium** (U.S.P. 1,743,524—5).—See I. **Joining metal to wood etc.** (B.P. 302,324).—See V. **White enamels** (B.P. 310,528). **Refractory article** (U.S.P. 1,743,803).—See VIII. **Smelting furnaces** (B.P. 302,308). **Separation of materials** (U.S.P. 1,744,989). **Chrome baths** (B.P. 327,293).—See XI. **Protection from mercury vapour** (G.P. 455,974).—See XXIII.

XI.—ELECTROTECHNICS.

Lead accumulators. E. DENINA (Giorn. Chim. Ind. Appl., 1930, 12, 67—72).—An investigation has been

made of the reactions occurring at the plates of an accumulator during discharge, by a potentiometric and gravimetric examination of artificial electrodes consisting of platinum plates coated electrolytically with lead peroxide and formed in sulphuric acid. Measurements of the *E.M.F.* and temperature coefficients of the single plates have been made in order to calculate the heat of the reaction occurring during discharge. The results in each case are in agreement with the theory of double sulphation during discharge. Measurements of the variation of potential and of the plate resistance during charge, discharge, and inversion show that the resistance in the cell depends primarily on the acid permeating the active material in the plates. The lead sulphate formed during discharge, contrary to expectation, has little effect on the resistance. F. G. TRYHORN.

Chemistry in the telephone industry. R. R. WILLIAMS (Ind. Eng. Chem., 1930, 22, 316—322).—A general account is given of work carried out in the chemical laboratories of the Bell Telephone Company.

H. INGLESON.

Electric-furnace iron. SIMPSON. **Corrosion of pipes.** KUHN. **Insulation of steel plates.** COURTNOT. **Electrolytic lead refining.** FINGLAND. **Electrolysis of gold and electroplating with bronze, cadmium, and copper-cadmium.** YAGI.—See X. **Polymerisation of oils.** GARDNER.—See XII. **Determination of copper in vegetables.** SPRINGER.—See XIX.

PATENTS.

Electric furnaces of the inductor type. E. F. NORTHROP (B.P. 316,662, 9.7.29. U.S., 2.8.28).—A magnetic core is inserted within the extension of the inductor coil unoccupied by the charge and the hearth or base of the crucible, so that the air gap traversed by the magnetic flux is reduced. J. S. G. THOMAS.

Electric induction smelting furnaces. J. HÄRDÉN (B.P. 302,308, 27.11.28. Swed., 14.12.27).—A main low-frequency furnace with closed iron core and an auxiliary high-frequency furnace without such core are connected, *e.g.*, by a duct, so that a charge, melted in the latter furnace, may be transferred to the former.

J. S. G. THOMAS.

Cathodes for electron-discharge tubes. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 319,652, 7.3.29. Holl., 25.9.28).—An indirectly heated cathode is introduced between the heating element and the emitting coating, the latter consisting, at least in part, of an alkali metal or compound, *e.g.*, caesium. J. S. G. THOMAS.

Determination of intensities of [ultra-violet] radiation. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 327,616, 15.5.29).—In a process carried out in accordance with B.P. 296,198 (B., 1928, 864), an optical filter, *e.g.*, composed of Uviol glass and cellophane coloured with picric acid, is inserted before the test liquids, so that a sharply-defined range of wave-lengths is transmitted through the filters, and is incident upon the test liquids. The radiations are preferably compared by observing the respective times in which the test liquids attain a standard coloration.

J. S. G. THOMAS.

organic salt) polymerised rapidly under these conditions and produced solid products. Tung oil treated with 0.05% S produced a wax-like mass resembling a hydrogenated fat. It is suggested that the tung oil (cobalt addition) product could be used in linoleum manufacture.

J. O. CUTTER.

Autoxidation of corn [maize] oil as related to its unsaponifiable constituents. H. A. MATTILL and B. CRAWFORD (Ind. Eng. Chem., 1930, 22, 341—344).—The initial stages of the oxidation, as measured by the decrease of the induction period of maize oil after being subjected to various commercial processes of manufacture, have been studied. The decrease of the induction period is, in general, parallel to the decrease in the amount of unsaponifiable matter (sterols) present. The unsaponifiable matter contains substances which act as antioxidising substances, and so lengthens the period of induction. This action disappears on acetylation of the unsaponifiable matter, and is probably associated with the presence of a hydroxyl group in this substance. Commercial corn oil has less satisfactory keeping qualities than the cold-solvent-extracted oil because of the loss of unsaponifiable matter during the processes of purification, and also to changes in this material rendering it less active as an antioxidant.

J. O. CUTTER.

Composition of commercial stearine and oleine. V. VESELY (Chim. et Ind., 1929, 22, 681—688).—The characteristics and requirements of technical oleines are described, and the presence of isomeric acids in distilled oleines is discussed. From the distillation products of 10-hydroxystearic acid, Δ^9 (elaidic) and Δ^{10} (isooleic) acids, *trans*-octadecenoic acids have been isolated (solid portion) and the presence of Δ^{10} -*cis*-octadecenoic accompanying the Δ^9 (oleic) acid has been demonstrated; it is therefore considered probable that distillation oleine represents a solution, saturated in the cold, of all the components of the "stearine" in the liquid Δ^9 - and Δ^{10} -oleic acids (cf. Vesely and Majtl, A., 1926, 47; Chem. Listy, 1928, 325). The possibility of the occurrence of two other isomerides (Δ^8) derived from 9-hydroxystearic acid (probably formed during the sulphuric acid treatment), or of other sets of isomerides from isomeric octadecenoic acids (e.g., petroselinic) in the original oil is not disregarded. E. LEWKOWITSCH.

Milkweed. GERHARDT.—See V.

PATENTS.

Preparation of [vitamin-containing] margarine from vegetable fats. W. W. TRIGGS. From VAN DEN BERGH'S MARGARINE GES.M.B.H. (B.P. 326,742, 25.7.29).—Vitamin-containing extracts from rice husks, malt, etc. are emulsified with vegetable mucilages or gums, egg yolk, etc. before being added to the margarine during churning. E. LEWKOWITSCH.

Neutralisation of free acidity of oils and fats. J. M. ZUMETA and A. MARTICORENA (YRASTORZA) (B.P. 308,250, 6.12.28. Spain, 20.3.28).—The oil is dissolved in carbon tetrachloride or trichloroethylene and neutralised by aqueous alkaline lye; the solvent is evaporated after washing the oil solution with brine.

E. LEWKOWITSCH.

Refining of wool fat. I. G. FARBENIND. A.-G. (B.P. 303,890, 3.1.29. Ger., 13.1.28).—Crude wool fat is esterified in excess of a lower aliphatic alcohol (e.g., by ethyl alcohol with concentrated sulphuric acid); the insoluble portion of the product is separated and constitutes an almost odourless product of low acid value and high m.p. (yield 65%, dropping pt. 45—46°).

E. LEWKOWITSCH.

Deodorisation of wool fat. M. GILLET (F.P. 569,624, 9.8.23. Belg., 2.8.23).—The wool fat, preferably in the form of aqueous emulsion, is treated with sulphur, sulphur compounds, sulphides, or polysulphides and an alkali; on stirring, mercaptans are liberated which are absorbed by the alkaline solution. After washing with water an odourless fat is obtained.

E. LEWKOWITSCH.

Production of vegetable fats from grape marc. SOC. ANON. FABR. CHIM. ARENELLA (F.P. 635,628, 8.6.27. It., 5.4.27).—By extracting the marc with sodium carbonate solution or carbon disulphide, 8—10% of fat, having f.p. 65° and containing about 50% of stearic acid, is obtained.

E. LEWKOWITSCH.

Bleaching of fatty acids [of marine origin]. A. GODAL (U.S.P. 1,740,012, 17.12.29. Appl., 11.2.26. Nor., 29.1.25).—The crude fatty acids are dissolved in a volatile solvent, e.g., benzine, benzene, tetrachloroethane, and agitated with small amounts of concentrated sulphuric acid at 15—20°. On keeping, the sulphonated acids settle out at the bottom, carrying with them the colouring matter; the oil is recovered by evaporation of the purified layer above.

E. LEWKOWITSCH.

Esterification of fatty acids and mixtures containing them. E. VECKER (F.P. 635,452, 2.6.27. Ger., 23.6.26).—Fatty acids are esterified in the usual way with deficiency of an alcohol, especially glycerol, the excess fatty acids being removed *in vacuo* by means of superheated steam or gases in the presence of liquid mists.

E. LEWKOWITSCH.

Manufacture of soaps. W. MANN (B.P. 325,865, 29.10.28).—Fatty acids (e.g., stearic, oleic) are saponified by milling with alkali carbonates, or fats with caustic alkalis, in the dry state, water being added afterwards to obtain the requisite moisture content for the finished soap. The saponification can be accelerated by the addition of a small proportion (5%) of alcohol.

E. LEWKOWITSCH.

Preparation of soaps containing a fat-solvent. S. ZIMMERMANN, and "HENRY" SEIFEN-, KERZEN-, UND FETTWARENFABR. G.M.B.H. (Aust.P. 109,404, 27.7.25).—Fats are saponified in the presence of, e.g., 3½% of isopropyl or *n*-propyl alcohol before the addition of benzine, or alkali may be added to a solution of the solvent, propyl alcohol, and fat.

E. LEWKOWITSCH.

Drying and conditioning of household soap. TOMLINSONS (ROCHDALE), LTD., and J. N. TOMLINSON (B.P. 325,807, 25.10.28).—Apparatus is described for conveying the raw soap through drying chambers against hot air currents and then through cooling chambers provided with circulating cold air.

E. LEWKOWITSCH.

Preparation of detergents and cleansers. D. D. BROOKS (B.P. 326,755 and 326,759, 24.9.29).—Tri-sodium phosphate and sodium sesquicarbonate and/or saponin are added to soaps, abrasives, etc.

L. A. COLES.

Extraction of [oleaginous] materials. H. G. C. FAIRWEATHER. From PROSCO OILS CORP. (B.P. 324,681, 1.8.28).—The material is subjected to continuous countercurrent contact, descending by gravity through the ascending solvent: in the first (uppermost) zone the solvent is relatively quiescent and saturates the material, which acts as a filter bed to remove fines from the solution which flows off above; in the second zone the material is agitated with the entering solvent and then accumulates at the bottom of the container, displaces by its weight any contained solvent, and is removed by suitable extracting apparatus.

E. LEWKOWITSCH.

Treatment of oil-containing fruits [especially palm fruits.] F. KRUPP GRUSONWERK A.-G. (B.P. 317,366, 25.6.29. Ger., 14.8.28).—The pulp is detached from the nuts by simultaneous dry heat treatment and mechanical stirring devices; and the pulp (with or without admixed nuts) is subjected to a second dry heat treatment in another larger, shallow pan, to remove the remaining water before subjection to oil-recovery processes.

E. LEWKOWITSCH.

Refining sulphur olive oil and the like [extracted oils]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 326,539, 14.12.28).—The oils are stirred for 10–20 min. at 60–80° with 1–4% of a concentrated volatile acid, *e.g.*, hydrochloric acid (*d* 1.19) or formic acid, which precipitates the slimes in the form of flocks; the oil is subsequently washed by showering.

E. LEWKOWITSCH.

Production of lubricating oil or grease. P. H. SNOW (B.P. 326,359, 19.3.29).—Equal amounts (*e.g.*, 12½ pts.) of castor oil and wool grease are mixed by gentle warming (60°), a much larger amount (75 pts.) of castor oil is added, and the mixture maintained at about 54–4° till evenly mixed.

E. LEWKOWITSCH.

Manufacture of products resembling linoxyn or rubber oil substitute. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,094, 17.9.28).—The unsaturated acids obtained by splitting off water from the oxidation products of paraffin hydrocarbons etc. (*e.g.*, by distillation) are esterified with, *e.g.*, glycerol, sorbitol; the esters so formed yield linoxyn-like products on heating in air with driers (*e.g.*, lead oxide, resins, linoleates) and rubber oil-like derivatives on treatment with sulphur.

E. LEWKOWITSCH.

Hydroxylated sulphonation product. B. KEISER (U.S.P. 1,745,221, 28.1.30. Appl., 21.1.29).—By sulphonating the fatty acids derived from linseed or perilla oils with sulphuric acid etc., at about 55°, neutralising, and washing in the usual way, a non-stringy, non-polymerised, water-soluble product of relatively low viscosity is obtained which is similar to Turkey-red oil.

E. LEWKOWITSCH.

Manufacture of new products resembling wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P.

324,631, 24.9.28).—Montan wax, bleached by oxidation, or a conversion product thereof (cf. B.P. 305,552, 308,996, and 320,854; B., 1929, 273, 425; 1930, 156) still containing free acids, together with other fatty, aromatic, or hydroaromatic acids (*e.g.*, coconut oil fatty acids, salicylic acid, naphthenic acids), are esterified with a polyhydric alcohol (or other polyhydroxy-compound which is liquid at the reaction temperature of 100–200°). Hydrocarbons or oily, fatty, waxy, or resinous compounds are added as desired. Any free acid remaining in the mixture may be converted into salts (*e.g.*, of calcium) or other compounds (esters, amides, etc.) wherein the carbonyl group still persists.

