# BRITISH CHEMICAL ABSTRACTS

# B.—APPLIED CHEMISTRY

AUG. 1 and 8, 1930.\*

# L-GENERAL; PLANT; MACHINERY.

Tests on a Birmingham [town's gas-fired oven-] furnace installation. C. M. WALTER (Gas J., 1930, 190, 576—577).—The developments in oven-furnace design which have occurred during recent years and the improvement of working efficiencies by the employment of special insulating materials, improved recuperator elements, the exact control of primary and secondary air, and the reduction of losses by leakage through doors and dampers are described. C. B. MARSON.

Critical relations between water and steam. H. L. CALLENDAR (Proc. Inst. Mech. Eng., 1929, 811-838) .- Equations hitherto proposed to represent the pressure-volume relations of gases near the critical point are based on the theory of the continuity of state. Discrepancies occur in all cases between theory and observation, and it is suggested that these are due to an error in the fundamental assumption of molecular identity between the two states. As the latent heat of fusion of ice is so much less than that of the vaporisation of steam, the author considers it reasonable to suppose that in water linkings exist approximating to the hexagonal-plate structure of ice on Bragg's model. On this view the Joule-Thomson equation for water becomes V - b = RT(V - c), in which c represents the defect of volume due to co-aggregation and is a function of V and T. The combination of this with the relation between total heat and volume gives very close values of the saturation pressures up to the critical point. Observation of the point of disappearance of the meniscus in a fresh quartz-glass tube gave a value for the critical temperature of  $374 \cdot 0^{\circ}$ , a figure higher than that previously recorded. This figure was verified by latent heat measurements in the critical region, and the results obtained were found to be in accordance with the co-aggregation theory. Beyond the critical point there are no longer distinct phases, but two mixtures of different density can still be observed and the latent heat does not entirely vanish at the critical temperature. Pressure-heat diagrams are given and the advantage to be gained by increasing pressure is discussed. This is less than has been generally supposed, and steam near the critical point would require a very high degree of superheat to be fitted for direct use in a turbine. The constants and equations on which an international system of defining the properties of steam can be based are discussed. C. IRWIN.

Submerged combustion. HAMMOND. Coal-tar oil for furnaces. LUHN. Graphite lubrication. CORDEBAS.—See II. Rubber linings. COURBIER and BOUSSAND.—See XIV. Boiling of worts. WORSSAM. —See XVIII.

# PATENTS.

**Furnaces.** I. W. FOLTZ (B.P. 329,010, 5.2.29).—In a boiler furnace having a fire-box, fire-bridge, and further combustion chamber, air is taken from the ashpit and preheated above the ignition point of the fuel in passages formed in the walls or floor of the combustion chamber and is then returned to the fire-box above the burning fuel and near the stoking door. B. M. VENABLES.

[Boiler] furnace and process of operating the same. H. J. KERR, ASST. to FULLER LEHIGH CO. (U.S.P. 1,747,676, 18.2.30. Appl., 8.10.27).—It is considered that the presence of incombustible dust in the combustion gases promotes transmission of heat to the boiler tubes (or other object to be heated). A dust catcher, of the type having vertical tubes which are slit longitudinally, facing the oncoming gases is placed in the flue and the dust collected therein transferred to a hopper, whence it is mixed with the pulverised fuel going to the boiler. B. M. VENABLES.

Apparatus [boiler furnace] for heating fluids. CARBORUNDUM CO., LTD. From CARBORUNDUM CO. (B.P. 329,316, 9.1.29).—The combustion space has walls and roof formed of water-tubes, with or without external walls of non-conducting brick; in the lower part an internal lining of good conducting material such as silicon carbide is provided. B. M. VENABLES.

Furnace fronts. J. HOWDEN & Co., LTD., and J. H. HUME (B.P. 329,879, 29.6.29).—A pulverised-fuel burner fitted to a furnace door is described. B. M. VENABLES.

Handling and removing furnace residues and other solid materials. ASH CO. (LONDON), LTD. (B.P. 329,840, 18.5.29).—The ash drops (continuously) into a large volume of water contained in a hopper; to remove the ash (intermittently) the water is first drained off, in order to prevent a sudden rush choking everything, and the self-supporting residue is sluiced away by jets of water. Suitable apparatus is described.

B. M. VENABLES.

**Continuous kilns.** C. Rost (B.P. 311,246, 7.5.29. Ger., 7.5.28).—In a kiln of the Hoffmann type fired with powdered fuel, the mixture of fuel and air is admitted to the kiln at the lowest possible speed through a number of long narrow slots in the walls and roof and the passages leading thereto are preferably flared towards the chamber, with the object of obtaining a cloud of flame and consequent uniform heating.

B. M. VENABLES.

Kiln or retort for continuous working. H. M. ROBERTSON (B.P. 317,066, 23.1.29. U.S., 10.8.28).—A tunnel kiln, stated to be suitable for recovering hydrocarbons from materials such as shale or briquettes, is

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

CL. I.-GENERAL; PLANT; MACHINERY.

constructed with preheating, heating, and cooling zones in line; each zone is provided with outlet conduits connecting with a suction device and is separately controllable. The walls of the preheating zone have projecting pillars built on them to cause the longitudinally travelling waste gases to eddy and circulate.

B. M. VENABLES.

Kiln bottoms. J. KNOWLES & Co. (WOODEN BOX), LTD., and H. J. TAYLOR (B.P. 329,443, 25.3.29).—The bottom of a downdraught kiln formed of perforated blocks has specially shaped perforations over the supporting walls so that gases may leave uniformly over the whole floor. B. M. VENABLES.

Heat exchangers. GRISCOM-RUSSELL Co., Assees. of K. B. RIS (B.P. 308,715, 19.2.29. U.S., 27.3.28).—In an exchanger of the bundle-of-tubes type, the outer fluid is caused to flow in an alternate rotating and axial manner by means of baffles strung on the tubes which are alternately (a) flat, having a clear passage through the centre; and (b) bent, having no perforations other than those occupied by the tubes and bolts, but being slit radially nearly to the centre; the sector-shaped blades thus formed are twisted so as to afford helical, circumferential passages for the outer fluid.

B. M. VENABLES.

Heat exchanger. L. S. QUENSEL and F. A. STEPHENS (U.S.P. 1,746,497, 11.2.30. Appl., 11.12.28).—A number of hollow, tapering drums are assembled on a hollow shaft forming a bellows-like structure which is rotated in the liquid to be heated (or cooled); the heating medium is supplied and removed through the hollow shaft, which has driving means and a stuffing box at the upper end and a bearing at the bottom.

B. M. VENABLES.

Heat interchangers. F. I. E. STENFORS (B.P. 329,714, 26.2.29).—In an exchanger of the conducting-plate-andframe (filter-press) type, in which the two fluids flow at right angles to each other in alternate frames, the transfer ports are made as long as the internal width, and as wide as the thickness, of the frames, the latter being bevelled internally to permit free flow of the fluids at the return bends. B. M. VENABLES.

Heat-interchange apparatus. G. CAHILL (U.S.P. 1,745,978, 4.2.30. Appl., 24.2.27).—An exchanger similar to a honeycomb radiator is provided with specially-shaped tubes for use round the sides to give a flat surface to which the frame may be attached. Adjacent the inlet and outlet connexions more space is provided for the fluid by reducing the size of the intermediate parts of the honeycomb tubes much below that of their polygonal ends, *e.g.*, by flattening them to cruciform, crescent, or other shape. B. M. VENABLES.

Plate apparatus for heat exchange. R. SELIGMAN (B.P. 310,411,15.3.29. Holl., 25.4.28. Addn. to B.P. 327,377; B., 1930, 490).—In an apparatus as described in the prior patent, the flow of vapour is zigzag vertically, but passages are left at the lower ends of the ribs for direct flow of condensate, these passages being small and increasing in size towards the outlet.

B. M. VENABLES.

Catalytic apparatus. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,741,309, 31.12.29. Appl., 13.12.27).—In

an apparatus suitable for gaseous reactions which are strongly exothermic or need close regulation of the temperature, the catalyst is in elongated annular form and is contained in large-bore tubes surrounded by a bath of liquid. The axial spaces in the catalyst masses are occupied by twin concentric tubes through which at least a part of the incoming gases passes to and fro. The liquid may be metallic or not, and may boil at, or slightly above or below, the required reaction temperature; or it may be non-boiling, in which case it should be mechanically circulated. B. M. VENABLES.

Gaseous exothermal catalyses. UNION CHIM. BELGE, SOC. ANON. (B.P. 329,551, 24.6.29. Belg., 13.11.28). —A portion only of the incoming gases is subjected to heat exchange with the outgoing gases, but the whole of the former is passed through the catalyst and through an annular space between the pressure-resisting wall and the heat-exchanging zone, the adjustable valve for dividing the gas coming after that pass. A heater is provided in the central catalysing zone, but is normally used only for starting. B. M. VENABLES.

Devices for carrying out exothermic catalytic reactions. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE, ASSEES. Of SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE & PROD. CHIM.) (B.P. 307,840, 13.3.29. Morocco, 14.3.28).—When the catalyst has to be cooled by the incoming gases (or other fluid) passing through an annular space round the catalyst chamber, that space is filled with metal gauze or other discontinuous material which improves heat transmission by forming eddies, steadies the flow, and prevents warping due to expansion and contraction.

B. M. VENABLES.

Apparatus for carrying out reactions continuously in the liquid phase under increased pressure. Imperial Chem. Industries, Ltd. (B.P. 329,260, 28.10.29. U.S., 29.10.28).-The apparatus comprises two concentric cylindrical chambers, the outer one being pressure-resisting and the inner having separate communication with external apparatus at the bottom, but opening at the top into the outer one, i.e., there is a vapour space common to them both. Part of the annular space is filled with Raschig rings (or similar device) and an inlet pipe is provided reaching to a distributor plate on top of the filling; pipes are connected to the bottom of each chamber for inlet or outlet according to circumstances, and at the top connexions are made for a pressure gauge and for drawing off permanent gases. A device to indicate when the central stand-pipe or chamber is full of B. M. VENABLES. liquid may be added.

Water-cooling towers. L. G. MOUCHEL & PARTNERS, LTD., and M. E. GERARD (B.P. 329,715, 26.2.29).—A cooling tower of hyperboloidal form is formed of straight timbers set at an angle (not perpendicular) to the ground with their lower ends on a circle and their intersections bolted together. B. M. VENABLES.

Drying apparatus. W. M. STRONG, ASST. to GEN. DRYING ENG. CORP. (U.S.P. 1,749,451, 4.3.30. Appl., 17.4.28).—The material, such as lucerne, is carried on a conveyor, which is enclosed above and below and is subjected to upward flows of hot air from a furnace and fan at the charging end. A proportion of the gases

when hottest pass through the material when wettest, but another regulable portion passes on through a door and percolates through the partly dried material at a lower speed, the length of conveyor forming the secondary zone being the longer. The moist gases from the primary zone are exhausted to a chimney, but the secondary gases are returned to the feed end and mixed with fresh gases from the heater. B. M. VENABLES.

Vacuum drying apparatus. A. E. JONSSON (B.P. 306,037 and 306,088, 11.2.29. Swed., [A] 14.2.28, [B] 15.2.28).—(A) A rotating or oscillating cylinder contains a number of hollow shelves arranged the full length of the cylinder and grouped in parallel on both sides of a diametral space, which affords communication to manholes for charging and discharging. The material being dried is intended to slide rather than tumble from shelf to shelf. The shelves, also the shell, if desired, are heated by a fluid in the spaces or jackets. (B) An apparatus similar in principle to that described in (A) is intended for non-powdered goods which are supported clear of the heat-radiating shelves by non-conducting ribs.

B. M. VENABLES.

Straining device for vacuum-drying apparatus. A. E. JONSSON (U.S.P. 1,748,955, 4.3.30. Appl., 21.3.28. Swed., 9.4.27).—A strainer for protecting the outlet pipe of a vacuum dryer is described. It comprises a cylindrical slotted surface, the slots being kept open by a comb that can be moved to and fro. B. M. VENABLES.

Centrifugal dryers. H. C. BEHR (U.S.P. 1,749,369 and 1,749,370, 4.3.30. Appl., [A] 3.8.28, [B] 23.8.28).-The cycle of operations of these centrifuges is controlled by a number of valves operated in sequence by a camshaft. A quantity of the pulp is drawn into a metering chamber by a vacuum and then propelled into the slowly rotating basket by air pressure. After centrifuging, a wash may be applied, and then, in (A), the basket, being cylindrical, is stopped and the material blown inwards off the screening surface by a pressure fluid, whence it drops out through bottom-discharge ports. In (B) the basket is the space between two cones of different angle, the upper one carrying the screening surface and the lower one the scrapers. It runs at the same speed as the upper one when centrifuging, but at a lower speed when discharging the collected cake, the change being effected by a hydraulic gear under control of the cam-shaft. B. M. VENABLES.

Absorption method and apparatus. F. G. WHEELER, Assr. to BLEACH PROCESS CO. (U.S.P. 1,747,687, 18.2.30. Appl., 5.12.25).—The apparatus may be constructed in stoneware and is suitable, *e.g.*, for absorbing chlorine in milk of lime. The liquid flows downward from the narrow end of an expanding pipe with a velocity insufficient to form a jet; the expanding portion of the pipe is pierced by downwardly inclined holes through which the gas is admitted from a surrounding T-piece. The liquid tends to break away from the wall of the pipe at the perforations, thus producing eddy currents which carry bubbles of gas into the body of liquid. B. M. VENABLES.

Absorber. J. A. CAMBPELL (U.S.P. 1,748,409, 25.2.30. Appl., 17.12.27).—In a bubble tower having decks composed of twin perforated plates, the space between becoming filled by a mass of froth which breaks

down above the upper plate of a pair, provision is made for running the liquid down from the top of an upper plate to the lower plate of the deck below through an annular space formed by a double shell. To avoid intermingling of the streams from alternate plates the flow is only permitted down one side alternately and the space is divided into semi-annuli by means of vertical ribs. B. M. VENABLES.

Bubble tower. W. E. TETER (U.S.P. 1,748,855, 25.2.30. Appl., 18.2.28).—The upflow pipes are of inverted **J**-shape, and on top of these is a perforated plate from the upper surface of which the downflow is taken, preferably by a single pipe which branches lower down. The cross-section of the tower may increase in the upper part. B. M. VENABLES.

Digestors. SILLER & RODENKIRCHEN GES.M.B.H., and W. SILLER (B.P. 329,600, 25.9.29).—The digestor, suitable for the extraction of glucose from wood by means of acid, rotates on a horizontal axis, the heating fluid to a jacket and the charge to the digestor being admitted through one trunnion and exhausted through the other. The inlet pipe for material is taken to the bottom and the outlet comes from the top of the internal space, both being curved in such a way that the rotation tends to keep the openings clear. (Cf. B.P. 325,760; B., 1930, 399.) B. M. VENABLES.

Disintegrator. W. M. DUNCAN (U.S.P. 1,745,478, 4.2.30. Appl., 1.7.27).—A rotor drives two sets of balls or rolls which make contact with the casing, each element being in a separate pocket on the rotor. The disintegration is effected by a combination of three actions, viz., rolling between the rolls and casing, attrition between the rolls and driving pockets, and impact; provided the centrifugal force is not too great, the rolls will make contact with alternate faces of the driving pockets, according to whether they are rising or falling.

B. M. VENABLES.

Apparatus for breaking and handling solid materials. ASH CO. (LONDON), LTD. (B.P. 312,057, 26.4.29. U.S., 19.5.28).—Under a boiler or similar furnace are a number of alined hoppers for discharge of ash, a transferable crushing unit is placed under each opening in turn, and the crushed product drops into a sluice, whence it is carried by water to an additional crusher which renders it fine enough for the pulp to be disposed of by pumps. B. M. VENABLES.

Crusher. W. I. BOWDEN, ASST. to AMER. ENG. Co. (U.S.P. 1,748,828, 25.2.30. Appl., 20.11.26).—An intermediate bearing for a long crusher roll suitable for boiler ash-pockets is described. B. M. VENABLES.

Crusher. R. BERNHARD, Assr. to TRAYLOR ENG. & MANUF. Co. (U.S.P. 1,748,102, 25.2.30. Appl., 24.5.29). —To prevent damage by uncrushable material, the upper end of the shaft of a gyratory crusher is yieldingly supported by a lever and spring. B. M. VENABLES.

Crusher. J. E. KENNEDY (U.S.P. 1,748,484, 25.2.30. Appl., 21.5.28).—The drive is transmitted to the shaft of a gyratory crusher by means of an electromagnetic friction clutch, to prevent damage by uncrushable pieces, to aid restarting, and to permit the use of a synchronous motor. B. M. VENABLES. Reinforced crusher plate. C. E. HARRISON, Assr. to AMER. ENG. Co. (U.S.P. 1,748,879, 25.2.30. Appl., 22.7.25).—A crusher plate or jaw for severe service, such as in the ash-pockets of pulverised-fuel furnaces, is formed with steel or wrought-iron rods embedded in the cast metal and is deeply ribbed on the back.

B. M. VENABLES. **Pulveriser.** G. D. HARRINGTON (U.S.P. 1,748,050, 18.2.30. Appl., 17.11.26).—The material is fed on to a horizontally rotating disc having upstanding radial beaters which fling the material against a toothed or corrugated stationary ring. Air enters with the feed, picks up fine material, and enters a fan above through a central port. Various adjustments are provided for the air current to determine the size of the delivered material. B. M. VENABLES.

Pulverising machine. E. H. ELZEMEYER, Assr. to AMER. PULVERIZER CO. (U.S.P. 1,748,214, 25.2.30. Appl., 9.10.25).—In a disintegrator having hammers in the form of loose rolling rings, the edges of the rings are made wavy (when viewed edgewise, but not when viewed axially) to prevent packing of the material upon the grids or other stationary surfaces. B. M. VENABLES.

Pulverising mills. INTERNAT. COMBUSTION, LTD., and F. H. ROSENCRANTS (B.P. 329,061, 1.3.29).—In an air-borne pulverising system, hot air is supplied by a heater of the type in which additional air is mixed with the products of combustion, the hot air passes through an explosion relief chamber which is constructed to serve also as a dust settler, and is drawn into the main grinding circuit by the action of the circulating fan, excess air being drawn from an earlier point of the same circuit through a dust separator by an additional fan. To prevent deposition of dew in the dust separator, hot air may be admitted thereto directly from the explosion chamber by means of a regulable by-pass.

B. M. VENABLES.

Pulverising mill. F. J. BULLOCK, ASST. to PAPEC MACHINE CO. (U.S.P. 1,748,046, 18.2.30. Appl., 5.7.28). —In a disintegrator the casing is supported in such a way that it can oscillate slightly in the plane of rotation of the beaters, and it is pulled back against the rotation by a spring; when grinding takes place the drag of the material will move the casing and extend the spring, this motion being utilised to vary the opening from a stationary feed hopper, so that the power required for grinding is held at a steady value. B. M. VENABLES.

Pulverisation of materials. M. W. ARROWOOD (U.S.P. 1,747,922, 18.2.30. Appl., 25.5.28).—A ball-, pebble-, or rod-mill is provided with a drum of grid-like structure and is surrounded by a fixed casing; interrupted helical vanes are attached to the outside of the drum and push material to one end of the casing where it is picked up by scoops and returned to the drum. The fine material is carried away in a current of air, which is blown in at one end of the casing and exhausted at the other, and travels in a spiral direction round the drum. The pulveriser is particularly suitable for coal.

B. M. VENABLES.

Comminution of material. R. C. NEWHOUSE, Assr. to Allis-Chalmers Manuf. Co. (U.S.P. 1,748,920, 25.2.30. Appl., 23.2.28).—In order to obtain a product containing a high proportion of very fine material from a tube-mill or similar grinder without correspondingly reducing the capacity of the air separators, the latter receives material from the last but one compartment of the mill and delivers its undersize to the last compartment for final grinding without subsequent air separation. B. M. VENABLES.

Grinding machine. H. C. SCHNEIDER, ASST. to FAIR-BANKS, MORSE & Co. (U.S.P. 1,745,646, 4.2.30. 18.11.26). —In a machine of the rotating hammer or knife type, the hammers, spacers, and inner races of the bearings are threaded on a shaft which is in the form of a bolt and the whole is clamped together by tightening up nuts on the end of the shaft. B. M. VENABLES.

Grinding bodies for tube- or drum-mills. Hell-PEBS, LTD., and R. McDOUGALL (B.P. 329,131, 24.4.29). —The bodies comprise square helices formed from square or rectangular metallic wire or rod.

B. M. VENABLES. Apparatus for testing materials [for hardness]. J. GOGAN (U.S.P. 1,746,891, 11.2.30. Appl., 28.2.27).— A machine suitable for rapidly effecting, without special preparation, a ball test on every piece of material manufactured, causes the test ball to advance under sufficient pressure to penetrate the skin and then from this point as zero to advance a predetermined distance, *e.g.*, 0.01 in., the extra pressure being the criterion of the hardness and the dent too small to spoil the piece for use. In practice a few pieces are checked by known refined methods and are then used as standards in the machine, the dial being set to zero for the maximum load on the standards, and the production pieces being passed if the reading on the dial does not go beyond prescribed limits.

B. M. VENABLES.

Means for measuring the hardness and thickness of [non-rigid] objects. H. A. DAYNES, E. B. JOHNSON, and RES. ASSOC. OF BRIT. RUBBER MANUFRS. (B.P. 329,629, 18.2.29).—The apparatus comprises a broad foot within the area of which is a loaded plunger which indents the test-piece and of which the advance relative to the foot is measured while under a definite load. Both the foot and the plunger rest on the testpiece, or, when taking the zero reading, on a rigid surface such as a sheet of glass. For measuring the hardness of a curved object such as a rubber-covered roller a pair of cylindrical rods suitably held in grooves take the place of the foot and the zero must be obtained on a rigid body of equal radius. For measuring the average thickness of a sheet the foot alone rests on the test-piece and the plunger on the rigid support. B. M. VENABLES.

Imparting compactness and coherence to a granular mass contained in a mould, and building block made therewith. R. LEJMARK (B.P. 329,063, 4.3.29).—The mould containing the mass (e.g., concrete) is subjected to jarring by allowing it to fall upon a heavy bed-plate and is then raised against a ram which effects smoothing of the upper portion. A machine is described which effects both motions by the same foot-operated mechanism. B. M. VENABLES.

Manufacture of materials in thin sheet form. W. H. PEASE (B.P. 329,691, 25.1.29).—A fluid material is supplied from a hopper through a slot, with micrometer adjustment, to a drum, and is spread thereon by adjustable knives. The temperature, humidity, etc. of the material, drum, and surrounding atmosphere (contained in an enclosure) are controlled so that the material leaves the drum in a solid or semi-solid state. An insoluble material may be interposed between the solution and the drum, thus forming a two-ply sheet. B. M. VENABLES.

Mixing machine. G. J. HICKS (U.S.P. 1,745,993, 4.2.30. Appl., 17.10.28).—A mixing machine suitable for confectioners' use comprises a trough-shaped container, one end of which is entirely removed for discharging purposes; the beaters are therefor journalled only at the other end. The solid material is fed from a hopper above by means of a screw-feeder and the liquid may be injected by compressed air either at the end of the feeder or upwards through the bottom of the trough. The temperature may be regulated by coils on the lid of the trough. B. M. VENABLES.

Automatic machines for mixing different products in specified proportions. Soc. des Sucreries TERNYNCK (B.P. 316,862, 22.4.29. Fr., 3.8.28).—Tipping dishes are provided for each material which are supported on scale beams in such a way that they are locked upright for filling, but when the counterpoise is balanced they overturn through 90° and discharge, which act transfers the centre of gravity to the other side of the pivot and causes the pan to turn back for filling again. The actual filing of the dishes is effected by any known device that can be instantly stopped, and the dish for the primary material governs the operation of the others in such a way that the latter can only return to the filling position when the primary is emptying; interlocks are provided to stop the whole mechanism in the event of deficiency of any material or if the receiving container becomes too full. B. M. VENABLES.

Separator. C. G. HAWLEY, Assr. to CENTRIFIX CORP. (U.S.P. 1,746,253, 11.2.30. Appl., 29.6.26).—An entrained substance (fluid or solid) is separated from a carrier fluid (gas, vapour, or liquid) by projecting the mixture downwards, deflecting it laterally then downwards, and allowing the cleaned carrier fluid to turn inwardly and upwardly and to pass out through the original stream where it is flowing laterally. The apparatus comprises a collecting hopper, above which are arranged deflectors to effect flow as described above, the outlet being situated annularly round the inlet.

B. M. VENABLES.

Separating apparatus and method. W. T. BIRD-SALL (U.S.P. 1,747,155, 18.2.30. Appl., 15.10.25. Renewed 25.6.29).—A separator for fluids, or fluids and solids, of different density or viscosity is based on the fact that the film on a rotating shaft has laminar motion with respect to the body of fluid which may or may not have eddying motion. The apparatus is intended to drag a film of a single constituent into another compartment; centrifugal force of the order of 200,000 times gravity may be developed tending to urge foreign matter out of the laminar film. Several forms of apparatus are described : one is like a plain journal bearing with one arc of bearing surface much longer than the opposite one; others have screw-threaded casings surrounding a plain or screwed shaft. B. M. VENABLES. Separation of intermixed divided materials. R. PEALE and R. PEALE, JUN. (B.P. 328,663, 31.12.28. U.S., 18.12.28).—An apparatus for the dry separation of particles which differ greatly in size but not much in sp. gr. comprises a shaking table which has a bed of varying porosity to upward air currents which can be controlled in zones. After stratification the products are delivered by inclined guides. B. M. VENABLES.

[Wet] separators [for ores etc.]. J. and J. O. SPROUL (B.P. 329,030, 15.2.29).—A separator of the type in which a conveyor carries the heavier constituents against a stream or streams of water has the conveyor composed of a number of shallow buckets or skips of which the bottom from the leading end is gradually inclined and at the back rises in a steep curve over the edge of the succeeding bucket. B. M. VENABLES.

Separation of minerals [containing flat and granular particles]. J. BLAND (U.S.P. 1,749,371, 4.3.30. Appl., 20.9.26).—Mixed minerals, such as mica and felspar, are dropped in an annular stream within the inner edges of a number of superposed, spaced, truncated, conical baffles, against an upward current of air which is insufficient not only to affect the downward flow of the granular particles, but also to lift the flakey particles of relatively large surface; it does, however, cause the latter to eddy and escape from the air current outwardly between the baffles. To obtain clean products of both constituents a number of units may be employed in series. B. M. VENABLES.

Separation apparatus. B. A. MILLER (B.P. 328,940, 7.1.29).—A pulp flowing down an inclined V-shaped sluice is given a side-to-side swirling motion by curved baffles attached to either side of the sluice alternately, thus causing the heavier concentrate to collect in the bottom of the sluice, whence it is removed through outlets controlled by internal lips. B. M. VENABLES.

Centrifugal separator. H. O. LINDGREN, Assr. to DE LAVAL SEPARATOR CO. (U.S.P. 1,749,291, 4.3.30. Appl., 24.8.28. Swed., 26.10.27).—The separator is suitable for the continuous separation of materials such as wool scourings, both dirt and fats being separated from the water. The bowl is slightly conical, and has an outlet for dirt (and water) leading from the largest circumference to a point nearer the axis, through which the water etc. passes before being finally discharged. The outlet for middle fluid (water) leads from the smallest circumference above the top separating disc to outlet jets at an intermediate radius. The lightest liquid leaves the spaces between the discs at their inner edges and flows upwards to an inner neck.

B. M. VENABLES.

Apparatus for separating solids from liquids. A. L. ARMENTROUT (U.S.P. 1,749,057, 4.3.30. Appl., 11.11.26).—The solid is settled centrifugally in a truncated conical rotor (or by gravity in a tank with sloping bottom), a carrier liquid of intermediate sp. gr. being added at the smaller circumference in such a manner that it will not emulsify with the lighter liquid. Separate outlets are provided for carrier liquid with dirt, excess carrier liquid, and lighter liquid, and the supply of carrier liquid is maintained by a pump rotating with the centrifuge. B. M. VENABLES.

Apparatus for the clarification of liquids and separation of finely-powdered solid substances [byflotation]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,013, 7.2.29).—The suspended particles are removed in a foam produced by the downward impingement of a jet of the cloudy liquid upon the surface of a column of the clarified liquid. A column of foam several metres high may be produced; it moves countercurrent to the jet, but is otherwise unobstructed. A wetting agent may be added previously, and the process may be applied to the separation of different solids; *e.g.*, in a gas-purification process a mixture of regenerated ferric hydroxide and sulphur may be treated so that the former will sink and the latter float. B. M. VENABLES.

Filter. J. E. OTIS, JUN., ASSR. to ALEMITE CORP. (U.S.P. 1,746,274, 11.2.30. Appl., 30.11.25).—A strainer suitable for connecting to the outlet of a tank is described. When the former is removed for cleaning, the supply of liquid is automatically cut off. B. M. VENABLES.

Filter leaf. E. J. SWEETLAND and J. V. ZENTHOEFFER, Assrs. to OLIVER UNITED FILTERS, INC. (U.S.P. 1,746,409, 11.2.30. Appl., 6.12.26).—Sectors for rotating-leaf filters, especially suitable for use with metallic cloth backed by heavier wire gauze, are constructed of cast metal (e.g., aluminium) with a cored central outlet, suitable for bolting to the rotating hub, with ribs in the drainage space and with twin flanges round the radial and circumferential edges, all cast in one piece. The flanges are drilled for securing the filter medium by rivets or other means, and the radial flanges of adjacent segments abut together and embrace spokes attached to the hub; the ends of the spokes are provided with nuts and bridge washers to clamp the sectors.

B. M. VENABLES.

Hydraulic filter press. W. H. MATHERS, ASST. to R. DUDGEON, INC. (U.S.P. 1,749,080, 4.3.30. Appl., 25.3.27).—The press is tightened by a hydraulic ram and the preliminary closing is by a rapid-acting screw.

B. M. VENABLES.

Apparatus for producing [edge-]filter members. W. L. KRAEMER and A. C. PURDY, ASSTS. to METAL EDGE FILTER CORP. (U.S.P. 1,745,426, 4.2.30. Appl., 10.6.27).—An apparatus is described suitable for the rapid manufacture of washers with slight projections on the surface, for use as filter packs from a long metal strip. The steps comprise cleaning, drying, applying printers' ink or other tenuous film to the unraised portions of the strip, plating the bare parts and thus producing the projections, cleaning off the ink, and punching out the washers. B. M. VENABLES.

Dialysing filter-presses. FILTRES PHILIPPE, and E. HEIBIG (B.P. 329,235, 14.8.29).—The construction is similar to that of an ordinary filter-press with additional frames substituted for the usual plates, and with the ports arranged to give series flow of two fluids, each one through every alternate frame, the general flow being countercurrent on opposite sides of dialysing diaphragms in the place of the usual filter cloths. Lattice work may be inserted in the frames to cause the liquid to travel in a longer course within the frames. B. M. VENABLES. Purification [of vapours in distillation] apparatus. V. SEEHOLZER (Swiss P. 124,999, 16.3.37).—At least one removable horizontal plate is arranged in the path of the vapours through the condenser to serve as a condenser for the less volatile portion of the vapours. A. R. POWELL.

Fractionating system. Method of fractionation. M. B. COOKE, Assr. to ATLANTIC REFINING CO. (U.S.P. 1,748,411 and 1,748,508, 25.2.30. Appl., [A] 24.7.25, [B] 21.3.29. Renewed [A] 20.2.29).—(A, B) In a fractionating column the decks are provided with bubbling caps, and with downflow pipes at alternate ends of a diameter, the downflowing liquid being kept away from the rising vapour. Above the caps is a mass of inert ceramic or other filling which catches any entrained liquid, the dried vapour then passing through a space for vapour only before reaching the next bubbler. The filling is supported on a perforated platform, which may be formed from the tops of the bubble caps themselves, suitably shaped and registering. B. M. VENABLES.

Method of fractionation. Fractionating column. R. B. CHILLAS, JUN., and A. G. PETERKIN, JUN., ASSTS. to ATLANTIC REFINING CO. (U.S.P. 1,748,595 and 1,748,704, 25.2.30. Appl., 24.11.25).—In the spaces between the decks of a bubbling column are placed moisture eliminators, permitting increased output owing to the fact that entrainment produced in the bubblers may be ignored and the velocity of the vapours raised to 2—6 ft./sec. The eliminators must not occupy much of the crosssection of the tower, and they return the liquid to the tray below in which the spray was produced ; they may consist of parallel V-shaped sheets. B. M. VENABLES.

Dephlegmator or fractionating column. L. C. HUFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,741,519, 31.12.29. Appl., 10.12.26).—The decks of a fractionating column are provided with the usual downflow pipes maintaining a layer of liquid; the upflows are in the form of ejectors, the rush of vapour through which entrains liquid, thereby lifting it in the form of a spray or film and returning it to the same tray.

B. M. VENABLES.

Fractional condensation of composite vapours. A. LOSEY, P. GOTTLIEB, and L. L. HAUPT (B.P. 329,418, 4.3.29. U.S., 31.1.29).—Elaborate systems involving expansion engines and the use of several different pressures are described. B. M. VENABLES.