E. LEWKOWITSCH.

Emulsification of substances of wax-like character. DEUTS. HYDRIERWERKE A.-G. (B.P. 307,472, 8.3.29. Ger., 8.3.28).—Neutral waxes and hydrocarbons of high mol. wt. are melted with alcohols of high mol. wt. (solid at ordinary temperatures) and higher fatty or resin acids (*e.g.*, 65 pts. of spermaceti, 25 pts. of cetyl alcohol, and 10 pts. of stearic acids); the products form stable emulsions with water or dilute alkaline solutions.

E. LEWKOWITSCH.

Bleaching of beeswax. P. MAHLER, ASSR. to DARCO SALES CORP. (U.S.P. 1,739,796, 17.12.29. Appl., 4.12.24).—Melted beeswax is agitated with 12–35% of a mixture of vegetable char and acid-treated bentonite or other inorganic colour-absorbent, at temperatures between 75° and 100° for about 30 min., and filtered. The wax in the filter-cake is recovered by agitating with hot water, and added to the next batch of crude wax.

E. LEWKOWITSCH.

Preparation of concentrated cod-liver oil. K. KAWAI (U.S.P. 1,753,790, 8.4.30. Appl., 28.9.26).—See B.P. 283,265; B., 1928, 273.

Mixing apparatus (U.S.P. 1,745,291). Presses (B.P. 306,931 and 326,741). **Distillation of glycerin** (U.S.P. 1,743,289). **Mixing of liquids and gas** (U.S.P. 1,740,441).—See I. **Preparations for finishing textiles** (B.P. 291,094).—See VI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Basic lead sulphate. H. WOLFF [with G. ZEIDLER and B. ROSEN] (Farben-Ztg., 1930, 35, 1307–1309).—The results of a series of normal and accelerated weathering tests on paints based on basic lead sulphate (of German and other origin), American "blue lead," and basic carbonate white lead are illustrated and discussed. Contrary to earlier statements (*e.g.*, those of Schulz), basic lead sulphate was not found to be noticeably inferior to basic lead carbonate; indeed the former shows better retention of colour and, under some conditions, less chalking than the latter. Although shown to be inferior to standard basic lead sulphate, "blue lead" gives satisfactory durability when correctly used. Further points of interest arising in the exposures were the advantages of a red lead priming coat and of the introduction of stand oil in the finishing coat.

S. S. WOOLF.

Durability tests of spar varnishes. C. L. CAME (Bur. Stand. J. Res., 1930, 4, 247–259).—Kauri

reduction values and accelerated weathering tests were found to give the same relative order for 50 commercial varnishes as was obtained by outdoor tests.

C. J. SMITHELLS.

Testing the softening point of resins etc. WALKER.
—See II.

PATENTS.

Production of priming composition [for paints and varnishes]. BERGOLIN-WERKE VAN DER BERGH, KOMM.-GES. AUF AKT. (G.P. 459,652, 1.12.22. Addn. to G.P. 427,411; B., 1926, 761).—In addition to the usual filling materials, about 10% of neutral metallic compounds of fatty acids are introduced. D. F. TWISS.

Production of paints and lacquers. H. E. PORTS. From H. PLAUSON (B.P. 326,216, 6.9.28).—Rubber, balata, or the like is dissolved in an organic volatile solvent, *e.g.*, a low-boiling saturated hydrocarbon or chlorinated hydrocarbon, and vulcanised under pressure at about 125°. The vulcanised product is colloidalised by heating at about 180° under higher pressure and pigment is suspended in the dispersion so formed. Organic anhydrides, *e.g.*, acetic or phthalic, or alkali or alkaline-earth peroxides may be added to improve dispersion. The addition of resins, soluble in the dispersion medium, *e.g.*, hardened rosin or coumarone resin, increases the ease of application of the lacquers produced, which are of special value as ships' paints.

S. S. WOOLF.

Rendering paint waterproof. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 300,580, 5.11.28. Ger., 15.11.27. Addn. to B.P. 288,601; B., 1929, 924).—Paints containing added fungicide, as claimed in the prior patent, are used to give a finishing coat only.

H. ROYAL-DAWSON.

Manufacture of artificial resins. E. VON HERZ (B.P. 301,429, 29.11.28. Ger., 29.11.27).—Penterythritol or the water-soluble condensation product obtained as a by-product in the manufacture of that compound from acetaldehyde and formaldehyde is condensed with phthalic acid or anhydride, the sublimed phthalic anhydride is reintroduced, and heating is continued until sublimation ceases. The reaction may be continued until the resin is insoluble and infusible, or it may be interrupted at an intermediate stage of solubility, *e.g.*, when the resin is insoluble in alcohol, but still soluble in acetone.

S. S. WOOLF.

Manufacture of hardened artificial masses. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 298,085 and Addn. B.P. 326,214, [A, B] 1.10.28).—(A) Phenol-formaldehyde condensation products containing carboxylic acid groups (obtained by reaction between a phenol-formaldehyde resin and a halogeno-fatty acid or salt thereof or between a phenoxy-fatty acid and formaldehyde or a substance yielding formaldehyde) are converted wholly or partly into a salt or derivative of ammonia and heated at above 100° in presence or absence of a polyhydric alcohol, and with or without the addition of a filling material. (B) The intermediate ammonia treatment may be dispensed with when a polyhydric alcohol is present.

S. S. WOOLF.

Manufacture of [horn-like] condensation products of arylamines [with formaldehyde]. P. HALLER and H. KAPPELER (B.P. 306,972, 28.2.29. Switz., 29.2.28. Addn. to B.P. 266,358 and 274,501; B., 1928, 224, 792).—A soluble inorganic salt which does not react with formaldehyde, *e.g.*, sodium chloride, ammonium chloride or nitrate, or calcium chloride, is added to the acid condensation mixture of 1 mol. of arylamine and more than 1 mol. of formaldehyde, either at the beginning or before solidification of the mass. The product is washed with water.

C. HOLLINS.

Manufacture of drying varnishes. H. BECKER, ASSR. to SIEMENS & HALSKE A.-G. (U.S.P. 1,754,186, 8.4.30. Appl., 7.10.26. Ger., 20.5.25).—See B.P. 275,813; B., 1927, 822.

Manufacture of horn-like material. J. SCHLINCK and G. MÜNCHMEYER (U.S.P. 1,753,626, 8.4.30. Appl., 17.2.26. Ger., 21.2.25).—See B.P. 247,955; B., 1927, 306.

Chromium oxide (B.P. 302,178).—See VII. **Rosin-core solder** (B.P. 327,337).—See X.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Cultivation and preparation of rubber in the United States. D. SPENCE (Ind. Eng. Chem., 1930, 22, 384—387).—The first steps for the cultivation of the guayule shrub, *Parthenium argentatum*, in the United States were taken in 1912. By suitable treatment of the seed, the proportion germinating has been raised from 5% to 96%. From about 1000 strains a few have now been selected for high rubber content, hardness, and adaptability to various conditions, the rubber content amounting even to 16%. Under normal conditions there is a progressive increase in the total amount of rubber per plant with each year of growth. In a year an acre of newly-sown seed may represent 1164 lb. of pure caoutchouc, a rate of production greatly exceeding that for an average *Hevea* plantation; the optimum growth for harvesting, however, is four years. The rubber is removed from the shrub by grinding in revolving tube-mills of the continuous-feed type; it is then screened, washed, and submitted to a second grinding. The rubber thus collected amounts to within 0.25% of the total rubber in the shrub. Guayule caoutchouc has the composition C_5H_8 , and shows the same behaviour as *Hevea* caoutchouc towards bromine, chromyl chloride, and vulcanisation. By submitting the shrub to a "retting" process, a product is obtainable still more closely resembling rubber from *Hevea brasiliensis* and, if desired, 75% of the acetone-soluble constituents can also be removed in this process, giving a crude rubber with only 5% of acetone-soluble matter. It is believed that guayule rubber will compete successfully in the United States with the product from the Far East, and the area of cultivated guayule is to be increased by 2500 acres annually.

D. F. TWISS.

Conditions affecting the vulcanisation of rubber.
IV. Heat reaction during vulcanisation. Y. TOYABE (J. Soc. Chem. Ind., Japan, 1930, 33, 96—98B;

cf. B., 1930, 339, 249).—Rubber obtained from latex by precipitation with acetone was mixed with various proportions of sulphur (14.7—47.1%), and vulcanised at 155°. An embedded thermocouple showed an internal rise in temperature in each mixture, a maximum being reached in all cases after 60—70 min., when the ratio of free sulphur to total sulphur was about 60%. Vulcanisation at 170° led to occurrence of a maximum internal temperature in 20 min., the proportion of the sulphur then in combination being about 40—50% of the total present. Assuming that maximum heat evolution is an index of maximum reaction velocity, the independence of the latter of the temperature of vulcanisation and its occurrence when approximately half the sulphur has entered into combination suggest that maximum reaction velocity coincides with maximum disintegration of the rubber micelles. D. F. TWISS.

Comparison of various organic reagents for accelerating the vulcanisation of rubber. Y. TANAKA and G. HARA (Bull. Osaka Inst. Ind. Res., 1929, 10, 1—29).—The order of accelerating action, and optimal percentage (on rubber), of the substances examined were: piperidyl pentamethylenedithiocarbamate 0.25—0.5, *p*-nitrodimethylaniline 0.25—1.0, diphenylguanidine 0.5—2.0, *p*-phenylenediamine 0.5—3.0, hexamethylenetetramine 0.5—4.0, triphenylguanidine 0.5—4.0, aldehyde-ammonia 0.75—4.0, thiocarbanilide 1.0—4.0. The breaking strength, elongation, hardness, and elasticity varied according to the accelerator used. The vulcanisation time is considerably shortened when a small, but often not when a large, quantity of reagent is employed. The sulphur may be decreased to 1% of the rubber. CHEMICAL ABSTRACTS.

PATENTS.

Preparation of pure caoutchouc from rubber latex. R. PUMMERER (G.P. 460,950, 26.1.26).—Rubber latex is caused to cream by treatment with dilute caustic alkali in the warm, preferably with absence of air; if desired, the cream may be re-diluted and re-treated. After removal of residual alkali, *e.g.*, by washing, filtration, dialysis, or centrifuging, pure caoutchouc is obtained which yields vulcanised products of super-normal technical qualities. D. F. TWISS.

Production of floating rubber resistant to mineral oil. TOKYO GOMU KABUSHIKI KAISHA (F.P. 634,628, 18.5.27).—An oil-resistant rubber of sp. gr. below that of water is produced by mixing rubber with factis, sulphur, alkali soap, and diphenylguanidine and vulcanising. Mineral fillers should not be used. D. F. TWISS.

Production of aqueous dispersions from rubber substitutes, more particularly factis. K.D.P., LTD. (B.P. 309,167, 15.3.29. Ger., 7.4.28).—Rubber substitutes are mixed with a peptising agent, such as gelatin, casein, or soap, on a roller-mill adapted to exert heavy pressure and thereby render the material plastic. As soon as intimate mixing has occurred, the material can be dispersed in water. D. F. TWISS.

Regeneration of vulcanised rubber. K.D.P., LTD., Assees. of METALLGES. A.-G. (B.P. 311,268, 15.3.29.

Ger., 8.5.28).—Vulcanised rubber is mixed with a peptising agent, *e.g.*, glue, saponin, soap, or casein (which, if desired, may be in solution), in a roller-mill or other suitable mixing apparatus adapted to exert substantial pressure and friction on the rubber. A product is obtained which on addition of water dissolves to a pulpy, finely-dispersed mass. D. F. TWISS.

Manufacture of accelerators for the vulcanisation of rubber and allied substances. S. J. PEACHEY (B.P. 326,256, 13. and 21.12.28, and 3. and 4.1.29).—Vulcanisation accelerators are prepared by heating *p*-nitrosodimethylaniline or one of its homologues, *e.g.*, *p*-nitrosodiethylaniline, with an arylamine of the benzene, naphthalene, or pyridine group and sulphur. The products are free from the objectionable staining properties of the parent nitroso-compound. D. F. TWISS.

Manufacture of synthetic rubber. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,869, 20.12.28).— α -Butadiene, or a homologue or derivative thereof, such as phenylbutadiene or styrene, is polymerised by an alkali metal in the presence of a small quantity of ammonia or a free cyclic or saturated aliphatic base (*e.g.*, 0.01—1% by wt. of the hydrocarbon to be polymerised). Substances favourable to the polymerisation, *e.g.*, ether, and diluents such as light petroleum may also be present. D. F. TWISS.

Manufacture of synthetic rubber. I. G. FARBENIND. A.-G. (B.P. 302,733, 20.12.28. Ger., 21.12.27).—Synthetic rubber-like masses are produced by separately polymerising quantities of the same butadiene hydrocarbon under different conditions and mixing the products. D. F. TWISS.