Congealing of liquid or semi-liquid substances. C. W. VOGT (B.P. 329,129, 23.4.29).—The liquid is sprayed on to a refrigerated surface and continuously removed therefrom. The atmosphere is also chilled by a refrigerating surface, not in contact with the spray, to such an extent that partial congelation takes place before the spray reaches the first-mentioned surface. Materials such as ice-cream are preferably pasteurised and homogenised before spraying, and the mist produced should be so fine that the frozen product is aerated by the atmosphere. B. M. VENABLES.

Electric hygrometer. W. LUBACH (U.S.P. 1,749,826, 11.3.30. Appl., 7.6.27. Ger., 8.6.26).—A pair of closelyspaced, convoluted conductors, preferably of nichrome, inserted in an electrical circuit including a source of constant potential and an indicating instrument, e.g., a galvanometer or cathode-ray oscillograph, are bridged by non-conducting material impervious to moisture, but upon which moisture can condense, e.g., glass-wool. A current will then flow between the conductors when moisture is deposited on the material, the magnitude of the current being a measurement of the humidity of the atmosphere surrounding the conductors.

# J. S. G. THOMAS.

**Refractometers for liquids.** C. ZEISS (B.P. 314,994, 1.7.29. Ger., 6.7.28).—A refractometer is provided with a knurled ring operating a deflection device by which the boundary line between the light and dark part of the field may be adjusted to a mark; when this is effected the scale showing refractive index (or concentration in the case of sugar solutions) appears superposed on a number of curved isothermal lines, so that the result can be read at once according to the known temperature. B. M. VENABLES.

Instrument for measuring fluids for turbidity, colour, and other characteristics of fluids. W. G. EXTON (B.P. 328,556, 24.1.29).-A cylindrical casing has at one end a source of light and at the others an eye-piece; intermediately are transverse plates with central apertures carrying, in order, a target, a test-tube containing the sample of liquid, a lens, a calibrated wedge of which alternative types are provided either absorbing or dispersing the light till the target disappears, and a set of colour screens. To prevent the final observation being made in darkness an annular space 18 left round all the plates, and the casing is made reflecting so that diffused light always reaches the eyepiece. Alternatively, the sample itself may be contained in a wedge-shaped container and adjusted till it extinguishes the target. When desired, a comparison field may be simultaneously viewed by means of a B. M. VENABLES. Fresnel rhomboid prism.

Air filtering, heating, humidifying, and like apparatus. T. ANDREW (B.P. 328,948, 6.2.29).— The air is passed from the inside outwards through a number of tubular fabric filters, which are supported in a horizontal position by means of spring tension applied to the closed end. The humidifying etc. may be done in a subsequent chamber. B. M. VENABLES.

Air filter. H. E. BIRKHOLZ, ASST. to AMER. AIR FILTER Co., INC. (U.S.P. 1,747,694, 18.2.30. Appl., 30.3.25).—The apparatus consists of a slowly rotating, drum-shaped screen formed from a spongy metallic mass through which the air passes inwards and leaves by one end which is open. The screen dips into a bath of oil, clean oil being supplied to the lower part of the interior by sprays so arranged that the screen is washed by a flow of oil in a direction opposite to that of the air. B. M. VENABLES.

Air filters. C. G. VOKES (B.P. 329,099, 28.3.29).— The filter medium is composed of several layers of wire gauze of the same or different mesh, with or without felt in between; the laminated sheet thus produced is pleated without substantial separation of the layers.

B. M. VENABLES. Air filter. W. M. REED and C. P. HEGAN (U.S.P. 1,746,283, 11.2.30. Appl., 3.3.26).—A method of fastening filter cells to supporting frames by means of latches is described. B. M. VENABLES.

Apparatus for extracting smoke and other solid particles from air. T. D. AUSTIN and C. DEWHURST (B.P. 329,501, 14.5.29).—The apparatus is of the dry centrifugal type, separate fans being used to draw in the air and to centrifuge it.

B. M. VENABLES. Air separator. T. J. STURTEVANT, ASST. to STURTE-VANT MILL Co. (U.S.P. 1,746,686, 11.2.30. Appl., 23.6.28).—The separation is effected in two upward, outward (through the blades of a fan), downward, inward circulations one above the other. The material is fed from a hopper to a distributing plate situated in the upward current of the upper circulation, and the oversize which is not carried up falls on to a similar distributor in the lower circuit, three products being thus obtained. B. M. VENABLES.

Gas and air cleaner. A. F. JENKINS (U.S.P. 1,746,774, 11.2.30. Appl., 10.9.27).—The gas etc. is admitted downwards through an axial tube nearly to the bottom of a cylindrical container, and on its return flow upward passes through a number of filter elements which may be composed of (lower) convex plates having small perforations surmounted by concave plates with larger perforations. Application to air for paint-spray apparatus is indicated. B. M. VENABLES.

Treating [cleaning and cooling] gases. A. G. McKee (U.S.P. 1,748,723, 25.2.30. Appl., 1.7.25).-A tower is provided with a number of separately driven, superposed, spraying devices each of which can be stopped and repaired without affecting the operation of the rest of the tower, access being afforded by workpassages. The earlier stages are supplied with hot water, which removes the solid matter, and is then passed to a settler where the bulk of the mud settles out. The water is returned to the same stages of the tower, though still containing sufficient solids to be a legal nuisance if it were discarded, but yet quite clean enough to use again; the elevated temperature promotes both the entrainment of the dust and the settlement in the thickener. All losses of water from this circuit are made up from the later cooling circuit in the upper stages, in which another supply of water is circulated through the sprays and a cooler, losses being made up with fresh water, the consumption of which is much reduced when compared with that from methods previously used.

B. M. VENABLES.

Apparatus for removing oil from gases. FREINS JOURDAIN MONNERET SOC. ANON. (B.P. 306,899, 18.2.29. Fr., 27.2.28).—The gases are passed in a zigzag path through a chamber composed of heat-radiating (external) and baffling (internal) sections; other baffles are also formed round the central pipe admitting the gas. B. M. VENABLES.

[Spectroscopes for] analysing gases or vapours. V. HENRI (B.P. 329,111, 9.4.29).—Two forms of spectroscope, suitable for the quantitative analysis of gases by ultra-violet light, are described. In one form the light rays from an electric lamp fitted with a quartz bulb are collected by a lens, passed in turn through a chamber for the gas, formed out of the tube of the

instrument and a pair of non-absorptive windows, then through a slit diaphragm, two prisms each of which can be rotated, to a fluorescent screen or photographic plate the angle of which can be varied and for the former a magnifying eyepiece may be provided. The other form makes use of a concave diffraction grating instead of the prisms. The determination of the concentration may be effected either by actual measurement of the energy absorbed by the bands, *e.g.*, by thermocouple, or, usually more conveniently, by comparison with a standardised gas. A convenient method of adjustment is to alter the pressure of the sample.

B. M. VENABLES. Fluid composition for treating brake and clutch linings. LIQUID VENEER CORP., Assees. of E. F. GINGRAS (B.P. 307,342, 14.2.29. U.S., 5.3.28).—Worn parts of a lining are built up by a suspension of particles of friction material in a vehicle comprising a gum in a suitable solvent. B. M. VENABLES.

**Production of high vacua.** KODAK, LTD., Assees. of K. C. D. HICKMAN (B.P. 329,918, 22.8.29. U.S., 19.2.29).—A vacuum pump of the vapour-jet type is described suitable for use with an organic liquid having a vapour pressure at 0°, which is less than the pressures used in high-vacuum work, and which is not decomposed by prolonged boiling, *e.g.*, *n*-dibutyl phthalate or other aromatic ester. The advantage over mercury is that the condensing vapour wets the surface of the apparatus, does not form globules, and uncondensed vapour is less likely to diffuse into the vessel being evacuated.

B. M. VENABLES.

[Pyrometer] device for measuring the temperature of hot fluids. R. HASE (U.S.P. 1,761,392, 3.6.30. Appl., 15.12.24. Ger., 28.1.24).—See B.P. 231,398; B., 1925, 482.

Liquid-circulating devices for absorption refrigerating apparatus. Refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRI-GERATING SYSTEM AKTIEB. (B.P. 309,545 and 312,359, [A] 5.4.29, [B] 16.5.29. Ger., [A] 12.4.28, [B] 25.5.28).

Apparatus for washing minerals by means of liquid streams. A. FRANCE (U.S.P. 1,759,940, 27.5.30. Appl., 30.12.26. Belg., 30.12.25).—See B.P. 285,987; B., 1928, 320.

[Dual] filter. J. ZWICKY (U.S.P. 1,759,927, 27.5.30. Appl., 26.9.25).—See B.P. 258,905 ; B., 1927, 176.

Separation of liquids of different specific gravities. W. A. WHITE, ASST. to WHITE OIL SEPARATORS, LTD. (U.S.P. 1,761,505, 3.6.30. Appl., 27.1.28. U.K., 7.6.27).—See B.P. 285,707; B., 1928, 507.

Method for atomising and drying liquids. A. NYROP (U.S.P. 1,762,026, 3.6.30. Appl., 3.8.26. Denm., 10.5.26).—See B.P. 275,404; B., 1927, 801.

Refractometer for liquids. F. PFEIFFER, Assr. to C. ZEISS (U.S.P. 1,760,209, 27.5.30. Appl., 29.6.29. Ger., 6.7.28).—See B.P. 314,994 ; preceding.

• Re-gasification of liquefied gases. C. W. P. HEY-LANDT (U.S.P. 1,747,366, 18.2.30. Appl., 16.2.28. Ger., 29.3.27).—See B.P. 287,909; B., 1929, 501. [Device for] controlling coal-dust furnaces [of locomotives etc.]. R. ROOSEN (B.P. 317,372, 7.8.29. Ger., 14.8.28).

Suspended roofs for furnaces and the like. A. REPPMAN (B.P. 330,326, 18.3.29).

Furnace walls. H. W. SPENCER, and LIPTAK FIRE-BRICK ARCH Co. (B.P. 330,351, 16.4.29).

Apparatus [valve devices] for aerating liquids under pressure. E. Ryles and J. Sopwith (B.P. 328,924, 5.2.29).

Carbonic anhydride  $(CO_2)$  [refrigerating] compressors. W. F. JENNINGS (B.P. 330,007, 5.3.29).

Fine powders (B.P. 327,979).—See II. Heatinsulating material (G.P. 461,889).—See IX. Heating of liquids (G.P. 461,567). Deoxidising system (U.S.P. 1,745,875).—See XI. Rubber-coated articles (B.P. 328,686). Porous body (B.P. 306,129). Brake liners (B.P. 329,021).—See XIV. Pasteuriser for liquids (B.P. 329,733).—See XVIII.

# II.—FUEL; GAS; TAR; MINERAL OILS.

Coal properties and its applications. T. KIDO-KORO and G. SHIRANE (J. Fuel Soc. Japan, 1930, 9, 43-49).—A method for the identification of the geological rank of coals, lignites, etc. by their treatment with oxidising agents followed by precipitation of the humic acids with caustic soda is described. Experiments on the oxidation and spontaneous combustion of coals, with special reference to their rank, as determined above, are also discussed. C. B. MARSON.

"Ball coals" (Mugelkohlen). S. von FINALY (Brennstoff-Chem., 1930, 11, 188-189) .- " Mugelkohlen" are round or elliptical masses of coal of varying size up to 40 cm. in diam., found in the coal seams in the neighbourhood of Pécs, Hungary. They are readily separated from the surrounding coal, and frequently, but not always, possess a more or less complete outer shell, 0.5-1.0 cm. thick. Slight but definite differences exist between the compositions (as shown by the ultimate analyses based on the ash-free, dry basis) of the "ball coal," the surrounding shell, and the coal-seam itself. In the two samples investigated, however, the ash of the " ball coal" (about 4%) was much less than that of the surrounding seam (about 19%). These coal masses have most probably been formed by the "rolling together" of particles of the original peaty material by the action of wind and water (cf. Gothan, Sitzungsber. Königl. Preuss. Akad. Wiss., 1910, 1, 136). A. B. MANNING.

Peat. III. Dry substance of peat. G. STADNIKOV (Kolloidchem. Beih., 1930, 31, 59—88; cf. B., 1930, 495).—Determinations of the dry residue of peat by drying in air at 105° give erroneous results, since simultaneous oxidation occurs and it is not possible to determine the end of the drying process. The weight is altered also by the decomposition of organic constituents at this temperature. The residue may be determined by heating at 80° in a stream of nitrogen, but a vacuum desiccator provided with electric heating is recommended. The weight of dry residue obtained from peat varies much with its nature, but lies between 9 and 15%. The ash content is obtained by ignition, and is found to

increase with the depth of layer from which the peat is obtained. Both the ash content and its composition vary greatly with the kind of peat; analysis have been made, and the principal constitutents found were calcium, iron, aluminium, potassium, silica, sulphate, and phosphate. The composition of the ash is closely connected with the suitability of the peat as a combustible, especially in determining the porosity of the burning material, and it is said that the iron content catalyses the oxidation of the organic matter. The elementary composition of the organic constituents does not vary appreciably with the depth of layer from which the peat is taken. The nitrogen content of peat is considerably higher than that of other fuels; it often reaches 2.5% and rarely falls below 1.5%. The products of distillation of peat at different temperatures have been investigated, and the yields of coke, tar, gas, water, ammonia, acetic acid, and methyl alcohol at 525°, 710°, and 850° are tabulated. E. S. HEDGES.

Further X-ray studies of carbonaceous and bituminous materials. C. MAHADEVAN (Indian J. Physics, 1930, 4, 457-466; cf. B., 1929, 965).-The X-ray diffraction patterns of fusain, graphitic anthracite, fibrous and non-fibrous peat, and elaterite have been examined. That of fusain shows a number of rings, much like durain (cf. loc. cit.), but with the interspaces fairly clear. There is also evidence of the fibre pattern in the diagram. Graphitic anthracite shows welldefined and fairly sharp halos for both the carbon and the ash, indicating that neither is in the colloidal state. In both fusain and anthracite the carbon seems to be in a free state. The prominent ash rings in both cases have been ascribed to silica and alumina. Fibrous peat gives a fibre pattern, whilst ordinary peat gives the diffuse and ill-defined diagram of a colloid. From the parallelism of the spacings in the peats and in the coals it is suggested that the difference between the two may be mainly one of structure rather than of composition. Elaterite gives a number of rings with spacings showing close identity with those of the higher members of the paraffin series, so it is concluded that elaterite is a mixture of the higher member of this series. J. W. SMITH.

Flash point of Polish coal dust. W. SWIENTO-SLAWSKI and M. GROCHOWSKI (Przemysl Chem., 1930, 14, 246—249).—The flash point of durain and vitrain dust lies between 330° and 360° for grains of mean diam. 1·5—2 mm., and between 190° and 220° for grains of less than 1 mm. diam. The flash point of fusain dust is 190—270°, both for grains of more and of less than 1 mm. diam. R. TRUSZKOWSKI.

Determination of soot. J. HIRSCH (Z. Hyg., 1929, 110, 399—412; Chem. Zentr., 1929, ii, 2800).—The soot is collected in a Dubsky combustion tube which is partly filled with alternate layers of copper oxide (as wire) and quartz splinters. Air is sucked through by an oil pump and measured. The soot is then heated in an oxygen stream and the carbon dioxide formed absorbed in 0.1N-barium hydroxide and determined by titration. The maximum error of the method is 4.85%. The small soot particles can be caught finally by means of a filter. L. S. THEOBALD. Humic acids. M. SAMEC [with B. PIRKMAIER] (Kolloid-Z., 1930, 51, 96—100).—The humic acid content of a number of coals has been determined by measurements of solubility in sodium hydroxide. Ammonium humate and hymatomelanate are retained by a collodion membrane and give a measurable osmotic pressure. Using different coals, the values 1445, 1235, and 1345 were obtained for the mol. wt. of ammonium humate and the values 855, 761, and 739 for ammonium hymatomelanate. The properties of these compounds are compared with those of ammonium lignate. E. S. HEDGES.

Softening of ash of solid fuel. H. A. J. PIETERS (Chem. Weekblad, 1930, 27, 331-334).—A modified apparatus for determining the softening point of ash is described, and a large number of results are recorded. Three classes are distinguished, with softening temperatures respectively below 1250°, and above 1250°, the third class showing softening but not melting above 1250°. The relationship between softening temperature and chemical composition is discussed.

S. I. LEVY.

History and development of submerged combustion. C. F. HAMMOND (J. Inst. Fuel, 1930, 3, 303—320).—The methods of submerged combustion are described and details are given of the Hammond system and its industrial applications, *e.g.*, evaporation of chemical solutions, heating of linseed oil, dyeing and washing processes, white-metal melting.

# H. E. BLAYDEN.

Effect of addition of dolomite on carbonisation and combustion of bituminous coal. (Sulphur distribution.) I. TRIFONOV and E. RASCHEVA-TRI-FONOVA (Brennstoff-Chem., 1930, 11, 185-188; cf. B., 1930, 592).-The addition of 10% of dolomite to coal has little effect on the yields of coke, tar, and gas obtained by carbonisation at 950°, and on the distribution of the sulphur between the coke and volatile products. The added dolomite, however, greatly increases the pro-portion of sulphur retained by the ash when the coke is burned, the effect indeed being greater than would be expected from the composition of the dolomite. This is attributed to the protective action of the calcium carbonate on the magnesium sulphate formed. In the combustion of coke produced at a normal rate of carbonisation, the addition of dolomite causes less sulphur to be retained in the ash than does that of calcium carbonate, whereas the reverse is true if a rapid rate of carbonisation is used, the proportion of sulphur retained depending on the relative rates of evolution of hydrogen sulphide and decomposition of the carbonates. Addition of dolomite produces a marked increase in the strength of the coke, an increase which, in general, cannot be brought about by the addition of an equivalent mixture of calcium and magnesium carbonates. By the direct combustion of a mixture of coal and dolomite almost the same proportion of the sulphur is retained in the ash as by carbonisation followed by combustion of the A. B. MANNING. coke.

Electric welding in carbonising plants and gasworks. W. KÜRSCHNER (Brennstoff-Chem., 1929, 10, 468-469).—Electric welding is recommended for the construction and repair of gas holders, boilers, pipes, etc. The process is rapid and repairs can often be done without interrupting the working of the plant. Electrically welded joints are less liable to leakage or to attack by corrosive fluids than riveted joints; they are, moreover, stronger and more economical in material. A. B. MANNING.

Corrosion at riveted and welded joints in fueltechnology practice. O. GROSS (Brennstoff-Chem., 1930, 11, 133—134. Cf. Kürschner, preceding abstract). —A number of cases are quoted, *e.g.*, sulphuric acid conduits and containers, pipes conveying steam containing ammonia and small quantities of sulphurous acid, copper centrifuges for draining acid ammonium sulphate, etc., in which much less corrosion occurs at riveted or flanged joints than at welded joints. A. B. MANNING.

Nomography in gas analysis. W. J. G. DAVEY (Gas World, 1930, 92, 694—695).—The application of nomographs to the determination of methane and hydrogen in coal gas when the percentages of the other constituents and the calorific value are known is described. C. W. GIBBY.

Coal-tar oil for heating industrial furnaces. H. G. LUHN (Brennstoff-Chem., 1930, 11, 192—193).— The objections which have been raised against the use of coal-tar oils in oil-fired furnaces are attributed principally to unsuitable choice of plant or faulty operation. The oil should always be preheated, not only to prevent separation of naphthalene or anthracene, but also to lower the viscosity and improve the atomisation at the burner. Coal-tar oil is lower in price than gas oil, and, owing to its higher carbon and lower hydrogen content, its use diminishes the heat lost in the flue gases. By preheating both the oil and the air rapid and efficient combustion is attained with a minimum excess air.

# A. B. MANNING.

Gum formation in cracked petrol. M. NAPHTALI (Chem.-Ztg., 1930, 54, 371).—The work of various investigators on the determination of gum, "preformed" and "potential," in cracked petrols and the comparative effect of inhibitors on its formation is summarised. Distillates cracked in the liquid phase contain much more resin than those cracked in the vapour phase.

## C. IRWIN.

Refining of benzol. Action of chlorine on crude benzol. T. I. RABEK (Brennstoff-Chem., 1930, 11, 189-192).-Bromine value determinations show that the unsaturated constituents of crude benzol occur principally in the lowest- and highest-boiling fractions. The intermediate fractions contain relatively small amounts of impurities. The sulphuric acid reaction is given not only by the unsaturated hydrocarbons, but also by some sulphur compounds of unknown constitution, and is unsuitable for the evaluation of benzol as a motor fuel. When the crude benzol is treated with chlorine the unsaturated constituents are attacked first and then the sulphur compounds, whilst the benzene itself remains unchanged. It is possible by such treatment to free the benzol completely from thiophen and its homologues. From the amounts of chlorine directly added, substituted, and unabsorbed, respectively, the unsaturation of the crude benzol has been determined and shown to correspond exactly with that deduced from the bromine values. A. B. MANNING.

Effect of refining agents and fused caustic alkali [alkali hydroxide] on [pure organic] sulphur compounds in naphtha solution. M. A. Yourz and P. P. PERKINS (Ind. Eng. Chem., 1930, 22, 610-611).-Treatment of solutions of n-, sec-, and iso-butyl sulphides in naphtha with sulphuric acid (cf. B., 1928, 78) causes almost complete removal of the sulphides. When aluminium chloride is used as the refining agent it is found that the n-sulphide is removed much less completely. Treatment of solutions of 13 sulphides with a mixture of sodium and potassium hydroxides at 343° results in the removal of most of the sulphide with heptyl and allyl sulphides, and ethyl and isoamyl disulphides; the lower alkyl and phenyl sulphides are not appreciably affected. H. BURTON.

Fuel-efficiency tests on batch oil stills. H. KREISINGER, W. R. ARGYLE, and W. E. RICE (U.S. Bur. Mines, Bull. 302, 1929, 94 pp.).-In order to promote the more efficient use of fuels in oil refining, 103 tests were made on batch oil stills fired with coal on chaingrate and underfeed stokers, with fuel oil, and with producer gas, various oil stocks being distilled. Comparable data are given for the three processes: (1) topping crude oil, (2) coking crude oil, and (3) re-running wax distillate. All distillations except the coking of the crude oil were made by the fire and steam method. Complete physical characteristics and chemical analyses of the fuels used are given, and the results are tabulated as weight of fuel fired per barrel of oil charged to each still, duration of the run, percentage of heat in the fuel lost in the flue gases, and the efficiency. The steam used in the stills was not considered in computing the heat accounts, and the amount of steam used was not measured except in a few tests on the re-running of the wax distillate for paraffin distillate. Samples of the flue gases were collected continuously and analysed every 30 min., their average carbon dioxide content for each test being recorded. Heat losses in unburnt carbonaceous matter in the ash and refuse, and losses due to radiation and convection are taken into account. The results obtained for each still or furnace are represented graphically. The first tests show how the efficiency might be improved by changes in the method of firing or operation, which are embodied in further tests. Data thus obtained point to further improvements that can be made by modifying the design or construction of the furnace. Such alterations are made step by step and their effect is determined by further tests. The development of the furnaces is traced by a series of drawings augmented by a discussion in the text. Temperature curves in connexion with heat distribution within the still during operation are recorded.

H. S. GARLICK.

Heavy distillates, fuel oils, asphalts, and residues. F. H. GARNER (J. Inst. Petroleum Tech., 1930, 16, 281–283).—A series of abstracts of recent publications on these subjects is presented.

H. S. GARLICK.

Special [petroleum] products. J. A. CARPENTER (J. Inst. Petroleum Tech., 1930, 16, 284-312).-A

review of recent literature and patents concerning paraffin wax: its crystallisation, structure, chemical constituents, physical properties, and behaviour of waxoil mixtures; the use of petroleum products in insecticides and larvicides; naphthenic acids; oxidation and oxidised products of petroleum; asphalt, pitch, and bituminous emulsions. H. S. GARLICK.

Analysis and testing [of petroleum products]. C. I. KELLY (J. Inst. Petroleum Tech., 1930, 16, 325— 333).—The new and modified methods of the Inst. Petroleum Tech., the B.E.S.A., the Bureau of Mines, and the A.S.T.M., for testing petroleum and its products, are reviewed. Methods of testing gasoline for knockrating and gum content are also included.

H. S. GARLICK.

Synthetic fuels. A. W. NASH (J. Inst. Petroleum Tech., 1930, 16, 313—324).—The progress of research during the last 2 years concerning (a) catalytic berginisation of coal and heavy tarry materials, and (b) production of liquid hydrocarbons from gases, e.g., methane, ethane, propane, etc., and their polmerisation to heavier oils is reviewed, and extensive references are made to the patent literature covering these processes.

H. S. GARLICK.

Lubricants. R. W. L. CLARKE (J. Inst. Petroleum Tech., 1930, 16, 255-265).—Progress made in this subject in the latter part of 1927 and during 1928—9 is reviewed. The chief improvements appear to be in the manufacturing and refining branches of the mineral oil industry. The problems of the cause of oiliness and the correlation of laboratory with practical tests have received attention. H. S. GARLICK.

Thermal decomposition of [lubricating] oils. J. DAMIAN and G. DIXMIER (Chim. et Ind., 1930, 23, 834-839).-The changes in viscosity, mol. wt., saponif. value, and iodine value of castor oil and two mineral lubricating oils, on being heated at 140° in contact with air, have been determined. The oils were heated in copper boats which were kept in oscillation ; the bottom of each boat was so shaped that in flowing from one end to the other the oil passed over a raised central portion, and was thereby exposed to the air in the form of a thin film. The viscosity, measured at 35° and at 100°, and the mol. wt. of each oil increased with the time of heating, whilst the iodine value fell. The rise in mol. wt. corresponded with the formation of a dark-coloured sludge, which, in the case of the mineral oils, was insoluble in the original oil, but was partially soluble therein in the case of the castor oil. The changes were similar to those occurring in the engine, but took place much more rapidly in the laboratory test.

A. B. MANNING.

Graphite lubrication. Colloidal graphite. R. CORDEBAS (Chim. et Ind., 1930, 23, 1092—1098).— Commercial graphite from Madagascar contains about 15% of siliceous impurities which render it quite unsuitable for lubrication purposes. The refined product should be wholly crystalline, powdered to pass 200-mesh, and free from amorphous graphite. It is used in the dry state, particularly at high temperatures, or incorporated in oil etc. At low pressures or high speeds a colloidal solution of graphite in water is an effective

lubricant, but it is only at low speeds and high pressures that the benefit of the use of graphite is realised. The object of graphite lubrication is to obtain a layer of graphite adsorbed in the metallic surfaces 1µ in thickness. This layer when formed will persist if oil alone is subsequently used. Colloidal graphite can be obtained by grinding, or by the action of potassium bichromate and sulphuric acid on graphite, but these methods are expensive. A stabiliser such as soap or gum arabic should be added to the colloidal oil solution. It is also possible to apply graphite by the use of a special antifriction metal containing it. Powdered graphite may be added to the amount of 1% to oil and kept in suspension by rapid circulation until graphitisation has occurred. This treatment should be repeated every two months. Graphite may also be incorporated in grease. C. IRWIN.

Heavy oils and lubricants. W. LEE (J. Inst. Petroleum Tech., 1930, 16, 266—279).—A progress report covering the years 1928—1929 is presented, together with a review of the literature and patents dealing with the subject. The production of heavy lubricants by polymerisation and the recovery of used oils is referred to, and attempts which have been made to correlate laboratory tests with results obtained in practice are noted. H. S. GARLICK.

Sulphur chloride from gas-purifier refuse. IPATIEV and VASILEVSKI.—See VII. Hot-patching of retorts. PARDOE.—See VIII.

## PATENTS.

Coke ovens. DR. C. OTTO & Co., G.M.B.H. (B.P. 315,668, 15.7.29. Ger., 14.7.28).—The lower ends of the vertical flues of a horizontal coke oven are divided by a perforated partition into passages for gas and air. The partitions are built of removable superimposed bricks formed with slanting channels through them, and designed to ensure that each brick will be permeable to gas and air and thus bring about any desired degree of pre-combustion. A. B. MANNING.

Apparatus for baking carbonaceous fuel briquettes. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 304,284, 14.1.29. U.S., 18.1.28).-A multiple-chamber baking oven comprises a battery of horizontal ovens vertically disposed one above the other, each provided with one or more fuel containers, removable from one end thereof, for charging and discharging purposes. Between the ovens are horizontal, transverse passageways through which the heating gases are passed from suitably arranged combustion chambers; the passageways are provided with baffles in order to give a turbulent flow to the gases. The temperature of the heating gases can be adjusted by returning a controlled amount of the flue gases to the combustion chambers. Means are provided for admitting air to the baking ovens and for leading the mixture of air and volatile products from the ovens to the combustion chambers. A condensing system is also provided for the recovery of condensible products evolved from the briquettes during the baking, the non-condensible products being led to the combustion chambers. The opparatus is adapted to carry out the

process in two stages as described in B.P. 320,814 (B., 1930, 7). A. B. MANNING.

Destructive hydrogenation of carbonaceous bodies. HOLZVERKOHLUNGS-IND. A.-G., and J. VARGA (B.P. 313,505, 12.6.29. Ger., 12.6.28).—Coal, tars, oils, etc. are treated with an excess of hydrogen at a high temperature and under pressure in the presence of catalysts containing molybdenum and/or tungsten, and in the presence of hydrogen sulphide, or a material liberating hydrogen sulphide under the conditions of the reaction, *e.g.*, elemental sulphur. An auxiliary catalyst or activator may also be used, *e.g.*, boric acid. The presence of the hydrogen sulphide brings about a marked decrease in the tar acid content of the product and an increase in the quality and yield of light oils. A. B. MANNING.

Manufacture of finely-divided active carbon. I. G. FARBENIND. A.-G. (B.P. 304,791, 25.1.29. Ger., 26.1.28).—Carbon monoxide is passed at atmospheric or higher pressures over a catalyst comprising a metal of group VIII, *e.g.*, iron, nickel, or cobalt, in a finelydivided form, in admixture with a difficultly reducible oxide, *e.g.*, magnesia or alumina. The reaction is carried out preferably at 300—350°. A hydrocarbon, *e.g.*, methane, may be mixed with the carbon monoxide. The catalyst is dissolved out from the finely-divided carbon formed and is recovered. A. B. MANNING.

Treatment of carbon black and other fine powderous materials. J. E. POLLAK. From W. B. WIEGAND and L. J. VENUTO (B.P. 327,979, 14.1.29).— Finely-powdered materials are converted into readily frangible, non-dusting agglomerates by treatment with two non-miscible liquids, both non-solvents for the material, one of which has a greater wetting power for the solid than the other. The agglomerates so formed are separated by filtration or decantation, and are then dried. Suitable liquids for use with carbon black are water and petrol. The agglomerates are denser than the original carbon and the industrially essential physical properties, *e.g.*, easy and uniform dispersion in oils, rubber, etc., are unaffected. A. B. MANNING.

Coolers or condensers for fuel gases. D. M. HENSHAW and W. C. HOLMES & Co., LTD. (B.P. 328,421, 12.4.29).—Below the condenser of a plant for the production of fuel gas are two chambers, one for accommodating mainly the condensed tar, and the other for cooling the aqueous distillate. A pipe from the condenser delivers the total condensate to the first chamber, whence the aqueous condensate passes through an overflow into the second chamber. The latter is provided with banks of tubes through which cold water is circulated. The condensate are preferably separated from the tar-collecting chamber by heatinsulating partitions. A. B. MANNING.

Production of water-gas or coal- and water-gas. BAMAG-MEGUIN A.-G., and O. HELLER (B.P. 304,139, 14.1.29. Ger., 12.1.28).—Finely-pulverised fuel and a mixture of gas and highly superheated steam are admitted to a reaction chamber, and the resulting gas is passed through a dry ash collector and a heat exchanger, after which part of the gas is passed through a wet dust filter, which serves as a steam generator, back to the steam superheater and the reaction chamber, while the surplus gas is passed through a second dust filter to a holder. The superheater is heated intermittently by the combustion of gas therein. The process may be made continuous by duplicating the superheater and reaction chamber, and arranging for each set to be operated alternately as described above.

# A. B. MANNING.

Gas purification. D. L. JACOBSON, ASST. to KOPPERS Co. (U.S.P. 1,741,113, 24.12.29. Appl., 11.1.27).— Gases are freed from hydrogen sulphide by washing with an alkaline solution containing in suspension a compound of a metal, the sulphide of which is insoluble therein, and containing also a compound of manganese. E.g., a liquid containing 1—3% of sodium carbonate, 2% of ferric oxide in suspension, and 0.5% of manganous chloride is suitable. The presence of the manganese diminishes the formation of sodium thiosulphate. The spent solution is revivified by aeration, and free sulphur is thereby recovered. A. B. MANNING.

Destructive hydrogenation of bars, oils, suspensions of carbonaceous materials in oils, etc. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 328,719, 21.2.29).—Heat exchangers used in connexion with high-pressure apparatus are built of concentric tubes, the outer one being thick enough to withstand the pressures, and the inner one thin-walled to allow of heat transference. The relatively cold material passes through the annular space to a reaction vessel in a countercurrent direction to hot material which is leaving the reaction vessel by means of the inner tube. T. A. SMITH.

Hydrogenation and distillation of (A) hydrocarbon oils and other carbonaceous material, (B) petroleum oils. STANDARD OIL DEVELOPMENT CO., Assees. of E. M. CLARK (B.P. 304,796-7, 26.1.29. U.S., 26.1.28).-(A) Preheated crude petroleum or reduced crude is mixed with heavy residuum from the stills and passed into a hydrogenation zone where the oil is held in contact with hydrogen or hydrogen-containing gases under a pressure exceeding 50 atm. (preferably about 200 atm.) and at a temperature of 400-510°, in the presence of a catalyst, preferably finely-pulverised metal oxides, e.g., 90% of chromium oxide and 10% of molybdenum oxide. Gases and vapours from the primary reactors are conducted into secondary reactors operating at 427-510° and above 25 atm. in the presence of the same catalyst, the requisite temperature being obtained by forcing in a supply of highly heated hydrogen or gas containing it. Vapour from the secondary reactors is cooled in preheating incoming crude oil and hydrogen in heat exchangers, and the liquid products are condensed in suitable condensers. The residual hydrogen is washed with alkali and water to remove hydrogen sulphide and with oil to remove hydrocarbons such as methane. The condensers may be of the simple type, or adapted to give any degree of reactification. The condensed hydrogenated oil is conveyed to a battery of stills, where it is separated into gasoline, naphtha, kerosene, gas-oil, and lubricating oil fractions. (B) The oil to be treated is preheated, and

a light fraction distilled from the oil in the first of a series of distillation zones, the residual oil being submitted to the above-mentioned process of destructive hydrogenation. H. S. GARLICK.

Recovery of motor fuel from crude oil, shale, lignite, coal, and other organic material. E. and E. L. SCHULTZ (B.P. 328,295, 22.1.29).-The crude oil or other material is vaporised in a retort or still, provided with the usual pressure- and temperature-recording instruments, up to a temperature of 270°, and the vapours are led off from the top to a pot or converter. The converter is fitted with heating means and a vertical shaft carrying agitating gear. The whole of the metal portions of the converter are formed preferably of a nickel-zinc-lead alloy or of steel. In the converter the vapours are subjected to the action of lime, zinc chloride, or zinc oxide, with or without sodium or magnesium chloride. The process consists in starting at a low pressure to recover the light oil, the temperature in the converter being regulated relatively to that in the still or retort, and then gradually raising the dual temperatures to treat the heavier oil, and thus obtain a high percentage of motor fuel fraction, the pressure being regulated by an expansion or needle valve as the temperature rises. The vapours in the converter are subjected to the action of the chemicals and pass through the valve to a dephlegmator and condenser from which the condensate is recovered, and, if required, treated with chlorine or alkali and fractionated with fuller's earth. The residue is then returned to be re-treated in a fresh batch of material.

H. S. GARLICK.

Cracking of petroleum oils. STANDARD OIL DEVELOPMENT Co., Assees. of N. E. LOOMIS (B.P. 304,798, 26.1.29. U.S., 27.1.28).-Heavy oil is heated to cracking temperature in a tube furnace and passed to a vertical reaction chamber fitted with an expansion valve. The oil leaving this chamber is diluted with a little fresh cracking stock or other oil before passing through the expansion valve. The vapours pass through a tar trap to a dephlegmator column which is fitted at top and bottom with coils, in which the cracking stock is heated by the cooling vapours. Condensate from the tower is added to the cracking stock and vapours from the tower are passed to a condenser. Coke deposition at the expansion valve is thereby reduced. T. A. SMITH.

Conversion of petroleums. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,741,509, 31.12.29. Appl., 21.10.21. Renewed 1.10.28).—Oil is continuously passed under superatmospheric pressure through a heating zone, where it is raised to cracking temperature, to an enlarged reaction zone where conversion occurs; the vapours are led to a dephlegmator and condenser from which the resulting condensate is collected. The reflux from the dephlegmator is separated into light and heavy fractions, the light fraction being returned directly to the heating zone for re-treatment, and the heavier passed to the reaction zone. H. S. GARLICK.

Treatment of petroleum products. F. B. MUHLEN-BERG (U.S.P. 1,750,420, 11.3.30. Appl., 15.12.25).— Gasoline, kerosene, head oil, etc. containing sulphur impurities are treated with an ammoniacal solution of copper hydroxide by spraying such a solution into the oil at atmospheric temperature, continuously or periodically removing the solution and impurities from the bottom of the oil and returning it to the sprayer, and repeating the operations until the oil is sufficiently purified. H. S. GARLICK.

Apparatus for treating crude petroleum. G. A. STAFFORD and C. D. CORK (U.S.P. 1,749,235, 4.3.30. Appl., 16.2.25).—Crude oil is stored in closed storage tanks fitted with drain valves for the removal of water. Gas given off from the oil or displaced from the storage tank is taken to another storage tank through a condenser. Liquid products condensed from the gas may be removed, and oil is displaced from the storage tank by returning the gas which is forced through the oil. Any light constituents are absorbed and the gravity of the oil is lowered. In addition, gas may be taken from a gas-well and pumped through the oil with the object of lowering the gravity of the oil. T. A. SMITH.

[Mineral] oil cracking process. J. B. JAQUA (U.S.P. 1,752,004, 25.3.30. Appl., 26.11.26).—Oil at a temperature slightly below cracking temperature is brought to reaction temperature by injecting small streams of a stable oil, heated externally to a temperature above that desired to effect an endothermic reaction of the new mineral oil, into an advancing stream of the oil. The additions are proportioned and so spaced, relatively to the dimensions and rate of advance of the treated oil, that a desired reacting temperature or temperatures is maintained for a predetermined period. H. S. GARLICK.

Treatment of emulsions of mineral oils. W. B. LERCH, Assr. to J. S. DEWAR (U.S.P. 1,747,119, 11.2.30. Appl., 31.5.28).—An improved demulsifying agent for resolving stable hydrocarbon emulsions consists of sulphonated, highly unsaturated hydrocarbons of the polyolefine, cyclopentene, and cyclohexene series, obtained by the addition of sulphuric acid to materials such as Blau or Pintsch gas tars or other residual tars containing an appreciable alumina content in their ash.

H. S. GARLICK.

Cracking of [hydrocarbon] oil. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,741,510, 31.12.29. Appl., 26.6.26).—Oil is raised to cracking temperature while it is flowing through a heating coil, and the evolved vapours are subjected to reflux condensation. A number of progressively lighter fractions of the condensate are collected and the lightest fraction is returned to the inlet of the heating coil, while progressively heavier fractions are separately returned to spaced points in the length of the heating coil, thereby passing the heavier fractions through a smaller portion of the heating coil than the lighter fractions. H. S. GARLICK.

Hydrocarbon oil conversion. C. J. PRATT, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,741,535, 31.12.29. Appl., 8.7.26).—Charging stock is subjected to non-cracking conditions of temperature and the vapours are separated into lighter and heavier portions. The separated fractions are then simultaneously subjected to independently controlled temperature and pressure conditions so that the lighter fractions are

treated at higher temperatures and pressures than the relatively heavier ones. The vapours from the fractions are dephlegmated in the zone where fractionation of the charging stock takes place.

#### H. S. GARLICK.

Cracking and distillation of heavy hydrocarbon oils. MIDLAND COAL PRODUCTS, LTD., and J. E. TRUZZELL (B.P. 328,639, 19.12.28).—Heavy hydrocarbon oil is mixed with up to 40% by wt. of bituminous coal slack and, while being agitated, is submitted to a distilling and cracking process under a pressure not above 150 lb./ in.<sup>2</sup> and a temperature not above 200°, this being sufficient to volatilise light hydrocarbons without distilling the coal. The residue is submitted to low-temperature carbonisation, giving a product suitable for fuel in Diesel engines and a semi-coke. H. S. GARLICK.

Distillation of heavy liquid hydrocarbons. J. H. DE LIGNAC (B.P. 328,875, 22.7.29).—Heavy oil, mixed with water, is distilled in a vessel containing in its lower portion a catalyst supported on grids. The catalyst consists of separate layers of nickel (2 pts.), copper (1 pt.), and iron (1 pt.). The vapours produced pass through similar beds of catalyst supported in the upper part of the chamber and through a further mass of catalyst in the vapour pipe before being condensed. T. A. SMITH.

Decolorising and colour-stabilising hydrocarbon oil. V. VOORHEES, ASST. to STANDARD OIL CO. (U.S.P. 1,747,806, 18.2.30. Appl., 31.10.27).—Pressure distillate, which has been treated in the vapour phase with clay, is treated with 1% by vol. of sulphuric acid (10—70% H<sub>2</sub>SO<sub>4</sub>). The product does not develop colour on storing. T. A. SMITH.

Production of stable hydrocarbon oils. B. T. BROOKS, ASST. to GRAY PROCESS CORP. (U.S.P. 1,748,507, 25.2.30. Appl., 10.10.25).-0.5-1.5 G. of ammonia are added per gal. of gasoline in order to stabilise the colour and prevent sedimentation. H. S. GARLICK.

Manufacture of hydrocarbons of low b.p. from those of higher b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,649, 1.2.29).-Tars, mineral oils, etc. are cracked in the presence of solid catalysts which maintain their shape unchanged under the conditions of working and which are dispersed in the liquid materials to be treated. Suitable catalysts are, e.g., bauxite, silica gel, etc., and those containing molybdenum or chromium. A part of the liquid material, together with the catalyst dispersed therein, is drawn off, without interruption of the cracking process, from the reaction chamber and, if desired, returned after partial or complete separation from the catalyst, which is regenerated outside the reaction chamber and then reintroduced either alone or together with fresh initial material. H. S. GARLICK.

Conversion of hydrocarbons of high b.p. into those of lower b.p. J. Y. JOHNSON. From I. G. FARB-ENIND. A.-G. (B.P. 329,959, 22.2.29).—Tars, mineral oils, etc. are subjected to consecutive restricted crackings in such manner that each time fractions having a comparatively narrow b.-p. range not exceeding 200° are treated, and at least one stage of the process is carried out in the presence of catalysts immune from sulphurpoisoning and under pressure of at least 50 atm.

H. S. GARLICK.

Manufacture of low-boiling hydrocarbon products. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,618, 13.10.28).-Carbonaceous materials which are liquid at ordinary temperatures (e.g., producer tar, tar from mineral coal) or middle oils obtained therefrom in the liquid state, are treated with a stream of hydrogen or hydrogen-containing gas at an elevated pressure and temperature, and in the presence of a catalyst (containing molybdenum or chromium) immune to sulphur-poisoning, under such conditions that products richer in hydrogen than the initial materials are obtained; these are then subjected to a high-temperature cracking process in the presence of gases having a reducing action, in order to obtain products of low b.p. [Stat. ref.] H. S. GARLICK.

Manufacture of hydrocarbons and oxygen derivatives of hydrocarbons, in particular those of low b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,586, 26.11.28).-Carbonaceous materials are treated with hydrogen at elevated temperatures and at pressures above 20 atm. in the presence of catalysts which are immune to sulphur-poisoning, and before or during the reaction a medium which is liquid under the conditions of working and which does not take part in the reaction, e.g., high-boiling organic solvents, in particular, oils of aromatic character, is added for the purpose of preventing the deposition of high-molecular substances on the catalyst. The medium is continuously removed from the reaction chamber, the lighter-boiling fractions are removed, and the residue is returned to the process. H. S. GARLICK.

Separation of hydrocarbons. W. K. LEWIS, ASST. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,746,197, 4.2.30. Appl., 16.8.22).—Mixtures of gasoline and kerosene are fractionated under a pressure of 15 lb., a temperature difference of approx. 80° being maintained between the top and bottom of the fractionating column used. T. A. SMITH.

[Production of gasoline from heavier hydrocarbons by] pressure distillation. R. E. HUMPH-REYS, ASST. to STANDARD OIL CO. (U.S.P. 1,745,931, 4.2.30. Appl., 9.4.21).—Gasoline is produced by distilling naphtha residues at about 340° and under 325 lb./ in.<sup>2</sup> T. A. SMITH.

**Production of gasoline from cracked distillate.** G. P. KOCH, ASST. to SHELL OIL CO. (U.S.P. 1,742,263, 7.1.30. Appl., 29.3.26).—Cracked distillate is treated with concentrated nitric acid in amount substantially equal in wt. to the sulphur content of the oil (as determined by previous analysis), without materially affecting the unsaturated hydrocarbons; it is then washed with water and further washed with caustic soda and sulphuric acid in amount considerably less than if the nitric acid had not been used. After a final water wash the gasoline and unsaturated hydrocarbons are distilled off from the distillate. H. S. GARLICK.

Recovery of gasoline from natural gas. F. E. HOSMER (B.P. 311,693, 14.1.29. U.S., 14.5.28).—" Wet"

natural or casinghead gas enters the bottom of a tower and flows upwardly past a number of bubble-plates before coming in contact with a refrigerating coil. The liquefied constituents fall back through the bubble-plates and are partially evaporated by the incoming gas, which is thereby cooled and saturated with the previously condensed constituents. Continuing upwardly, the now lean gas leaves the system after passing through a second series of bubble-plates over which a stream of brine is pumped from a reservoir at the base of the tower. The brine stream is cooled by the lean cold gas and picks up the condensed constituents of the gas, washing them downwardly past the lower bubble-plates, where some of the gaseous constituents are evaporated, into the reservoir at the base of the tower, which is provided with a trapped line through which the separated and "weathered " gasoline is removed. H. S. GARLICK.

Apparatus for salvaging used cleaners' gasoline. W.S. BAYLIS, ASST. to FILTROL CO. OF CALIFORNIA (U.S.P. 1,751,613, 25.3.30. Appl., 21.12.25).—The used solvent passes through a trap to remove any large particles and is pumped through a primary filter consisting of a shell provided with layers of filtering material separated by perforated plates. From this filter the solvent passes to a washer where it is agitated with an absorptive material, *e.g.*, fuller's earth. After settling, the solvent is drawn off to a secondary filter capable of removing very fine particles and is then passed to storage. Arrangements for cleaning the primary filter by backwash are described. H. S. GARLICK.

Manufacture of lubricating oils. G. H. TABER, JUN., Assr. to SINCLAIR REFINING CO. (U.S.P. 1,746,916, 11.2.30. Appl., 15.12.28).—Vapours from a lubricating oil still are passed to a tower down which alkali solution is sprayed. Vapours which are not condensed in the tower are passed through a cooling coil. Condensate and alkali from the treating tower are received in a tank where they may be separated, or recirculated if desired. The apparatus is arranged to work under ordinary or reduced pressure. T. A. SMITH.

Recovery of entrained [petroleum lubricating] oils from filter cakes. G. F. OLSEN (U.S.P. 1,745,837, 4.2.30. Appl., 22.1.27).—Entrained oil is recovered from a hot filter-press cake which is simultaneously cooled by displacing and dissolving the entrained oil with a petroleum solvent intermediate in gravity and viscosity between kerosene and lubricating oil of 75 sec. viscosity (Saybolt). The solution is reduced by partial distillation to the consistency of a lubricating oil, the recovered distillate being utilised in a succeeding operation.

H. S. GARLICK.

Purification of used lubricating oils. J. G. FORD, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,746,641, 11.2.30. Appl., 6.8.27).—Used transformer etc. oils are treated with 25—50% by vol. of liquid sulphur dioxide. After separation the residual sulphur dioxide is removed by treatment with alkali or a stream of air or nitrogen. It is then treated with 5% by wt. of infusorial earth at 100° and filtered. T. A. SMITH.

Refining of used [lubricating oi J. C. PATRICK (U.S.P. 1,750,350, 11.3.30. Appl., 28.10.27).—Free water is allowed to settle from used crank-case oi which is then heated to 49° and agitated at that temperature with 2% by vol. of sulphuric acid ( $d \ 1.83$ ). After settling, the oil is removed from the resultant sludge and heated under vacuum to 232° to remove diluents. The hot oil is treated with 15—18% of fuller's earth to which has been added an anhydrous oxide in order to clarify, decolorise, and neutralise the oil, and the resultant purified oil is drawn off. H. S. GABLICK.

Reclaiming used lubricating oils. H. W. CHETWIN and W. MANN (B.P. 328,558, 25.1.29).-Used lubricating oils (flash point under 177°), if emulsified, are brought to the b.p. with about 20% of a 10% alum solution before proceeding with the same treatment as with non-emulsified oil, which consists in heating it for 4-6 hrs. at 82-88°, allowing to settle for 10 hrs. without further heating, centrifuging or filtering the oil, passing it through a continuously working steam-heated still wherein light fractions are volatilised, clarifying by means of an acid, washing with alkali, and finally filtering. The still consists of one or more pipes each having downwardly extending baffle plates subdividing the interior, with an uptake pipe in the roof communicating with each subdivision and uniting in a common condenser pipe leading to one or more water-cooled receivers.

H. S. GARLICK.

Apparatus for treating used lubricating oil. R. M. DILWORTH and F. G. NIECE, Assrs. to ELECTRO-MOTIVE Co. (U.S.P. 1,749,571, 4.3.30. Appl., 4.11.27).—A vertical still is connected to a heating coil, a charging tank, and a condenser. The arrangement is such that oil pumped from the charging tank through the coil to the still returns to the charging tank when the oil in the still reaches a determined level. Steam is passed into the oil stream before it enters the heating coil. Vapours from the still are taken to a condenser. The oil is circulated until all the volatile impurities have been removed. A small draw-off line is so arranged that any water condensed in the still is drawn into the oil stream to the heating coil and not into the storage tank. After sufficient treatment the oil is filtered before re-use.

T. A. SMITH.

**Reclaiming [mineral insulating] oil.** D. C. Cox, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,752,238, 25.3.30. Appl., 18.3.25).—Sludged transformer oil is passed through a series of tanks, the oil flowing from the bottom of one into the top of the next. Each tank contains a charge of fuller's earth supported on a perforated plate with a suitable filter-cloth interposed between the earth and the plate, and a second fabric is placed on top of the earth. Means are provided for separating any one or more tanks from the series for cleaning and recharging without interfering with the rest. To the cleaned oil is added an approximately equal volume of new oil before returning it for use in a transformer.

H. S. GARLICK.

Apparatus for refining [mineral] oil. G. H. TABER, JUN., Assr. to SINCLAIR REFINING Co. (U.S.P. 1,746,915, 11.2.30. Appl., 15.12.28).—Lubricating oils are produced in a plant consisting of a series of stills each provided with its own fractionating tower, treating tower connected to the fractionating tower, condenser, and cooler for receiving the condensate from the fractionating tower. Caustic alkali solution is supplied to at least one of the

treating towers of the series, the condensate from the associated cooler being delivered to a succeeding treating tower of the series. H. S. GARLICK.

Purification of hydrocarbon distillates. R. C. OSTERSTROM (B.P. 328,309, 9.10.28) .- Cracked hydrocarbon oils containing unsaturated compounds are treated with an absorbent capable of polymerising gumforming and colour-imparting constituents normally present in such oils by introducing the absorbent into a stream of oil or oil vapours which carry the absorbent with them in their subsequent continuous flow before or after vaporising the oils. The effective treatment takes place while the oil is in the vapour phase, the heat generated by the reaction causing an increase of pressure and temperature sufficient to cause polymerisation, but insufficient to cause cracking. The mixture is discharged into an enlarged separating or vaporising and fractionating zone the upper part of which is supplied with means for applying heat in order to facilitate the separation of the light oils from the heavier polymerised oils and absorbent brought about by the release of pres-H. S. GARLICK. sure.

Refining of benzol. Soc. DU GAZ DE PARIS (B.P. 307,935 and Addn. B.P. 330,045, [A] 15.3.29, [B] 2.4.29. Fr., [A] 17.3.28, [B] 29.11.28).—(A) The vapours of crude benzol, b.p. 75—180°, are passed through porous masses, impregnated with sulphuric acid ( $d \ 1.83$  or less) and used in the proportion of 1-2% of the benzol treated, placed in a suitable heat-insulated apparatus capable of being heated, if necessary. The refined benzol is washed free from acid, and the porous mass as it becomes inactive is replaced by fresh acidified material. (B) The fraction of crude benzol distilling over the range 65—120° is caused to flow through a mass of refractory material maintained at 90—95° countercurrent to a stream of dilute sulphuric acid, and is washed with water and alkali and finally rectified in a reflux condenser.

H. S. GARLICK.

Vacuum distillation and rectification of paraffin distillate. N. E. LOOMIS and W. K. LEWIS, ASSTS. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,746,198, 4.2.30. Appl., 2.6.24).—Paraffin-base crude oil is heated to not above 340° under an absolute pressure not exceeding 30 mm. Hg, and the vapours are led to a group of fractionating zones where liquid and vapour phases exist in partial equilibrium, the vapours being subsequently led away and those of the paraffin distillate condensed, cooled to a point at which the paraffin precipitates in crystalline form, and filtered to produce a residue of paraffin wax and a filtrate of petroleum lubricating oil. H. S. GARLICK.

Purification of wax distillates. E. PETTY, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 1,750,646, 18.3.30. Appl., 24.8.21).—Purified paraffin and wax-free oils of different gravities are produced by separately distilling from a petroleum crude oil a number of wax distillates and subjecting each fraction separately to the following treatment: dilution with a light-petroleum distillate, agitation of the diluted stock with acid, removal of the acid sludge, agitation of the stock with alkali, and centrifuging the resultant emulsion to separate the soap and alkaline solution. The diluted stock is then chilled sufficiently to precipitate the wax and centrifuged, thereby separating the wax from the oil. The wax-free oil is then further treated with a clarifying and decolorising agent, the last traces of which are finally removed by centrifuging, and the diluent is eliminated from the oil by distillation. H. S. GARLICK.

Manufacture of motor fuels. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,587, 22.12.28).— The stability to compression of motor fuels consisting substantially of saturated hydrocarbons and containing, if desired, iron carbonyl, lead tetraethyl, alcohols, amines, or benzol, is increased by the addition of up to 30% of liquids consisting only of polymerides of the lower olefines. Examples are diisobutylene, alone or with diisoamylene; mixed polymerides of isobutylene and trimethylamine. H. S. GARLICK.

Motor fuels and their manufacture. CARBIDE & CARBON CHEM. CORP. (B.P. 303,505, 13.12.28. U.S., 6.1.28).—Alkylbenzenes, the alkyl groups of which contain two or more carbon atoms, are added to motor fuels in order to produce an antidetonating fuel for use in high-compression engines. A. B. MANNING.

Purification [desulphurisation] of [combustible] gases. E. A. PRUDHOMME, ASST. to SOC. INTERNAT. DES PROC. PRUDHOMME (S.I.P.P.) (U.S.P. 1,760,522, 27.5.30. Appl., 7.4.26. Fr., 4.3.26).—See B.P. 267,138; B., 1928, 220.

Treatment of [asphalt] dispersions. F. R. Moser (U.S.P. 1,759,913, 27.5.30. Appl., 27.7.25. Holl., 2.1.25).—See B.P. 245,418; B., 1927, 101.

Treatment of hydrocarbon oils. J. B. WEAVER, Assr. to Gyro Process Co. (Re-issue 17,681, 27.5.30, of U.S.P. 1,601,786, 5.10.26).—See B., 1927, 6.

Protecting the interiors of oil-cracking retorts. A. C. HOLZAPFEL (U.S.P. 1,761,343, 3.6.30. Appl., 30.3.27).—See B.P. 291,585; B., 1928, 560.

Burners for gaseous, liquid, or powdered solid fuel. P. J. J. ANDRIEUX, Assee. of Soc. GÉN. DES CHAUX ET CIMENTS (B.P. 310,881, 29.4.29. Fr., 2.5.28). Miners' safety lamps. G. MÜLLER (B.P. 330,469, 5.9.29. Ger., 6.7.29. Addn. to B.P. 318,481).

Grates more especially for gas producers. A. L. GALUSHA (B.P. 315,887, 20.7.29. U.S., 20.7.28).

[Valve for] retort-house gas governors. METERS, LTD., and W. T. GLOVER (B.P. 330,189, 23.8.29).

Retorts for continuous working (B.P. 317,066). Bubble tower (U.S.P. 1,748,855). Pulveriser (U.S.P. 1,747,922). Separation of solids by flotation (B.P. 329,013). Fractionation (U.S.P. 1,748,411, 1,748,508, 1,748,595, and 1,748,704). Removing oil from gases (B.P. 306,899).—See I. Purified ethylene (U.S.P. 1,741,559). Cuprene from acetylene (B.P. 303,797). Humous colloids (B.P. 312,233). Dispersing etc. agents (B.P. 300,574).—See III. Neutral ammonium sulphate (B.P. 310,536). Thiocyanates from gas liquors etc. (U.S.P. 1,751,274). Phosphorusfree carbon monoxide (G.P. 462,521).—See VII. Refractories (B.P. 304,729).—See VIII. Construction of roads etc. (B.P. 329,569).—See IX. Purification of hot gases (B.P. 329,962).—See XI. Lyophile products (B.P. 329,305). Emulsions (B.P. 329,266). —See XII. Lacquers etc. (B.P. 329,694).—See XIII. Paraffin emulsions (G.P. 446,598).—See XX.

# III.—ORGANIC INTERMEDIATES.

Petroleum products. CARPENTER.—See II.

#### PATENTS.

Purification of ethylene [for anæsthesia]. H. H. DAWSON, ASSR. to OHIO CHEM. MANUF. Co. (U.S.P. 1,741,559, 31.12.29. Appl., 7.1.25).-Relatively warm ethylene is expanded from a pressure of about 1500 lb./ in.<sup>2</sup>, whereby the resulting cold causes condensation of moisture and other impurities, which are removed; the cold gas is then passed in heat-exchange relation with warmer compressed ethylene, treated with sulphuric acid of sufficient concentration to remove olefinic impurities without reaction with the ethylene itself, neutralised (preferably with caustic potash solution), compressed, and dried. The product is again treated with sulphuric acid and neutralised, and the gas finally passed in heat-exchange relation with cold ethylene and collected at a pressure less than that from which it was initially expanded. H. S. GARLICK.

Manufacture of acetic anhydride. O. Y. IMRAY. From A.-G. F. STICKSTOFFDÜNGER (B.P. 328,572, 29.1.29). —Concentrated acetic acid is treated with carbonyl chloride in presence of about 10% of aluminium chloride, magnesium acetate, or an oxide, chloride, or acetate of a metal of group II or III. C. HOLLINS.

Preparation of highly adsorbent cuprene from acetylene. N.V. "ELECTRO" ZUUR- EN WATERSTOF-FABR. (B.P. 303,797, 8.1.29. Holl., 9.1.28).—Acetylene is passed at 200—400° over copper, nickel, iron, or oxides of these, to which less than 1% of magnesium powder has been added. C. HOLLINS.

Manufacture of  $1:3-[\gamma-]$ butyleneglycol. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,083, 9.3.29).—Aqueous aldol is hydrogenated in presence of a copper catalyst, *e.g.*, reduced copper on silicic acid or on vitreous alumina, reduced copper silicate, copper and nickel on silica gel, etc., at 40—90° and 50—200 atm. C. HOLLINS.

Manufacture of aliphatic or cyclic primary amines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,381, 11.3.29).—Unsaturated aldehydes or ketones are passed in vapour phase with ammonia and hydrogen over catalysts as in B.P. 265,960 (B., 1928, 360). Saturated amines are produced; e.g., crotonaldehyde gives *n*-butylamine; acraldehyde, *n*propylamine; and cinnamaldehyde,  $\gamma$ -phenyl-*n*-propylamine (b.p. 102—104°/118 mm.). C. HOLLINS.

Production of humous colloids. R. EBERHARD (B.P. 312,233, 16.5.29. Ger., 22.5.28).—Humous substances (powdered peat) are boiled with aqueous solutions of neutral or weakly basic organic compounds containing nitrogen or nitrogen and sulphur, other than aliphatic amides; *e.g.*, hexamethylenetetramine, carbamide, formanilide, formaldoxime, acetaldehydeammonia, piperazine, etc. C. HOLLINS.

Manufacture of esters of polymerised carbohydrates. I. G. FARBENIND. A.-G. (B.P. 305,661, 29.1.29.

Ger., 10.2.28).—A polymerised carbohydrate (cellulose, starch, agar-agar) is allowed to swell in caustic alkali solution of at least 30% concentration, and then treated preferably in suspension in a solvent (benzene, chlorobenzene, etc.) with a halide of a higher fatty acid or cyclic carboxylic acid (lauric acid, coconut oil acids, naphthenic acid, phenylacetic acid), without any cooling; if desired, heat may be applied. The resulting esters are soluble in organic solvents. Mixed esters may be prepared. C. HOLLINS.

Catalytic molecular association [esterification] of organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 308,582, 26.2.29. U.S., 23.3.28).—Baseexchange substances (artificial zeolites) containing esterification catalysts, such as zirconia, titania, alumina, or thoria, are used to catalyse the production of esters from vapours of alcohols and organic acids at 280— 350°. C. HolLINS.

Manufacture of dispersing, emulsifying, and stabilising agents, and of dispersions and emulsions. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 300,574, 11.10.28. Holl., 15.11.27).—The aromatic constituents extracted from mineral oil by liquid sulphur dioxide, furfuraldehyde, aniline, methyl sulphate, or similar solvents are sulphonated to give products similar to those of B.P. 291,393 (B., 1929, 805). C. HOLLINS.

Manufacture of wetting, cleansing, dispersing agents, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,675, 31.1.29).—Salts, amides, esteramides, or esters of high-molecular organic acids, which are wetting agents etc. are improved by incorporation with wetting agents of the sulphonic acid type. Examples are :  $\beta$ -diethylaminoethylamide of oleic acid and sodium propylnaphthalenesulphonate; stearamide and sulphonated oleic acid; oleic  $\beta$ -hydroxyethylamide, or anilide, or cyclohexylamide and diisopropylnaphthalenesulphonic acid. C. HOLLINS.

Sulphonation of aromatic hydroxy-derivatives. MAJOR & CO., LTD., and H. H. HINCHCLIFFE (B.P. 328,220, 19.1.29).—Phenol, o- and p-cresols,  $\alpha$ - and  $\beta$ -naphthols are sulphonated in the form of their aryl-sulphonates with concentrated sulphuric acid at 40—70° or oleum at 15—20°. On diluting the reaction mixture the arylsulphonyl group is removed and the phenol-sulphonic acid is obtained in solution. The process is ineffective with nitrophenols. C. HOLLINS.

Manufacture of m-2-xylidine. IMPERIAL CHEM. INDUSTRIES, LTD., and L. J. ALLCHIN (B.P. 328,418, 11.4.29).—After removal of the bulk of m- and p-xylidines by known methods, the crude oil is dissolved in hydrochloric acid and zinc chloride is added to the hot solution. The double salt of m-2-xylidine separates on cooling. C. HOLLINS.

Manufacture of aromatic amines from mixtures thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,210, 18.1.29).—Dry hydrogen chloride is led into the mixture of aromatic amines, preferably in an organic solvent, and successive fractions of hydrochloride are removed. Crude xylidine in benzene gives a 90% recovery of *m*-xylidine hydrochloride, and the *p*- and *o*-salts separate in that order by application of more

hydrogen chloride. The method is also used for the separation of m-4- from m-2-xylidine, o-4- from o-3-xylidine, p- from o-toluidine, 5- from 8-chloro- $\alpha$ -naphthylamine, aniline from toluidines, etc. C. HOLLINS.

Manufacture of hydroxysulphamic acids and salts thereof. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,032, 13.1.28).—An aminonaphthol, aminoarylpyrazolone, acetoacetic or 2:3-hydroxynaphthoic aminoarylamide is treated with chlorosulphonic acid or ester, sulphur trioxide in tetrachloroethane, or pyridinesulphuric anhydride, preferably in presence of pyridine or other acid-binding agent. The N-sulphonic acids of 2:7-aminonaphthol, 1-m-aminophenyl-3-methylpyrazolone, acetoacetic p-aminoanilide, and 2:3-hydroxynaphthoic p-aminoanilide are described. C. HOLLINS.

Production of nitrosodiazo solutions. I. G. FARBENIND. A.-G. (B.P. 307,890, 15.3.29. Ger., 15.3.28). —A solid, stabilised diazo compound made from an aminodiphenylamine is mixed with sodium nitrite and, if necessary, an acid or acid salt. On dissolution in water the nitrosodiazodiphenylamine is formed at once. Examples are diphenylamine-*p*-diazonium hydrogen sulphate or zinc chloride double salt; 4'-methoxydiphenylamine-4-diazonium hydrogen sulphate or benzenesulphonate. C. HOLLINS.

Preparation of N-substituted cyanoformarylamides. DEUTS. GOLD- U. SILBER-SCHIEDEANSTALT, VORM. ROESSLER (B.P. 306,450, 5.2.29. Ger., 20.2.28).— Hydrogen cyanide is added to a pyridine solution of a "urea chloride," ArNR·COCl, at 50—80°, to give cyanoformarylamides ("urea cyanides"). Examples are products from hydrogen cyanide and the carbamyl chlorides prepared by the phosgenation of ethylaniline (m.p. 51°, b.p. 156—158°/16 mm.), methylaniline (m.p. 64—65°, b.p. 154—156°), phenylglycine nitrile (m.p. 143°), p-bromomethylaniline (m.p. 95°), tetrahydroquinoline (m.p. 74°), diphenylamine (m.p. 132°), ethyl- $\alpha$ -naphthylamine (m.p. 64°), ethyl- $\beta$ -naphthylamine (m.p. 104°), and tetrahydro- $\beta$ -naphthaquinoline (m.p. 134°). C. HOLLINS.