Manufacture of resilient [rubber] tyres and treads therefor. I. G. FARBENIND. A.-G. (B.P. 326,202, 30.8.28. Addn. to B.P. 299,037; B., 1930, 71).—In the manufacture of tyres from the plastic polymerisation product of butadiene, carbon-black is used as a compounding ingredient in addition to any other customary materials. D. F. TWISS.

Manufacture of rubber articles. MORGAN & WRIGHT, Assees. of (A) E. HAZELL, (B) A. W. KEEN (B.P. 307,789 and 307,792, 7.1.29. U.S., 13.3.28).—In the manufacture of rubber articles from aqueous dispersions of rubber by direct deposition of the rubber on to a form of the desired shape comprising a perforated metallic foundation with a coating of filtering material, (A) fabric or fibrous material such as paper pulp in one or more layers is interposed between the foundation and the external filtering coating which may consist of kieselguhr, clay, etc. Sufficient of this filtering composition is applied over the fabric layers to give a smooth continuous coating. (B) Using a similar arrangement, provision is made for heating the form (*e.g.*, by introduction of a heating medium) and the rubber deposited on it. The form may be immersed in the latex and withdrawn with a deposit of the desired quantity of rubber; heat is then applied for the substantial removal of water from the deposit, vulcanisation being completed subsequently. D. F. TWISS.

Production of rubber articles. DUNLOP RUBBER CO., LTD., C. HAYES, and E. A. MURPHY (B.P. 326,496, 14.11.28).—Rubber articles, particularly inner tubes or treads and other articles of irregular cross-section, are produced from aqueous dispersions of rubber or similar material in concentrated and/or compounded condition, by passing a mandrel downwards through a bath of thick creamy consistency, the thickness of the resulting coating being regulated by a spreading device, and then passing the coated mandrel downwards through an air-gap into a fixing bath containing, *e.g.*, a dehydrating and setting medium. Vulcanisation is effected subsequently. D. F. TWISS.

Manufacture of articles of rubber or similar material. DUNLOP RUBBER CO., LTD., D. F. TWISS, E. A. MURPHY, and W. G. THORPE (B.P. 326,282, 5.1.29).—Rubber articles of moderate thickness are produced by dipping a former into latex which preferably has been concentrated and/or compounded, and then bringing the uncoagulated deposit on the former into contact with an atmosphere containing, or consisting of, a volatile coagulant or with a dilute aqueous solution of such a coagulant. D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER CO., LTD., W. H. CHAPMAN, D. W. POUNDER, E. A. MURPHY, and F. T. PURKIS (B.P. 326,210, 7.12.28).—Silicofluorides have a variable effect on a dispersion of rubber or similar material, according to the proportion added. With a proportion in excess of a certain minimum, *e.g.*, 0.1–1% for a particular latex containing 60% of rubber and 0.5% of ammonia, the salts render the latex heat-sensitive, so that gelling occurs when the temperature is raised. With quantities below these, *e.g.*, 0.05–0.1%, the viscosity of the latex may be increased without inducing marked heat-sensitiveness. With greater proportions, *e.g.*, 1–5%, uniform gelling can be effected at ordinary temperature; the presence of zinc oxide assists the gelling process. The salts may be added as a powder or an aqueous dispersion. D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER CO., LTD., W. H. CHAPMAN, and D. W. POUNDER (B.P. 326,497 and 326,782, 14.11.28).—(A) Aqueous dispersions of rubber or similar material, compounded or otherwise, which have been rendered heat-sensitive, are brought to the desired shape, *e.g.*, by spreading, moulding, or electrophoresis, and are then gelled by contact with a fluid heating medium such as hot air or water. (B) Rubber sheet, tyre treads, and similar articles are produced by calendaring a heat-sensitive latex mixing between two non-adhesive sheets and causing the latex layer to gel by leading the sheets into a heating medium; after gelling, the rubber is removed from the sheets and dried. Using sheets which have not been pretreated to prevent adhesion, a double-texture, waterproofed material is obtained. By modified procedure it is possible to provide cloth or fabric with a permanent rubber coating on one or each side. D. F. TWISS.

Manufacture of rubber-fibre articles. W. B. WESCOTT, ASSR. to RUBBER LATEX RES. CORP. (U.S.P. 1,737,133, 26.11.29. Appl., 12.7.27).—The cord fibre,

ranging from 0.5 to 1.5 in. in length, remaining after the mechanical removal of the bulk of the rubber from comminuted tyre scrap and containing, *e.g.*, 3–5% of adherent vulcanised rubber, is wetted thoroughly with latex to which a protective colloid such as haemoglobin may have been added. Fillers and vulcanising agents may also be present. The roughly moulded mixture is dried to a pervious mass and, preferably while still warm, is compressed in the final mould and vulcanised. The minimum proportion of latex rubber necessary for satisfactory results is 10–15%. D. F. TWISS.

Rubbering of fabrics containing copper and manganese. W. ESCH (G.P. 461,134, 11.11.26).—The fabric is given a preliminary coating of a rubber mixture resistant to copper and manganese and capable of being vulcanised at a moderate temperature; additional coats are then applied of a rubber mixture which can be vulcanised with a solution of sulphur chloride. A suitable copper- and manganese-resisting mixing comprises rubber, zinc oxide, lithopone, calcium carbonate, sulphur, cotton-seed oil, organic colour, and tetramethylthiuram disulphide. D. F. TWISS.

Preservation and packing of rubber goods. M. G. GRAICHEN (G.P. 461,060, 16.9.26).—Rubber articles such as finger-stalls are packed in air-tight containers filled with a mixture of water, glycerin, and borax, whereby the quality of the material is retained and after-vulcanisation prevented. D. F. TWISS.

Treatment of gutta-percha, balata, and like thermoplastic natural and artificial products and manufacture of moulded articles therefrom. W. S. SMITH, H. J. GARNETT, and J. N. DEAN (B.P. 326,481, 12.12.28 and 14.5.29).—Gutta-percha is subjected to heat treatment at a temperature below that causing melting or softening; this raises the normal melting or softening point and improves the mechanical, electrical, and water-resistant properties. Articles made of or containing gutta-percha, *e.g.*, insulated wires, may be so treated. On account of the induced hardening, the temperature of the heat-treatment may be raised gradually. Occasional "quenching" in cold water during the treatment accelerates the hardening process. D. F. TWISS.

Rubberising fibres (B.P. 325,916).—See V. **Electrical insulation** (U.S.P. 1,736,899).—See XI. **Rubber oil substitute** (B.P. 327,094).—See XII. **Paints and lacquers** (B.P. 326,216).—See XIII.

XV.—LEATHER; GLUE.

Effect of post-mortem action on the nitrogen distribution of animal skin. E. R. THEIS (J. Amer. Leather Chem. Assoc., 1930, 25, 92–102).—Small cubes of animal skin cut immediately after flaying were kept in stoppered bottles for definite periods at 5°, 20°, 30°, and 37.5°, respectively. There was a gradual formation of ammonia at 5°, which must be due to enzyme activity. A normal amount of ammonia was evolved at 20° in the first 48 hrs., after which there was a greater increase probably due to the combined activity of bacteria and autolytic enzymes. A much increased amount of ammonia was evolved at 37.5°. The presence

of very active autolytic enzymes in the skin was demonstrated. It was shown that the amide nitrogen increased progressively as the post-mortem period was increased, whilst the melanin nitrogen decreased. The amount of basic nitrogen was constant during the early stages, but diminished with more prolonged action. The total non-basic nitrogen remained practically constant, but the amino-nitrogen gradually decreased, thus indicating progressive deamination during post-mortem action.

D. WOODROFFE.

Formaldehyde tanning. B. ZIROULSKY (Halle aux Cuirs, Suppl. tech., 1929, 163—170; Chem. Zentr., 1929, ii, 2527).—The tanning action of formaldehyde is rapid; the absorption increases with the concentration of the formaldehyde and is markedly increased by the presence of alkali.

A. A. ELDRIDGE.

PATENTS.

Depilation of hides. E. A. TAYLOR, ASSR. to GRASSELLI CHEM. CO. (U.S.P. 1,743,938, 14.1.30. Appl., 4.2.28).—Hides are treated with an aqueous solution prepared by dissolving in water an alkaline-earth oxide (25 pts. of lime), an alkaline sulphide (6.5 pts. of sodium sulphide), and an alkaline hydrosulphide (16.5 pts. of sodium hydrosulphide per 1000 pts. of green hide), the molecular amount of which is greater than that of the sulphide. A mixture of sodium sulphide and sodium hydrosulphide (mol. ratio of which is within the limits 1:2 and 1:5) may be used.

D. WOODROFFE.

Unhairing of hides or skins. A. E. WHITE. From TANNERS' COUNCIL OF U.S.A. (B.P. 327,132, 27.10.28).—Hides or skins are treated at about 20° with a saturated lime liquor, to which has been added about 1% of a primary alkylamine not containing other polar groups, e.g., ethylamine and/or methylamine.

D. WOODROFFE.

Treatment of skins or hides previous to tanning. W. H. ALLEN, ASSR. to LEATHER MAKERS' PROCESS CO. (U.S.P. 1,743,647, 14.1.30. Appl., 2.7.28).—Hides etc. are disinfected by immersion in a solution of sodium chloride and hydrochloric acid, the solution with the hides therein is neutralised with sufficient alkaline material, e.g., calcium hydroxide, to neutralise the free acid, and the hides are then removed from the liquor and washed free from soluble salts.

D. WOODROFFE.

Tanning of hides. HEIN & Co. (G.P. 459,617, 5.4.23).—Chlorine is passed into solutions of sulphite-cellulose waste liquor or ligninsulphonic acid until no more is absorbed, the excess chlorine is removed by treatment with alkali, and the product is combined with aromatic sulphonic acids, e.g., *p*-toluenesulphonyl chloride, and used for tanning hides.

D. WOODROFFE.

Tanning of hides. W. HILDT and R. MALACHOWSKI (G.P. 458,338, 9.3.23. Poland, 24.3.23).—The alkali naphthenates obtained by the alkali treatment of mineral oils are sulphonated, after which they may or may not be combined with acid resins and neutralised with alkali. The product, with or without the addition of other tanning materials, is used for tanning hides.

D. WOODROFFE.

Treatment of leather. B. RUBENS, ASSR. to A. BRICK, W. HALLNER, I. PRITSKER, S. KAGAN, N. L.

COHEN, and S. HOROWITZ (U.S.P. 1,744,506, 21.1.30. Appl., 27.4.27).—Ground oak bark (5 pts.) is extracted with boiling water (95 pts.), filtered, sodium hydroxide (0.25 pt.) is added, the solution allowed to cool, and the leather sponged or saturated with the solution and then moulded or shaped as desired.

D. WOODROFFE.

Tanning and other processes for treating solids with liquids. V. A. COLLINS (B.P. 327,146, 28.9.28).—Hides are suspended in tanning liquor in a closed container on the top of which is a series of cylinders, each containing a piston; the pistons are suitably reciprocated by means of a crankshaft to provide a continuous series of variations in the pressure in the container. A storage tank and pressure accumulator are connected with the container and the liquor is circulated by means of a pump.

D. WOODROFFE.

XVI.—AGRICULTURE.

Chemical composition of colloidal material isolated from the horizons of various soil profiles.

I. A. DENISON (J. Agric. Res., 1930, 40, 469—483).—A number of detailed chemical analyses are recorded. In some profiles the colloids of various horizons were of practically constant composition; in others wide variations existed. The most variable constituents were silica, iron, aluminium, organic matter, and combined water. Where the colloid composition changed with depth, variations in silica and aluminium contents were regular, but not always in the same direction in all soils. Weathering may not be the chief factor in reducing the silica: sesquioxide ratio of colloids, since in some instances this ratio decreased with the depth of horizon. Evidence was obtained of the presence of free alumina in the colloids of some of the lower (C) horizons.

A. G. POLLARD.

Composition of soils of the Nile delta. P. KOENIG (Ernähr. Pflanze, 1929, 25, 417—428; Chem. Zentr., 1929, ii, 2597).—The soils, which vary considerably in mechanical composition, seldom contain more than N 0.1, K₂O 0.5—1, CaO 3, P₂O₅ 0.2% (of which 10—20% is soluble in citric acid); the content of assimilable potassium is small.

A. A. ELDRIDGE.

Base exchange and acidity [in soils]. II. Preliminary experiments with permutits. P. VAGELER and J. WOLTERSDORF (Z. Pflanz. Düng., 1930, 16A, 184—204; cf. B., 1930, 295).—The results of experiments with soils and permutits are recorded in confirmation of the authors' mathematical treatment of the course of the exchange of calcium and hydrogen ions in relation to the anions with which they are associated.

A. G. POLLARD.

Relation of the amount and nature of exchangeable cations to the structure of a colloidal clay. L. D. BAUER (Soil Sci., 1930, 29, 291—309).—The properties of electrodyalysed clay saturated to varying extents with different cations are examined. The reaction of clay sols containing different cations varied according to the lyotropic series Li > Na > K > Mg > Ca > H, whilst their specific conductivities were in the order Na > Li > K > Mg > Ca, and flocculation values in the order Li = Na > K > Mg = Ca. Univalent

cations increased the charge on the particles to a maximum at the saturation capacity of the colloid. Still higher concentrations of ions decreased the charge. Bivalent cations decreased the charge. The average radius of the aggregates varied with the cation from 136 $\mu\mu$ for hydrogen to 148 $\mu\mu$ for calcium. Pore sizes in calcium-clay were larger than in a sodium-clay. Addition of increasing amounts of univalent cations to clay at first decreased its viscosity, which subsequently increased to a maximum corresponding to the saturation point of the clay with bases. Still higher proportions of cations caused a sharp decline in viscosity, followed by an increase at the point of flocculation. Bivalent ions decreased the viscosity of clay to the point of flocculation, where a sharp increase occurred. The hydration and viscosity of the aggregates were in the order $\text{Li} > \text{Na} > \text{K} > \text{H} > \text{Ca} > \text{Mg}$. Changes in these properties of clay sols are associated with the number of "active" ions on the particle (*i.e.*, dissociated ions existing on the particle surface as distinct from those of the undissociated clay "salts"). The size, stability, and low hydration of the calcium and magnesium aggregates are important for maintaining good soil structure.

A. G. POLLARD.

Replaceable bases of irrigated soil. W. H. METZGER (Soil Sci., 1930, 29, 251—260).—The proportions of replaceable bases in a soil in the air-dry, moistened, and flooded condition are compared. No appreciable change in the amount of replaceable sodium and potassium with moisture conditions was observed. Flooding largely increased the replaceable calcium, magnesium, aluminium, iron, ammonia, and manganese. Moistening to 20% produced increases only in the exchangeable manganese and magnesium. Comparison is also made of the distribution of replaceable bases in the various horizons of irrigated and non-irrigated soils.

A. G. POLLARD.

Transition of quicklime and its influence on the solubility of phosphoric acid and potash in heavy soils. H. HEUSER (Z. Pflanz. Düng., 1930, 16A, 204—226).—Changes resulting from the addition of quicklime to soils are examined by electro-ultrafiltration methods. In heavy soils quicklime is not converted quantitatively into calcium carbonate. Quicklime does not affect the solubility of soil potash. The easily soluble phosphate content of some soils was decreased by liming, but in others the transition of insoluble to easily soluble phosphate was increased. Electro-ultrafiltration methods may be used to follow the initial podsolisation processes in soils, which cannot be detected by macroscopic processes.

A. G. POLLARD.

Colorimetric determination of soil reaction. C. J. SCHOLLENBERGER (Science, 1930, 71, 220).—A simple form of percolator giving a clear water extract of soil is described.

L. S. THEOBALD.

Biochemical researches on soil. A. J. J. VANDE VELDE and A. VERBELEN (Compt. rend., 1930, 190, 977—979).—The method devised by Skar (B., 1929, 263) for the determination of the number of micro-organisms in milk has been applied to soil. The earth is weighed aseptically and shaken with sterile milk for 30 min.

in the presence of 4% formaldehyde. No relationship was established between the p_{H} , which varied from 5.11 to 7.95, and the number of micro-organisms, which varied from 1280 to 21,600 millions per g. of the soil from 30 fields. Cultivation of barley or radish in the same soil, with or without sterilisation or treatment with lime, showed that the number of micro-organisms diminished during germination, except where sterilised soil was used, in which case the number increased. The p_{H} increased in the cultures of the radish and decreased in those of the barley. Liming raises the alkalinity of the soil, but the reaction of the plant juices is dependent more on the life of the plant than on the reaction of the substrate.

C. C. N. VASS.

Effect of leaking natural gas on the soil. C. J. SCHOLLENBERGER (Soil Sci., 1930, 29, 261—266).—The exchangeable manganese content of soil is considerably increased by the leakage to it of natural gas. There is a smaller increase in exchangeable ammonia and a decrease in hydrogen-ion concentration. It is suggested that the gas displaces air from the soil and the resulting biological reduction processes tend to render active the higher oxides of manganese known to be present in the soil. Aeration of the soil leads to rapid reversion of the manganese to an inactive form and subsequent plant growth is normal.

A. G. POLLARD.

Soil and crop studies with ammonium sulphate. A. L. PRINCE and A. W. BLAIR (Soil Sci., 1930, 29, 267—279).—Pot cultures of barley, rape, and lettuce fertilised with varying amounts of ammonium sulphate are described. On a heavy loam rape and barley were not injured by heavy dressings (up to 1800 lb./acre) of ammonium sulphate, and the nitrogen content of the crop was increased. On sandy soil dressings exceeding 350 lb./acre caused decreased crops and poor germination. Below this limit the dry weight and nitrogen content of the crops were increased by ammonium sulphate. With the larger amounts of ammonium sulphate applied, the p_{H} value of the soils decreased to an extent which was greater on heavy soil than on sand. Plant injury, however, was more marked on the lighter soil. The p_{H} values of treated soils were found to be correlated with the proportion of "active" aluminium present. The hydrolysis of the aluminium sulphate produced by base exchange probably explains the changes in reaction.

A. G. POLLARD.

Action of ammonium salts [on plants] in relation to hydrogen-ion concentration. H. ENGEL (Z. Pflanz. Düng., 1930, 16A, 226—233).—The work of Mevius and Engel (*cf.* B., 1928, 278, and Planta, 1929, 9, 1) is further discussed. Addition of ammonium salts of the stronger acids to plant-nutrient solutions results in the penetration of hydrolytically produced ammonia into the root cells, in quantities increasing with the p_{H} value and ammonia tension of the solutions. Within the root cell the ammonia is neutralised by organic acids and its subsequent conversion into asparagin, amino-acids, and proteins is controlled by the carbon dioxide assimilation of the plant. Because of its physiologically amphoteric nature, ammonium nitrate is suitable for the study of the ammonia absorption of plants, in which no "acidosis" (Pranischnikov) is caused.

A. G. POLLARD.

Determination of inorganic nitrogen in dried plant tissue. A. C. SESSIONS (Soil Sci., 1930, 29, 285—289).—The method of Sessions and Shive (A., 1929, 960) is modified to deal with dried plant matter. The sample is ground to pass 30-mesh and 2–5 g. are placed in a 300-c.c. aspirating flask with 70 c.c. of water. After setting aside for 1 hr. with occasional shaking, material adhering to the sides of the flask is washed down with not more than 30 c.c. of water. Then 15 g. of sodium chloride and 3–4 g. of Devarda's alloy (100-mesh) are added and sufficient sodium hydroxide to make the solution approx. $N/7$. Air is drawn through the apparatus for 12 hrs., the ammonia being trapped in acid. Frothing is checked by the addition of 2–3 c.c. of light paraffin oil or 2–3 drops of decyl alcohol. A. G. POLLARD.

New basis for fertiliser experiments. W. J. SPILLMAN (Science, 1930, 71, 135—136).—In field experiments on a soil containing n , p , and k available units per acre of nitrogen, phosphoric acid, and potash, respectively, the yield per acre $y = A(1 - R_1^{n+x_1})(1 - R_2^{p+x_2})x \times (1 - R_3^{k+x_3})$, where A is a maximum yield constant, x_1 , x_2 , and x_3 are quantities of growth factors, and R_1 , R_2 , and R_3 are their related constants. L. S. THEOBALD.

Comparative [fertiliser] values of different phosphates. C. A. MOOERS (Tenn. Agric. Exp. Sta. Bull., 1929, No. 141, 1—18).—Basic phosphates were suitable for unlimed land and were superior to superphosphate. Under liming, Thomas slag and superphosphate were of equal value. Rock phosphate was in both cases inferior. The test crops were maize, oats, potatoes, and hay. CHEMICAL ABSTRACTS.

Effect of burning on the accumulation of organic matter in forest soils. R. M. BARNETTE and J. B. HESTER (Soil Sci., 1930, 29, 281—284).—Soil analyses show that considerable losses to forest soils of organic matter and nitrogen are incurred by the periodic burning of undergrowth etc. A. G. POLLARD.

Preparation and effectiveness of basic copper sulphate as a fungicide. E. B. HOLLAND, C. O. DUNBAR, G. M. GILLIGAN, and W. L. DORAN (Mass. Agric. Exp. Sta. Bull., 1929, No. 252, 124—149).—Commercial basic copper sulphate was effective for cucumbers and celery. The concentration of copper necessary may be greater than that in Bordeaux mixture owing to lower dispersion. The concentration of basic sulphate necessary in sprays differs from that in dusts owing to the large proportion of inert vehicle in the latter. For sprays the following concentrations of copper and arsenic, respectively, are recommended: apples and grapes 0.05, 0.07; cucumber 0.025, 0.09; potatoes 0.30, 0.09; celery 0.30%. CHEMICAL ABSTRACTS.

Determination of the content of toxic substance in insecticides etc. V. Volumetric determination of formaldehyde in seed fungicides. J. BODNÁR and W. GERVAY (Z. anal. Chem., 1930, 80, 127—134; cf. B., 1928, 619).—In fungicides containing phenol, glycerol, soaps, or sodium chloride, formaldehyde may be determined directly by the hydrogen peroxide method of Blank and Finkenbeiner (B., 1899, 614) or by the sulphite method of Lemme (cf. B., 1903, 1107). If the material contains mercuric chloride, 5 g. of sodium

chloride are added to the solution and the formaldehyde is separated from the mercury by distillation, the distillate being analysed by either of the above-mentioned methods. A. R. POWELL.

Pneumatic transport. NEU.—See I. Removal of ions from calcium phosphate solutions by gels. GHOSH and BHATTACHARYYA.—See VII. Cultivation of rubber. SPENCE.—See XIV. Plant colloids. KAVČIČ.—See XVII. Ripening of fruits. OVERHOLSER. Foods treated with arsenical preparations. VON FELLEBERG.—See XIX.

PATENTS.

Insecticides. A. CARPMAEL. From I. G. FARBEN-IND. A.-G. (B.P. 325,910, 12.12.28).—Benzene derivatives containing one or more methoxyl groups and at least 2 thiocyano-groups are used for the destruction of plant lice. Examples are: 4-methoxy-*m*-xylylene dithiocyanate, m.p. 71—72°, 2:5-dimethoxy-*p*-xylylene dithiocyanate, m.p. 173°, and 6-chloro-4-methoxy-*m*-xylylene dithiocyanate. The product may be dissolved, with an acetone-soluble wetting agent, in acetone and diluted with water to give an emulsion ready for use. C. HOLLINS.

Insecticide and fungicide. R. H. MCKEE (U.S.P. 1,744,324, 21.1.30. Appl., 28.3.25).—The shale oil fraction distilling at 150—300°, consisting of bases and saturated and unsaturated hydrocarbons, is emulsified with soap solution; the product on dilution with water is used for spraying plants or for impregnating wood. L. A. COLES.

Manufacture of insecticides. W. H. GROOM-BRIDGE, Assr. to A. J. DICKINSON, LTD. (U.S.P. 1,753,887, 8.4.30. Appl., 23.11.27).—See B.P. 301,186; B., 1929, 106.

XVII.—SUGARS; STARCHES; GUMS.

Purification of sugar-cane juice. A. LITJAGO (Weissruss. Staatl. Akad. Landw., 1929, 7, 315—320; Chem. Zentr., 1929, ii, 2611).—By the use of magnesium sulphate in the clarification of diffusion juice according to the equation $C_{12}H_{22}O_{11} \cdot CaO + MgSO_4 + H_2O = C_{12}H_{22}O_{11} + CaSO_4 + Mg(OH)_2$, almost complete separation of the organic, and to some extent of the inorganic, non-sugar substances takes place owing to the formation of colloidal magnesium hydroxide. The sugar, however, contains 0.4% of calcium sulphate. A. A. ELDRIDGE.

Determination of the sugar content of carbonated scums. F. HERLES; also J. HAMOUS (Z. Zuckerind. Czechoslov., 1929, 54, 23).—Polemical (cf. Kadlev, B., 1930, 29).

Free water necessary to change β -anhydrous lactose into α -hydrous lactose. R. W. BELL (Ind. Eng. Chem., 1930, 22, 379—380).—Of the three known forms of lactose, viz., α -hydrate, α -anhydride, and β -anhydride, the first-named is stable at temperatures below 93° in the presence of small amounts of water (cf. Hudson and Brown, B., 1908, 699, 1169). Experiments are described in which the amount of free water necessary to convert the β -anhydride into the α -hydrate has been determined. This was found to be between 0.5 and 1.0%. H. INGLESON.

Plant colloids. XXV. Potato starch from different varieties of *Solanum tuberosum*. J. KAVČIČ (Kolloidchem. Beih., 1930, 30, 406—415).—Using potato starch from four different varieties of *Solanum tuberosum*, measurements were made of the mean diameter of the grains, water content in the air-dried state, ash content, content of phosphoric acid, swelling temperature, viscosity, electrical conductivity, hydrogen-ion concentration, capacity for iodine adsorption, optical rotatory power, aggregate weight, and fraction diffusible through a collodion membrane. Different values of these properties were obtained for the different varieties and the variations were consistent, indicating that potato starch cannot be regarded as a definite colloid-chemical individual. E. S. HEDGES.