Manufacture of products of the anthracene series [intermediates and vat dyes]. I. G. FARBENIND. A.-G. (B.P. 303,375, 31.12.28. Ger., 30.12.27).—o-α-Naphthoylbenzoic acid is converted by sodium aluminium chloride at 100° into a compound (probably having annexed formula), m.p. 177°, which yields a violet vat dye when fused with alkali at 230—260°. C. HOLLINS.

Manufacture of derivatives of the anthracene series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,128, 26.9. and 1.10.28. Cf. B.P. 324,661; B., 1930, 452).— $\alpha$ -Naphthaquinone derivatives hydrogenated in the benzene nucleus are condensed with  $\alpha\gamma$ -butadiene or its homologues to give hydrogenated anthracenes, which may be converted by the method of B.P. 320,375 (B., 1930, 233) into anthraquinones. The hydrogenated naphthaquinone from benzoquinone and  $\alpha\gamma$ -butadiene is heated with  $\alpha\gamma$ -butadiene or its  $\beta\gamma$ -dimethyl derivative at 60—100° under pressure; the conversion of the product from  $\alpha\gamma$ -butadiene into 9:10-dihydroxy-1:4:5:8-tetrahydroanthracene is described. C. HOLLINS.

Manufacture of leuco-indigo. IMPERIAL CHEM. INDUSTRIES, LTD., and K. H. SAUNDERS (B.P. 328,104, 8.4.29).—An alkaline indigo paste is passed continuously with hydrogen under pressure over a nickel catalyst on a rigid support at 120—140°.

C. HOLLINS. Production of primary [aliphatic and cyclic] amines. W. REPPE, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,762,742, 10.6.30. Appl., 11.2.27. Ger., 9.2.26). —See B.P. 265,960; B., 1928, 360.

Introduction of an aldehydic group into heterocyclic nitrogen compounds. G. KALISCHER, H. SCHEYER, and K. KELLER, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,763,557, 10.6.26. Appl., 30.1.28. Ger., 2.2.27).—See B.P. 311,208; B., 1929, 747.

Manufacture of condensation products from hydroxybenzenes and hydroaromatic ring-ketones. E. KORTEN, ASST. to GEN. ANILINE WORKS, INC. (U.S.P. 1,760,758, 27.5.30. Appl., 5.1.28. Ger., 20.1.27).— See B.P. 310,825; B., 1929, 747.

Preparation of trihalogenobenzene sulphochloride. R. HERZ, E. RUNNE, and E. ALBRECHT, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,763,556, 10.6.30. Appl., 15.3.28. Ger., 18.3.27).—See B.P. 287,178; B., 1929, 235.

Preparation of chlorine-substitution products of 4-nitro-1:3-dimethylbenzene [chlorinated 4nitro-m-xylene]. E. HOFFA and E. THOMA, ASSIS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,762,018, 3.6.30. Appl., 21.9.27. Ger., 11.10.26).—See B.P. 298,761; B., 1928, 225.

Preparation of condensation products of the naphthastyril series. G. KRÄNZLEIN, M. HEYSE, and P. OCHWAT, ASSIS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,762,021, 3.6.30. Appl., 21.11.27. Ger., 30.11.26).—See B.P. 289,692; B., 1928, 475.

Preparation of arylides of 2-hydroxy-3-naphthoic acid. E. B. HIGGINS, ASST. to BRIT. SYNTHETICS, LTD. (U.S.P. 1,762,474, 10.6.30. Appl., 26.11.26. U.K., 28.12.25).—See B.P. 262,958; B., 1927, 102.

Separation of hydrocarbons (U.S.P. 1,746,197). Refining of benzene (B.P. 307,935 and 330,045).— See II. Table salt (B.P. 312,088).—See VII. Emulsion (B.P. 329,266).—See XII.

# IV.—DYESTUFFS.

#### PATENTS.

**Preparations useful for production of dyes.** O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 328,383, 12.3.29).—Dyeing preparations of the Rapid Fast series containing nitrosamine alkali salts and diacetoacetic tolidide etc. are rendered more stable to long storage by addition of feebly acid substances (sodium hydrogen carbonate) and a water-binding salt (sodium acetate, anhydrous sodium sulphate).

C. HOLLINS.

Manufacture of vat dye preparations. J. R. GEIGY A.-G. (B.P. 308,730, 26.3.29. Ger., 27.3.28).—

In solid preparations containing vat dye, hyposulphite, and alkali, the alkali is added in the form of its compound with a polyhydric alcohol, or an aliphatic or aromatic hydroxy-acid, *e.g.*, the sodium compound of glycol, of sodium glycollate, or of sodium salicylate. Alkali carbohydrate compounds are not included.

C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 327,967, 12.1.29).—The acridone from 2:4dibromo-1-aminoanthraquinone and anthranilic acid is halogenated in the benzene nucleus by treatment with halogenating agents in the usual way; or 2:4-dibromo-1-aminoanthraquinone is condensed with 3:5-dichloroanthranilic acid and the product cyclised. Blue-green vat dyes of good tinctorial power are obtained.

#### C. HOLLINS.

Manufacture of vat dyes of the indigo series. I. G. FARBENIND. A.-G. (B.P. 305,592, 7.2.29. Ger., 7.2.28).—5: 5'-Dihalogeno-4: 4': 7: 7'-tetramethylindigos, obtained from p-3-xylidine (with halogenation of the tetramethylindigo) or its 6-halogeno-derivatives containing hydrogen or a carboxyl group in position 2, give pure blue shades fast to washing, boiling, and chlorine. C. HOLLINS.

Manufacture of polymethine dyes. R. KUHN and A. WINTERSTEIN (B.P. 328,357, 13.2.29).—Formic acid or a formate is condensed with a cyclic ammonium organic salt derived from a heterocyclic compound containing a reactive methyl group, or with the corresponding methylene ( $\psi$ -) base. Examples are: formic acid with  $\beta$ -naphthaquinaldine methiodide, 2:3:3-trimethylindoline methiodide, 2-methylbenzthiazoline methiodide, etc., or the corresponding  $\psi$ -bases. The method is applicable where other processes fail. C. HOLLINS.

Preparation of 2:3-benzocarbazole-1:4-quinones [vat dyes of the  $\alpha$ -naphthaquinone series]. H. T. BUCHERER, ASST. to GEN. ANILINE WORKS, INC. (U.S.P. 1,763,216, 10.6.30. Appl., 26.6.28. Ger., 3.6.26).— See B.P. 317,928; B., 1929, 890.

Indigoid vat dyes. K. THIESS, T. MEISSNER, and C. J. MÜLLER, ASSIS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,760,797, 27.5.30. Appl., 17.12.27. Ger., 24.12.26).—See B.P. 282,805; B., 1928, 849.

Manufacture of azo dyes insoluble in water. L. LASKA and A. ZITSCHER, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,762,022, 3.6.30. Appl., 29.1.29. Ger., 1.2.28).—See B.P. 323,937; B., 1929, 366.

Anthracene dyes (B.P. 303,375).—See III.

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

Examination of the fine structure of vegetable fibres by means of concentrated sulphuric acid. A. P. SAKOSTSCHIKOV (Textilber., 1930, 11, 441—443).— The fine structure of vegetable fibres such as linen, hemp, ramie, and cotton is conveniently revealed by covering them with a few drops of sulphuric acid  $(d \ 1.83)$  on a glass microscope slide and then arresting the dissolution of the fibre by adding glycerin; greater clarity is obtained by then adding a few drops of a solution of zinc iodochloride and washing the stained fibres with lactic acid. After such treatment the fibres appear to consist of small units bounded by parallel planes at right angles to the fibre axis. Numerous photomicrographs are given. A. J. HALL.

Comparison of various brands of cellulose acetate silks. H. STADLINGER (Textilber., 1930, 11, 450-458).-A comprehensive comparison is made of the physical and dyeing properties of Setilose (Fabr. de Soie Artificielle de Tubize), Celanese, Fr. Rhodiaseta (Soc. pour la Fabr. de la Soie Rhodiaseta, Rousillon), G. Rhodiaseta (Deutsche Acetat-Kunstseide A.-G. "Rhodiaseta," Freiburg), Aceta (Aceta Ges.m.b.H., Berlin-Rummelsberg), Apex, and "Drya" (N.V. Nederlandsche Kunstzijdefabriek) silks. The acetyl contents (%) of the silks are: Setilose (53.2), Celanese (53.8), Fr. Rhodiaseta (54.4), G. Rhodiaseta (54.3), and Aceta (54.9), and the viscosities of their 2% solutions in acetone are 5.70, 5.80, 5.55, 5.75, and 8.85, respectively. The dry and wet tensile strengths, and also the dry tensile strength after immersion for 0.5 hr. in boiling water (g. per 1 denier at 20° and R.H. 60%), are, respectively: Setilose (1.22, 0.64, 1.08), Celanese (1.23, 0.64, 1.08), Fr. Rhodiaseta (1.25, 0.76, 1.12), G. Rhodiaseta (1.33, 0.78, 1.22), and Aceta (1.34, 0.90, 1.36). Under similar conditions the extensibilities were  $28 \cdot 2$ ,  $31 \cdot 7$ , and  $20 \cdot 5\%$ ;  $26 \cdot 0$ ,  $32 \cdot 2$ , and  $19 \cdot 4\%$ ; 27.7, 38.5, and 21.5%; 24.7, 32.8, and 19.9%; 26.4, 36.4, and 19.9%, respectively. Celanese and Fr. Rhodiaseta silks lost much lustre and became woolly, whereas Setilose and G. Rhodiaseta silks lost but little lustre and became only slightly woolly, and Aceta silk was almost unaffected when boiled in water for 0.5 hr. Setilose silk had the highest affinity for Cellit (cellulose acetate) dyes, Celanese having the next highest affinity, and the other silks considerably less affinity; Celanese showed an abnormally high affinity for Cellit Fast Blue A when dyed with a mixture of dyes containing this dye. Setilose and Aceta silks have the softest handle, and G. Rhodiaseta the harshest. The fluorescence of the silks in ultra-violet light was affected by means of lubricating oils added to the silk, but after extraction with ether Setilose and Aceta silks showed the strongest fluorescence (violet). The cross-sections of Apex, Enka-Drya, and La Soie de Clairoix are similar in that they are ribbon-like and free from indentations; those of Celanese, Aceta, Setilose, and Fr. and G. Rhodiaseta are circular and slightly indented.

A. J. HALL.

Properties of cellophan and transparite. F. LENZE amd L. METZ (Kunststoffe, 1929, 19, 217-219, 247-250, 271-277; Chem. Zentr., 1930, i, 773).—Chemically, the differences are small, values being: glycerol 13-14, water 7-8, ash 0.5, cellulose 77-78%, and acid coefficient 7-8. Physical properties were determined. A. A. ELDRIDGE.

Testing paper for bursting strength. B. SCHULZE (Papier-Fabr., 1930, 28, 267-271, 377-382).—The Schopper-Dalén bursting-strength tester is discussed, and experiments carried out with the instrument are described. It is found that a rate of application of load equal to 0.2 kg./sec. is most suitable for testing, and

that the arithmetic mean of ten tests gives accurate results. The effect of increasing the surface under test is to reduce the bursting pressure and increase the height of the diaphragm at the point of rupture. A testing area of 10 sq. cm. is recommended. The correlation of bursting strength with tensile strength has been investigated; it is considered impracticable to formulate any definite relationship between the two. The elongation under tensile strain may be approximated from the curvature of the sample at bursting point, and curves are given which show the relationship for different testing areas. T. T. Ports.

Xylose from cellulosic waste. HALL and others.— See XVII.

#### PATENTS.

Manufacture of yarns. C. LIÉNARD-FIÉVET (B.P. 304,606, 21.1.29. Fr., 21.1.28).—Short, waste fibres of wool, which have been lubricated with sulphonated castor oil, are bound together into a solid yarn by incorporation with vegetable fibres which have been given a cork-screw form by the lanification treatment described in B.P. 304,602 (B., 1930, 709.)

F. R. Ennos.

[Manufacture of] cloth-like fabric. C. F. BURGESS LABS., INC., Assees. of M. J. SHOEMAKER (B.P. 317,330, 12.8.29. U.S., 13.8.28).—A water-absorbent sheet of unwoven fibres of cellulose material (wood pulp) is held together by discontinuous films of water-absorbent material (regenerated cellulose), preferably in the form of parallel strips at right angles to one another on the two sides of the sheet, the fibres of which are securely anchored to the adjacent strips.

F. R. ENNOS.

Utilisation of waste wool. S. R. and E. R. TROTMAN, and WOLSEY, LTD. (B.P. 329,766, 4.4.29).—Waste wool is heated under pressure with an aqueous solution of a weakly alkaline salt (borax, sodium phosphate) to extract the protein in a form which is soluble in water, but which has undergone as little degradation as possible. F. R. ENNOS.

Manufacture of artificial silk etc. from viscose. DEUTS. ZELLSTOFF-TEXTILWERKE GES.M.B.H., Assees. of K. LEUCHS (G.P. 461,749, 13.1.21).—The precipitation bath comprises a sulphuric acid solution of sodium and aluminium sulphates, preferably in equimolecular ratio, the content of  $Al_2(SO_4)_3$ ,  $18H_2O$  being greater than 15%. A. R. POWELL.

Spinning of tubular bodies from cellulose solutions such as viscose. KALLE & Co. A.-G. (B.P. 306,851, 25.2.29. Ger., 23.2.28).—Viscose is spun through an annular nozzle open at both ends, and the tubular body formed is distended by introducing the precipitant in a strong jet into its interior by means of a pipe projecting into the nozzle. A core consisting of a bar carrying one or more rings and provided at its end with a triangular member, the width of which can be adjusted as desired, is caused to descend gradually into the tubular body, and precipitation is completed by passing the precipitant through the supporting rings.

F. R. ENNOS. Manufacture of non-fibrous cellulose flour. E. R. DARLING, Assr. to CORNSTALK PRODUCTS CO., INC. (U.S.P. 1,751,267, 18.3.30. Appl., 7.6.28).—Cottonseed hulls are heated in an oxidising solution for a predetermined time and at a predetermined temperature to oxidise the constituents other than cellulose, which are removed by washing with water and heating under pressure with caustic soda and sodium sulphite ; the product is finally washed, bleached, again washed, and dried. F. R. ENNOS.

Manufacture of cellulose acetate. U.S. INDUS-TRIAL ALCOHOL CO. (B.P. 329,718, 22.1.29. U.S., 24.2.28).—After acetylating cellulose in the presence of liquid sulphur dioxide as in B.P. 306,531 (B., 1930, 609), the ester produced is partially hydrolysed in the same medium to the required solubility by the addition of water, with or without acetic acid or alcohol. By abrupt liberation of the sulphur dioxide as gas the product is discharged from the reaction vessel in a light or fluffy form. F. R. ENNOS.

Utilisation of cellulose ester waste. R. SPELLING (B.P. 311,368, 2.3.29. Ger., 10.5.28).—After removal of their chemical coating, worn-out films etc. are dissolved in a suitable solvent and spun into thread, new spinning solution being added, if necessary, to bring the prepared solution to the desired viscosity. [Stat. ref.] F. R. ENNOS.

Apparatus and process for separating or extracting cellulose or paper pulp. E. SPENCER (U.S.P. 1,761,543-4, 3.6.30. Appl., 10.2.28. Brit. India, 28.5.27).—See B.P. 283,910; B., 1929, 13.

Cementitious product [from cellulosic material]. R. A. MARR, Assr. to RAMAR SYND., INC. (U.S.P. 1,762,481, 10.6.30. Appl., 12.1.24. Renewed 31.7.26).— See B.P. 266,168 ; B., 1927, 295.

Manufacture of cellulose derivatives. W. H. GLOVER and C. DIAMOND, Assr. to COURTAULDS, LTD. (U.S.P. 1,763,428, 10.6.30. Appl., 3.2.27. U.K., 10.4.26).—See B.P. 268,552; B., 1927, 473.

[Abrasive-covered] rollers for drawing, spinning, or otherwise treating fibrous materials. D. Mol. PROCTOR, and CARBORUNDUM Co., LITD. (B.P. 330,323, 16.3.29. Addn. to B.P. 275,762).

Materials in sheet form (B.P. 329,691). Centrifugal separator (U.S.P. 1,749,291).—See I. Esters of carbohydrates (B.P. 305,661).—See III. Laminated glass (B.P. 306,397).—See VIII. Insulating materials (B.P. 308,224).—See XI. Products from wool fat (B.P. 305,597). Emulsions (B.P. 329,266).—See XII. Impregnation of paper etc. with varnishes etc. (B.P. 329,066).—See XIII. Rubbered fabrics (U.S.P. 1,749,743). [Filaments from] chlorinated rubber (B.P. 328,818).—See XIV.

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

X-Ray study of mercerisation. G. VON SUSICH and W. W. WOLFF (Z. physikal. Chem., 1930, **B8**, 221–230: cf. A., 1929, 1354).—The process of mercerisation has been followed by means of X-ray photographs taken during the course of the reaction, and hydrated sodapulp has been detected as an intermediate compound. Diagrams of untreated ramie, hydrated and nonhydrated soda-pulp, and mercerised cellulose are given.

The X-ray diagram of the intermediate compound is identical with that obtained under similar conditions by Katz and Mark (A., 1925, ii, 660). Concentration and temperature conditions for mercerisation by lithium, sodium, or potassium hydroxides are tabulated.

F. L. USHER.

Submerged combustion. HAMMOND.--See II. Comparison of acetate silks. Stadlinger.-See V.

# PATENTS.

Dyeing of esters or ethers of cellulose or of its transformation products. I. G. FARBENIND. A.-G. (B.P. 328,308, 25.1.29. Addn. to B.P. 304,739; B., 1930, 506).—Cellulose acetate etc. is dyed with 4-amino-1:8-naphthalimides carrying as substituent in the imide group a hydroaromatic residue, *e.g.*, the *cyclohexyl* derivative, m.p. 240—242°. C. HOLLINS.

Dyeing of regenerated cellulose materials. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 328,706, 13.2.29).—Level shades on viscose are obtained by the use of disazo dyes from tetrazotised 1:5-naphthylenediamine coupled with (a) a phenol, naphthol, a phenolcarboxylic acid, a naphtholsulphonic acid, or an aminonaphtholsulphonic acid, and (b) another coupling component, J-acid and its derivatives being excluded in both cases. Preferably there is present one sulphonic group for each naphthalene residue. Examples are 1:5-naphthylenediamine with 1:8-aminonaphthol- 2:4-disulphonic acid and  $\beta$ -naphthol (blue), with salicylic acid and methyl  $\beta$ -naphthylamine-7-sulphonic acid (red), or with N.W.-acid and  $\gamma$ -acid (violet).

C. HOLLINS.

Dyeing of chrome leather with basic dyes. ORANIENBURGER CHEM. FABR. A.-G. (B.P. 304,608, 21.1.29. Ger., 19.1.28).—Chrome leather is dyed with basic dyes with the aid of highly sulphonated neutral fats, fatty acids, resin acids, naphthenic acids, petroleum distillates, brown-coal tar, or sulphonated condensation products, such as are described in B.P. 269,917, 275,267, 288,126, 308,280, 313,861, and 289,841 (B., 1927, 650; 1929, 292, 529, 727, 708; 1930, 276). C. HOLLINS.

Production of a wool finish on cotton goods. R. G. KNOWLAND and G. T. LOVERIDGE, Assrs. to NASHUA MANUFG. CO. (U.S.P. 1,751,089, 18.3.30. Appl., 11.5.26. Renewed 13.8.29).—Cotton goods are treated with caustic alkali of mercerising strength until the fibre has become permanently swollen, the material being allowed to shrink freely during the process. After removal of a considerable part of the alkali by washing, the goods are dried by ironing while still retaining a part of the caustic soda, which is finally washed out with water. F. R. ENNOS.

Conversion of textile vegetable fibres into threads having the texture of wool. F. G. MAILLARD (B.P. 329,792, 24.4.29).—Fibres of jute, hemp, etc., after mechanical treatment in a greasy bath, are spun into thread, doubled, and treated with a solution of alkaline carbonate at 60° followed by a solution of sodium sulphoricinoleate and alkaline carbonate at ordinary temperature in order to separate the ligneous constituents, which are then hydrolysed by means of caustic soda. The threads are finally bleached, finished, and rendered impermeable by electrolytic treatment in a bath of aluminium sulphoacetate. F. R. ENNOS.

Treatment of vegetable fibres to effect their lanification. C. LIÉNARD-FIÉVET (B.P. 304,602, 21.1.29. Fr., 20.1.28).—Vegetable fibres, which have been freed from pectic substances by steeping in a 1% sodium carbonate solution and then in an alkaline solution of vegetable oil ("Tetrapol"), are crimped or waved by treatment with a solution of sodium carbonate and Monopol soap. After washing, the fibres are neutralised, bleached by the action of sulphur dioxide and trioxide, and finally dried by hot air.

F. R. ENNOS. Production of lustre effect on textile fabrics [by calendering]. K. WESSEL, Assee. of A. BOSSHARD (B.P. 328,175, 8.8.29. Ger., 11.3.29).—Fabric is dried by passing over drying cylinders, the last two of which are electrically insulated and driven at a slightly slower rate than the remaining ones, so that the fabric becomes charged electrostatically by friction, and are then calendered between two jute rollers each heated to about 180° by contact with a gas-heated steel roller.

A. J. HALL.

Dressing of textile materials. ORANIENBURGER CHEM. FABR. A.-G. (B.P. 320,018, 30.9.29. Ger., 29.9.28).—The material is treated with an aqueous solution or emulsion of a drying oil, *e.g.*, linseed oil, wood oil, preferably boiled or mixed with a siccative, or of a varnish made therefrom. F. R. ENNOS.

Waterproofing of sailcloth and similar textiles. H. LANDMARK and W. NAGEL (Nor.P. 42,619, 4.6.25).— The material is impregnated with an emulsion of a drying or semi-drying oil (linseed oil, whale oil, etc.) in a solution containing chromium salts and glue, preferably together with asphalt, paraffin, pigments, cellulose, wood-pulp, or similar fillers or colouring materials. A. R. POWELL.

Increasing the lustre of goods made of animal hair or wool. E. BÖHM (U.S.P. 1,760,738, 27.5.30. Appl., 18.9.28. Austr., 1.12.26).—See B.P. 281,646; B., 1928, 520.

**Dyeing and like machines.** C. S. BEDFORD (B.P. 330,377, 8.5.29).

Salvaging cleaners' gasoline (U.S.P. 1,751,613).— See II. Wetting agents etc. (B.P. 300,574 and 328,675).—See III. Dyeing rubber (U.S.P. 1,742,757). Colouring rubber (B.P. 329,761).—See XIV.

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

Determination of water by distillation with tetrachloroethane. W. LEPPER (Z. Unters. Lebensm., 1930, 59, 79-81).—The use of tetrachloroethane as the distillation medium in the apparatus of Pritzker and Jungkunz (cf. A., 1929, 1029) is unsatisfactory owing to its corrosiveness, its sensitivity towards alkalis, and its tendency to split off hydrogen chloride in the presence of water. When the method was applied to the determination of the water content of sucrose, decomposition occurred resulting in very high values. For flour and starch-containing materials a liquid having British Chemical Abstracts-B.

a b.p. lower than that of tetrachloroethane is essential, and the apparatus of Spiehl and Striemann (cf. Z. angew. Chem., 1927, 40, 464), using benzene, toluene, and xylene mixtures, is recommended.

H. J. DOWDEN.

Synthetic preparation of ammonia under hyperpressures and production of the necessary hydrogen. G. CLAUDE (Z. angew. Chem. 1930, 43, 417-423). -A historical account of the author's work. He early reached the conclusion that hydrogen for ammonia synthesis could never be sufficiently freed from carbon monoxide by liquefaction of the latter at  $-190^{\circ}$ , its b.p. A liquefaction method of separation, using lower temperatures, was gradually developed which yielded hydrogen of 94% purity, but the process was tedious. The author's earlier experiments on ammonia synthesis were conducted with electrolytic hydrogen which was in France too expensive for commercial use, but in 1917 the dissolution process for the removal of carbon monoxide from water-gas made it possible to obtain hydrogen containing less than 0.2% CO. The complication of the dissolution process caused the author to return to liquefaction, and he solved the principal difficulty, viz., that of finding a lubricant capable of use below -200°, by employing liquid nitrogen. This yielded a hydrogen of 99% purity. The final success was the removal of the remaining 1% by catalytic reaction  $(CO + 3H_2 = CH_4 + H_2O)$ at 350°. In 1922 the Claude process was first successfully applied to coke-oven gas. In this case ethylene is obtained as a by-product and serves for the manufacture of ethyl alcohol, and the traces of carbon monoxide left in the hydrogen can be used for the synthesis of methyl alcohol. The advantages of the use of hyper-pressures are discussed. The thermal conditions are, however, entirely different from those of the Haber process. Whilst in the latter electric heating is often necessary, in the Claude process there is an excess of heat which must be removed. This is effected by cooling the catalyst with the reaction gases without the use of a heat exchanger. The total energy consumption of the Claude process as realised in existing plants is below 2.5 kw.-hrs. per kg. of anhydrous ammonia. C. IRWIN.

Types of common salt and its standardisation. I. K. EROSCHKIN (Ann. Inst. Anal. Phys. Chem., 1930, 4, 291-305).—The necessity of the adoption of standards for the common salt industry is urged.

R. TRUSZKOWSKI.

Mechanism of the azide-iodine test for thiosulphate and its quantitative application. H. BAINES (Phot. J., 1930, 70, 235-236).—The azide test (cf. Jelley and Clark, B., 1930, 55) depends on the formation, during the reaction between thiosulphate and iodine in very dilute solution, of a substance, other than tetrathionate, which is capable of giving a positive reaction with sodium azide. Attempts to identify this substance as an unstable sulphur compound, such as sulphoxylic acid, were not successful. A method is outlined for the application of this test to the determination of traces of thiosulphate. J. W. GLASSETT.

Water solubility of the phosphoric acid of monocalcium phosphate. W. Stollenwerk (Z. Pflanz. Düng., 1930, 17A, 42-47).—In concentrated solutions

monocalcium phosphate, both pure and in the form of superphosphate, undergoes decomposition with the formation of free phosphoric acid. The extent of the decomposition decreases with increasing dilution. (Cf. Schneider, Diss. Giessen, 1929.) A. G. POLLARD.

Rapid opening-up of calcium cyanamide. W. LEPPER (Z. anal. Chem., 1930, 80, 331—334).—The crude cyanamide (5 g.) is mixed in a 500-c.c. flask with copper sulphate (1 g.) and powdered potassium sulphate (10—20 g.), and 50 c.c. of concentrated sulphuric acid are added. The flask is heated for 15 min. by a small flame until the initial reaction is over, and the liquid then boiled for a further 15 min. The contents of the flask are then made up to 500 c.c. and an aliquot part of the solution is distilled with excess of sodium hydroxide in the usual way. Concordant results cannot be assured if less than 5 g. of the material be taken for analysis. H. F. HARWOOD.

Volumetric determination of citric acid in calcium citrate. G. ROMEO (Riv. Ital. Prof., 1929, 11, xxiii—xxvi; Chem. Zentr., 1930, i, 265).—The calcium citrate is converted into the potassium salt and treated with an excess of standard silver nitrate solution, the excess being titrated with thiocyanate. Oxalic, tartaric, hydrochloric, and phosphoric acids, but not calcium sulphate nor pectic substances, interfere. A. A. ELDRIDGE.

Antimony pentasulphide. E. SCHÜRMANN and W. Вöнм (Kautschuk, 1930, 6, 70-73, 91-94, 136-141).—After a review of earlier work on the methods of formation and the composition of antimony pentasulphide, an account is given of selected methods for the complete analysis of this raw material for the rubber industry, followed by a record of experiments attempting the production of pure pentasulphide in various ways, wet and dry. Commercial samples always contain more or less free sulphur, after the removal of which the sulphur content of the residue falls below that for the composition Sb<sub>2</sub>S<sub>5</sub>, and indicates a mixture of pentaand tri-sulphides. Pure pentasulphide probably has never yet been obtained. It is now found that by passing hydrogen sulphide into a carefully prepared solution of antimony pentachloride containing 12-15% of hydrogen chloride and conducting all operations, including drying, as expeditiously as possible, a product is obtained which, after the removal of the small proportion of free sulphur by purified carbon disulphide, is free from any reducing action on ammoniacal silver solution, and represents substantially pure antimony D. F. Twiss. pentasulphide.

Manufacture of sulphur chloride from pyrites, alkaline-earth sulphates, and refuse from gas purifiers. V. N. IFATIEV and V. V. VASILEVSKI (J. Appl. Chem., Russia, 1929, 2, 689—701).—When chlorine is passed through refuse from gas purifiers a mixture of sulphur mono- and di-chlorides (e.g., 58:42) is obtained; iron catalyses the formation of the latter, whilst decrease in the rate of passage of the gas or distillation in presence of excess of sulphur favours that of the former. Gaseous cyano-compounds are also formed. In the similar preparation (e.g., 89:11) from pyrites the maximum yield is obtained at 200° (based on the amount of pyrites reacting) or  $300^{\circ}$  (on the amount of pyrites present). At  $500^{\circ}$  activated carbon does not react with barium sulphate in absence of chlorine, and chlorine has no appreciable action on calcium sulphate in absence of carbon; by joint action on either sulphate, the metal chlorides are formed in yields 91% and 81% of the theoretical, respectively. Increase in the amount of carbon diminishes the yield of barium chloride to 76—85\%, whilst 27—30% of the sulphur is converted into the monochloride. CHEMICAL ABSTRACTS.

Welded joints and corrosion. KÜRSCHNER; GROSS.—See II.

## PATENTS.

Manufacture of phosphoric acid and hydrogen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,711, 15.2.29).-The formation of red phosphorus in a coherent form during the production of phosphorus acids from liquid phosphorus and water at elevated temperatures and pressures is prevented, e.g., by bringing the phosphorus introduced into the pressure-tight reaction vessel into immediate intimate contact with the water. Thus phosphorus and water may be continously or periodically introduced in small quantities into the vessel heated at such a temperature that interaction occurs in a short period of time. Alternatively, larger quantities of phosphorus and water may be introduced and finely dispersed and intermixed by stirring; or the phosphorus may be quickly heated to a temperature (300-400°) at which it reacts rapidly with water. It is also advantageous to supply the phosphorus in a state of fine subdivision, e.g., as a fine spray. S. K. TWEEDY.

Preparation of substitutes for table salt suitable for flavouring purposes. CHEM. PHARM. A.-G. (B.P. 312,088, 17.5.29. Ger., 19.5.28).—The flavour of sodium formate, lactate, acetate, or other organic salts or of potassium chloride is improved by addition of glutamic acid, sodium hydrogen glutamate, or mixtures of aminoacids obtained by hydrolysis of albumins, so that the  $p_{\rm H}$  value is moved towards the acid side (about + 5). The products have a purely saline taste. C. HOLLINS.

Production of alkali [sodium carbonate] from natural brine. R. CUMMINGS, ASST. to R. D. PIKE (U.S.P. 1,751,132, 18.3.30. Appl., 14.12.27).—The brine from Green River, Wyoming, is concentrated in saltingout evaporators until about 86% of the water has evaporated and about 44% of the total Na<sub>2</sub>O is precipitated and removed as sodium carbonate monohydrate; the mother-liquor, after cooling to  $30-35^{\circ}$ , is carbonated to precipitate most of the remaining sodium carbonate as the bicarbonate which, as well as the monohydrate, is calcined to yield soda ash. The final liquor is treated for the recovery of sodium chloride, bromine, etc.

#### L. A. COLES.

Working natural sodium sulphate deposits. G. F. ANDERSEN, ASST. to WELTHA PROCESS CORP. (U.S.P. 1,751,901, 25.3.30. Appl., 12.11.28).—In lakes containing water saturated with sodium sulphate, siltproof walls are built enclosing a portion of the lake; from this portion the silt is removed and the clear solution allowed to flow into it in such a manner as not to disturb the bottom. In due course long columnar-crystals of pure sodium sulphate grow up from the bottom, and may readily be collected free from entrained silt.

A. R. POWELL. **Precipitation of potassium cobaltinitrite.** "SACHTLEBEN" A.-G. F. BERGBAU U. CHEM. IND., and H. NITZE, Assees. of H. NITZE (G.P. 461,898, 5.5.27).— The precipitation is effected in a solution containing alkali hydrogen sulphate and a smaller proportion of acetic acid than that usually employed. A. R. POWELL.

Recovery of neutral ammonium sulphate from the mixture of crystals and liquor obtained in an ammonia sulphate saturator. C. STILL (B.P. 310,536, 25.4.29. Ger., 28.4.28).—An enclosed rotary plane or drum filter is described, adapted so that gases or air containing ammonia can be led over and/or through the layer of salt deposited on the surface of the filter for the purpose of neutralising the acid motherliquor adhering to the filtered salt. S. K. TWEEDY.

Mixtures of nitrate of ammonia and nitrate of lime. APPARELLS ET EVAPORATEURS KESTNER (B.P. 316,664, 11.7.29. Fr., 2.8.28).—Dry, non-deliquescent mixtures of the compounds are prepared by concentrating the calcium nitrate to about 82% before mixing it with the ammonium nitrate. L. A. COLES.

Manufacture of alkali, alkaline-earth, and ammonium nitrates from chlorides. E. E. NAEF (F.P. 638,551, 9.12.26).—The chlorides are heated at 50—80° with brown, fuming nitric acid preferably in the presence of manganese dioxide or sulphuric acid.