Yield of potato starch. W. BIELICKI (Przemysl Chem., 1930, 14, 145—149).—Maximum yields of starch from potato flakes were obtained at p_H 5.8. The yields obtained from flakes containing 18.4—35.5% of water are the same, provided that the duration of drying does not exceed 30 min. at a sufficiently low temperature, and that the water used for extraction of the starch has a low mineral content. R. TRUSZKOWSKI.

Soluble starch. H. INUKAI (Bull. Kyoto Ind. Res. Inst., 1927, 1, 1—23; 1928, 3, 13—42).—The properties of soluble starch prepared by various methods are recorded. The use of glacial acetic and concentrated sulphuric acids results in partial esterification but gives a good product, whilst bleaching powder causes oxidation. CHEMICAL ABSTRACTS.

Polysaccharide of larchwood. WISE and PETERSON.—See V.

PATENT.

Glycerol from dextrose (B.P. 299,373).—See III.

XVIII.—FERMENTATION INDUSTRIES.

Simple connecting device for the pure culture of yeast. H. FINK and R. KÜHLES (Woch. Brau., 1930, 47, 152—153).—Two flasks, of 1 and 5 litres capacity, respectively, with side tubes similar to those of Pasteur flasks, are connected by short rubber tubes and a T-piece. The rubber connexions can be closed by screw clips, and the other branch of the T-piece by a clip and a glass stopper. The larger flask is filled with wort, a small quantity is placed in the smaller flask, and the whole sterilised. The smaller flask is opened once for inoculation, and the T-piece filled with alcohol and closed. When fermentation is well established in the smaller flask, this alcohol is removed, the clips between the two flasks are opened, and wort is run from the large to the small flask. Portions of culture can be drawn off through the T-piece without risk of infection, or the contents of the large flask can be inoculated to obtain a culture for inoculating technical fermentations. F. E. DAY.

Staining of yeast by methylene-blue and its relation to hydrogen-ion concentration and the problem of permeability. H. FINK and F. WEINFURTER (Woch. Brau., 1930, 47, 89—93, 110—116, 124—127).—The proportion of cells stained and the

intensity of staining are greater in suspensions in alkaline than in acid media. The effect is more marked in dilute solutions of electrolytes than in wort. Acid media extract the dye from stained cells more readily than do alkaline media. Yeast washed with distilled water and suspended in conductivity water is "sensitised" to staining, and a similar result is obtained if the latter medium be replaced by 20% solutions of dextrose, lævulose, or sucrose in distilled water. Maltose (2 specimens) was less effective, glycerin much less, and mannitol had no effect. The presence of salts protects the cells against "sensitisation" even in the concentration present in (Munich) supply water, and treatment with such dilute salt solutions after sensitisation and before staining destroys the sensitisation. The production of carbon dioxide in fermentation experiments is affected in a similar manner, parallel fermentations in media prepared with distilled and supply water giving 7.7 and 14.4 c.c. of carbon dioxide, respectively, in absence of methylene-blue and 1.1 and 9.4 c.c. in presence of the stain. Pure water and carbohydrates appear to increase the permeability of the cell, and thus to facilitate the entry of the dye, which then acts as a poison. This has been confirmed by plate counts of variously treated yeasts, and a parallel is drawn between these results and the protective action of salts against the yeast poison of wheat flour, especially as bakers' and factory yeasts are less susceptible of "sensitisation." In this respect yeasts show considerable variation, and this may be associated with high salt content of the previous culture medium, and consequent storage of salts by the cells. The relative protective action of a number of salts has been studied, and it appears that the differences are relatively small, but that if the effects of the ions are separated they tend to follow Hofmeister's series; e.g., the degree of protection increases from lithium through sodium and potassium to ammonium. Reference is made to work in hand on the influence of peptones, and on a modification of the staining test for yeast, and the possible effects of the protective action of salts in fermentation technology is discussed (cf. B., 1929, 792, 1028). F. E. DAY.

Graphical evaluation of sieve analyses [of grist]. O. HALTMEIER (Woch. Brau., 1930, 47, 133—137, 149—151).—If the results of sieve analyses are plotted graphically—percentage of residue against diameter of perforations—curves are obtained which are independent of the sieves employed. By comparing the curves given by the products from each pair of rolls, the effect of each on the final grist can be studied and the grinding correctly adjusted. F. E. DAY.

Separation of the agitation effect in beer fermentation into aeration and effective yeast-surface factors ("spärende Wirkung"). F. WINDISCH (Woch. Brau., 1930, 47, 94—96).—Wort agitated during fermentation gives a higher degree of attenuation even when the aeration effect is eliminated by increasing the pitching rate till no yeast increase occurs. At normal pitching rates the addition of 20 g. of wood-wool to 1.5 litres lessens the difference of attenuation between unagitated and agitated fermentations. In the presence of wood-wool and at high pitching rates, stirred and

unstirred fermentations reach the same degree of attenuation. F. E. DAY.

Gasometric method for the determination of carbon dioxide in beer. H. LUNDIN, O. ÖHLIN, and J. ELLBURG (*Woch. Brau.*, 1930, 47, 121—124, 137—140).—The method previously described (B., 1928, 725) has been improved. The Van Slyke apparatus and that for rendering the beer alkaline without escape of gas have been modified in certain details, and a borer for piercing crown-cork capsules and withdrawing the contents of the bottle quantitatively is described. The contents of the bottle are rendered alkaline and made up to 750 c.c., and the carbon dioxide liberated on acidifying 1 c.c. of this is measured, which may be done in one extraction if the re-adsorption factor for the Van Slyke apparatus is known. This may be determined by comparing the results on a few beers with those obtained by Langer and Schultze's method, or by making a second extraction. For measuring under reduced pressure the small volumes of gas obtained in the latter case, a device consisting of a light rod supported vertically by a float on the mercury reservoir and carrying a pointer 500 mm. above the mercury surface is described. When the pointer is adjusted to the corrected level of the mercury in the pipette, the gas is at a pressure of 500 mm. below atmospheric pressure. Full details of technique and calculation are given, together with examples of the increase of the amount of carbon dioxide in beer during fermentation and storage. F. E. DAY.

Importance of acidity in brewing processes. P. KOLBACH (*Woch. Brau.*, 1930, 47, 145—149, 163—168).—A review and discussion of recent work. Since a correct acidity is of importance at every stage of the brewing operations, artificial acidification of mash or wort is specially considered. In the author's view, the former is best achieved by addition of cultures of *B. Delbrücki* or, alternatively, of lactic acid to the mash rather than to the water. Acidification of the wort improves colour and economises hops without affecting the buffering. F. E. DAY.

Change of buffer action on saccharisation of rice malt. K. MATSUMOTO and Y. KISE (*Bull. Res. Inst. Fermentation*, 1929, No. 102, 63—84).—When rice malt is saccharised at 55° the buffer action of the solution first increases and then decreases (apparently owing to the presence of lactic acid bacteria), the *pH* remaining constant. The buffer action differs with different acids, and is weaker towards alkalis than towards acids. The titratable acidity normally increases gradually during saccharisation. The buffer capacity is increased by addition of calcium salts, and is maintained constant by addition of lactic acid. When soluble starch or rice paste is saccharised by diastase or malt extract the buffer capacity remains constant.

CHEMICAL ABSTRACTS.

Investigation and evaluation of wine distillates and wine brandies. II. G. BÜTTNER and A. MIERMEISTER (*Z. Unters. Lebensm.*, 1929, 58, 628—635; cf. B., 1929, 373).—Eight French wines, compounded with wine distillates, have been subjected to analysis, with particular reference to their contents of

aldehydes, esters, and higher alcohols. The samples containing significant amounts of furfuraldehyde were low in acetaldehyde, whereas those containing no furfuraldehyde had a high acetaldehyde content. By studying the course of the distillation of wine, it was found that acetaldehyde distils early and furfuraldehyde largely at the middle of the run, but the esters and higher alcohols distil more or less regularly and no appreciable partition is effected. The opinions of Wüstenfeld and Luckow (cf. B., 1929, 574) and of Ruppig (cf. B., 1929, 573), that tasting should be an essential part of the testing of wine brandies and distillates, are further criticised. H. J. DOWDEN.

Composition of wines from Baden, with special reference to their arsenic content, and the use of Bezssonoff's reagent for the detection of vitamin-C. E. REMY and F. RICHTER (*Z. Unters. Lebensm.*, 1929, 58, 624—628).—A systematic analysis of eight wines of different origin has shown that arsenic is not a normal constituent and occurs only in insignificant amounts. The authors have confirmed the observation of Glassmann and Posdeev (cf. B., 1929, 575) that tannic acid gives Bezssonoff's reaction, and a further colorimetric test has been evolved for vitamin-C in the presence of tannin. When lemon juice treated with Bezssonoff's reagent, after keeping for 1 hr., is made feebly alkaline with 10% caustic soda and shaken, the violet colour changes to blue, which develops a violet tinge on re-acidifying carefully with 10% hydrochloric acid. Tannin solution with the reagent, when made alkaline, turns reddish-yellow, with a further change to green on re-acidifying. When a mixture of lemon juice and tannin solution is similarly treated, the colours produced are blue-green and dirty green, respectively. That the reaction is a test for vitamin-C has been confirmed by experiments on other antiscorbutic materials such as the juice of oranges, tomatoes, vegetables, etc. H. J. DOWDEN.

Improved manufacture of absolute alcohol. H. GUINOT (*Z. Spiritusind.*, 1930, 53, 101—102).—Absolute alcohol can be obtained directly from fermented liquids by the azeotropic method of distillation with the same expenditure of steam as is required to manufacture raw spirit, the costs of the two processes being about equal. Most of the water is separated, together with the slop, from the fermented liquid at the foot of the first of three distillation columns. Further amounts are removed at the bottom and top of the concentration column in which the dehydration hydrocarbon functions. In this column there is an intermediate zone from which the vapour consisting of 98.5—99.0% of alcohol mixed with a small amount of the dehydration liquid is led to the third column, which is smaller. The remaining moisture and hydrocarbon are removed and 99.8% alcohol is collected at the foot of the column. Dehydration is accompanied by purification, and the final alcohol is almost free from aldehyde and ethers. C. RANKEN.

Examination of brewery products with ultra-violet light. T. KOANA (*Bull. Res. Inst. Fermentation, Japan*, 1929, 102, 85—118).

Biochemistry of breadmaking. ELION. [Alcohol from] rice husks. DE MONGEOT.—See XIX.

PATENTS.

Fermentation process. W. L. OWEN, Assr. to CITIZENS OF U.S.A. (U.S.P. 1,744,001, 14.1.30. Appl., 21.12.27).—The seed yeast is retained for about 30 min. in the seed vat in contact with an amount of vegetable carbon equal to 5% by vol. of the wort to be fermented. The vat is then filled with freshly sterilised wort and, after the density of the wort is reduced by one half, the entire contents of the seed vat are transferred to a fermenter which is operated at 30–40° Brix until the fermentation is complete. C. RANKEN.

Treatment of wine residues. H. L. RICARD (F.P. 631,824, 30.3.27).—The wine residues, after pressing, are dried by an air current at 80–100°, and broken up into the separate constituents (skins etc.). C. RANKEN.

Jellification and solidification of alcoholic beverages, medicines, and alcohol. C. A. and G. COFMAN-NICORESTI (B.P. 326,447, 10.12.28).—Jellification etc. is effected by means of an alcoholic solution of pectin; gum arabic and gum tragacanth may also be added. H. ROYAL-DAWSON.

Cooling of metal vessels (Austr.P. 109,381).—See X. **Fats from grape marc** (F.P. 635,628).—See XII.

XIX.—FOODS.

Storage of food-grain. G. R. DUTT and A. N. PURI (Agric. J. India, 1929, 24, 245–250).—Attack of wheat by insects is prevented in closed containers by the presence of a small quantity of mercury or tin amalgam; no appreciable amount of mercury is absorbed by the wheat, the germinating power of which is not depressed. CHEMICAL ABSTRACTS.

Detection of flour bleached with chlorine or oxides of nitrogen. J. KULMAN (Z. Unters. Lebensm., 1929, 58, 635–649).—Solutions of potassium chromate or dichromate having been found unsuitable for colorimetric measurements on benzene extracts, a graduated scale was prepared from the most strongly coloured flour extract by dilution in stages of 10% with benzene, each dilution being evaluated in terms of a dye, with which the undiluted extract (No. 1 on the scale) was compared. Freshly milled flour had a colour value of 1–2, whilst flours bleached by chlorine or oxides of nitrogen had values between 5 and 9. To differentiate between chemically bleached flours and those bleached by natural ageing, it was found that when the benzene extract was evaporated at 110–115°, that from unbleached flour retains its original yellow colour, whilst that from chemically bleached flour changes to a reddish-brown. Spectroscopic investigations failed to reveal any differences in the absorption spectra of bleached and unbleached flours, as might be expected if the former contained nitro- or chloro-derivatives of carotene, and accordingly the behaviour of the extracting medium was examined. It was found that the brown coloration produced by evaporation of the extract at 110–115° was due to the action of chlorine or nitric oxide on toluene or xylene. Accordingly, it is recommended that the sample of flour (50 g.) be extracted with a mixture of benzene 85 c.c., toluene 10 c.c., and xylene 5 c.c., followed

by evaporation of the extract at 110–115° if the colour value on the scale described is within the limits 5–9. A brown coloration denotes a flour that has been bleached by chlorine or nitric oxide. H. J. DOWDEN.

Biochemistry of breadmaking. L. ELION (Chem. Weekblad, 1930, 27, 219–227).—The fermenting powers of brewers' yeast and bakers' yeast at different temperatures and for different flours are compared. The various factors influencing the fermentation and the baking are considered, and the importance of actual baking for testing purposes is stressed. The influence of bleaching agents, and of calcium and ammonium dihydrogen phosphates, is discussed. S. I. LEVY.

Utilisation of rice husks. L. B. DE MONGEOT (Giorn. Chim. Ind. Appl., 1930, 12, 74–76).—Rice husks consist of 60.04% of volatile matter, 24.79% of carbonaceous matter, and 15.17% of ash. Analysis of the ash gives SiO₂ 93.95, Fe₂O₃ 0.01, CaO 2.28, K₂O 3.15%, Mn₂O₄ and Al₂O₃ traces. The use of this silicious ash is suggested in the glass industry, the estimated annual production in Italy being 16,900 tons. Dry distillation of the husks gives a carbon of low density, and a liquid in which acetic and propionic acids, furfuraldehyde, phenol, and cresols were identified. Microscopical examination of the husks show them to be rich in cellulose, and they are suggested as a source of industrial alcohol. F. G. TRYHORN.

Milk. IV. Effect of heating on the constituents of milk. I. MATSUO (Osaka J. Med., 1929, 28, 555–562).—Raw and sterilised milk contained, respectively, total nitrogen 0.47, 0.449; albumin 0.410, trace; globulin 0.022, 0.014; caseinogen 0.370, 0.398%. Human milk (4–5 days after parturition) contained 1.41–1.66 mg. of ferric oxide per litre, in 13–44 days 1.12–1.57 mg., in 201–218 days 1.05–1.43 mg. Raw and boiled cow's milk contained 2.02 mg. (0.33 mg. only in a glass receiver); goat's milk contained 1.17 mg. CHEMICAL ABSTRACTS.

Heat-coagulation of milk as a function of acidity. A. TAPERNOUX and K. KATRANIEV (Compt. rend. Soc. Biol., 1929, 101, 828–829; Chem. Zentr., 1929, ii, 2953).—A definite relationship was observed. A. A. ELDRIDGE.

So-called low pasteurisation of milk. H. ZELLER, W. WEDEMANN, L. LANGE, and E. GILDEMEISTER (Arb. Reichsgesundh.-Amt, 1929, 61, 1–72; Chem. Zentr., 1929, ii, 2953).—Chemical and bacterial changes are recorded; the acidity is diminished, albumin and globulin are partly coagulated, the casein is altered, the catalase diminished, and the diastase destroyed. A. A. ELDRIDGE.

Analysis of sheeps'-milk cheese produced in the Tatra mountains. H. RUEBENBAUER (Przemysl Chem., 1930, 14, 169–178).—The fat content of the milk of sheep bred in the Tatra mountains varies from 6.86% in May to 9.12% in August, whilst the content of solids-not-fat remains constant (10.99–11.33%). An analysis of cheese made from the milk gives (%): water content 36.81, fat 33.5, ash 4.54 (including 2.47% NaCl), casein 15.17, albumin 1.32, amino-compounds 6.69, diamino-compounds 0.72, ammonia 0.08, and lactose

1.17%. The fat has m.p. 28.2°; f.p. 18.9°; critical dissolution temperature (alcohol) 53°; refractivity 44.1°; acidity corresponding with a free oleic acid content of 0.7191%; saponif. value 229.1; soluble and insoluble volatile acid value, respectively, 28.2, 3.8; non-volatile acid content 86.4%; iodine value 35.2; thiocyanogen value 35.2; octoic acid value 2.4; lauric, stearic, and oleic acid contents, respectively, 6.95, 4.7, 38.2%; palmitic and myristic acid content 33%; unsaponifiable residue 1.8%. The fat gave a positive aldehyde reaction and a blue luminescence on exposure to the mercury-vapour lamp. Sheeps' cheese thus differs from that of cows chiefly in its albumin content and in the properties of the fat, which is distinguished by its low refractive index, high ratio of soluble to insoluble volatile fatty acids, and by the high saponification value. The microscopical flora of sheeps' cheese consists chiefly in *Oidium lactis*, together with *Penicillium glaucum*, *B. lact. acid.* *Leichm.*, and *B. casei*.

R. TRUSZKOWSKI.

Determination of moisture in cheese. F. H. McDOWALL (New Zealand J. Sci. Tech., 1930, 11, 293—294).—A suggested standardised procedure as to (a) sampling, (b) preparation of samples, and (c) determination of moisture in cheese by direct heating in aluminium or nickel dishes, as recommended by the New Zealand Dairy Science Association, is outlined.

B. W. TOWN.

Effects of temperature on the ripening and keeping of fruits. E. L. OVERHOLSER (Proc. Int. Cong. Plant Sci., 1929, 2, 999—1011).—The course or character, as well as the rate, of metabolism, both in storage and in the field, may be modified by temperatures above the optimal. CHEMICAL ABSTRACTS.

Arsenic content of foods and of foods treated with arsenical preparations. T. VON FELLEBERG (Biochem. Z., 1930, 218, 300—317).—Ordinary foods and drinks contain only harmless amounts of arsenic, and no danger is to be feared from arsenic consumed with fruit which has been sprayed with arsenical compounds, although lead from lead arsenate sprays is a source of danger.

W. McCARTNEY.

Influence on the softening of peas of calcium and magnesium hardness of the cooking water. A. MÜLLER (Z. Unters. Lebensm., 1929, 58, 608—623).—An instrument has been devised by which the hardness of peas may be measured by the loading required to drive a needle (0.7 mm. in diam.) through the peas. By means of this apparatus a study has been made of the rate of softening of peas, previously swollen, when boiled in distilled water and in water of various degrees of hardness due to magnesium chloride, calcium chloride, or calcium bicarbonate. An increase in the time required by the peas to become tender was caused by all types of hard water, the influence of calcium hardness being more pronounced than that of magnesium. The observations support the hypothesis of van der Marel (cf. Pharm. Weekblad, 1922, 59, 82) that sparingly soluble pectin compounds are formed with the salts present in the water.

H. J. DOWDEN.

Electrolytic determination of copper in preserved vegetables. J. W. SPRINGER (Z. Unters. Lebensm.,

1929, 58, 651—652).—The copper salts used for conserving the green colour of preserved peas, beans, etc. may be quickly determined by heating a sample of the pulped material (100 or 150 g.) on a water-bath with 10 c.c. of nitric acid (*d* 1.4). To the mash, made faintly alkaline with ammonia, are added 20 c.c. of sulphuric acid (50%), the mixture being then diluted to 300 c.c. with hot water and electrolysed at 80—90° by the method previously described for the determination of zinc (cf. B., 1925, 175). With a current of 4—5 amp., the deposition is complete in 10—20 min. and, after washing the electrode, the deposit is redissolved in 10 c.c. of nitric acid. This copper solution is made feebly alkaline with ammonia, acidified with sulphuric acid, and then re-electrolysed, using a freshly ignited, weighed, platinum electrode. The weight of copper deposited on the electrode yields results in good agreement with those obtained by ashing processes. H. J. DOWDEN.

Use of benzoic acid and sodium benzoate as ingredients of fresh sausages and fresh minced meat. H. EICHLER, G. ENDRES, G. GMINDER, O. MEZGER, and J. UMBRECHT (Z. Unters. Lebensm., 1929, 58, 553—584).—A collaborative study has been made of the effects of incorporating sodium benzoate and benzoic acid in the preserving salt used in the manufacture of sausages. The mixture, which consisted of benzoic acid 8.5 pts., sodium benzoate 8.5 pts., and common salt 83 pts., was added in the proportion of 4 g. of preservative per kg. of sausage meat to various sausages and to raw minced beef. Controls of identical composition were prepared without preservative and the samples were then stored at three different laboratories, in the dark, at room temperature, and in the ice chamber. Examinations of colour, taste, smell, bacterial content, and decomposition (Eber's test) were made at frequent intervals. A survey of the results showed that (i) a concentration of 0.06% of benzoic acid approximately doubles the keeping qualities of the sausages; (ii) the red colour of the meat is not enhanced, but is in some cases slightly diminished; (iii) the growth of all types of bacteria is retarded although none is entirely eliminated. H. J. DOWDEN.

Determination of sulphurous acid in minced meat. G. STEINHOFF (Z. Unters. Lebensm., 1929, 58, 649—650).—A suspension of benzidine sulphate undergoes quantitative hydrolysis and may be titrated with caustic soda (cf. Raschig, B., 1903, 1066). The sample of meat (25 g.) is digested on a water-bath with dilute alkali and filtered off, and to the filtrate, acidified with acetic acid, are added 15 c.c. of benzidine hydrochloride solution (20 g. of benzidine dissolved in 35 c.c. of hydrochloric acid [*d* 1.19] and diluted to 1 litre). Albumin is removed from the extract by Carrez's method (cf. A., 1909, ii, 625) and the resultant solution is diluted to 250 c.c. and filtered. To 100 c.c. of the filtrate are added 10 c.c. of 3% hydrogen peroxide, whereby the sulphurous acid is oxidised and precipitated as benzidine sulphate, which is collected, washed, suspended in water, and titrated with 0.1N-caustic soda against phenolphthalein (factor 32). The method is found to be unsuitable for the examination of gelatin or dried fruit. H. J. DOWDEN.

Detection of the commencement of putrefaction of flesh, and a method for the determination of ammonium salts of flesh. B. GLASSMANN and F. ROCHWARGER (Z. Unters. Lebensm., 1929, **58**, 585—592).—The ammonium salt content of meat and fish has been found to provide a useful indication of the onset of decomposition and a method is described for the determination of ammonium salts in the presence of protein degradation products. A representative sample (100 g.) is washed, dried, and minced finely, 5 g. of the minced meat being then ground with 50 c.c. of water and collected on a filter. 10 c.c. of the filtrate are shaken with sodium permutit previously purified by treatment with 2% acetic acid, to absorb the ammonium salts. The permutit is washed several times by decantation and then treated with 5 c.c. of 10% caustic soda solution followed by about 150 c.c. of water and 10 c.c. of Nessler's reagent, the whole being finally diluted to 200 c.c. The colour is compared in a Duboscq colorimeter with that of a solution of ammonium sulphate (1 c.c. containing 0.121 mg. NH_3) mixed with identical volumes of caustic soda and Nessler's reagent and diluted to 200 c.c. The critical value for meat was found to be 0.02% NH_3 , and for fish 0.02—0.025%, according to the conditions of storage. H. J. DOWDEN.

Carbon dioxide preservation of meat and fish. D. H. KILLEFFER (Ind. Eng. Chem., 1930, **22**, 140—143).—Meat of various kinds, sausages, butter, cheese, and eggs were in a good condition after storing at 4—7° for 2 weeks in an atmosphere of carbon dioxide and did not show spoilage until after 3 weeks, but in air many of the meat samples were spoiled after 2 weeks. Meat and fish stored in carbon dioxide keep 2—3 times as long as in air at the same temperature; at temperatures above the f.p. they showed a slight fall in p_{H} , which rose again on exposure to the air. Many bacteria (but not *B. typhosus*) show much slower growth on nutrient-agar plates in an atmosphere of carbon dioxide than in air; this is attributed to the lowering of the p_{H} of the medium. E. B. HUGHES.

Food preservatives. T. SABALITSCHKA (Pharm. Ztg., 1930, **75**, 454—456, 466—468).—Methods used for the determination of the activity and efficiency of preservatives and their bactericidal action under varying conditions are discussed, together with the influence of different physical factors and the character of the foodstuffs on the action of a substance as a preservative. The relative activity of a large number of substances including phenols and esters of *p*-hydroxybenzoic acid is described. Methyl *p*-hydroxybenzoate ("Nipagin") and particularly the higher esters of this acid have a much stronger action against a large number of organisms, especially moulds, than any of the usual preservatives. E. H. SHARPLES.

Digestibility trials on Indian feeding stuffs. VI. Green fodders and silage. P. E. LANDER and P. L. C. DHARMANI (Mem. Dept. Agric., India, 1930, **10**, 193—208).

Milkweed. GERHARDT.—See V. Effect of cooking

salt on butter. BUDAGJAN and PAVLOV. Butter fat in cooking fats. PRITZKER.—See XII. Saccharisation of rice malt. MATSUMOTO and KISE. Detection of vitamin-C. REMY and RICHTER.—See XVIII.

PATENTS.

Whole-grain flour and the like. J. SCHNEIBLE, Assr. to C. B. SCHNEIBLE and K. F. SCHREIER (U.S.P. 1,744,581, 21.1.30. Appl., 7.12.23).—In the preparation of flour, the moistened whole grain is allowed to germinate and is then dried at 21—30° in such a manner that the enzymes are not injured, but insect eggs, larvæ, and moulds are destroyed. The product, containing less than 6% of moisture, is then ground. E. B. HUGHES.