A. R. POWELL.

Obtaining alkaline-earth thiocyanates [from gas-purification liquors etc.]. R. R. FULTON, Assr. to KOPPERS Co. (U.S.P. 1,751,274, 18.3.30. Appl., 23.12.26).—The liquor is treated with sufficient calcium chloride to precipitate thiocyanates, thiosulphates, carbonates, and ferrocyanides as their calcium salts and the mixture is heated at 110—120° to decompose the calcium thiosulphate into calcium sulphite and sulphur; after removal of the precipitated material, the solution is concentrated and cooled to remove sodium chloride, treated with clarifying agents (lime, bleaching powder, etc.) to remove colloidal and coloured impurities, and finally concentrated to crystallise calcium thiocyanate. L. A. COLES.

Production of hypochlorite compositions. MATHIESON ALKALI WORKS, Assees. of (A-C) R. B. MACMULLIN and (A, C) M. C. TAYLOR (B.P. 317,437, 319,727, and 329,896, 12.7.29. U.S., [A] 16.8.28, [B] 27.9.28, and [c] 29.12.28).-(A) A new triple salt, Ca(OCl)2, NaOCl, NaCl, 12H2O, crystallising in hexagonal prisms and stable below 22°, is obtained by chlorinating a mixture of 37 pts. of calcium hydroxide, 40 pts. of sodium hydroxide, and 180 pts. of water at 0-16°, or by adding sodium chloride to a saturated solution of calcium hypochlorite below 16°. Agitation of the triple salt with a chlorinated milk of lime slurry in suitable proportions at 10° followed by heating at 16° causes the formation of a rigid mass of interlocking crystals of calcium hypochlorite, Ca(OCl)2,2H2O, and sodium chloride, which can be dried in vacuo to give a product containing 65-70% of calcium hypochlorite. (B) The triple salt is obtained by chlorinating a mixture of sodium and calcium hydroxides in a saturated solution of sodium chloride at  $10^\circ$ , separating the precipitated sodium chloride, adding more of the hydroxides, and again chlorinating at  $0^\circ$ . (c) The chlorinated lime slurry to which the sodium chloride is to be added is produced by a two-stage chlorination at (a) above and (b) below  $10^\circ$ , so that calcium hypochlorite dihydrate does not separate. A. R. POWELL.

Simultaneous production of precipitated dicalcium phosphate and nitrates. F. C. and F. PALAZZO (B.P. 316,605, 14.2.29. It., 1.8.28).-Finely-divided raw phosphate mixed to a paste with water is stirred at the ordinary temperature with mitric acid of 30-50% concentration; after removal of insoluble sludge, the solution is treated with sodium or potassium hydroxide or ammonia to precipitate dicalcium phosphate, which is removed, washed, and dried at 50° under reduced pressure. The solution is evaporated to yield a mixture of sodium or ammonium and calcium nitrates, or, where potassium hydroxide has been used for neutralisation, it is concentrated so that, on cooling, the potassium nitrate crystallises out and a residue of calcium nitrate is obtained on further evaporation. The liquors obtained by washing the insoluble sludge and the dicalcium phosphate are used for the production of nitric acid for use in the initial stage of the process.

#### L. A. COLES.

Separation of the constituents of mineral sili-S. W. SCOFIELD, Assee. of J. B. LA RUE (U.S.P. cates. 1,751,663, 25.3.30. Appl., 7.2.24).-Potash felspar ground to 35-mesh is calcined for 1-2 hrs. at 800-1050°, then ground to pass 200-mesh, and digested for 1-2 hrs. in an autoclave at 275-325° with 2.5-4 times its weight of a 45% solution of potassium hydroxide. The mixture, after cooling, is treated with 1.4 times its volume of water and carbon dioxide is passed through the liquor to convert all the potassium into potassium hydrogen carbonate and to precipitate aluminium hydroxide and gelatinous silica. The filtered solution is evaporated to recover part of the potassium as carbonate and the mother-liquor is diluted and treated with slaked lime to regenerate potassium hydroxide for use again in the process. A. R. POWELL.

Treatment of greensand or glauconite for rendering it an efficient water-softening agent. F. K. LINDSAY, ASST. to ARIZONA MINERALS CORP. (U.S.P. 1,750,847, 18.3.30. Appl., 19.2.25. Renewed 8.1.30). —Greensand etc. is stirred into a solution containing sodium silicate and a soluble salt (e.g., sodium aluminate) so that the particles are held in suspension in the slowly gelling mass; when gelation is complete the product is drained for 12 hrs. at the ordinary temperature, and then dried in a current of air at about 60°.

#### L. A. Coles.

Manufacture of decomposition products from iron sulphates. I. G. FARBENIND. A.-G. (B.P. 303,808, 9.1.29. Ger., 9.1.28).—The dehydration and decomposition is effected in the presence of, e.g., 10% of a solid decomposition product obtained by heating ferric sulphate, e.g., ferric oxide, which product usually acts as a catalyst and also diminishes the plasticity of the mass so that the heating can be effected in a rotary furnace. Small quantities of alkali or alkaline-earth oxide, hydroxide, or carbonate may also be added, *e.g.*, when adhering sulphuric acid is present. Should no catalytic action occur a catalyst which promotes the oxidation of sulphur dioxide to sulphur trioxide may be added. S. K. TWEEDY.

Separation of iron from solutions containing cobalt and rich in iron, such as those obtained by lixiviating roasted pyrites. H. E. POTTS. From ORKLA GRUBE-AKTIEBOLAG (B.P. 329,543, 18.6.29).— The ferrous iron, prior to precipitation, is oxidised to ferric iron by treatment with an alkali (sodium) chlorate. L. A. COLES.

Production of pure aluminium compounds suitable for the production of aluminium. W. BACHMANN (B.P. 307,345, 25.2.29. Austr., 5.3.28).-Clay is digested with nitric acid to obtain a neutral solution of aluminium nitrate which, after filtration, is treated with sodium fluoride to produce cryolite or aluminium fluoride. Either of these may be converted into sodium aluminate by digestion with sodium hydroxide, and aluminium hydroxide may be recovered from the aluminate by known means. Alternatively, the fluoride may be heated with silica to produce alumina and silicon fluoride; the latter is then caused to react with water and the hydrofluosilicic acid produced is mixed with the sodium nitrate solution previously obtained to regenerate nitric acid. The precipitated sodium fluosilicate is heated to produce sodium fluoride and silicon fluoride for use again in the process.

A. R. POWELL.

Conversion of cobalt and nickel carbonates into hydrated sesquioxides. I. G. FARBENIND. A.-G., Assees. of F. MEIDERT (G.P. 462,350, 22.4.27).—The carbonate is suspended in water and the suspension treated with an alkali hypochlorite solution containing no free alkali and at least 150 g. of active chlorine per litre. Alternatively, chlorine may be passed into the suspension and alkali hydroxide added from time to time in such a way that an excess is never present.

A. R. POWELL.

Manufacture of a readily soluble complex silver compound [thiosulphate]. REMEDIUM CHEMISCHES INSTITUT G.M.B.H. (G.P. 462,782, 14.11.24).—Silver nitrate (2 mols.) is added to a solution containing sodium chloride or bromide (2 mols.) and sodium thiosulphate (3 mols.) Addition of alcohol precipitates the compound Ag<sub>2</sub>S<sub>2</sub>O<sub>3</sub>,2Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>,2H<sub>2</sub>O. A. R. POWELL.

Intumescent compositions. A. E. WHITE. From UPSON Co. (B.P. 329,474, 16.4.29).—A mixture of "light magnesium carbonate," sodium silicate solution (d < 1.51), and sand, slag, shale, limestone, etc., ground so that 75% passes 30-mesh, is moulded, heated, e.g., by being passed through steam-heated platens, and treated with hot air at about 210°. L. A. COLES.

[Apparatus for absorption of oxides of nitrogen for] production of nitric acid. HERCULES POWDER Co., Assees. of J. H. SHAPLEIGH (B.P. 329,427, 9.3.29. U.S., 28.1.29).—High, relatively narrow towers constructed, *e.g.*, of chrome iron are packed with hollow glass cylinders each containing an irregular lump of quartz etc., are cooled by an external stream of water, and are provided at the top with a water inlet and a spent-gas outlet and at the bottom with a gas inlet and an acid outlet. L. A. COLES.

Purification of gases [e.g., nitrogen and hydrogen for ammonia synthesis]. Soc. D'ELECTROCHIM., D'ELECTROMÉTALL., ET DES ACIÈRIES ELECTR. D'UGINE (F.P. 638,520, 6.12.26).—The compressed gases are bubbled through molten sodium or, preferably, molten sodium-potassium alloy. A. R. POWELL.

Separation of phosphorus vapours from carbon monoxide. W. KYBER (G.P. 462,521, 19.7.25).—The gases evolved from the furnace in which phosphates are reduced with carbon are passed through a tower packed with porous earthenware and coke, through which water is allowed to trickle and in which the phosphorus condenses, leaving a gas of high calorific value for industrial use. The phosphorus is recovered from the tower packing by burning it in a current of air and collecting the phosphorus pentoxide in an electrical fume-precipitating plant. A. R. POWELL.

Production of chromates with simultaneous recovery of alumina. H. SPECKETER and G. HENSCHEL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,760,788, 27.5.30. Appl., 23.6.27. Ger., 1.7.26).—See B.P. 273,666; B., 1928, 191.

Manganese-compound recovery. N. A. LAURY, Assr. to J. C. WIARDA & Co. (U.S.P. 1,761,133, 3.6.30. Appl., 22.7.25).—See B.P. 284,098 ; B., 1928, 235.

Purification of pearl essence. J. PAISSEAU (U.S.P. 1,760,771, 27.5.30. Appl., 9.2.25. Fr., 3.10.24).— See B.P. 240,858; B., 1926, 665.

Absorption apparatus (U.S.P. 1,747,687).—See I. Welding flux (U.S.P. 1,750,301).—See X. Lithopone and zinc sulphide (G.P. 462,372).—See XIII. Fertilisers (B.P. 312,169, 329,326, 329,641, and 329,883). Dusting powders (U.S.P. 1,749,575). Manure from final liquors of potash industry (B.P. 329,495).— See XVI.

#### VIII.—GLASS; CERAMICS.

System lime-alumina-silica. E. BERL and F. LÖBLEIN (Arch. Wärmewirtsch., 1929, 10, 339-342; Chem. Zentr., 1930, i, 574).—The behaviour of a series of mixtures heated at and above 1600° was investigated. Mixtures of composition approaching that of aluminium silicate are regarded as refractory. Determinations of resistance to pressure were made. The softening temperature falls with increasing content of lime. The thermal conductivity is but slightly dependent on the chemical composition and changes little with rise of temperature. A. A. ELDRIDGE.

Constitution of silicate fusions. H. SALMANG (Glastech. Ber., 1929, 7, 277—279; Chem. Zentr., 1930, i, 423).—The scorifiability, viscosity, etc. of refractory materials are influenced by oxides in the fused mass in the following order: calcium, lead, potassium, sodium, magnesium, aluminium, zinc, silicon, boron.

A. A. ELDRIDGE.

Manufacture of ceramic structures. L. MELLERSH-JACKSON. From W. TAYLOR (B.P. 329,011, 5.2.29).— In the drying and firing of clay structures, *e.g.*, water closets, special supports are used which are shaped to correspond with the desired final shape of that portion of the piece in contact with the support. To facilitate uniform drying, the support may vary in thickness and may be so arranged that thin portions of the piece are in contact with thicker portions of the support.

F. SALT.

Hot-patching [of high-temperature retorts]. H. PARDOE (Trans. Ceram. Soc., 1930, 29, 87-92).-The methods and materials for closing leaks caused by thermal expansion in high-temperature retorts are outlined. The spraying of material of a fluxing nature on to the refractory walls is effective as a preventative. Hot-patching may be carried out by the "paddle" or "peel" method or by the aid of air-guns or detonator guns. A device capable of firing on to a definite target from outside the structure is briefly described. A suitable material for hot-patching contains 3 pts. of silica sand (200-mesh), 2 pts. of precalcined bauxite grog (10-mesh), 3 pts. of powdered plate glass (100-mesh), 2 pts. of powdered vegetable glue, and  $1\frac{1}{2}$  pts. of lime. This mixture has m.p. 1550°. F. SALT.

Behaviour of soluble salts during the burning of clays : the problem of effloresence. IV. Conditions for the formation of sulphate efflorescences in ceramic materials. A. SIMON and K. SCHMIDER (Z. angew. Chem., 1930, 43, 380-382; cf. B., 1930, 326).--Clay material containing quartz, or quartz material, was admixed with varying quantities of lime (containing magnesia) and heated at 1000° for 3 hrs. and then maintained at this temperature for 5 hrs. in air containing 0.5% or 2% SO<sub>2</sub>. For the same lime content the amount of sulphate formed rises with increasing sulphur dioxide concentration, the amount of sulphur dioxide absorbed being directly proportional to its concentration. Similarly, for the same sulphur dioxide concentration the quantity of this gas absorbed and of sulphate formed increases with the rise in lime content, but at a much lower rate than the latter. The temperature interval  $300-600^{\circ}$  is most favourable for the absorption of sulphur dioxide, maximum absorption probably occurring between 400° and 500°. In the firing of ceramic materials, therefore, the interval 300-600° should be passed as quickly as possible in order to avoid absorption of the sulphur dioxide produced by the combustion of the sulphur of the coal. No absorption occurs at 750° and above; the sulphate detected in the experiments carried out at 1000° is formed during the heating up to this temperature, and although the dissociation pressure of calcium sulphate in presence of silica is already exceeded at 750°, greater energies than are here present are necessary before decomposition occurs. The drying and preheating of ceramic material, therefore, should be completed as far as possible in absence of sulphur dioxide, whilst for sharp firing coal containing sulphur may be used without adverse effect. S. K. TWEEDY.

Microstructure of some West of England siliceous bricks. J. M. Coon (Trans. Ceram. Soc., 1930, 29, 125— 137).—Five kinds of bricks made, by similar manufacturing processes, from mixtures of material resulting from kaolinisation of granite were studied. The bricks were fired to a maximum temperature of 1300°. Data are presented on the inversion of quartz to tridymite and crystobalite, the formation of sillimanite or mullite by dissolution of silica in the glassy melt, and on slag formation on exposed surfaces. A number of photomicrographs are reproduced and described. F. SALT.

Decoration of glazed tiles. J. W. THOMPSON (Trans. Ceram. Soc., 1930, 29, 99—111).—Saturated solutions of salts commonly used in the manufacture of pottery were applied with a brush to tiles coated with a soft, lead majolica glaze. After crystallisation had taken place, the tiles were fired to about cone OSA. The effects of using unsaturated solutions, of adding colourless salts to simple solutions, and of mixing simple solutions were also studied. The results are tabulated. F. SALT.

#### PATENTS.

Production of glasses with changing colour effects. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H., Assees. of F. WEIDERT (B.P. 329,946, 29.10.29. Ger., 15.12.28). —Praseodymium and neodymium oxides in the ratio of from 3: 4 to 5:3 are added to the glass frit to produce glasses which exhibit colours varying from pale ruby-red to greyish-blue and brownish-green, according to the incidence of the light. A. R. POWELL.

**Production of heat- and chemical-resisting glass.** C. G. BROOKS (B.P. 328,432, 22.4.29).—A borosilicate glass suitable for cooking or chemical ware is made by melting a batch containing <70% SiO<sub>2</sub>, >6% Al<sub>2</sub>O<sub>3</sub>, and a moderate amount of boric oxide, together with at least three oxides each of mono- and bi-valent metals, the proportions being determined to give a very low thermal expansion. A preferred composition is : SiO<sub>2</sub> 68, Al<sub>2</sub>O<sub>3</sub> 7.5, B<sub>2</sub>O<sub>3</sub> 10, Na<sub>2</sub>O 3, K<sub>2</sub>O 4, Li<sub>2</sub>O 1.5, MgO 1, ZnO 2.5, BaO 1.5, CaO 1%. M. PARKIN.

Manufacture of [eye-protecting] glass. H. SEA-BROOK. From DEUTS, SPIEGELGLAS-A.-G. (B.P. 328,520, 2.8.29).—A glass, absorbing strongly in the infra-red and ultra-violet, besides considerably reducing the visible transmission, is produced by adding cobalt and iron oxides (at least 0.3% on the weight of finished glass) to a batch for producing a colourless glass, and melting the batch under reducing conditions (e.g., with carbon or stannous chloride). Addition of arsenic oxide or antimony oxide increases the absorption of the heat rays. Suitable proportions are Fe<sub>3</sub>O<sub>4</sub> 1—5%, CoO 0.3—1%, As<sub>2</sub>O<sub>3</sub> or Sb<sub>2</sub>O<sub>3</sub> 0.5—1.5% on the weight of glass. M. PARKIN.

Laminated sheet glass. BRIT. CELANESE, LTD. (B.P. 306,397, 6.2.29. U.S., 18.2.28).—Splinterless glass permeable to ultra-violet rays is produced from pure quartz glass, or glass having a high quartz content, and cellulose acetate, preferably by using the ester as a powder (cf. B.P. 282,723; B., 1928, 853) mixed with sufficient plasticiser to produce the sheet *in situ*, the whole being pressed under 300—500 lb./in.<sup>2</sup> at 50—80° (preferably 70°) until consolidated. Other cellulose esters or ethers may be substituted for the acetate. M. PARKIN.

Manufacture of yellow glazes. DEUTS. TON & STEINZEUG-WERKE A.-G., Assees. of P. SCHNEIDER (G.P. 462,284, 3.9.27).—Stoneware is coated with any ordinary loam glaze containing more than 30% of calcium carbonate and fired in a salt-glaze furnace.

A. R. POWELL.

[Refractory floating rings for use in] glass manufacture. R. E. THRELFALL (B.P. 329,468, 15.4.29).— Clay rings which float readily on glasses of relatively low density are prepared by enclosing a core of suitable material (asbestos, papier maché, cardboard) in the clay before firing, or by building the rings up from hollow sections which are joined together by clay or refractory cement. L. A. COLES.

Manufacture of refractory insulating material. METALLGES. A.-G. (B.P. 304,729, 24.1.29. Ger., 25.1.28). —Natural minerals rich in magnesium orthosilicate, or hydrosilicates, such as serpentine, capable of being converted into orthosilicates by preliminary heat treatment at 800—1000°, are crushed, graded, and used to prepare insulating materials, suitable for metallurgical furnaces, coke ovens, etc., that retain both their insulating and mechanical properties at high temperatures. The material together with binders (sawdust, cork waste, etc.) and magnesium nitrate or carbonate, aluminium nitrate, or other gas-producing substances which do not sensibly reduce the refractoriness of the product are moulded and fired at a temperature insufficient to cause fusion.

M. PARKIN.

Manufacture of refractory materials. L. MEL-LERSH-JACKSON. From HARTFORD-EMPIRE Co. (B.P. 329,803, 27.4.29).—Grog for the manufacture of refractory materials is prepared by compressing the raw materials, e.g., fireclay, with a moisture content of about 12%, into compact blocks under 200 lb./in.<sup>2</sup>, firing the blocks under the usual conditions, and breaking them down to the desired grade for incorporation in fireclay mixtures for the manufacture of bricks for glass-melting tanks. A. R. POWELL.

Porcelain articles and their manufacture. A. S. WATTS (B.P. 330,397, 1.6.29).—See U.S.P. 1,728,382; B., 1928, 919.

Artificial plagioclase compounds. F. SINGER (U.S.P. 1,759,919, 27.5.30. Appl., 9.12.27. Ger., 17.12.26). —See B.P. 282,402 ; B., 1928, 817.

Methods and apparatus for manufacture of tubes made of a fusible material which can be drawn out, in particular glass tubes. P. A. FAVRE (B.P. 304,664, 22.1.29. Belg., 23.1.28).

Mechanism for feeding molten glass. L. Mel-Lersh-Jackson. From Hartford-Empire Co. (B.P. 330,366, 27.4.29).

[Vacuum apparatus for manufacture of splinterless] sheet glass. J. JEFFRAY (B.P. 327,949, 12.11.28).

Apparatus for coating glass sheets. R. W. MUNRO, LTD., and J. F. WILSON (B.P. 330,290, 5.3.29).

#### IX.—BUILDING MATERIALS.

Significance of fineness and chemical composition in the water requirement of cement. H. KÜHL (Tonind.-Ztg., 1929, 53, 1509—1510; Chem. Zentr., 1930, i, 882).—The effect of fine grinding in diminishing the setting time was examined quantitatively.

A. A. ELDRIDGE.

Petroleum products. CARPENTER.-See II.

# PATENTS. another in another of

Rotary kiln for burning cement. KLÖCKNER-WERKE A.-G. ABT. MANNSTAEDTWERKE (G.P. 461,431, 7.1.25).—The furnace head is joined firmly to the cooler so that both rotate simultaneously, and compressed air is blown through the cooler to assist the chimney draught. A. R. POWELL.

Manufacture of a heat-insulating material from gypsum. J. and H. SCHEIDEMANDEL (G.P. 461,889, 9.6.25).—Hot milk of lime is added to hot sulphuric acid, whereby a vigorous action ensues and the water is removed by the heat evolved, leaving a fine porous mass of gypsum of d < 0.35. A. R. POWELL.

**Production of moulded articles.** BELL'S UNITED ASBESTOS CO., LTD., J. A. CANN, and E. R. HARRAP (B.P. 329,330, 16.11.28).—A mixture of basic and acid compounds which react to form difficultly soluble compounds is moulded under the influence of heat and pressure; *e.g.*, Portland cement, calcined magnesite, or lime may be mixed with citric, tartaric, oxalic, tannic, lactic, or boric acid. Citric or oxalic acid is particularly suitable for Portland cement, and boric or oxalic acid is preferred for magnesite. (Cf. B.P. 326,825; B., 1930, 511.)

C. A. KING.

Manufacture of moulded or rolled articles. W. H. BOORNE (B.P. 329,382, 19.2.29).—Tiles, slabs, etc. may be moulded by hot-pressing a mixture composed of an acid binder, *e.g.*, resin (or pitch) and clay, a basic compound, *e.g.*, lime, a filler, *e.g.*, sawdust or sand, and 2—8% of water. C. A. KING.

Manufacture of blocks, bricks, slabs, etc. for the construction of roads, buildings, etc. A. F. BERRY (B.P. 329,291, 9.2.29).—A mixture composed of a hard base material, comminuted cement, a metal or alloy powder, and a chemical which will react with the other constituents with the generation of gas to cause the brick to be porous is moulded and allowed to harden. Such a mixture may contain broken vitreous clayware, Portland cement treated with a 10% solution of sodium thiosulphate, and a ferrosilicon alloy (Stalloy).

#### C. A. KING.

Manufacture of materials for use in the construction of roads, roofs, etc. SOUTH METROPOLITAN GAS Co., H. PICKARD, and C. M. C. HUGHES (B.P. 329,569, 23.7.29).—A binder (15-18%), made by mixing coal with coal tar at 250—300°, so that the mixture has m.p. 115-122° and a penetration of 1—5, is mixed with stone dust (26-30%) and hard chippings (55-58%). The warmed mass may be pressed into blocks or spread over a road foundation. C. A. KING.

Preservation of wood. GRUBENHOLZIMPRÄGNIER-UNG. GES.M.B.H. (B.P. 314,380, 18.6.29. Ger., 26.6.28). —Wood is saturated with a solution under pressure, the solution is drained, and the container evacuated so that only the cell walls are impregnated. A second suitable solution is then forced into the cells and an insoluble protective compound is formed by interaction between the two solutions. The liquids are employed in the form of their aqueous solutions; for the first, zinc, mercury, or copper salts may be used: for the second, alkali fluorides, arsenates, or arsenites. C. A. KING. Immunising agents for wood, grain, or the like. I. G. FARBENIND. A.-G. (B.P. 304,741, 25.1.29. Ger., 25.1.28).—An *o*-dihydroxybenzene is treated with a solution of arsenious oxide in hydrochloric acid

to give products of the type  $C_6H_4 <_O^O > AsCl$ . These and

the corresponding hydroxides are used, alone or with other fungicides, for protecting wood, grain, etc. against plant pests. C. HOLLINS.

Grinding of cement materials etc. C. PONTOP-PIDAN, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,762,241, 10.6.30. Appl., 30.12.27. U.K., 8.10.27).—See B.P. 283,091; B., 1928, 194.

Building slabs for walls, floors, pavings, etc. R. G. Ross and F. A. WILLIAMS (B.P. 330,418, 25.6.29).

[Bull-nose] tiles. J. B. BLAKEY (B.P. 330,383, 16.5.29).

Roof coverings. S. FACDOUELLE (B.P. 330,340, 5.4.29).

Manufacture of [felt-base] floor coverings [from waste]. J. LUNAN (B.P. 330,384, 16.5.29).

Building block (B.P. 329,063).—See I. Insulating masses (G.P. 461,493).—See XI. Paint for cement (U.S.P. 1,749,923). Impregnation of wood etc. with varnishes etc. (B.P. 329,066).—See XIII.

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

Nitrogen in technical iron. W. KÖSTER (Arch. Eisenhüttenw., 1929-1930, 3, 637-638; Stahl u. Eisen, 1930, 50, 629-631).-The solid solubility of nitrogen, as iron nitride, in a-iron is 0.001% N at 20°. 0.005% N at 200°, 0.01% N at 300°, and 0.02% N at 400°. Relatively slow cooling of mild steel containing nitrogen from below the A1 point does not result in the attainment of a state of equilibrium at room temperature as, by ageing at temperatures up to 100°, considerable changes occur in the magnetic and mechanical properties. After 12 hrs. at 65° the coercivity begins to rise, and at 100° it increases by 3.2 gauss for every 0.01% N precipitated over a period of 14 days. The conductivity also increases proportionally with the amount of nitride precipitated. The reaction can readily be followed in micro-sections etched with Fry's copper reagent, precipitation of nitride being accompanied by the appearance of minute black dots and needles in the ferrite crystals. These needles become coarser and more numerous with rise of ageing temperature, reaching a maximum at 110°, and then decreasing in size and number as the temperature is raised to 250°. The separation of nitride causes a slight increase in the density and torsion value, a slight decrease in the tensile strength, and a considerable increase in the rate of dissolution in acids. The differences in the properties of Thomas steel and open-hearth steel are ascribed to their different nitrogen contents. On ageing a nitrogenous mild steel which has been quenched from just below the Al point, maxima occur in the hardness curve at 150° and at 300° showing that, although the separation of excess nitride from its supersaturated solid solution takes place independently of the precipitation of carbide to  $p_{\rm H} 2.5$  is added; this is followed by addition of calcium chloride, filtration, and electrolysis in a cement cell having graphite anodes and a rotating-disc cathode. CHEMICAL ABSTRACTS.

Production of electrolytic iron printing plates. C. T. THOMAS and W. BLUM (Amer. Electrochem. Soc., May, 1930. Advance copy. 13 pp.).-An investigation has been made of the best conditions for the electrodeposition of iron from a bath of the Fischer-Langbein type to form a backing for nickel electrotypes. From a bath which was 3N with respect to ferrous chloride and 6N to calcium chloride the most satisfactory deposits were obtained at about 90°, using a cathodic current density of 5-7 amp./dm.2, provided that the bath was moderately agitated. Mechanical agitation produced by motion of the cathodes themselves is preferred. Air agitation leads to somewhat brittle deposits and lowers the current efficiency. The calcium chloride concentration can be reduced to 2-4N, but the acidity of the bath is important and should correspond to a free hydrochloric acid concentration of about 0.01N. Hourly additions of acid should be made to make up for loss due to chemical action on the anodes. The anodes may be of Armco iron, but since the impurities lead to roughening of the cathode deposits when the thickness of the latter exceeds about 1 mm., it was found necessary to enclose each anode in a rectangular alundum pot. The possibility of avoiding this need for using a diaphragm is being examined. The electrolyte is contained in a rubber-lined steel tank with a thin inner lining of cypress wood to protect the rubber against impact or abrasion. Methods of analysis for control of the bath composition are given. The deposited iron has a tensile strength of about 4000 kg./cm.<sup>2</sup> and an elongation of about 20%. The finished plates are chromium-plated. H. J. T. ELLINGHAM,

Hydrometallurgy of copper at the Bagdad property. C. T. BAROCH (Amer. Electrochem. Soc., May, 1930. Advance copy. 25 pp.).—A pilot plant has been operated at Bagdad, Arizona, for the extraction of copper from low-grade sulphide ores by electrolysis of the liquors obtained by the acid leaching of roasted concentrates. A three-stage roasting process has been developed which renders iron practically insoluble, whilst about 90% of the copper can be extracted. The important point is that between the first and second stages the temperature must not be raised above 455° until all sulphide sulphur has been oxidised. The calcine carries about 30% of acid-soluble copper, including about 8% which is water-soluble. A flow sheet of the leaching cycle is given. The filtered leach liquor is mixed with spent electrolyte and the resulting solution, which contains about 50 g. of copper and 25 g. of free sulphuric acid per litre, is electrolysed at 2.1 amp./dm.<sup>2</sup> The cells are of redwood lined with lead, each containing nine copper starting sheets and 10 cast antimonial lead anodes. The cells are arranged in groups of 16, each group comprising 4 cascade units. Cathodes are drawn after 14 days. The copper content of the electrolyte is reduced to about 20 g./litre. A portion of the spent electrolyte is diverted from the cycle to prevent undue accumulation of impurities, and after plating out the copper in special stripping cells it is

discarded. The cathode deposited in the main cells is 99.98% pure, and the energy consumption is 2.56 kw.-hr./kg. of copper. The concentration of precious metals in the ore is too small to warrant recovery.

## H. J. T. ELLINGHAM.

Electrolytic zinc plant of the Evans-Wallower Co. at East St. Louis, Ill. U. C. TAINTON and D. Bosqui (Amer. Electrochem. Soc., May, 1930. Advance copy. 10 pp.) .- A plant for the electrolytic extraction of zinc from Joplin ores, which were formerly treated entirely by the retort process, is now operated at this company's works at Illinois. Flotation concentrate containing 19-58% Zn is dried and roasted, the roaster gases which contain at least 5% SO2 passing to the neighbouring sulphuric acid plant of the Monsanto Chemical Co. The calcine is separated into a magnetic (ferrite) portion and a non-magnetic (oxide) portion. The ferrite portion is first agitated with the spent electrolyte, and, after adding manganese dioxide to oxidise all dissolved iron to the ferric state, the oxide portion is added until the acid is neutralised and all the iron precipitated. The residue is shipped to the lead smelter. The liquor is filtered, treated with zinc dust to precipitate impurities, and refiltered. The precipitate is agitated with acid, giving a solution from which cadmium is electrodeposited and a residue which goes to the copper smelter. The purified leach liquor passes to the zinc-extraction room, which contains 190 cells in series, each containing 12 aluminium cathodes and 24 grid-shaped anodes composed of lead alloyed with 1% of silver. The cathodic current density is 100 amp./ft.<sup>2</sup> (10.7 amp./dm.<sup>2</sup>). Cathodes are stripped every 12-16 hrs. and the zinc is melted and cast : it is guaranteed 99.99% pure. Manganese dioxide is formed at the anodes and settles on the bottom of the cells, whence it is periodically removed, washed, dried, and marketed. Steam-generated power supplied from a neighbouring station is used. The daily capacity of the plant is 50 tons of zinc and 450 kg. of cadmium. H. J. T. ELLINGHAM.

Germanium in relation to electrolytic zinc production. U. C. TAINTON and E. T. CLAYTON (Amer. Electrochem. Soc., May, 1930. Advance copy. 8 pp.).—In the electrolysis of zinc sulphate solutions derived from the leaching of roasted Joplin concentrate (cf. preceding abstract) violent fluctuations of current efficiency occurred, zinc deposits showed a great tendency to redissolve, and sometimes no zinc was deposited at all. The ordinary impurities such as antimony, cobalt, nickel, and arsenic, which are known to produce such effects, had been removed, so that the influence of some other element in the solution was suspected. This proved to be germanium, which is found to cause serious loss in efficiency at concentrations as low as 1 mg./litre. Experiments showed that treatment of the solution with precipitated ferric hydroxide removed the germanium. Normally the ores contain sufficient iron to prevent this element entering the solution, but some of the Joplin ores are very low in iron and apparently high in germanium. The trouble was remedied by starting with a mixture of concentrates containing a sufficient total proportion of iron to effect complete removal of germanium. H. J. T. ELLINGHAM.

Electrodeposition of zinc on aluminium and its alloys. H. C. COCKS (J. Electroplaters' Dep. Tech. Soc., 1930, 5, 83—90).—An account is given of the influence of  $p_{\rm H}$ , addenda, and current density on the nature of the deposits from zinc sulphate and cyanide baths. With sulphate baths gum arabic (1 g. per litre), and sulphuric acid (to  $p_{\rm H}$  4) as addenda were preferred, with 10—30 amp. per sq. ft. In cyanide baths the concentrations of cyanide and hydroxyl ions should be low. Sulphate baths are preferred, except when irregularly shaped articles are being plated.