Improvements of cereals, cereal flours, and food products therefrom. A. S. POLLAK (Austr.P. 109,163, 31.7.26).—The finely-divided material is placed in a sieve, and the sifted material, as it falls from the sieve, is treated with a vaporised solution of hydrogen peroxide. C. RANKEN.

Production of bread. A. M. J. SOREL (F.P. 569,990, 22.8.23).—Bread is manufactured without addition of leaven or yeast by introducing carbon dioxide under pressure into dough prepared with water saturated with carbon dioxide. C. RANKEN.

Preservation of eggs. T. M. RECTOR, Assr. to EMULSOL CORP. (U.S.P. 1,744,575, 21.1.30. Appl., 28.9.26).—Egg yolk is preserved in a suitable condition for use in mayonnaise manufacture by saturating it with salt (about 10%) and then storing it at a temperature slightly above the f.p. of the mixture.

E. B. HUGHES.

Egg-preserving compositions. (Mrs.) C. C. ALEXANDER (B.P. 326,325, 25.2.29).—A mixture of lime, liquid petrolatum, paraffin wax, and formaldehyde is claimed. H. ROYAL-DAWSON.

Sausage products and their manufacture. MODERN FOOD PROCESS CO. (B.P. 326,847, 21.12.28. U.S., 10.11.28).—A protective membrane is produced on sausages by exposing the moulded sausage mixture to a damp atmosphere at about 35°, drying at 43° for a short time, and then smoking at 43—74°. The sausages may then be boiled or canned in the usual way.

E. B. HUGHES.

Manufacture of food products. G. GRINDROD (B.P. 326,156, 29.8.28).—Liquids containing protein (pre-heated to about 43°) are placed in a closed container fitted with a hollow revolving arm carrying a large number of small nozzles. Steam at 25—50 lb. is passed through the nozzles into the liquid, which attains a temperature of about 110° by maintaining the container pressure at 6—10 lb./in.² It is claimed that in 3 min. the contents become sterile and their physical state is considerably changed. In the case of milk the globulin is destroyed, the albumin re-dispersed in some other colloidal form, and the casein becomes hydrated and resistant to coagulation. After this treatment milk may be concentrated to 50—65% of total solids, and when cooled it sets to a translucent yellowish gel which remains unchanged for months in an open container.

On dilution this gel reverts to ordinary milk having its appearance and flavour unchanged and complete solubility, but without the usual property of coagulation. By this process also (1) a concentrated ice-cream mix may be made as a gel; (2) gelatin may be sterilised without affecting its gelatinising power; (3) preparations of chocolate and candy products may be made as jellies; (4) preservation of ground meat, sausages, etc. may be effected by embedding in gelatin and subjecting the mixture to the treatment. E. B. HUGHES.

[Puffed] cereal foods. JERSEY CEREAL CO., ASSEES. OF C. E. LUKE (B.P. 299,803, 17.10.28. U.S., 1.11.27).

Margarine (B.P. 326,742).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Relationship between enzymes and alkaloids of *Atropa belladonna*. L. J. P. TODD (J. Roy. Tech. Coll. Glasgow, 1930, 2, 353—365).—Fresh belladonna leaves may be stabilised by heating in alcohol vapour at a pressure of 1.5—2.5 atm. with only slight loss of alkaloid, but this process has no advantage over careful drying, and the appearance is impaired. Losses of alkaloid of up to 20% occur when the leaves are allowed to ferment in presence of antiseptics at 32°. At 55° no loss occurs. The emulsification of the solvent in the process of determining the alkaloid content is most troublesome in carefully dried and enzyme-free leaves, and an improved method is described in which ether-soluble inert matter is first extracted from the powdered leaves moistened with acid. R. K. CALLOW.

Chemical and photochemical oxidation of commercial adrenaline solutions. T. VACEK (Compt. rend. Soc. Biol., 1929, 109, 583—584; Chem. Zentr., 1929, ii, 3026—3027).—The p_H values of commercial adrenaline solutions varied between 1.6 and 4.0. The rate of oxidation with hydrogen peroxide and in light is independent of the p_H , whilst an increase in the rate with increasing p_H was observed with pure solutions of adrenaline hydrochloride. The intensity of photo-oxidation depends on the intensity of the light and on the wave-length; irradiation with ultra-violet light alone causes less change than that with visible and invisible rays at the same time. A. A. ELDRIDGE.

Significance of the degree of dissociation of acids in the oxidation of adrenaline. T. VACEK (Compt. rend. Soc. Biol., 1929, 100, 585—586; Chem. Zentr., 1929, ii, 3027).—The speed of oxidation of adrenaline solutions is directly proportional to the hydrogen-ion concentration only when the same solvent is employed. A. A. ELDRIDGE.

Alkaloid determination in ergot. H. OETTEL (Arch. exp. Path. Pharm., 1930, 149, 218—239).—For testing ergot and its preparations the methods of D.A.B. VI (cornutin reaction and precipitation with Mayer's reagent) are inapplicable. A simple and reliable method using sodium carbonate is described for testing qualitatively the therapeutically active alkaloid content of *Secale cornutum* and preparations from it. For quantitative purposes the methods of Keller-Fromme

and of the German pharmacopœia are unreliable. Forst's method, slightly modified, gives unobjectionable results. The drug is first extracted by percolation with alcohol, the crude product purified by means of chloroform-ether, precipitated from a sulphanic acid solution with sodium carbonate, and weighed. The alkaloid contents of various commercial preparations varied greatly. T. R. SESHADRI.

Determination of morphine in opium. E. STUBER and B. KLJATSCHKINA (Arch. Pharm., 1930, 268, 209—223).—Morphine is most completely precipitated from its hydrochloride by ammonia at p_H 9.2, or in 2% ammonium chloride solution at p_H 8.9. For opium extracts the optimum p_H of precipitation varies with the sample. Precise directions are given for the determination of morphine in crude opium or opium powder by this method. The sample is extracted with water at 55° for 4 hrs. and kept overnight. After narcotine etc. have been removed from the extract by exact neutralisation with ammonia, three trials are sufficient to determine the maximum amount of morphine which can be precipitated by further addition of ammonia. The results, corrected for solubility, are higher than those given by Dieterich's method, and agree with those obtained when calcium hydroxide or borax is the precipitant. H. E. F. NOTTON.

Determination of morphine in tablets and pills. J. LAURENCE (J. Pharm. Chim., 1930, [viii], 11, 336—337).—About 10 g. of the powdered tablets are rubbed in the mortar with a mixture of 1 g. of calcium hydroxide and 15 c.c. of distilled water, the mixture is filtered, and the filtrate and washings are made up to 35 c.c. Hydrochloric acid is added until the liquid is just acid and then 1 g. of potassium iodide. When this is dissolved, exactly 15 c.c. of 0.1N-iodine are added and the mixture is agitated gently for 2 min. and set aside for 1 min. An aliquot of the supernatant liquid is filtered off and titrated with 0.1N-sodium thiosulphate. One mol. of morphine combines with 3 atoms of iodine.

E. H. SHARPLES.

Detection of nickel in drugs. L. ROSENTHALER (Pharm. Zentr., 1930, 71, 241—242).—The ash from 10 g. of the drug is warmed with aqua regia, the solution filtered, and the filtrate made alkaline with ammonia. The precipitate is filtered off and alcoholic dimethylglyoxime solution (Tschugaev) is added to the filtrate. The presence of nickel is indicated by a red colour. Results of the examination of a large number of drugs and herbs are given. E. H. SHARPLES.

Determination of the alcohol content of tincture of iodine. K. FEIST and F. KLATT (Pharm. Ztg., 1930, 75, 424).—The iodine is taken into combination by addition of water and iron powder; after reaction, the mixture is distilled, and the distillate analysed in the usual way. S. I. LEVY.

Detection of isopropyl alcohol in alcoholic pharmaceutical preparations. T. BOEHM and K. BODENDORF (Arch. Pharm., 1930, 268, 249—262).—Previously described methods are criticised (cf. Reif, B., 1928, 686).

When an aqueous solution of isopropyl alcohol is superposed on concentrated sulphuric acid containing *m*-nitrobenzaldehyde, a carmine-red ring appears at the point of contact. Methyl and ethyl alcohols and acetone neither give nor vitiate the reaction, but *n*-propyl, isobutyl, and amyl alcohols give a similar colour. The application of the test to tinctures and spirits is described. The detection of acetone in presence of acetaldehyde by means of sodium nitroprusside in presence of ammonia and ammonium chloride has been studied, and a method is given for the colorimetric determination of isopropyl alcohol, after oxidation, by this reaction.

H. E. F. NOTTON.

Chemical evaluation of Extractum Filicis maris. Z. CSIPKE (Magyar Gyó. Társ. Ert., 1929, 5, 425—438; Chem. Zentr., 1929, ii, 3045).—The pure filicic acid content is not an exact measure of the activity of the drug; the determination of the crude filicic acid content gives inaccurate values. The value of the extract is best expressed in terms of phloroglucin or phlorobutyrophenone. The extract is dissolved in ether, shaken twice with baryta water, the aqueous solution is filtered, and an aliquot portion treated with ammoniacal silver nitrate. After 6—10 hrs. in the absence of light the silver is collected, washed, dissolved in nitric acid, and titrated; 0.0108 g. corresponds with 0.00217 g. of phloroglucinol or 0.0196 g. of phlorobutyrophenone.

A. A. ELDRIDGE.

Adulteration of Flores Tiliae, D.A.B. VI. H. KAISER and K. EGGENSEPERGER (Süddeut. Apoth.-Ztg., 1929, 69, 450—451; Chem. Zentr., 1929, ii, 3045).—A taste test is needed; capillary and fluorescence analysis is insufficient.

A. A. ELDRIDGE.

[Biological] evaluation of digitalis. II. Comparison of Mansfeld's sinus method with the 6-hr. frog or cat method. A. STASIAK and B. ZBORAY (Magyar Gyó. Társ. Ert., 1929, 5, 400—406; Chem. Zentr., 1929, ii, 3045).

Detection of vitamin-C. REMY and RICHLER.—See XVIII. **Preservatives.** SABALITSCHKA.—See XIX.

PATENTS.

Extracting nicotine from tobacco in continuous operation. S. E. PAGE. From BIGOT SCHÄRFE & Co. CHEM. FABR. G.M.B.H. (B.P. 327,034, 22.4.29).—A mixture of tobacco with an excess of an alkali hydroxide is passed by means of a screw-conveyor through a horizontal, steam-jacketed cylinder in which it is treated with a countercurrent of superheated steam and/or hot, non-reacting gases, *e.g.*, furnace gases; nicotine is recovered from the gases and vapours leaving the cylinder.

L. A. COLES.

Treatment of tobacco. W. W. TRIGGS. From AMER. MACHINE & FOUNDRY Co. (B.P. 326,400, 6.5.29).—An apparatus is described whereby the volume of cured tobacco is increased (about 15%) by treatment (at 110°) under pressure (20 lb.) with air or steam, with subsequent sudden release of pressure.

E. B. HUGHES.

Preparation of thymol. RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 298,600, 325,856, and 326,215,

30.8.28. Ger., 12.10.27).—*m*-Cresol, either (A) liquid, or (B) gaseous, is condensed with propylene, or substances which yield propylene by dehydration, in presence of dehydration catalysts such as alumina, zinc chloride, etc. Thymol is separated by distillation from two isomerides (m.p. 69°, b.p. 228.5°, and m.p. 112°, b.p. 245—246°, respectively). (C) The isomeric propyl- and isopropyl-*m*-cresols obtained as in (A) are converted largely into thymol by heating with dehydration catalysts (aluminium silicate) at 230°.

C. HOLLINS.

Separation of alkoxyisoeugenol from alkoxyisochavibetol and production of isoeugenol from the separated compounds. GRAESSER-MONSANTO CHEM. WORKS, LTD., Assees. of T. S. CARSWELL (B.P. 303,021, 24.12.28. U.S., 22.12.27).—The mixture of methoxymethyl ethers of 3:4-dihydroxyisopropenylbenzene, obtained, *e.g.*, from safrole or isosafrole by heating with methyl-alcoholic alkali at 135°, is converted into calcium salts by adding lime to the alcoholic solution. The insoluble portion is filtered off and acidified to yield the 4-ether ("methoxyisochavibetol"), and the 3-ether ("methoxyisoeugenol") is recovered from the filtrate. Each is then converted into isoeugenol, in one case by methylation and hydrolysis of the methoxymethyl group, and in the other by hydrolysis and methylation.

C. HOLLINS.

Manufacture of aqueous solutions of barbituric acids. A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 325,847, 30.11.28).—Monoalkylamides of 5:5-disubstituted barbituric acids are sufficiently soluble in water to be useful for intravenous injection.