#### CHEMICAL ABSTRACTS.

Addition agents in bismuth electrolytes. E. F. KERN and T. R. JONES (Amer. Electrochem. Soc., May, 1930. Advance copy. 19 pp.).—The suitability of fluosilicate, fluoborate, cresolsulphonate, perchlorate, 1930. fluoride, sulphate, and chloride electrolytes in the electrolytic refining of bismuth has been examined. The most satisfactory was a chloride electrolyte containing 70 g. of bismuth, 50 g. of hydrochloric acid, and either 100 g. of sodium or calcium chloride or 65 g. of magnesium chloride per litre. The solubility of bismuth chloride in the hydrochloric acid solution is notably raised by the presence of these high concentrations of alkali or alkaline-earth chlorides. The character of the cathode deposit is improved by the addition of 0.1g. of resorcinol, pyrogallol, or benzoic acid per litre. The current density employed was 1 amp./dm.2 If the anodes contain lead, sulphuric acid may be added to the electrolyte (except when it contains calcium chloride), and as much as 20 g./litre may be added without altering the nature of the cathode deposit.

#### H. J. T. ELLINGHAM.

Oven-furnace installation. WALTER.—See I. Submerged combustion. HAMMOND. Electric welding. KÜRSCHNER. Corrosion at welded joints. GROSS.— See II. Hot-patching of retorts. PARDOE.—See VIII. Rubber linings. COURBIER and BOUSSAND.— See XIV.

#### PATENTS.

Moulds for the manufacture of castings from metals or alloys. J. Y. JOHNSON. From I. G. FARB-ENIND. A.-G. (B.P. 330,018, 11.3.29).—The moulds are made from, or lined with, sheets made from a pure metal powder compressed and sintered to a porous mass permeable to gases. The metal powder is preferably obtained by the thermal decomposition of metal carbonyls. A. R. POWELL.

Production of cast iron containing little gas or oxides. C. BRACKELSBERG (B.P. 306,141, 12.2.29. Ger., 16.2.28).—The charge is smelted in a rotary furnace fired by a coal-dust flame produced directly in the furnace to avoid the formation of pointed flames which cause local overheating. A. R. POWELL.

Compound for filling recesses in metal castings and the like. Z. M. MARR (U.S.P. 1,749,541, 4.3.30. Appl., 16.4.28).—The material comprises a molten mixture of 7.5 pts. by vol. of sulphur, 1 pt. of graphite, and 1 pt. of antimony. A. R. POWELL.

Preparation of [an iron] aluminothermic mixture. R. W. Hyde (U.S.P. 1,751,550, 25.3.30. Appl., 29.9.28).—A mixture of iron oxide or iron scale is mixed with a solid carbonaceous material, e.g., coal or coke dust or sawdust, moistened with water, and blast-roasted in a thin layer to convert the greater part of the iron into granular, porous magnetite. The mass is then crushed through 14—20-mesh and mixed with the requisite amount of granular aluminium of the same degree of fineness. The mass reacts rapidly with the evolution of intense heat when fired with the usual primer. A. R. POWELL.

[Iron] thermit mixture. J. H. DEPPELER (U.S.P. 1,750,162, 11.3.30. Appl., 8.9.28).—A mixture of iron oxide and aluminium with 15% of ground slag from a previous thermit operation is claimed.

A. R. POWELL.

Welding flux [for iron]. R. R. FULTON, ASST. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,750,301, 11.3.30. Appl., 16.9.26).—The flux comprises a mixture of 10 pts. of an alloy of 92% Cu, 7% Si, and 1% Fe, 50 pts. of ammonium borate, 6 pts. of manganese, and 24 pts. of 50% ferrosilicon, all ground to pass 20-mesh. A. R. POWELL.

Soldering [and brazing] flux. C. D. LAWHON, Assr. to FULTON SYLPHON CO. (U.S.P. 1,748,180, 25.2.30. Appl., 3.6.29).—Aniline hydrochloride is employed, either as such or in solution (e.g., in *iso*propyl alcohol). It is adapted for use with soft solder. S. K. TWEEDY.

Increasing the elongation limit and tensile strength of low-carbon steels. VEREIN. STAHL-WERKE A.-G. (B.P. 300,553, 12.11.28. Ger., 14.11.27).— The steel is annealed above 900° and cooled slowly, it is then reheated at a temperature below the Arl point, *e.g.*, at 650°, quenched, and aged for some weeks at the ordinary temperature or for a short time at not above 250°. [Stat. ref.] A. R. POWELL.

Decarburisation of ferrochromium containing carbon. F. KRUPP A.-G., Assees. of B. STRAUSS and P. KLINGER (G.P. 461,945, 12.11.26).—Finely-powdered ferrochromium is treated at 1200° with a current of atomic hydrogen prepared by passing hydrogen through a high-tension arc or by subjecting it to a glow discharge under reduced pressure. A. R. POWELL.

[Copper-chromium] alloy steels. VEREIN. STAHL-WERKE A.-G. (B.P. 307,492, 27.2.29. Ger., 10.3.28).— A structural steel contains less than 0.2% C, less than 2% (Cr + Cu), and up to 1% Ni, W, Mo, Ti, and/or V. The preferred copper and chromium contents are 0.5— 1.5% and 0.25—0.5%, respectively. A. R. POWELL.

Manufacture of alloy [tungsten] steels. A. D. SHANKLAND, ASST. to BETHLEHEM STEEL Co. (U.S.P. 1,749,678, 4.3.30. Appl., 19.5.26).—A plastic, impervious mass of partly fused pig iron and steel scrap is produced in an acid open-hearth furnace and on this mass is placed a mixture of scale, containing tungsten trioxide equivalent to 10% W, and ferrosilicon, containing sufficient silicon to reduce the tungsten trioxide. The whole is covered with a charge of scrap steel and the mixture is heated until fusion occurs. The carbon and silicon contents are then adjusted to the desired values by the addition of iron ore, complete deoxidation being finally effected by means of ferromanganese. A. R. POWELL.

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

Production of titanium steel. W. and H. MATHE-SIUS (B.P. 329,705, 25.2.29).—Steels containing not more than 1% C and 0.8% Ti with small additions of silicon, manganese, chromium, and other metals which improve the properties are claimed, a preferred composition being 0.1% C, 0.2% Ti, 0.5% Si, 0.5% Mn, and 0.5% Cr. The steel is produced by deoxidising a bath containing less than 0.1% C by the addition of carbon-free silicides of ferromanganese, ferrochromium, or ferromanganese– chromium having such a carbon content that 0.1% C remains in the bath ; the requisite titanium is then added as carbon-free ferrotitanium. A. R. POWELL.

Elimination of silicon in ferrophosphorus. H. E. WHITE, ASST. to FEDERAL PHOSPHORUS CO. (U.S.P. 1,751,783, 25.3.30. Appl., 4.9.25. Renewed 11.1.30).— Phosphate rock is smelted with silicon and scrap iron or with a high-grade ferrosilicon, using a 10% excess of phosphate to ensure absence of silicon in the resulting ferrophosphorus. A. R. POWELL.

Production of a high lustre on articles made of stellite alloys. VEREIN. STAHLWERKE A.-G. (G.P. 461,866, 22.10.27).—The articles are first pickled in a strong acid to remove oxide scale, then in a weak acid (preferably organic) which does not dissolve the alloy nor appreciably corrode it. A. R. POWELL.

Two-step sulphatising roast [for copper-zinclead sulphide ores. F. A. BRINKER, Assr. to J. P. RUTH, JUN. (U.S.P. 1,749,125, 4.3.30. Appl., 21.10.26). -The ore is passed downwards through a multiplehearth roasting furnace through which air is passed in such a quantity and under such conditions countercurrent to the ore stream that the temperature in the lowest, hottest hearth never exceeds 550°. In this operation the iron sulphide minerals are oxidised to a highly-active ferric oxide and part of the copper and lead minerals are desulphurised, but the blende remains unattacked. The discharge from this furnace together with the sulphur dioxide gas mixture therefrom and more air are passed downwards through a rotary tubefurnace maintained at 600-700°, whereby the zinc sulphide and the copper and lead compounds present are converted into sulphates by the sulphur trioxide formed by the catalytic oxidation of sulphur dioxide in the presence of active ferric oxide. This procedure avoids the formation of zinc ferrite. A. R. POWELL.

Continuous blasting of granular or briquetted mixtures of ores and metallurgical products in thin layers. VEREIN. STAHLWERKE A.-G. (G.P. 462,411, 14.7.23).—The charge is treated on a water-cooled grate, the blast air being admitted at such a height above the grate that it cannot penetrate downwards; the charge just above the grate becomes only tacky (without melting), thus forming a bed on which the subsequent blast operations are conducted. A. R. POWELL.

Refining of copper. O. NAESER (G.P. 462,264, 4.5.27).—Arsenic, antimony, and tin are removed from copper by melting the metal under a slag comprising alkali or alkaline-earth sulphates and carbonates. This mixture acts more energetically than do the carbonates alone. A. R. POWELL. Production of composite metals suitable for hotrolling, by casting-on coatings of copper-zinc alloys. METALLGES. A.-G., H. VON FORSTER, and E. LAY (B.P. 329,572, 30.7.29).—The brass used for the production of composite metals with an iron core by the castingon process contains 68-72% Cu, 0.05-1% Mn, and a small proportion of iron, but is substantially free from lead (less than 0.01%). The brass is cast at 1000° around the iron core heated at 1200—1250°, whereby appreciable diffusion of the surface layers occurs and a welded article suitable for hot-working is obtained. A. R. POWELL.

Aluminium alloy. H. C. HALL and T. F. BRADBURY (B.P. 328,138, 21.5.28. Addn. to B.P. 323,353; B., 1930, 198).—The magnesium content is raised to 0.05— 2% in the alloy described in the prior patent.

H. ROYAL-DAWSON.

Manufacture of aluminium or aluminium alloy wires. VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 306,898, 15.2.29. Ger., 27.2.28).—Wire bars are cooled by blowing air on the surface during or before their passage through the rolls, in which their diameter is reduced to 10—12 mm. In this way the temperature is prevented from rising above 300°. The wire is then drawn down to 6 mm., during which operation the temperature rises to 150°; to prevent recrystallisation the wire is then chilled prior to coiling on reels. A. R. POWELL.

Forming articles from heat-treatable aluminiumbase alloys. (A) R. S. ARCHER and (B) W. C. WINTER, Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,751,468 and 1,751,500, 25.3.30. Appl., [A] 24.12.27, [B] 13.12.27). —(A) An aluminium alloy containing 3— $5 \cdot 5\%$  Cu,  $0 \cdot 75\%$ Mn, and  $0 \cdot 75\%$  Si is heated at 515° to obtain a homogeneous solid-solution structure, forged or rolled into shape at 440°, and quenched to retain the greater part of the copper in solid solution. (B) An aluminium alloy containing 3—5% Cu,  $0 \cdot 5\%$  Mg, and  $0 \cdot 6\%$  Mn is heated to cause the alloying elements to enter into solid solution, then die-pressed while still hot, and cooled rapidly in the mould. A. R. POWELL.

Insulating coating on aluminium or aluminium alloys. L. McCulloch, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,751,213, 18.3.30. Appl., 19.11.27).—The articles are boiled for 5—30 min. in a suspension of 5 g. each of lime and gypsum in 1 litre of water, whereby a film is produced having the composition : 76.8% Al<sub>2</sub>O<sub>3</sub>, 8.1% CaO, and 15.1% SO<sub>3</sub>. After drying, the film may be made non-porous and more adhesive by impregnation with tung oil, linseed oil, or a synthetic or natural resin. A. R. POWELL.

Non-corrodible metal [aluminium bronze]. W. HADDON and J. WINFIELD (B.P. 329,361, 16.1.29).— The alloy contains 88—90% Cu,  $7 \cdot 5$ —10% Al,  $0 \cdot 25$ —  $0 \cdot 4\%$  Mn, and 1— $3 \cdot 75\%$  Ni; the nickel may be wholly or partly replaced by iron. The preferred composition is  $89 \cdot 3\%$  Cu,  $9 \cdot 1\%$  Al, 1% Ni, and  $0 \cdot 27\%$  Mn, with small quantities of tin and/or lead. A. R. POWELL.

Articles of high resistance to wear, well polishable, and neutral to chemical agents. F. HAUPT-MEYER (B.P. 329,953, 27.1.30).—The articles comprise a mixture of up to 50% of a corrosion-resistant nickelchromium alloy or a rust-resisting nickel-chromium steel in powder form with sulphur, tar, rubber, resin, or other inert plasticising material. A. R. POWELL.

Casting of light metals, more particularly magnesium and its alloys, in sand moulds. I. G. FARBENIND. A.-G. (B.P. 319,272, 30.8.29. Ger., 19.9.28). —Green-sand moulds for use in making magnesium castings are heated internally by passing a current of hot air through them until the chills embedded in the mould attain the desired temperature for casting.

A. R. POWELL.

Manufacture of [gold-beryllium] alloys, particularly for production of sheets, membranes, etc. permeable to cathode, Röntgen, and like rays. ROHM & HAAS A.-G. (B.P. 313,527, 12.6.29. Ger., 12.6.28).—The alloy comprises 3—15% Au and 97—85% Be melted together in a vacuum furnace. The gold is preferably introduced as an amalgam.

A. R. POWELL.

Alloys [e.g., barium-nickel alloys for radiovalve filaments]. WESTINGHOUSE ELECTRIC & MANUF. Co., Assees. of P. H. BRACE (B.P. 317,785, 8.8.29. U.S., 22.8.28).—An alloy of nickel with 5-25% Ba is obtained by mixing finely-divided nickel with barium azide, compressing the mixture into bars, and heating the bars in a vacuum furnace, first at 200° to remove the nitrogen, then at a red heat to cause the barium to diffuse into the nickel. The resulting sintered bar is swaged in an inert atmosphere into a rod which can be drawn down to fine wire. The nickel may be wholly or partly replaced by iron or cobalt and the barium by any other alkaline-earth metal, by magnesium, or by an alkali metal. A. R. POWELL.

Reduction of tin and other metals from their salts and purification of molten baths of metal salts. E. A. ASHCROFT and F. E. ELMORE (B.P. 329,159, 26.2.29).—Lead, stannous, and cuprous chlorides are reduced to metal by passing hydrogen or a mixture of hydrogen and steam through the fused chlorides at 800°, 600°, and 500°, respectively. A bath of molten lead or tin is preferably maintained below the fused chloride in the first two cases. Fused zinc chloride may be purified from copper, lead, and tin by treatment with hydrogen at a temperature below the volatilisation point of the chloride. [Stat. ref.] A. R. POWELL.

Production of shaped bodies having a predominating percentage of tungsten or molybdenum. F. KRUPP A.-G. (B.P. 310,876, 22.4.29. Ger., 2.5.28).—Shaped bodies containing tungsten or molybdenum carbides and nickel, cobalt, or iron are prepared roughly by casting and the shaping is finished by applying pressure at 2200—2400°, *i.e.*, just below the m.p.

A. R. POWELL.

Treatment of vanadiferous ores. P. ASKENASY and E. ELÖD (G.P. 461,959, 21.4.25).—The finely-ground ore is digested with nitric acid and oxygen under pressure, sulphuric acid also being added when the ore is free from sulphur. A. R. POWELL.

Annealing of metal bands. ELECTRIC FURNACE CO., LTD., Assees. of HIRSCH KUPFER- U. MESSINGWERKE A.-G. (B.P. 305,951 and 312,988, [A] 11.2.29, [B] 8.3.29. Ger., [A] 11.2.28, [B] 4.6.28).—(A) The material is run continuously through an electric strip-annealing furnace the maximum temperature of which, controlled by a thermostat, is reached near the inlet end. The pitch of the resistance heating coils is so arranged that from the region of maximum temperature there is a gradual and regulated fall in temperature towards the outlet end of the furnace. (B) The strip hangs freely in the furnace in a catenary curve and is kept in position away from contact with the walls by passing through a clamping device before entering the furnace.

A. R. POWELL. **Production of easily soldered metal coatings.** M. SCHLÖTTER (B.P. 329,346, 16.2.29).—Coatings of tinlead alloys may be obtained from baths containing 100 g. each of lead and tin *p*-benzenedisulphonates, 50 g. of the free acid, 5 g. of gelatin, and 5 g. of phenol in 1 litre of water. The anodes used consist of lead-tin alloys containing the metals in the same relative properties as in the bath. More readily fusible deposits are obtained by adding 20 g. of cadmium perchlorate to the bath and incorporating a corresponding proportion of cadmium in the anodes.

A. R. POWELL.

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Production of electrolytic iron from scrap. R. D. PIKE (U.S.P. 1,751,099, 18.3.30. Appl., 20.7.26).—The scrap is dissolved in a mixture of ferric and ferrous chlorides, the solution is purified by treatment with hydrogen sulphide from the action of sulphuric acid on ferrous sulphide, calcium chloride is added occasionally to compensate for losses of chlorine and ferrous sulphate (from the hydrogen sulphide generator) to precipitate calcium, and the solution is electrolysed to obtain pure iron and a ferroso-ferric chloride anolyte for dissolving more scrap. A. R. POWELL.

Electroplating process [deposition of coppernickel alloys]. R. B. P. CRAWFORD and R. D. SNYDER (U.S.P. 1,750,092, 11.3.30. Appl., 26.11.21. Renewed 23.1.30).—An alloy resembling monel metal is deposited from baths having the composition (a) 600 g. of nickel sulphate crystals, 20 g. of copper acetate, 40 g. of nickel acetate, and 10 g. of sodium chloride per litre, or (b) 97.5 g. of nickel ammonium sulphate, 3.75 g. of copper oxalate, 7.5 g. of nickel chloride, and 20 g. of boric acid per litre, with enough ammonia to dissolve the copper oxalate. About 0.05% of glue may be added to the baths to improve the lustre of the deposits.

A. R. POWELL.

Chromium[-plating] bath for the production of white, soft, and easily polishable chromium deposits. J. BAUER (B.P. 310,427, 25.4.29. Ger., 25.4.28).—A cerium or yttrium salt is added to the ordinary chromium-plating bath in such quantity that the bath contains 5—15 g. Ce or 2—10 g. Yt per litre. A. R. POWELL.

Electrodeposition of chromium. ELEKTRO-CHROM GES.M.B.H. (G.P. 461,987, 19.2.26. Addn. to G.P. 398,054).—The electrolyte comprises a solution of ter- and bi-valent chromium compounds obtained by fusion of chromium trioxide with finely-divided carbon, cellulose, paper, leather, or proteins, or with aluminium or magnesium powder. A. R. POWELL.

Production of protective coatings of chromium. ELEKTRO-CHROM GES.M.B.H. (G.P. 462,275, 30.10.24.

Addn. to G.P. 458,632; B., 1930, 290).—The intermediate layer between the iron or steel and the chromium consists of manganese or tantalum.

A. R. FOWELL.

Electrolytic deposition of heavy metals, [e.g., lead and copper]. M. SCHLÖTTER (B.P. 329,324, 14.2.29. Ger., 9.11.28).—The electrolyte comprises a solution of salts of the metal with one or more inorganic acids and with one or more non-carboxylic organic acids. For the deposition of lead the bath contains 200 g. of lead *p*-benzenedisulphonate and 100 g. of lead perchlorate per litre, and for the deposition of copper it contains 200 g. of copper *p*-benzenedisulphonate, 100 g. of copper sulphate, 5 g. of gelatin, 5 g. of tannin, and 10 g. of *p*-benzenedisulphonic acid per litre. A. R. POWELL.

Production of firmly adherent electrolytic metal [lead] deposits upon articles plated in the molten bath. H. STEFKE (B.P. 330,040, 26.3.29).—Iron or copper alloys are plated with nickel or copper and then immersed in a bath of molten lead. The lead-coated material is made the anode in an acid plating bath, containing lead fluosilicate, acetate, or phenolsulphonate until the crystal structure of the lead coating is fully developed; the current is then reversed and the coating built up to the desired thickness by electrodeposition of lead. A. R. POWELL.

Preparation of alkali metals by fusion electrolysis. Dr. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (G.P. 461,694, 18.6.25).—The raw material used in the preparation of metallic sodium is the sodium chloride obtained by the evaporation of solutions of sodium hydroxide produced by the electrolysis of brine. The crystals so obtained are washed with brine until free alkali is removed or they are neutralised directly with hydrochloric acid. A. R. POWELL.

[Multiple-hearth] roasting furnace construction. H. J. HARTLEY, ASST. to NICHOLS COPPER Co. (U.S.P. 1,760,694, 27.5.30. Appl., 10.11.28).—See B.P. 324,471; B., 1930, 379.

Working up complex ores and metallurgical products. F. JOHANNSEN, ASST. to F. KRUPP GRUSON-WERK A.-G. (U.S.P. 1,762,867, 10.6.30. Appl., 21.1.27. Ger., 26.1.26).—See B.P. 265,162; B., 1927, 659.

Iron-nickel alloy. F. STÄBLEIN, ASST. to F. KRUPP A.-G. (U.S.P. 1,760,326, 27.5.30. Appl., 31.10.28. Ger., 24.12.27).—See B.P. 302,994; B., 1929, 561.

Removing, purifying, and recovering oil from tinplate. J. H. HOLDEN, T. O'BRIEN, J. C. WHETZEL, and R. E. ZIMMERMAN, ASSIS. to AMER. SHEET & TIN PLATE CO. (U.S.P. 1,763,430, 10.6.30. Appl., 15.6.26).— See B.P. 283,614; B., 1928, 235.

Separators for ores etc. (B.P. 329,030). Separation of minerals (U.S.P. 1,749,371). Edge-filter members (U.S.P. 1,745,426).—See I. Separating iron from roasted pyrites (B.P. 329,543). Aluminium compounds for production of aluminium (B.P. 307,345).—See VII. Refractories (B.P. 304,729).—See VIII. Purification of hot gases (B.P. 329,962).—See

XI. Degreasing castings etc. (B.P. 329,156).— See XII. Oligodynamic metals for water sterilisation (B.P. 306,547).—See XXIII.

# XI.—ELECTROTECHNICS.

Electrolytes for lead accumulators. N. KAMEYAMA and T. TAKASHIO (J. Soc. Chem. Ind., Japan, 1930, 33, 179B).—Measurements have been made of the E.M.F.of lead accumulators in which various sulphates were added to the electrolyte in order to increase the E.M.F.without causing excessive corrosive action on the plates. The sulphates of lithium, potassium, sodium, magnesium, and zinc were tried. In most cases the E.M.F. of the cell containing m mols. of sulphuric acid and n mols. of the metallic sulphate was greater than that of the cell containing only m mols. of sulphuric acid, but less than that of the cell containing (m+n) mols. of acid.

#### O. J. WALKER.

Electric welding in gas-works etc. KÜRSCHNER.— See II. Production of alloy steels. DÖRRENBERG and BROGLIO. Electrolytic iron. PIKE and others. Iron printing plates. THOMAS and BLUM. Hydrometallurgy of copper. BAROCH. Electrolytic zinc. TAINTON and BOSQUI; TAINTON and CLAYTON. Zinc on aluminium. COCKS. Bismuth electrolytes. KERN and JONES.—See X. Solidification of fatty oils. HOCK. —See XII. Rubber for electrotechnical work. HEERING.—See XIV.  $p_{\rm H}$  of tan liquors. HEPBURN.— See XV.

# PATENTS.

[Electric] heat-treating furnace. H. O. BREAKER (B.P. 307,301, 4.3.29. U.S., 3.3.28).—An electric resistance furnace for the treatment of goods which may contain occluded moisture is charged and discharged by means of a drop-bottom which is hydraulically raised and wedged into place ; a form of sand or other fluid seal is described which in conjunction with a welded metallic casing forms a substantially gastight furnace. Moisture is removed (to avoid oxidation) as soon as it is evolved, by means of a condenser forming an extension of the casing and surrounding the electric leads, the only communication for gases to the interior of the furnace being passages along or near the conductors. B. M. VENABLES.

Electrical heating of liquids. I. G. FARBENIND. A.-G., Assees. of F. WINKLER (G.P. 461,567, 12.2.26).— The electric resistor comprises a mass of granular conducting material kept in motion by a current of gas which passes through the liquid in which the resistor is immersed. Heating is rapid and no decomposition of the liquid occurs. A. R. POWELL.

Purification of hot gases. LODGE-COTTRELL, LTD. From METALLGES. A.-G. (B.P. 329,962, 27.2.29).—Hot gas, after passing through a heat-exchange cooler, enters an electrical precipitator having an electric field sufficiently long to precipitate successively spray or mist and dust or fume particles, then traverses an irrigating cooler in which remaining traces of dust are wetted and the gas is cooled, and finally passes through one or more electrical precipitators.

J. S. G. THOMAS.

Manufacture of self-baking electrodes. M. O. SEM and C. W. SÖDERBERG, ASSTS. to NORSKE AKTIE-SELSKAB FOR ELECTROKEM. IND. OF NORWAY (U.S.P. 1,751,177, 18.3.30. Appl., 6.12.28. Ger., 26.9.28).— Raw electrode paste containing a relatively high proportion of binder, *e.g.*, tar or pitch, is filled into the top end of an enclosing mantle, *e.g.*, of sheet iron, and heated to 100—200°, whereby it is caused to flow and form a solid electrode. Alternatively, the paste may be rammed or pressed after heating. J. S. G. THOMAS.

Deoxidising system [for transformers]. C. A. STYER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,745,875, 4.2.30. Appl., 5.4.28).—Air above the oil in a transformer casing is maintained substantially free from oxygen by attaching this apparatus to the casing and allowing the air to circulate through it by convection. The apparatus comprises a block of carbon resting on a metallic resistor which is maintained by electric currents at a temperature above the ignition point of the carbon. The heat produced is negligible compared to the losses in the transformer, and when oxygen arrives, due to the breathing of the transformer, the heat in the deoxidiser increases owing to combustion, which thereby increases the convection currents; to that extent the apparatus is self-regulating.

B. M. VENABLES.

(A, E) Asymmetric electric couples. (B-D) Electric-current rectifiers. S. RUBEN, Assr. to RUBEN RECTIFIER CORP. (U.S.P. 1,751,359, 1,751,360, 1,751,362 -3, and 1,751,460, 18.3.30. Appl., [A] 20.8.25, [B] 29.4.26, [c] 17.6.26, [D] 20.12.29, [E] 16.11.25).-(A) An asymmetric rectifying couple comprises an electropositive electrode of magnesium and an electronegative electrode formed of an alloy of copper with an element of the oxygen series of group VI in such proportions that, when current is passed, an inverse currentblocking film is formed and maintained at the junction of the electrode elements. (B) An electropositive metal, e.g., aluminium, is subjected to the action of a solution of sodium sulphide at about 100° and maintained at a positive electrical potential with respect to the solution until the current flowing attains approximately a minimum value, whereby the aluminium becomes coated with a dense homogeneous layer containing sulphide. (c) A rectifier comprising electropositive and electronegative electrodes composed, respectively, of magnesium and of a fused compound of the oxygen family of group VI having a base of copper, and provided with external and internal, independent, electric heating devices is claimed. Other rectifiers described comprise the following electropositive and electronegative electrodes, respectively : in (D) magnesium; a compound of copper and sulphur; and in (E) one of the lighter elements of groups II and III; a compound of an element of the oxygen family of group VI, e.g., sulphur or selenium, with an alloy of copper with zinc, silver, lead, tin, or antimony.

J. S. G. THOMAS.

Manufacture of oxide cathodes for discharge tubes. VEREIN. GLÜHLAMPEN U. ELEKTRICITÄTS A.-G. (B.P. 311,705, 25.4.29. Austr., 14.5.28).—An alkalineearth metal vapour and oxygen simultaneously produced within the tube, under conditions which prevent direct oxidation of the cathode core-wire, combine to form a deposit of alkaline-earth oxide on the cathode, which is then heated above its working temperature so that the oxide layer adheres firmly.

J. S. G. THOMAS.

Magnetic insulating materials. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of COMP. FRANÇ. POUR L'EXPLOIT. DES PROC. THOMSON-HOUSTON (B.P. 308,224, 19.3.29. Fr., 19.3.28).—Homogeneous sheets of textile material, impregnated with a mixture of synthetic or natural resin and filings or fine powder of magnetic material, are piled and consolidated under pressure and heat. [Stat. ref.] J. S. G. THOMAS.

Electrical insulating materials [comprising rubber]. W. S. SMITH, H. J. GARNETT, J. N. DEAN, B. J. HABGOOD, and H. C. CHANNON (B.P. 329,275, 11.2.29).-Unmasticated raw rubber is heated over the range 50-250° in a vacuum or an inert fluid; it is then dried, if necessary, and then treated with a solvent such as benzene or petroleum spirit. The dissolved fraction, after removal of the insoluble residue, is substantially free from non-rubber substances. The heat treatment results in a greater yield of dissolved rubber than is obtainable from unheated, unworked material. The product has superior electrical properties, but it is mechanically weak, and is preferably strengthened by mixing with gutta-percha, balata, or bitumen of high m.p., but low content of mineral matter and of free D. F. Twiss. carbon.

Electrical insulating materials [comprising rubber]. W. S. SMITH, H. J. GARNETT, H. C. CHANNON, and J. N. DEAN (B.P. 329,981, 28.2.29. Addn. to B.P. 307,390; B., 1929, 400).—Ordinary rubber (20—50%) is mixed with the dirt- and resin-free gutta-percha and/or balata claimed in the prior patent. J. S. G. THOMAS.

Electrical insulating masses for the construction of separation walls. I. G. FARBENIND. A.-G., Assees. of C. THODE and A. BENISCHEK (G.P. 461,493, 7.11.26).— Asbestos, slag wool, or other inorganic fibrous material is mixed with fillers, *e.g.* magnesia or alumina, and nitrocellulose, with or without the addition of a plasticiser, *e.g.*, a phenol ester of phosphoric acid.

A. R. POWELL.

Apparatus for electrometric analysis. M. TRÉNEL (G.P. 462,754, 15.9.25).—The comparison liquid is separated from the solution being titrated by a porous diaphragm saturated with quinhydrone or other reagent capable of evolving hydrogen. In this way continual evolution of hydrogen at the electrodes may be avoided, and no reducing reagent need be added to the electrolyte. A. R. POWELL.

Magnetic-core induction furnace. P. E. BUNET, Assr. to Soc. Actèries de Gennevilliers (U.S.P. 1,760,741, 27.5.30. Appl., 25.5.28. Fr., 2.6.27).—See B.P. 291,455; B., 1929, 823.

[Electric induction] apparatus for treatment of gases at high temperatures. M. FOURMENT (U.S.P. 1,763,229, 10.6.30. Appl., 28.10.25. Fr., 22.12.24).— See F.P. 593,298; B., 1926, 145.

Method of arc-welding. G. A. WHITING and R. A. HOLLAND, ASSTS. to E.M.F. ELECTRIC CO. PROPRIETARY, LTD. (U.S.P. 1,760,534, 27.5.30. Appl., 23.6.27. Austral., 9.7.26).—See B.P. 296,509; B., 1928, 790.

Glow-discharge lamps. TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 314,898, 3.7.29. Ger., 4.7.28).

Manufacture of light-emitting elements for electric lamps. A. SCHMID, and METROPOLE DEVELOP-MENTS, LTD. (B.P. 327,951, 12.12.28).

[Zinc container for] electric dry cells. SIEMENS BROTHERS & CO., LTD. From GEN. DRY BATTERIES, INC. (B.P. 330,165, 8.7.29).

[Inert-type] galvanic batteries. L. and G. J. A. FULLER (B.P. 330,444, 26.7.29).

**X-Ray apparatus [and safety devices therefor].** E. J. WARD-WATKINSON (B.P. 330,318, 14.3.29).

[Semi-portable] X-ray apparatus. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of VICTOR X-RAY CORP. (B.P. 329,978, 28.2.29).

[Bulbs for] incandescence electric lamps. J. L. РЕСН (В.Р. 305,227, 1.2.29. Fr., 3.2.28).

Hygrometer (U.S.P. 1,749,826).-See I. Lubricating oils (U.S.P. 1,746,641). Reclaiming insulating oil (U.S.P. 1,752,238) .- See II. Conversion of fibres into threads resembling wool (B.P. 329,792).-See VI. Separation of phosphorus vapours (G.P. 462,521).-See VII. Ferrochromium (G.P. 461,945). Insulating coating on aluminium alloys (U.S.P. 1,751,213). Gold-beryllium alloys (B.P. 313,527). Alloys for radio-valve filaments (B.P. 317,785). Annealing of metal bands (B.P. 305,951 and 312,988). Easily soldered metal coatings (B.P. 329,346). Electrolytic iron (U.S.P. 1,751,099). Copper-nickel alloys (U.S.P. 1,750,092). Chromium-plating bath (B.P. 310,427). Chromium coatings (G.P. 461,987 and 462,275). Heavy metals (B.P. 329,324). Adherent lead deposits (B.P. 330,040). Alkali metals (G.P. 461,694). —See X. Insulating materials etc. (B.P. 329,694). See XIII. Separation of rubber from Jatax (B.P. Separation of rubber from latex (B.P. 307,830 and 309,947). Electrophoretic deposition of solids from dispersions (B.P. 305,630 and 307,912). Preparation of rubber goods (B.P. 328,980). Porous body (B.P. 306,129).-See XIV. Treatment of seaweed (F.P. 633,696).-See XIX.