C. HOLLINS.

Manufacture of esters of [methyl-substituted] 2-phenylquinoline-4-carboxylic acids [atophans]. A. J. STEPHENS. From R. VON WÜLFING and E. MÜLLER (J. A. WÜLFING) (B.P. 325,985, 8.2.29).—Methyl-substituted atophans are heated at about 100° with 3—4 times the theoretical proportion of *n*-propyl, *n*-butyl, or isobutyl hydrogen sulphate. The three 2-phenyl-6-methylquinoline-4-carboxylates have m.p. 79—80°, 64—65°, and 74—75°, respectively; the 6:8-dimethyl compounds melt at 73—74°, 80°, and 83°, respectively.

C. HOLLINS.

Manufacture of alkylamine derivatives of organic compounds. A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 326,553, 17.12.28).—The usual methods are applied to the production of β -diethylaminoethylamino-anthracenes or -phenanthrenes containing hydroxyl groups. *E.g.*, 1-bromoanthraquinone is condensed with β -diethylaminoethylamine and reduced to 1- β -diethylaminoethylamino-9:10-dihydroxyanthracene; 3-nitrophenanthraquinone is similarly converted into 3- β -diethylaminoethylamino-9:10-dihydroxyphenanthrene, b.p. above 250°/1 mm. The products have therapeutic value.

C. HOLLINS.

Manufacture of organic mercury compounds. A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 325,846, 30.11.28).—Aromatic hydrocarbons (benzene) are refluxed with mercuric oxide or acetate in glacial acetic acid for 2—3 hrs. at 90—95°.

C. HOLLINS.

Manufacture of metal-organic complex salts. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 326,209, 4.12.28).—Carboxylic or sulphonic derivatives of resorcinol, particularly resorcinol-4:6-disulphonic acid, form therapeutically useful complex salts when treated with solutions of metal salts (silver nitrate, thallium chloride) and ethylenediamine. C. HOLLINS.

Production of a composition [which emits β -rays]. M. WRESCHNER and L. F. LOEB, Assrs. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,752,826, 1.4.30. Appl., 4.3.26. Ger., 6.3.25).—See B.P. 248,765; B., 1926, 769.

Introduction of iodine into pyridine derivatives. G. KOCHENDOERFER, Assr. to DEUTS. GOLD- u. SILBERSCHEIDENSTALT VORM. ROESSLER (U.S.P. 1,753,170, 1.4.30. Appl., 1.2.26. Ger., 24.1.25).—See B.P. 246,501; B., 1927, 507.

Preparation of solutions of the active principle from animal organs. W. LUDWIG and O. SCHAUMANN, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,752,916, 1.4.30. Appl., 6.10.27. Ger., 15.10.26).—See B.P. 279,123; B., 1929, 226.

Distillation of glycerin (U.S.P. 1,743,289).—See I. N-Substituted arylaminesulphonyl chlorides (B.P. 326,226).—See III. Solidification of medicines etc. (B.P. 326, 447).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photosensitive preparations of selenium and tellurium. T. PAVOLINI (Giorn. Chim. Ind. Appl., 1930, 12, 72—74).—Mixtures of tartaric acid and ferric salts with selenious acid or with potassium tellurite are reduced in the presence of light with the formation of amorphous selenium or tellurium. The reaction proceeds in two stages, according to the equations $C_4H_6O_6 + 4FeCl_3 \rightleftharpoons 4FeCl_2 + 4HCl + C_4H_2O_6$, $H_2SeO_3 + 4FeCl_2 + 4HCl \rightleftharpoons 4FeCl_3 + 3H_2O + Se$, but in the case of tellurium the second reaction is easily reversible and the precipitated tellurium redissolves in the presence of excess ferric chloride solution. Details are given of the preparation of sensitive papers with these reactions as a basis. The selenium paper gives a bright red and the tellurium a black to brown tone. Development and fixation may be carried out with a dilute solution of hydrochloric acid, with the addition, in the case of the tellurium paper, of stannous chloride to prevent the destruction of the image by the unchanged ferric chloride. F. G. TRYHORN.

PATENTS.

Manufacture of light-sensitive surfaces for use in colour photography. K. BIALON (B.P. 326,287, 15.1.29).—Certain dyes form with silver bromide complex compounds which are sensitive to the light rays absorbed by the dye (acid-green, brilliant-blue, acid-violet, violamine, ponceau, azo-yellow), which, however, is not itself appreciably affected. Aqueous potassium bromide (1%) is added to a mixture of dye solution (0.5%) and silver nitrate (1%); the

precipitate, after washing with acetic acid, is mixed with gelatin solution (5%), a little ammonia is added, and the mixture, heated to 33°, is used for coating glass plates. C. HOLLINS.

Production of photographic images. H. D. MURRAY, Assr. to NORTON & GREGORY, LTD. (U.S.P. 1,753,059, 1.4.30. Appl., 14.9.29. U.K., 14.7.28).—See B.P. 317,199; B., 1929, 872.

[Apparatus for] colour photography, in particular, cinematography. P. VEROLA (B.P. 308,973, 27.3.29. Fr., 2.4.28).

XXII.—EXPLOSIVES; MATCHES.

Nitrocellulose of low viscosity. M. G. MILLIKEN (Ind. Eng. Chem., 1930, 22, 326—328).—The reduction in viscosity of nitrocellulose from 500 to $\frac{1}{2}$ sec. by heating with water is carried out by a continuous process, the mixture passing through a tube 4 in. in diam. and 4000 ft. long, which is heated by a steam-jacket for a distance at the entrance, is heat-insulated along most of its length, and is cooled by a water-jacket near the exit end. Flashing of the water into steam is prevented by the back-pressure of a stand-pipe 200 ft. high connected to the exit, from which the treated mixture overflows into receiving tubs below. The process is safer and yields a more uniform product than that of batch-digestion in autoclaves. F. R. ENNOS.

PATENTS.

Blasting cartridge, percussion cap, detonator, detonating fuse, and the like. O. TUREK (U.S.P. 1,743,739, 14.1.30. Appl., 25.2.29. Czechoslov., 28.3.28).—A detonator contains as secondary charge compressed trinitrotoluene, tetryl, or 2:4:6-trinitro-1:3:5-triazidobenzene, and above this a priming charge of 0.05—0.2 g. of 2:4:6-trinitro-1:3:5-triazidobenzene pressed at a pressure not exceeding 300 kg./cm.² A perforated cap is finally inserted.

W. J. WRIGHT.

Explosive powder. W. F. VAN VACTOR (U.S.P. 1,743,941, 14.1.30. Appl., 30.8.28).—The explosive is prepared by boiling sugar with water at 112.7—135° without stirring, and adding Irish moss jelly, potassium chlorate, and crude fuel oil. It contains potassium chlorate 40—60%, sufficient sugar to make the total with the chlorate 97%, Irish moss jelly 0.5%, fuel oil 0.5%, and water 2%.

W. J. WRIGHT.

Low-velocity ammonia-dynamite. W. H. WARD, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,743,172, 14.1.30. Appl., 14.8.23).—For the manufacture of non-inflammable blasting explosives, containing nitroglycerin, ammonium nitrate, sodium nitrate, and a combustible, "relatively coarse" ammonium nitrate is used, its fineness being such that not more than 5% is retained by a 10-mesh, and not more than 20% passes a 60-mesh sieve. The strength of these dynamites lies between 10 and 60%, and their velocity of detonation does not exceed 2000 m./sec. A 20% dynamite contains nitroglycerin 4—7%, "relatively coarse" ammonium nitrate 23—27%, and sodium nitrate and combustible 68—74%.

W. J. WRIGHT.

Percussion fuses for projectiles. SCHNEIDER & CIE. (B.P. 315,852, 23.5.29. Fr., 20.7.28).

Distillation of glycerin (U.S.P. 1,743,289).—See I.
Separation of mixed acids (B.P. 326,977).—See VII.

XXIII.—SANITATION; WATER PURIFICATION.

Effect of certain acids on [sewage] sludge digestion. H. W. CLARK and G. O. ADAMS (Sewage Works J., 1929, 1, 393—397).—Lactic acid is less readily attacked by the bacteria of digesting sludge than is acetic, butyric, formic, or oxalic acid, but under certain conditions it is readily attacked. Unless present at the beginning of fermentation organic acids are not necessarily injurious to digesting sludge.

CHEMICAL ABSTRACTS.

Effect of iron compounds on [sewage] sedimentation, digestion, and ripe-sludge conditioning. W. RUDOLFS, L. R. SETTER, and W. BAUMGARTNER (Sewage Works J., 1929, 1, 398—410).—The addition of 5% of ferric chloride to raw, screened, domestic sewage increased the rate of settling of suspended solids from 24% to 53%. Small quantities of ferric chloride shortened the digestion time, but decreased the quantity of gas formed and the percentage reduction in volatile solids. Large quantities were beneficial to sludge digestion. The drying of digested sludge was accelerated by addition of ferric chloride. CHEMICAL ABSTRACTS.

Comparison of diffused-air and stream-flow aeration in purification of packing-house wastes. M. LEVINE, H. N. JENKS, and F. G. NELSON (Sewage Works J., 1929, 1, 425—430).—The latter gave a more rapid reduction of organic nitrogen, oxygen consumed, and five-day biological oxygen demand, whilst the former gave an effluent of better appearance.

CHEMICAL ABSTRACTS.

Engineering studies of municipal zeolite water-softening. H. N. JENKS (J. Amer. Water Works' Assoc., 1930, 22, 342—356).—The efficiency of a zeolite water-softening plant depends largely on the intimacy of the contact of the water and the medium. Experimental studies with a practical-scale plant indicate that at low rates of flow an upward-filtration plant loses efficiency owing to "channeling," whilst at high rates a downward-filtration plant suffers from an excessive loss of head. It is suggested that large plants should be designed so that they can be operated in either direction, according to the rate of flow, or if the latter is constant they should be either shallow beds employing downward filtration or deep beds with an upward flow. The zeolite may be regenerated more economically if the brine solution can be recirculated and the volume of wash-water used can be considerably reduced by replacing the usual 18—22-in. bed of gravel on which the sand rests by a bed of asphalt-bound gravel about 8 in. thick. For use in a large plant the asphalt-gravel bed may be made of slabs instead of being constructed *in situ*. In the discussion of the paper a simple form of apparatus is described in which the softening efficiency of the zeolite may be determined. C. JEPSON.

Scientific control [of water-purification plant]

from the operator's viewpoint. R. G. YAXLEY (J. Amer. Water Works' Assoc., 1930, 22, 387—395).—The plant at Waterford, N.Y., obtains a supply of soft water from the Hudson River which is badly polluted with paper-mill wastes and sewage. The effective coagulation range is limited, a divergence of 0.15 either way from the optimum p_H value of 5.95 being noticeable in the basin reactions. As these changes occur without any apparent alteration in flow, colour, turbidity, etc., p_H determinations are regularly made and sulphuric acid is used to reduce any excessive alkalinity. By this means it has proved possible to obtain excellent coagulation over a wide range of water conditions without a radical increase in the rate of alum feed. C. JEPSON.

Colour test of floc in treating coloured water. G. G. NASMITH (J. Amer. Water Works' Assoc., 1930, 22, 396—398).—The length of filter runs in an experimental plant at Ottawa was shown to be directly dependent on the quantity of floc leaving the settlement basins. As the colour removed by the floc constitutes part of the floc, the quantity present may be estimated by replacing the colour in solution by acidifying and comparing it with a sample similarly treated but containing the whole of the floc. By this test much time was saved in discontinuing unsuccessful experiments, and filter runs were increased finally from 5 to 65—70 hrs.

C. JEPSON.

Disposal of waste liquors from textile plants. COPSON and CURTIS.—See V.

PATENTS.

Removal of odours from gases and air. J. T. TRAVERS, Assr. to TRAVERS-LEWIS PROCESS CORP. (U.S.P. 1,738,543, 10.12.29. Appl., 18.5.28).—Waste gases from packing houses, fertiliser or rendering plants, etc. may be deodorised by passing through an absorption tower in countercurrent to a creamy mixture containing calcium hydroxide, calcium sulphate, ferrous hydroxide, and marl, a suitable mixture containing 1 pt. of solids to 4 pts. of water. The proportions of the solid ingredients may be varied within wide limits, but the marl should be 50—80% of the whole, should contain 70—95% of calcium or magnesium carbonate and not less than 5% of matter insoluble in hydrochloric acid (1:1), and should have a "flotation value" (rate of settling in still water) of less than 4 in./min. The amount of lime used may be 15—35% provided there is always sufficient to react completely with ferrous sulphate (5—15%) to produce the remaining ingredients.

C. JEPSON.

Protection against the noxious effect of inhaling mercury vapour. GEHR. MERZ MERZ-WERKE (G.P. 455,974, 6.5.26).—Air to be breathed is passed through permeable material having a large superficial area, e.g., fibrous or powdered material, covered with a very thin layer of metallic gold, and contained in a gas-mask.

J. S. G. THOMAS.

Mixing apparatus (U.S.P. 1,745,291). **Filtration medium** (U.S.P. 1,743,524—5).—See I. **Moth-proofing agents** (B.P. 326,137). **N-Substituted arylamine sulphonyl chlorides** (B.P. 326,226).—See III. **Water-softening material** (U.S.P. 1,744,703).—See VII.