# XII.-FATS; OILS; WAXES.

Metallic soaps—their uses, preparation, and properties. W. F. WHITMORE and M. LAURO (Ind. Eng. Chem., 1930, 22, 646—649).—Heavy-metal salts of fatty acids are prepared, usually from an alkali salt of the fatty acid and the metal acetate or nitrate, in aqueous-alcoholic solution at 0° or 60°. The following are described (m.p. in brackets): Oleates: silver, mercury, lead (about 50°), zinc (about 70°), copper (fuses below 100°), and nickel (about 18—20°); stearates: silver, mercury (112.2°), lead (115°), zinc (about 130°), copper (about 125°), and nickel (about 100°); palmitates: silver, mercury (above 105°), lead (113°), zinc (129°), copper (120°), and nickel (about 80°); erucates: silver, mercury (below 50°), lead (about 100°), zinc (94°), copper (about 100°), and nickel (about 35°); laurates: silver, mercury (106°), zinc (128°), copper (111—113°), and nickel (44°). All the above silver salts have m.p. above 200° (decomp.). A brief *résumé* of the uses of the heavy-metal salts is given. H. BURTON.

Solidification of fatty oils by silent electric discharges (" Voltolfactis "). L. HOCK (Kautschuk, 1930, 6, 142-143).-Removal of unchanged oil from the gelatinous flocks obtained by submitting fatty oils in an atmosphere of hydrogen to the silent discharge yields a bulky, yellowish-white mass resembling white factis. It is possible to vary the character of the product by modifying the treatment. Röntgen diagrams reveal no indication of evidence of fibrous structure, but the weakness of the material prevents considerable extension. Use of hydrogen sulphide instead of hydrogen as atmosphere leads to a sulphurated "voltolfactis" of dark colour and unpleasant odour, and it would be preferable to defer introduction of sulphur as such or as hydrogen sulphide until nearly the end of the electrical treatment. The mol. wt. of "voltoloil" and also its heat of dissolution vary considerably with different solvents. D. F. Twiss.

Determination of unsaturation of fats and fatty acids. II. J. VAN LOON (Chem. Umschau, 1930, 37, 135—137; cf. B., 1929, 567).—Methods for the determination of iodine value are discussed. The Wijs method has been tested under varying conditions on a linseed oil. When a solution containing a small excess (3%) of iodine (no free chlorine) was used, no substitution occurred, constant values being obtained in from  $2\frac{1}{2}$  hrs. to 7 days. The iodine value varied slightly with the halogen excess used, not on account of substitution, but because the reaction proceeds to an equilibrium; with 150% excess of halogen the true iodine value (cf. hydrogen value) may be obtained in  $2\frac{1}{2}$  hrs. E. LEWKOWITSCH.

Composition of tung oil. J. VAN LOON (Farben-Ztg., 1930, 35, 1767—1769).—On the assumption that elæostearic acid (the glyceride of which constitutes 70-80% of tung oil) is an isomeride of linolenic acid, the values (150-170) quoted in the literature for the iodine value of tung oil are too low. It is shown that, under usual conditions, this oil gives an apparent iodine value only, considerable excess of iodine monochloride and a reaction period of at least 7 days being necessary for the attainment of the " true iodine value." By this method values of 217, 274, and 232 were obtained as the iodine values of tung oil, elæostearic acid, and tung oil total fatty acids, respectively, thus confirming the postulated degree of unsaturation of elæostearic acid. On the basis of these figures and earlier information, the following composition is given for tung oil: glycerin residue 4.7%, unsaponifiable matter 0.5%, volatile matter 3.4%, saturated acids 4.9%,  $\Delta^9$ -oleic acid 13.6%, and elæostearic acid 72.8%. S. S. WOOLF.

Production of pure ricinoleic glyceride. P. PAN-JUTIN and M. RAPOPORT (Chem. Umschau, 1930, 37, 130-135).—Attempts were made to isolate pure ricinoleic glyceride by repeatedly extracting castor oil with light petroleum (containing 10% of aromatic hydrocarbons) in order to remove other constituents: analyses were made after each extraction of the residual oil, the extract, and the fatty acids from each fraction. Oleic, linoleic, and stearic acids disappeared almost completely after three extractions, and dihydroxystearic acid, occurring mainly as the free acid, was removed by an alkaline wash. The unsaponifiable matter (1.8%) consisted largely of glycerin, which is not removed by the solvent. A ricinoleic glyceride was thus obtained containing only 0.5-1% of other substances. E. LEWKOWITSCH.

Hydrolysis in aqueous dispersions of sulphonated oils. W. SCHINDLER and R. SCHACHERL (Collegium, 1930, 108—116).—When dispersions of various sulphonated oils at different  $p_{\rm H}$  values, respectively, were boiled, sulphuric acid was, in most cases, found to be split off at  $p_{\rm H}$  values < 4.0 and in strongly alkaline solutions. There was practically no hydrolysis between  $p_{\rm H} 4.0$  and 7.0. The hydrolysis of a highly sulphonated castor oil was small when boiled for 30 min. at  $p_{\rm H} 4.2$  and diminished to zero at  $p_{\rm H} 6.8$ .

D. WOODROFFE. Examination of oils in ultra-violet light. J. A. PIERCE (Z. Unters. Lebensm., 1930, 59, 94-99).-The fluorescence phenomena exhibited under ultra-violet radiation by 48 different types of oil, principally crude and refined olive oils, have been studied under standardised conditions. Refined olive oil always gave a blue fluorescence and the crude oil a yellow, thus establishing a means for their differentiation. The filtration of cod-liver oil through fuller's earth produces a change in the fluorescence from yellow to blue. This change cannot be ascribed to the change in the colour of the oil, since by addition of carotene and chlorophyll the yellow fluorescence is not restored, although it is restored when refined oil is irradiated with ultra-violet light. This phenomenon and the observation that irradiation of unrefined olive oil produces an intensification of the yellow fluorescence support van Raalte's theory that fluorescence phenomena are attributable to the presence of vitamins. Bleaching by exposure to sunlight, or by heating for 3 hrs. at 150°, produced no change in the fluorescence, thus proving that the colour of the oil bears no relationship to the fluorescent appearance. H. J. DOWDEN.

Submerged combustion. HAMMOND.—See II. Sulpholeates in tanning. GERONAZZO.—See XV. Xylene value of butter. HANKE and DEUTSCHMANN. Soya-bean residues. MALKOMESIUS and SCHRAMM.— See XIX. Oil from cascara sagrada. JERMSTAD. —See XX.

#### PATENTS. , snimula

Preparation of fine granules on soar T-Cols and PALMOLIVE-PEEr 1003, BEP: 3143482,0118:22:29:1100:91, 28.6.28).—Hot, semi-fluid soap is mixed with air under pressure and beaten to a froth in an agitator; the mixture so formed is passed through a shredder and discharged as small granules in a semi-liquid condition into a hot-air drying chamber. E. LEWKOWITSCH.

Manufacture of water-soluble products from commercial fatty acids obtained from raw wool fat. I. G. FARBENIND. A.-G. (B.P. 305,597, 7.2.29. Ger., 7.2.28. Addn. to B.P. 307,776; B., 1929, 442).— The fatty acids are treated with oxygen or air at 100— 300° (with or without a catalyst such as cobalt resinate) prior to the sulphonation treatment described in the prior patent. E. LEWKOWITSCH.

Degreasing of articles [castings etc.]. CARRIER ENG. Co., LTD., and J. R. ROBERTSON (B.P. 329,156, 10.5.29).—The articles are submitted first to the action of a vapour grease-solvent, e.g., trichloroethylene, which condenses on them and effects removal of soluble matter, and subsequently to sprays of liquid solvent under pressure ; a bath open to the air may be used if provided with a suitable canopy or cooled zone to limit the height of the vapour bath. Suitable apparatus, embodying conveyors, stills for regeneration of clean solvent, etc. is described. E. LEWKOWITSCH,

**Production of emulsions.** A. L. MOND. From T. GOLDSCHMIDT A.-G. (B.P. 329,266, 5.11.28).—The higher fatty acid esters of polyhydric alcohols in which the alcoholic radical still contains unesterified hydroxyl groups, *e.g.*, glyceryl distearate, form stable aqueous emulsions in the presence of caustic alkalis or soaps. By the use of these esters aqueous emulsions of fats, oils, waxes, latex, zinc oxide, etc. are produced, suitable for pharmaceutical preparations, lubricants, wetting agents, wool-oiling, etc. E. LEWKOWITSCHOOP

Treatment of tung oil and mixtures containing the same. IMPERIAL CHEM. INDUSTRIESPIETD, OH. 9H. MORGAN, and A. A. DRUMMOND (B.P.1929)685, 14110(28). —After heating a solution of rawibuilg(oil=invavvolatile organic solvent, e.g., cyclohexantd, hethahydronaphtlithene, for several hours at above 1509, æsinall ambund of a dehydrating agent. (dyh@zing of balciam dhlorido, sulphuric acid, preferably dissolved in aviolatile solvent) is added. The product gives a chiar dy film bir stoving at steam temperatures? Thisecob sit, resins? etterliary be added before the relationship of the solvent of the solvent

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Use of ethylene dichloride in lacquer formulation. R. B. FRAZIER and E. W. REID (Ind. Eng. Chem., 1930, 22, 604-608).-Determinations of the solubility of nitrocellulose and cellulose acetate in various threecomponent solvent mixtures (one component being ethylene dichloride) show that nitrocellulose is practically completely soluble in a mixture of ethylene dichloride (80%) and methyl or ethyl alcohol (20%); a very small amount of an active solvent is added to the mixture to effect complete dissolution. A mixture of the dichloride (70-80%) and alcohol (20-30%) has the highest solvent power for cellulose acetate. The dichloride and the 80: 20-mixture are also good solvents for the usual gums and resins. The dichloride is exceptionally stable in lacquers, showing no tendency to hydrolysis. H. BURTON.

Practical evaluation of nitrocellulose lacquers. F. ZIMMER (Farben-Ztg., 1930, 35, 1766-1767).--Simple tests on raw materials and finished products, *e.g.*, absence of water or acid, viscosity, sp. gr., general spraying properties, rate of drying of film and its behaviour to finger-nail scratching, are briefly summarised. S. S. WOOLF.

Formation of synthetic resins. R. H. KIENLE (Ind. Eng. Chem., 1930, 22, 590-594).-Synthetic resins and highly polymerised compounds are classified (cf. B., 1929, 444) as follows : (1) heat-convertible (polyolefines, phenol-aldehydes), (2) heat-non-convertible (styrene, acidic type of phenol-aldehydes), and (3) element-convertible (glycerol polyunsaturated esters). From a survey of the literature and a study of the varying reactivity of alcohols and acids in the formation of polymeric esters and " alkyd " resins (loc. cit.), three postulates are advanced : (a) high-molecular compounds are formed only when the interacting molecules are poly-reactive, i.e., the molecules must be able to combine with one another through more than one primary valency linking; (b) the combination of molecules is determined by the probability of contact of mutually reactive, primary valency linkings; and (c) molecular size and shape, and the position of the reactive centres determine the physical properties of the resulting polymeride. A practical application of these postulates is illustrated by a study of the reactions between phenolphthalein, formaldehyde, and glycerol. H. BURTON.

Rinmann's green and rubber. DITMAR and PREUSSE.—See XIV.

# PATENTS. Jiw Ho bool a door of

Coating compositions, plastic compositions, etc. containing cellulose ethers. BRIT. CELANESE, LTD. (B.P. 313,133, 7.6.29. U.S., 7.6.28).—Compositions containing aralkyl ethers of cellulose, e.g., benzyl cellulose, one or more synthetic resins, e.g., resins of the phenol-aldehyde, glyptal, zinc butyl phthalate, polymerised lactic acid, or coumarone type, and, if desired, solvents, softeners, pigments, dyes, etc. are claimed.

S. S. WOOLF. Coating composition [for painting cement etc.]. J. V. REARDON, Assr. to REARDON Co. (U.S.P. 1,749,923, 11.3.30. Appl., 9.11.26).—A mixture of approx. equal parts of a hydraulic cement and slaked lime, together with relatively small proportions of a hygroscopic salt (sodium chloride), a sugar, and a water-repelling soap (e.g., calcium or aluminium stearate) is worked up with water to a paste suitable for brushing or spraying. L. A. COLES.

Manufacture of dope material for producing nontransparent coatings. I. G. FARBENIND. A.-G. (B.P. 302,280, 7.12.28. Ger., 13.12.27).-A cellulose ester, e.g., nitrocellulose, a gelatinising agent, e.g., camphor, a substantial proportion of oil, e.g., castor oil (preferably at least equal to the quantity of cellulose ester), colouring materials, fillers, etc. are thoroughly subdivided and mixed by working together, e.g., on rollers under pressure, in the presence of a volatile solvent, e.g., alcohol, the temperature being such that a large proportion of the solvent is evaporated during the operation, but without bringing the colouring matter into the colloidal condition. The material produced is capable of withstanding pressures of 200 atm. without separation of its constituents, contains a small proportion of lowboiling solvent, and is soluble in such solvents to form a lustrous varnish. S. S. WOOLF.

Impregnation of articles of wood, tissue, paper, cardboard, etc. with varnishes of synthetic resins. MANUF. DE MACHINES AUXILIAIRES POUR L'ÉLECTR. ET L'IND., and D. A. L. TEXIER (B.P. 329,066, 6.3.29).— The stages of solvent-extraction and resin-polymerisation in the customary synthetic-resin varnish-impregnation processes are carried out in a gaseous atmosphere of high sp. heat, *e.g.*, in dry steam, capable of absorbing or of carrying along solvents and of being condensed at ordinary temperatures. Suitable apparatus is described and the process is detailed. The use of relatively low temperatures and the complete recovery of solvents and excess synthetic resin components are claimed.

S. S. WOOLF. Paint, varnish, and enamel, and their production. W. F. BLEECKER and E. A. CLARK (U.S.P. 1,750,087, 11.3.30. Appl., 12.11.23).—Rubber, sulphur, and an oil, preferably lard oil, although vegetable or mineral oil will suffice, are heated together to above 177° to form a composition applicable as a paint or varnish or useful for impregnating porous substances to withstand chemical corrosion. Disintegrated vulcanised rubber can be used to supply both rubber and sulphur, a mixture with one ninth of its weight of oil being well adapted for the purpose. Asphaltum, fillers, pigments, and thinners may be incorporated. D. F. Twiss.

Production of painting materials from castor oil. J. SCHEIBER (B.P. 306,452, 9.2.29. Ger., 20.2.28). —Ricinoleic acid (castor oil fatty acids) is heated in vacuo at about 200° (with or without catalysts such as alumina, silica gel), whereby water is split off; the isomeric linoleic acid with conjugated double linkings which is formed is subsequently esterified with glycerol or other polyhydric alcohol by heating. The esters so produced can be used for varnishes etc. which dry to brilliant films highly indifferent to the action of water. E. LEWKOWITSCH.

Manufacture of lithopone or zinc sulphide resistant to light. E. MAASS and R. KEMPF (G.P. 462,372, 26.8.24).—The pigment prepared by any of the usual precipitation processes is mixed, while still wet, with a small quantity of a soluble thiosulphate, polythionate, sulphite, or hyposulphite and an ammonium salt, or with a soluble, readily dissociated metal sulphide, and the mixture is dried. The mass so obtained is ground and leached with water until both wash water and pigment react neutral to phenolphthalein. The pigment is again dried and calcined in the absence of air.

A. R. POWELL.

Manufactures of lacquers, coating materials, insulating and impregnating materials, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,694, 23.2.29).—The residues from the distillation of the condensation products of olefines (cf. B.P. 291,137 and 309,199; B., 1928, 560; 1929, 424) are dissolved in volatile organic solvents, with or without the addition of resins, rubber, etc. E. LEWKOWITSCH.

Production of rapidly hardening, odourless, phenol-aldehyde resins and mixtures of these with fillers. BAKELITE GES.M.B.H. (B.P. 305,237, 2.2.29. Ger., 3.2.28).—Metal oxides, hydroxides, or salts,  $e.g._i$ lime, magnesia, zinc hydroxide, calcium stearate, in quantity not much in excess of that necessary for complete binding of free phenol and uncombined by-products, are added to fusible, water-insoluble, resinous phenolaldehyde condensation products. The time of hardening of the resin is shortened and the thermal and electrical properties of the finished product are improved.

S. S. WOOLF.

Moulding mixtures. H. WADE. From BAKELITE CORP. (B.P. 329,628, 18.2.29).—A urea-formaldehyde resin is produced in the presence of fillers including a protein substance of the type of casein and a polyhydric alcohol, *e.g.*, glycol. Cellulose esters, plasticisers, etc. may be added as desired. E. LEWKOWITSCH.

Manufacture of resinous plastic material [by dry process]. A. V. KELLER (B.P. 328,938, 2.1.29).—Dry urea, thiourea, or derivatives thereof are caused to react with solid anhydrous polymerides of formaldehyde, e.g., paraformaldehyde, so as to obviate the presence of free formaldehyde in the final condensation stage. A neutral salt, e.g., sodium chloride, may be present as catalyst for the initial condensation, and such salts or solid organic acids may be added as accelerators for the final conversion into the insoluble form under heat and pressure. S. S. Woolf.

Manufacture of condensation products of urea and aldehyde. K. RIPPER, Assr. to F. POLLAK (U.S.P. 1,762,456, 10.6.30, Appl., 27.9.24, Austr., 25.8.24).— See B.P. 238,904; B., 1925, 891.

Materials in sheet form (B.P. 329,691). Air cleaner (U.S.P. 1,746,774).—See I. Carbon black (B.P. 327,979).—See II. Esters of carbohydrates (B.P. 305,661).—See III. Dressing of textiles (B.P. 320,018). —See VI. Coatings on aluminium alloys (U.S.P. 1,751,213).—See X. Foils from rubber-like substances (B.P. 329,000). Chlorination of rubber (B.P. 328,818).—See XIV.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Structure of rubber; swelling experiments in various liquids. P. BARY (Kautschuk, 1930, 6, 121-126).—Rubber probably consists of an intimate mixture of different polymerides of isoprene, the lowest degree of polymerisation present being dependent on the temperature and pre-history of the sample. The polymerides can be separated into two groups by their relative diffusibility, and unvulcanised rubber consists of a solution of the more easily diffusible polymerides in those of lower diffusibility. Harries' conception of the stability of the rubber molecule is out of accord with recent investigations, and the theory of true solutions is not applicable to jellies such as rubber gives with solvents. If a piece of vulcanised rubber, swollen with a liquid such as benzene, is transferred to the vapour space above the liquid, it shrinks gradually to the volume it would have attained by direct swelling in the vapour. This is not due to greater vapour tension in the jelly than in the pure liquid, but arises owing to liberation of heat by syneresis causing a temperature difference which actuates evaporation of the solvent from the jelly. The swelling of rubber is a two-fold phenomenon comprising entry of the solvent by diffusion and subsequent solvatation which accounts for its alteration in character from exothermic initially to endothermic later. Swelling of rubber, whether vulcanised or not, in a liquid such as toluene, is accompanied by a contraction of the system during the earlier stages, corresponding presumably with the exothermic period; the subsequent endothermic course of the swelling is probably associated with the solvatation process.

D. F. Twiss.

Viscosity of rubber solutions. C. M. BLOW (Trans. Inst. Rubber Ind., 1930, 5, 417-425).-Solutions of over-masticated rubber show small structural viscosity, but introduction of carbon black into the rubber before dissolving causes an increase in viscosity with development of structure viscosity and thixotropy which become more marked on storage. This effect is attributable to orientation of adsorbed rubber molecules on the carbon particles. The rubber which diffuses out of such mixtures as swell to a jelly has the original viscosity. It is believed that the viscosity of rubber solutions bears no relation to the mol. wt. of the rubber and that mastication does not effect real depolymerisation, changes in physical structure being responsible for the phenomena. D. F. Twiss.

Viscosity of rubber solutions. C. M. BLOW (Kautschuk, 1930, 6, 126—129; cf. preceding abstract).— In addition to the previous investigation, experiments on the viscosity of rubber after various periods of mastication and subsequent storage are reported. Any explanation of the effect of mastication as solely the result of depolymerisation induced by the action of oxygen would be difficult to reconcile with the above results. — D. F. TWISS.

Sun-cracking of vulcanised rubber. B. B. Evans (Trans. Inst. Rubber Ind., 1930, 5, 442-450; Indiarubber J., 1930, 79, 853-854).—Using mixtures containing (a) rubber (100 vols.), sulphur, and filler (20 vols.); (b) as in (a), but coloured with carbon black; and (c) similar to (a), but with vulcanisation aided by diphenylguanidine, and exposing vulcanised samples, bent double, (i) in the open facing south, (ii) indoors near a south window, and (iii) to a mercury-vapour lamp, it is found that zinc oxide is of little advantage and mixtures containing it are inferior to those containing clay or magnesium carbonate in resistance to sun-cracking. The addition of 1% of carbon black is found generally to cause increased tendency to sun-cracking, the results of Shepard, Krall, and Morris (B., 1926, 598) being thereby confirmed; the divergence of other observations on this point may arise from variation in the character of the carbon black used. Cracking tends generally to occur at right angles to the direction of mechanical strain, calender grain being of minor influence. The organic accelerator appears to have little effect on the cracking behaviour, but smoothness of surface is beneficial to the resistance of the sample. Exposure to the mercury-vapour lamp gives results qualitatively in line with natural exposure, but with less marked differences. D. F. Twiss.

Evolution of hydrogen sulphide from vulcanised rubber. E. WOLESENSKY (Bur. Stand. J. Res., 1930, 4, 501-513).-Samples of vulcanised rubber were heated at various temperatures in a current of hydrogen and the hydrogen sulphide evolved was determined as lead sulphide. In one case a sample heated for 5 hrs. at 285° yielded 8.4% of its total sulphur content in this form. At 25° hydrogen sulphide could be qualitatively identified, and if the temperature of destructive distillation was reached (about 300°) the yield exceeded 50%. Removal of free sulphur, or of non-rubber constituents, did not affect the result, nor is the presence of moisture in the nitrogen necessary. The reaction affords an additional explanation of the formation of hydrogen sulphide during vulcanisation and may be a factor in ageing. C. IRWIN.

Rinmann's green in accelerated rubber mixtures. R. DITMAR and K. H. PREUSSE (Gummi-Ztg., 1930, 44, 1842).—Rinmann's green maintains its colour during vulcanisation in the presence of accelerators of the dithiocarbamate or thiuram disulphide class, but loses its colour with most other organic accelerators; it activates the accelerator and effects a stiffening of the vulcanite. D. F. TWISS.

Rubber as material for electrotechnical work. H. HEERING (Kautschuk, 1930, 6, 129–136).—The knowledge of the dielectric characteristics of rubber covering, e.g., the influence of vulcanisation, compounding, moisture, and pressure on the electrical resistance, insulating power, power factor, and dielectric strength, is reviewed. The use of a layer of unvulcanised rubber between compounded rubber insulation and a metal conductor, which is frequent in England, is incidentally criticised as ineffective. (Cf. also Curtis and co-workers, B., 1926, 503; 1927, 916). D. F. Twiss.

Rubber lining in the chemical industry. J. COUR-BIER and T. BOUSSAND (Chim. et Ind., 1930, 23, 1099— 1100).—The method of producing an adherent rubber or ebonite lining on iron or steel is described and the limitations of dimensions of pipes etc. to which a satisfactory lining can be applied are mentioned. A table gives the limits of concentration and temperature within which resistance to different reagents can be assured. Soft rubber linings have a high resistance to abrasion, and are used on the interiors of tube-mills, breakers, etc. C. IRWIN.

Antimony pentasulphide. Schürmann and Böhm. —See VII.

### PATENTS.

Separation of rubber from latex. SIEMENS-ELEK-TRO-OSMOSE GES.M.B.H. (B.P. 307,830, 2.3.29. Ger., 14.3.28).—Latex is mixed with a liquid, such as glycerin, of low electrical conductivity so that it is possible to use continuous high-tension current, *e.g.*, at above 250 volts, to effect deposition of the rubber on an electrode or on a porous mould in front of the electrode. If desired, compounds yielding multivalent cations may be added, such as lead, prior to the introduction of the poorly conducting liquid, to deposition at the cathode instead of at the anode. D. F. Twiss.

Electrical separation of rubber from rubber latex. SIEMENS & HALSKE A.-G. (B.P. 309,947, 4.3.29. Ger., 18.4.28. Addn. to B.P. 307,747; B., 1930, 471). — In the process of the prior patent the support (either the electrode or the adjacent porous mould), bearing its rubber deposit, is dipped into a weak acid solution immediately after removal from the latex. This treatment increases the firmness of the deposit and facilitates its removal from the support.

D. F. Twiss.

Electrophoretic deposition of solids from dis-SIEMENSpersions, e.g., caoutchouc from latex. ELEKTRO-OSMOSE GES.M.B.H. (B.P. 305,630 and Addn. B.P. 307,912, 5.2.29. Ger., [A] 8.2.28, [B] 17.3.28).-(A) In the ordinary anodic deposition of rubber or yeast from aqueous dispersions by electrophoresis, the concurrent anodic liberation of oxygen or of acids may be disadvantageous. This feature is avoided by the addition of inorganic salt solutions containing multivalent cations, e.g., thorium nitrate, or of positively charged colloids, e.g., bovine serum, so that the originally negatively charged particles alter the direction of their migration and are deposited on the cathode or on a porous cathodic diaphragm of non-conducting material. It is advisable first to adjust the dispersion to  $p_{\rm H}$  7. (B) In connexion with the preceding process the  $p_{\rm H}$  of a dispersion such as latex is adjusted to a value 7 by initially bringing it into contact with a gas or gaseous mixture of feebly acid reaction, e.g., carbon dioxide, preferably after the addition of a protective colloid such D. F. Twiss. as globulin.

Preparation of goods of rubber or similar material from rubber latex or the like. DUNLOP RUBBER Co., LTD., R. F. McKAY, and E. W. MADGE (B.P. 328,980, 9.11.28).—Homogeneous, non-porous, anodic rubber deposits are obtained by immersing two dissimilar electrodes, *e.g.*, a zinc anode and a depolarised carbon cathode, in natural, preserved, or artificial latex, which may have been concentrated and/ or compounded, without the independent addition of electrolytes. D. F. TWISS.

Manufacture of rubber articles. MORGAN & WRIGHT, Assees. of W. A. GIBBONS (B.P. 307,790, 7.1.29. U.S., 13.3.28).—A hollow, annular form comprising a number of sections, one of which is relatively short and one at least of which has a conduit communicating with the interior and also with sources of fluid pressure, has several small, evenly distributed perforations over its surface. The form, covered with a filtering material, e.g., fibre coated with clay, is immersed in rubber latex, filtration being effected by evacuating the interior of the form. The rubber deposit so obtained is dried and vulcanised on the form, the sections of the latter being subsequently removed. D. F. Twiss.

Manufacture of rubber articles. GOODYEAR TIRE & RUBBER Co., Assees. of B. W. ROWLAND (B.P. 329,897, 12.7.29. U.S., 10.11.28).—A rubber suspension, which may have been concentrated and compounded, is formed into a film which is coagulated on one side, *e.g.*, by applying a quantity to a cylindrical surface which has been treated with coagulant such as 40% acetic acid. A form or mandrel which also may have been coated with coagulant, is then rolled across this film, thereby causing the layer of freshly coagulated latex to adhere to it. D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER CO., LTD., C. HAYES, E. W. MADGE, and F. C. JENNINGS (B.P. 329,381, 19.2.29).-If an aqueous solution of phenol is added to alkalipreserved latex and the alkalinity of the latter is then reduced, e.g., in the case of ammoniacal latex, by the addition of formaldehyde, the latex retains its mobility for a short time during which it may be formed into any desired shape by moulding, spreading, etc., and then changes into a firm continuous coagulum. Although tannic acid alone will not cause the formation of a continuous coagulum, one third of the phenol can be replaced by tannic acid without affecting the result. For latex of 60% concentration produced by a centrifuging process, the proportion of phenol necessary is about 5% on the rubber, but in the presence of compounding ingredients the proportion is smaller.

D. F. Twiss.

Manufacture of rubber-coated articles. DUNLOP RUBBER Co., LTD., and D. F. TWISS (B.P. 328,686, 5.2.29).—Chemical plant, coils, pipes, and perforated or woven sheet are coated with soft or hard rubber by spraying with an aqueous dispersion of rubber or similar material, drying, and vulcanising. [Stat. ref.]

D. F. TWISS. Liner [for rubbered fabrics] and treatment of same. L. B. SEBRELL, ASST. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,749,743, 4.3.30. Appl., 6.4.27).— Liners, for use between the adhesive surfaces of freshly rubbered fabrics, are coated with nitrocellulose which has been treated previously, or is treated subsequently, with certain softening agents such as phthalic esters. Old liners may advantageously receive similar treatment. The softening agent checks hardening, cracking, and peeling of the nitrocellulose film. D. F. TWISS.

Manufacture of foils and the like [from synthetic rubber-like substances]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,000, 5.1.29).— Hard flexible foils, such as films, bands, lacquer coatings, or filaments of great strength, low extensibility, and considerable resistance to water and chemical influences, are obtained from soft films of the desired form prepared from synthetic polymerisation products of diolefines, especially butadiene, by treating them with oxygen or oxygen-containing gases or with agents supplying oxygen, *e.g.*, peroxides. The films can, if desired, contain additional substances, such as fillers, pigments, and softening agents, and may also have been vulcanised before the described treatment. The process may be accelerated by raising the temperature, *e.g.*, to between 50° and 200°. D. F. Twiss.

**Porous body [containing rubber].** H. BECKMANN (B.P. 306,129, 31.1.29. Ger., 17.2.28).—Porous bodies suitable for filter-plates, electrolytic diaphragms, lubricating pads, etc. are made by mixing granular or fibrous substances with latex (sufficient to give approx. 15% of rubber in the finished product) and sulphur. A coagulant is then added, *e.g.*, a magnesium salt or sulphurous acid, so as to form a rubber gel permeating the entire mass. The mass is then moulded and vulcanised while moist to any desired shape. A porous, moulded body is thus obtained in which the grains or fibres are bound together by a highly permeable but chemically resistant material. D. F. TWISS.

Treatment of rubber and products obtained thereby. J. McGAVACK, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,742,609, 7.1.30. Appl., 20.9.28).—Increased flexing life can be imparted to rubber, *e.g.*, in pneumatic tyres or hose, by the addition, either singly or mixed, of derivatives of thymol, *e.g.*, thymoquinone, nitrosothymol, aminothymol, or of  $\alpha$ - or  $\beta$ -naphthaquinone. Such treatment also improves the flexing qualities of spray-dried rubber to the level of ordinary plantation rubber. D. F. TWISS.

Dyeing of rubber. E. G. CROAKMAN, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,742,757, 7.1.30. Appl., 8.11.27).—Certain basic dyes of the triarylmethane series, e.g., Victoria-blue B base, are normally unable to withstand hot vulcanisation, but in the additional presence of an aromatic nitro-compound, particularly a nitrophenol such as 2:4-dinitrophenol, coloured finished products are obtainable having shades more or less of the same order as the dye or mixture of dyes, before vulcanisation. D. F. TWISS.

Colouring rubber and like materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,761, 28.3.29).—Rubber and similar materials are coloured by mechanically working with an aqueous paste or suspension of a water-insoluble colouring matter (e.g., Brilliant Indigo 4G) containing an added organic substance or substances, such as amyl alcohol, glycerin, glyceryl acetates, triaryl phosphates, mineral oil, etc., which are less volatile than water. It is often advantageous initially to produce a coloured stock containing an excessive proportion of colour, e.g., 10% or more, and then to use this for colouring further quantities. D. F. TWISS.

Rubber composition and method of preserving rubber. P. C. JONES, ASST. to B. F. GOODRICH CO. (U.S.P. 1,741,778, 31.12.29. Appl., 21.1.29).—Tetrasubstituted hydrazines, especially tetraaryl hydrazines, such as tetraphenylhydrazine, are effective in retarding the deterioration of rubber with age. D. F. TWISS.

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Incorporation of lampblack into rubber and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,812, 13.5.29).—Lampblack is advantageously incorporated into rubber, either natural or synthetic, after first mixing it with a fluid, incompletely polymerised substance, such as butadiene, or a vinyl ester. The proportion of the liquid polymerisable, polymerised material may be 20—500% of the lampblack.

# D. F. Twiss.

Manufacture of rubber pastes. I. G. FARBENIND. A.-G. (B.P. 305,490, 4.2.29. Ger., 3.2.28).—Concentrated latex pastes are obtained without evaporation of water by mixing the latex with a water-soluble cellulose ether. On heating, the cellulose ether is precipitated in a gelatinous form, and carries with it the rubber globules. By suitable means, such as clarification, filtration, or centrifuging, the water can then largely be removed from the mixture, whereby a paste is produced containing a high proportion of rubber. Such pastes can be mixed with compounding ingredients and then dried or may be re-diluted. In the case of artificial rubber emulsions such as those obtainable by polymerisation of emulsified butadiene hydrocarbons it is desirable, before the concentration process, wholly or partially to remove the emulsifying agent. The cellulose ether, which in an example quoted for ordinary preserved latex amounted to 10% on the rubber, forms an excellent filler for the latter. D. F. Twiss.

Vulcanisation of rubber and the like. IMPERIAL CHEM. INDUSTRIES, LTD., S. COFFEY, W. J. S. NAUNTON, and A. SHEPHERDSON (B.P. 329,456, 4.4.29).—Vulcanisation is accelerated by the N: N-dithio-derivative of a secondary amine (e.g., dimethylamine, piperidine), which can be produced from the latter by the action of sulphur chloride. D. F. TWISS.

Rubber or like products [anti-agers]. GOOD-YEAR TIRE & RUBBER Co., Assees. of A. M. CLIFFORD (B.P. 313,486, 19.4.29. U.S., 12.6.28).—The 2-hydroxyand 2-anilino-1: 4-naphthaquinone 4-anils obtained by the action of aniline on  $\beta$ -naphthaquinone-4-sulphonic acid, as also 2: 4-dianilino- $\alpha$ -naphthol, are anti-agers for rubber. C. HOLLINS.

Chlorination of rubber. M. DESENISS and A. NIEL-SEN (B.P. 328,818, 16.5.29).—Stable chlorination products of rubber suitable for the manufacture of films, filaments, varnishes, etc. are produced by chlorinating dissolved or dispersed rubber at about 80°, e.g., by passing chlorine into a boiling solution of rubber in carbon tetrachloride under reflux until hydrogen chloride is no longer formed. Vulcanised rubber chlorinated as a dispersion in carbon tetrachloride yields similar products, the whole of its sulphur being eliminated as sulphur chloride. Films prepared from the chlorinated solutions, unlike those from cellulose esters, are practically noninflammable. D. F. Twiss.

Manufacture of brake liners and the like. J. E. POLLAK. From RUBBER LATEX RES. CORP. (B.P. 329,021, 11.2.29).—A wire reinforcement of very open structure is filled with a doughy composition of asbestos and rubber latex (or aqueous emulsion of rubber), the mass is dried by vacuum, and the product—a mass much reduced in volume but still open-textured and containing gelled rubber—is compressed and finally vulcanised. To prevent coagulation of the latex by the asbestos, the latter may be washed with dilute hydrochloric acid or the former stabilised by the addition of blood.

#### B. M. VENABLES.

**Treatment of latex and product obtained thereby.** J. McGavack, Assr. to NAUGATUCK СНЕМ. Co. (U.S.P. 1,762,729, 10.6.30. Appl., 14.10.27).—See B.P. 298,628; B., 1929, 864.

Composition of matter comprising resins and rubber in aqueous dispersion and their preparation. Treatment of aqueously dispersed colloidal substances [rubber latex]. A. BIDDLE, ASST. to UNITED PRODUCTS CORP. OF AMERICA (U.S.P. 1,762,153–4, 10.6.30. Appl., [A] 30.6.26, [B] 4.11.26).—See B.P. 283,686; B., 1928, 309.

Vulcanisation of caoutchouc and product derived therefrom. L. B. SEBRELL, ASST. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,763,619, 10.6.30. Appl., 12.6.28).— See B.P. 297,051 ; B., 1929, 949.

Antioxidant or age retarder [for rubber]. A. M. CLIFFORD, ASST. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,763,579, 10.6.30. Appl., 1.3.28).—See B.P. 307,013; B., 1930, 432.

Painting [rubber] balls and apparatus therefor. DUNLOP RUBBER Co., LTD., J. WRIGHT, and H. TREVASKIS (B.P. 330,365, 27.4.29).

Hardness etc. of non-rigid objects (B.P. 329,629). Materials in sheet form (B.P. 329,691).—See I. Carbon black (B.P. 327,979).—See II. Insulating materials (B.P. 329,275 and 329,981).—See XI. Emulsions (B.P. 329,266).—See XII. Paint, varnish, etc. (U.S.P. 1,750,087).—See XIII.

### XV.—LEATHER; GLUE.

South African tanning materials. II. Trees and plants other than the black wattle. C. O. WILLIAMS (Union S. Africa, Dept. Agric., Div. Chem. Series 93, 1930, Sci. Bull. 74, 68 pp.; cf. B., 1929, 405).-The average percentage of tannin in the bark of Acacia decurrens (green wattle) increased with the age of the tree, was greatest in the bark near the base of the tree, and decreased regularly the higher up the tree the bark was obtained. The rate of decrease was not so great as with A. mollissima. The tannin content, which was less than that of the bark of A. mollissima of the same age, varied directly with the thickness of the bark. The colour of the infusion increased with the age of the tree. A much lower yield of bark, approximately the same amount of tannin in trees of the same age, and a deepercoloured infusion were obtained from A. decurrens than from A. mollissima. A number of barks of exotic trees were analysed and contained on a 10% moisture basis : tans, soluble non-tans (%), degrees red and yellow, respectively: Green wattle (A. decurrens), 23.5-42.1, 10.8, 2.7-6.6, 2.8-9.3; silver wattle (A. dealbata), 14.3-28.2, -, 7.3-16.9, 9.1-17.9; golden wattle (A. pycnantha),  $35 \cdot 0 - 43 \cdot 4$ ,  $10 \cdot 4$ ,  $4 \cdot 4 - 6 \cdot 1$ ,  $7 \cdot 0 - 8 \cdot 8$ ; Blackwood (A. melanoxylon), 6.8-18.1, 10.1, 4.2-7.9, 6.4-11.7; saligna wattle (A. saligna), 12.1-

 $30\cdot3, 8\cdot8-12\cdot2, 1\cdot7-5\cdot2, 2\cdot5-6\cdot3; A.$  cyclopis, 6.7-13.0, 8.1-8.7, 2.8-10.0, 4.6-14.7; A. longifolia, 7.6, 10.1, 4.7, 6.6; Eucalyptus astringens, 27.5- $45 \cdot 4, 11 \cdot 6 - 13 \cdot 7, 2 \cdot 9 - 5 \cdot 6, 4 \cdot 9 - 21 \cdot 6$ ; E. sideroxylon,  $7 \cdot 1 - 30 \cdot 1, 6 \cdot 3 - 9 \cdot 1, 3 \cdot 1 - 16 \cdot 6, 5 \cdot 6 - 49 \cdot 0; E. diversi$ color, 3.6-16.3, -, 2.2-8.1, 3.5-11.0; E. Smithii, 11.8, -, 6.4, 13.3; E. paniculata, 8.6, -, 6.9-12.6; E. cladocalyx,  $8 \cdot 1$ , -,  $6 \cdot 2$ ,  $16 \cdot 8$ . The tannin content of other eucalyptus trees was very low. Other results were : Pinus halepensis, 17.9, -, 3.6, 6.8; P. longifolia, 16.6, -, 5.2, 14.1; P. insignis, 13.2, -, 3.9, 7.1; P. patula, 8.6, -, 4.3, 7.0; Callitris calcarata, 22.1, -, 7.1, 9.9; C. rhomboidea, 15.2, 10.3, 3.3, 5.5; C. robusta,  $21 \cdot 9$ ,  $7 \cdot 3$ ,  $6 \cdot 9$ ,  $12 \cdot 2$ ; Quercus pedunculata, 9.9, -, 7.3, 17.8; Casuarina cunninghamiana, 9.2, -, 5.7, 9.5; Salix babylonica, 7.3, -, 9.3, 26.7. The following indigenous trees and plants had : Acacia karroo, 16.7, 8.2, 6.7, 8.8; A. arabica (pods), 21.5, 24.9, 30, 8.8; bark, 15.3, 8.7, 5.4, 17.1; A. caffra,  $13 \cdot 1, 13 \cdot 8, 7 \cdot 8, 10 \cdot 3$ ; Elephantorrhiza burchelli (roots), 15.6-17.3, 16.4-23.6, 2.9-11.8, 4.9-38.4; Schotia speciosa, 16.3, 10.7, 12.2, 23.1; Rhus Thunbergii, 35.0, 17.7, 6.8, 17.7; R. incana, 19.2, 7.9, 14.7, 26.8; R. lævigata, 18.5, 6.2, 11.4, 18.1; R. natalensis, 15.2, 7.3, 8.2, 14.6; Protorhus longifolia, 10.2, 4.3, 11.4, 16.9; Harpephyllum caffrum, 18.2, 8.7, 9.1, 23.7; Sclerocarya caffra, 10.7, 6.5, 15.8, 19.1; Cape sumac (Osyris compressa) leaves and bark, 15.7-26.9, 15.4-25.4, 3.7-10.8, 6.9-21.3; O. abyssinica (leaves), 21.3, 19.1, 4.1, 9.5; Rhizophora mucronata, 33.6, 11.4, 7.7, 17.0; Bruguiera gymnorrhiza, 27.9, 10.0, 6.5, 9.5; Curtisia faginea, 21.5, 10.3, 15.2, 28.9; Olinia radiata, 17.2, 15.4, 3.7, 8.8; Eugenia Gerrardii, 16.7, 7.1, 13.0, 24.6; Elæodendron croceum, 16.4, 8.2, 14.9, 22.9; Pterocelastrus variabilis, 13.6, 20.6, 5.7, 7.1; Protea mellifera, 16.4, 12.3, 5.6, 14.7; P. grandiflora, 13.6, 16.9, 4.8, 7.4; P. caffra, 12.7, --, 7.7, 11.7; Brabeium stellatifolium, 13.3, 9.7, 3.5, 5.2; Leucadendron argenteum, 13.2, 7.6, 6.9, 8.8; Leucospermum conocarpum, 12.6, 5.2, 5.0, 6.6; Zizyphus mucronata, 13.9, 7.3, 6.1, 9.0; Jatropha zeyheri (roots), 20.5, 15.6, 9.4, 22.5; Phyllanthus discoideus, 9.9, --, 7.2, 19.9; Myrsine melanophleos, 11.6, 5.7, 7.3, 9.7; Garcinia Gerrardii, 11.3, 10.2, 4.9, 11.7; Terminalia sericea (galls), 10.2, 7.1, 24.5, 42.1; Baikiaa plurijuga 19.9, 4.9, 12.5, 17.0; Brachystigia spiceformis, 19.6, 9.1, 5.5, 9.4; Lonchocarpus mossambicensis, 21.1, 14.2, 19.0, 47.6. The tannin content of the yellow-D. WOODROFFE. wood barks was low.

Automatic apparatus for extraction of tanning materials for [tannin] analysis. G. PARSY (J. Soc. Leather Trades' Chem., 1930, 14, 117—119).—The tanning material is placed in an inverted, narrownecked bottle, from which the bottom has been cut and replaced by a two-holed stopper fitted with a delivery tube from a steam generator and a reflux condenser. The neck of the bottle is provided with a stopper and U-tube, to which is attached a siphon and delivery tube drawn out to a fine nozzle. The U-tube is supported by a flat-bottomed porcelain basin. The extraction is commenced with water at 50—60°, and gradually the temperature rises until finally it is at 100°.

D. WOODROFFE.

Apparatus for cooling tannin solutions during filtration for anaylsis. L. SHEARD (J. Amer. Leather Chem. Assoc., 1930, 25, 248-249).-Two shallow, rectangular troughs are arranged one above the other. The upper one is provided with holes which are fitted with rubber stoppers each carrying a funnel, the stem of which reaches to just above the collecting bottles, which stand in the lower trough. A rubber band is fixed on the lower part of the funnel stem and on it rests a conical-shaped glass or metal cover. The upper trough is fitted with a hinged cover, which may be closed during filtration, and with a water-inlet pipe at one end; at the other end is an outlet pipe connecting with the lower vessel, which is similarly fitted with an exit pipe. This apparatus is used in filtering tannin solutions in accordance with the regulations of the official method of tannin analysis, except that the temperature is regulated to  $20-21^{\circ}$ , as the author considers the range of 5° allowed in the official method is too great, especially for crude quebracho extracts.

D. WOODROFFE.

Determining the  $p_{\rm H}$  of tan liquors by the hydrogen electrode. T. F. G. HEPBURN (J. Soc. Leather Trades' Chem., 1930, 14, 261).—Poisoning of the electrode is reduced in the following way. A freshly-coated Rideal electrode is immersed in distilled water and hydrogen is bubbled through so that the whole cylinder is sprayed by it as the hydrogen emerges. After 3 min. the water is replaced by the tan liquor, so that the electrode is completely immersed therein, and the hydrogen is sprayed over it. A constant reading is attained in 2 min. D. WOODROFFE.

Measurement of the colour of tanning extracts. G. BALDRACCO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1930, 8, 100—109).—For this measurement the Lovibond tintometer gives inexact results, but if the tanning liquid is used in suitable concentration, so that tanning is complete, practical tests with hides give useful data. T. H. POPE.

Analytical controls of sulpholeates used in tanning. II. Sulphoricinoleates (II). III. Sulpholeates. M. GERONAZZO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1930, 8, 142—144, 144—150).—II. The results of a more strict chemical analysis of 37 samples of sulphoricinoleates are in agreement with the indications of the simple technical analysis proposed (B., 1928, 580), the value of which is thus confirmed.

III. Sulpholeates with a basis of fish oils should remain clear and liquid at the ordinary temperature, such properties not being due to dilution with mineral oil or other organic solvent. Fish and cetacean oil sulpholeates are brownish-yellow or brownish-red, bluish-green fluorescence usually indicating the presence of mineral oil. Most sulpholeates exhibit the odour due to sulphonation, which becomes more evident in their aqueous emulsion, as well as the characteristic odour of the original fatty material and that of any solvent added. The reaction towards litmus paper is usually acid, but the use of , highly acid products may be harmful in certain tanning operations. The characters of technical importance in an emulsion of sulpholeates are colour, intensity, duration, emulsive capacity, and behaviour towards ammonia, these being observed after shaking 2 c.c.

of the sulpholeate with 20 c.c. of water for 2 min. The value of the sulpholeate is directly related to the duration of the emulsion and to the emulsive capacity (loc. cit.), which should be determined by a tanning test. The behaviour towards ammonia is examined by adding ammonia until the emulsion has a distinct odour of it, and then shaking; destruction of the emulsion with formation of a clear liquid indicates derivation from sulphonated matter only, whilst persistent turbidity shows the presence of extraneous matters such as emulsive oils (mineral or resin) or of other emulsifying agents insoluble in ammonia. The titre is determined as previously described (loc. cit.). The above method of technical examination is applied to 47 samples of sulpholeates of various types, the results being in agreement with those of more accurate chemical analysis. T. H. POPE.

Tanning problem. L. MEUNIER and K. LE VIET (Cuir tech., 1929, 22, 432—439; Chem. Zentr., 1930, i, 785).—The terms tanning agent, intensity of tanning, and astringency are defined, and the effect of centrifuging on the capillary water is considered. Determinations of the astringency of various leathers were made. A. A. ELDRIDGE.

Limits of acidity and temperature in "clearing" semi-tanned sheepskins. P. S. BRIGGS (J. Soc. Leather Trades' Chem., 1930, 14, 244—253).—Elongation-temperature curves were derived for pieces of semi-tanned sheepskins (Egyptian, Smyrna, Syrian, and Mosul tannages) in water,  $0 \cdot 1N$ -,  $0 \cdot 02N$ -, and  $0 \cdot 01N$ -sulphuric acid. Maxima were observed in one case at 80° in water, diminishing as the concentration of the acid "clearing" solution was raised to 60° for  $0 \cdot 1N$ -sulphuric acid. The maximum was as low as 37° for some tannages, indicating that the temperature of "clearing" should not exceed 30°, otherwise the skins may be damaged, producing harsh leather and loss in area. D. WOODROFFE.

"Red stains " and " red heat " on salted hides. F. STATHER (Collegium, 1930, 151-153; cf. B., 1930, 433).-The author observes that "red stains" and "red heat" are not caused by different bacteria as suggested by Lloyd and co-workers (cf. B., 1930, 251). The characteristics of the bacteria which cause "red heat" agree closely with those of the bacteria which cause "red stains" viz., *Micrococcus roseus*, *Sarcina lutea*, and *S. auriantica*. The chief difference is their reaction to common salt. The "red heat" bacteria give optimum growth on a medium which contains 16-30% of sodium chloride, and only poor growth when only 6-8% was present. The "red stain" bacteria showed no growth on a medium containing 16% of sodium chloride, but when grown on media increasing in salt content to 20% practically no difference was found between them and the "red heat" bacteria, except that the latter developed much better and more quickly. Cultures of "red heat" bacteria were made on media of decreasing salt contents. D. WOODROFFE.

Violet specks on salted raw hides. F. STATHER, G. SCHUCK, and E. LIEBSCHER (Collegium, 1930, 153-161). —Small reddish- or bluish-violet specks are often observed on the flesh side of salted raw skins. They

are caused by chromogenic micro-organisms, and disappear during soaking and liming. No increased loss of hide substance was observed on stained skins, but violet specks were found on the grain side of skins which had been stained on the flesh side, and they rendered such skins unfit for pale shades. D. WOODROOFE.

Salt stains. II. Grain damage on limed hide and chrome-tanned leather. F. STATHER and G. SCHUCK (Collegium, 1930, 161-169; cf. B., 1929, 140). -Determinations of the calcium, phosphoric acid, and sulphuric acid contents of a number of salt-stained and unstained raw calf-skins showed them to be the greater in the salt-stained skins. The chromium contents of salt-stained and unstained pieces were determined after tannage and found to be the same except for one finished leather in which the chromium content was the greater in the tanned salt-stained piece. The stains are not removed by liming, bating, nor pickling. They are fixed by tannage and generally have a darker green colour than the normal tanned leather, but sometimes they are brighter coloured. Dyes are absorbed differently by the stained and the unstained portions. Fat and season are only slightly or not at all absorbed by the salt-stained grain, which cracks on glazing if badly stained. D. WOODROFFE.

Bacteriology of the violet specks on salted raw hides. F. STATHER and E. LIEBSCHER (Collegium, 1930, 170—175; cf. preceding abstract).—Micrococcus roseus, M. luteus, and M. pyogenes albus have been identified amongst the bacteria on salted raw hides showing violet specks. Several harmless forms of Proteus were found and also a species of bacteria (Actinomyces) which formed long threads, coloured the medium reddish, and liquefied gelatin.

D. WOODROOFE. Effect of heat on wetted vegetable-tanned leathers. VI. W. J. CHATER (J. Soc. Leather Trades' Chem., 1930, 14, 133-153; cf. B., 1930, 574). Shrinkage curves have been derived for a number of commercial sole leathers in different stages of tannage. The initial shrinkage temperature (I.S.T.) of the fullytanned leathers was in every case less than that of the half-tanned products, and was increased by the removal of water-soluble matter from the fully-tanned leathers. It is shown that the I.S.T. is increased by treating the fully-tanned leather with buffer solutions, attaining a maximum at  $p_{\rm H} 5.0$ , and then diminishing slightly with further increase in the  $p_{\rm H}$  of the buffer solution. The  $p_{\rm H}$  of the half-tanned leather is diminished as the tannage becomes more complete, which would explain the change in I.S.T. Samples of pelt after being tanned with quebracho, valonia, oakwood extract, and myrobalans, respectively, were found to have varied I.S.T. D. WOODROFFE. values.

Sodium hydrogen sulphide in the manufacture of glove leather. V. CASABURI (Cuir tech., 1929, 22, 489-494; Chem. Zentr., 1930, i, 784).---A study of conditions for depilation. A. A. ELDRIDGE.

Testing of patent leather. F. FEIN (Collegium, 1930, 117—118).—Tests were carried out on two patent leathers A and B. The tensile strength and stretch of the patent film on A were slightly less than those

of the leather itself, whereas the same properties of the film on B were about 30% less than those of the leather. Of the two samples, A was better than B. This affords a useful method of testing patent leathers. Sample B would crack the more readily.

D. WOODROFFE.

Identification of formaldehyde in leathers. P. CHAMBARD (J. Soc. Leather Trades' Chem., 1930, 14, 258-261).-Different leathers were tested for formaldebyde by the Schiff and the Grosse-Bohle (Z. Unters Nahr. Genussm., 1907, 14, 89) reactions. A positive reaction was obtained with the Schiff test on pelt, chamois, alum-tanned and formaldehyde leathers; it is consequently useless on leather. Positive reactions were given by the Grosse-Bohle test on undyed formaldehyde-tanned leathers, whether freshly tanned or aged, but not with dyed formaldehyde leathers. The colour was developed slowly on aged leathers. This test is therefore satisfactory for detecting formaldehyde in leathers. D. WOODROFFE.

Effect of different methods of preparation of sole leather on resistance to wear. A. GOLDENBERG (Cuir tech., 1929, 22, 414—422; Chem. Zentr., 1930, i, 786).

Determining the insoluble matter [in tan liquors] by the sedimentation method. V. KUBELKA and V. NĚMEC (J. Soc. Leather Trades' Chem., 1930, 14, 254—258).—Cf. B., 1929, 991.

#### PATENTS.

(A) Production of a bate or drench for skins. (B) Bating or drenching limed, unhaired hides or skins. I. G. FARBENIND. A.-G. (B.P. 304,214 and 304,294, [A] 16.1.29, [B] 18.1.29. Ger., [A] 16.1.28, [B] 18.1.28).—(A) Bacteria capable of hydrolysing albumin and fat are cultivated on a medium containing these substances (e.g., blood and animal fat) and are then removed, the liquor being used for bating or drenching purposes. (B) The above bating liquor  $(0\cdot3-0\cdot5\%)$  is allowed to react for 1 hr. on rinsed, limed, unhaired calf skin at 35-37°.

D. WOODROFFE.

Manufacture of glutinous products. J. Y. JOHN-SON. From I. G. FARBENIND. A.-G. (B.P. 328,197, 22.12.28. Addn. to B.P. 313,101; B., 1929, 653).— Finely-ground dry yeast (10 pts.) or products of yeast autolysis are treated with aldehydes (e.g., paraldehyde, 1 pt.), their polymerisation products, or with substances capable of developing them, prior to, during, or after treatment with weakly alkaline or acid reacting agents or salts or a strong alkali (e.g., 30 pts. of 8% sodium hydroxide solution). The product, a glue, is left for 3 hrs. D. WOODROFFE.

Treatment of fish skins. A. EHRENREICH (U.S.P. 1,762,053, 3.6.30. Appl., 24.1.28. Belg., 26.1.27).— See B.P. 281,919; B., 1928, 133.

Process for hardening colloids. A. MILLER, Assr. to AGFA ANSCO CORP. (U.S.P. 1,763,533, 10.6.30. Appl., 2.2.24. Ger., 27.11.23).—See B.P. 231,564; B., 1925, 479.

Dyeing of chrome leather (B.P. 304,608).—See VI.

## XVI.—AGRICULTURE.

Soil evaluation and examination. A. von Nostrrz (Landw. Jahrb., 1930, 71, 837—854).—In the mechanical analysis of soils, the effects of the use of hard water for sedimentation, of the various methods of dispersion, and the fluctuations of temperature during the process are considered, together with the relationship between the mechanical composition and water relationships of soils. A. G. POLLARD.

Improvements in the Kühn method for the mechanical analysis of soils. L. PINTO (Z. Pflanz. Düng., 1930, 17A, 102-112).—Kühn's method is modified by determining the chalk and organic matter in the sand and clay fractions. Boiling the soil suspension for 2 hrs. did not appreciably affect the proportion of clay recorded, but markedly decreased the necessary washing period in the elutriation cylinder.

A. G. POLLARD.

The phosphate question [in soils]. O. ARRHENIUS (Z. Pflanz. Düng., 1930, 16A, 307—310; cf. B., 1930, 386).—Evidence from culture experiments indicates that silica cannot, even partially, replace phosphate as a plant nutrient and actually tends to reduce the efficiency of phosphates in this connexion.

A. G. POLLARD.

Determination of the root-soluble phosphate in soils by the seedling method. A. NÉMEC (Z. Pflanz. Düng., 1930, 16A, 354—363).—Low values for the assimilable phosphate in soils, determined by the seedling method as compared with field trials and other methods, were due to deficient nitrate in the soils and consequent abnormal assimilation by the plants. Effects of increasing applications of nitrogenous fertilisers on the Neubauer phosphate values of soils are recorded. A. G. POLLARD.

Examination of the Neubauer method and its application to easily assimilable nitrogen [of soils]. R. THUN (Z. Pflanz. Düng., 1930, 16A, 257–283).— Changes in the Neubauer values for assimilable potash and phosphate in soils with season and with fertiliser treatment are recorded and discussed together with the selection of "limiting values" for particular crops. Qualitative examination of the nitrate content of soils may be made by immersing the cut stems of seedlings grown by Neubauer's method in diphenylamine solution and observing the intensity of the blue colour produced (cf. Niklas and Vogel, B., 1924, 142). Comparison tests with fertilised soil form a basis for the calculation of the nitrogen requirement of the soil. Results of such tests are confirmed by field trials. A. G. POLLARD.

Neubauer analyses and their relation to field trials. K. HESSE (Landw. Versuchs-stat., 1930, 110, 1-31).—Irregular results of Neubauer tests are ascribed to lack of uniformity of experimental conditions, notably the lighting of the growing seedlings, moisture conditions of the soil before and during the experiment, and the season during which the trials are made. With comparable conditions Neubauer results agree well with those of field trials; in general, better agreement is obtained for potash than for phosphates. The amount of minerals assimilated by plants in seedling trials are 734

dependent on the time of application of the fertilisers. The intake of potash and phosphate by the seedlings is not affected by nitrogenous manuring.

A. G. POLLARD.

Lime requirement of lime-deficient moor soils. B. TACKE [with T. ARND, W. SIEMERS, A. POOCK, I. SAFFRON, and A. SPIECKER] (Z. Pflanz. Düng., 1930, 9B, 216-234).-The effect of increasing quantities of lime on crop production in moor soils is examined in pot cultures. Maximum crops corresponded in most cases with partial neutralisation of the total acidity, the actual proportions varying with the crop. Excessive liming produced markedly decreased yields. The repeated use of physiologically acid or alkaline fertilisers changes the "lime condition" of these soils to a considerable extent. A. G. POLLARD.

Von Csiky's method for determining the lime requirement [of soils]. J. WOLTERSDORF (Z. Pflanz. Düng., 1930, 16A, 290-297).—In determinations of the hydrolytic acidity of soils by leaching with calcium acetate solution (von Csiky, B., 1929, 788), the relationship between the amount of calcium acetate used and the acidity developed is represented by a hyperbolic curve. Thus the end-point of the reaction, i.e., the true hydrolytic acidity, is not actually observed, but must be calculated after experimental determination of at least two points on the curve. The retentive power of the absorbing complex of soils for hydrogen ions is sufficiently great to demand relatively high proportions of other ions for its complete displacement. The calcium ion exhibits relatively greater displacing power than the ions K', Na', Mg', or NH'<sub>4</sub>, and is more strongly absorbed by the soil. Von Csiky's method does not include consideration of these observations and gives results which although not generally far in error must nevertheless be suspect. A. G. POLLARD.

The chlorine question [in soils]. O. ARRHENIUS (Z. Pflanz. Düng., 1930, 16A, 310-314) .- Sand cultures are described showing the effects of varying concentrations of sodium chloride on the crop yields of wheat, barley, oats, clover, sugar beet, and sugar cane.

A. G. POLLARD.

Determination of exchange acidity in potassium chloride extracts of soils. R. Albert (Z. Pflanz. Düng., 1930, 16A, 305-307).-Ferrous compounds may occur in potassium chloride extracts of soil and cause unsatisfactory results in  $p_{\mathbf{H}}$  determinations with the quinhydrone electrode. The mechanism of the reactions involved is discussed. A. G. POLLARD.

 $p_{\pi}$  determinations [of soil] in water and potassium chloride solutions. H. ELLEDER (Z. Pflanz. Düng., 1930, 16A, 283-289 ; cf. B., 1930, 630).-Examination is made of the factors causing differences in the recorded  $p_{\mathbf{H}}$  values of soils as determined in aqueous and in N-potassium chloride suspensions. The more alkaline values for potassium suspensions of chalky soils cannot be correlated with the calcium content (whether carbonate or total), but are more closely related to the lime and magnesia content (cf. Goy and von Burow, B., 1929, 830). A. G. POLLARD.

Physiological reaction of potash salts. W. ZIEL-STORFF and K. NEHRING (Z. Pflanz. Düng., 1930, 17A,

67-79).-In pot cultures neither "40% potash salts" nor kainite affected soil reaction, but potassium and magnesium sulphate mixtures tended to produce slight acidity. In field trials none of these materials affected soil reaction. A. G. POLLARD.

Relationships of the different acids and bases in soil to the electrometric titration. S. Gov, P. MÜLLER, and O. Roos (Z. Pflanz. Düng., 1930, 16A, 342-354 ; cf. B., 1930, 254).-Comparative determinations of acidity,  $p_{\rm H}$  values, and electrometric titration values of soils are recorded. Exchange acidity does not exist in soils with  $p_{\rm H} > 5.5$ . For practical purposes electrometric titration can be utilised for determinations of hydrolytic and exchange acidity, neutralisation values, and lime requirement. The authors' system of soil classification is preferable to individual determinations of  $p_{\mathbf{H}}$  value, titratable acidity, or buffer capacity for characterising the acid injury of heavy soils. The assimilable potash and phosphate contents of soils may be determined from electrometric titration values. A. G. POLLARD.

Nitrogen losses from "Kalkammonsalpeter" by the volatilisation of ammonia. W. SELKE (Z. Pflanz. Düng., 1930, 9B, 207-215).-Laboratory experiments indicate possible losses of free ammonia from "Kalkammonsalpeter" when mixed with moist soil. Under field conditions, unless the fertiliser is applied as a top dressing or used on a very light chalk soil, the absorptive power of the soil is sufficient to retain any free ammonia formed. A. G. POLLARD.

Nitrogen fertilisers. A. N. VOLSKAYA (Udobren. Urozhai, 1929, No. 4, 221-226).-On sandy and heavy loam soils ammonia fertilisers are as good as, or better than, nitrates with potatoes and oats ; ammonium nitrate ranked first, followed by the sulphate, whilst calcium nitrate, calcium cyanamide, and urea gave erratic results. CHEMICAL ABSTRACTS.

[German] supervisory department for ammoniacal and phosphatic fertilisers. ANON. (Z. Pflanz. Düng., 1930, 9B, 234-235).-Official notification is given of changes in the naming and marking etc. according to the regulations for the sale of fertilisers.

A. G. POLLARD.

Preparation and action of "artificial stall manure." O. FLIEG (Z. Pflanz. Düng., 1930, 9B, 193-207).—During the rotting of straw the pentosan content steadily decreases and serves as a measure of the extent of the rotting process. Satisfactory rotting of straw composts results if the straw is first wetted with water and when heating begins (a few days) it is stacked with the admixture of nitrogenous materials. Addition of lime, potash, or phosphates to the stack is unnecessary. After about 3 months the mixture should contain approx. 80% of water and 0.35% N. If the customary period of time is allowed before use the compost causes no plant injury due to temporary binding of soil nitrate, but exerts a fertilising effect corresponding to its nitrogen content. By addition of soluble nitrogen to the compost in quantity to give a total nitrogen content similar to that of fresh stall manure, an "artificial stall manure " is obtained with a greater efficiency than

the natural material. On light soils similar results are obtained by ploughing-in straw with the addition of an adequate proportion of soluble nitrogen (2%).

# A. G. POLLARD.

Physical chemistry of cultivated soils. II. Influence of manuring on the properties and adsorption potential of low-lying loams. R. A. HERZNER (Z. Pflanz. Düng., 1930, 16A, 315-331; cf. B., 1930, 294).-Elutriation and sedimentation processes for the mechanical analysis of soils give higher values than pipette and hydrometer methods. Kozeny's equation  $(h/\sqrt{t} = K)$ , representing the rate of capillary rise (h) in soils, is examined and its relation to other physical properties discussed, together with its possible use in showing the effects of fertilisers on soilwater movements. Measurements of the adsorption potential of soils (loc. cit.) are valuable in studies of soil texture and water relationships. Changes in the adsorption potential of soils following manuring result not only from the increased electrolyte content, but also from the altered degree of dispersion of the soil colloids. No definite relationships were observed between adsorption potential and crop increases of manured soils. A. G. POLLARD.

Relationship between soil reaction, distribution of meadow plants, type and yield of meadows. KLAPP (Landw. Jahrb., 1930, 71, 807—835).—Optimum ranges of soil reaction for the growth of numerous species of meadow grasses etc. are recorded. For the majority of species the growth- $p_{\rm H}$  range is of such width that the characterisation of soil reaction by classification of the natural flora is indecisive. Highest hay yields are associated with soil reactions of  $p_{\rm H} 5 \cdot 0 - 6 \cdot 8$ , with a preponderance of plants of better feeding value in the less acid areas. A. G. POLLARD.

Lime-sensitiveness of yellow lupins and the rôle of the nodule bacteria in lupin sickness. R. REINCKE (Z. Pflanz. Düng., 1930, 17A, 79-102).-Chlorosis in yellow lupins is characteristic of a definite early stage in the growth of the plants and is distinct from and independent of injury produced by nitrogen deficiency, although both may result from excessive liming. Nitrogen deficiency following liming is due to decreased activity of the nodule bacteria. Chlorosis is rare in blue lupins, but lime injury to the nodule bacteria occurs. The latter is not caused by the increased alkalinity of soil following liming, but is the result of increased calcium concentration of the soil solution, which in turn changes the ionic ratios within the plant root. A. G. POLLARD.

Influence of silicic acid, peat, and humus on the solubility and intake of phosphates [by plants]. W. JESSEN and W. LESCH (Z. Pflanz. Düng, 1930, 17A, 48— 66).—Both colloidal silica and peat increase the solubility of dicalcium phosphate in water, but the effects are modified by the presence of other fertiliser salts. In sand cultures silica and peat increased the phosphate assimilation of plants only when phosphorus was supplied in a difficultly soluble form or in mixed fertilisers tending to render the phosphate insoluble. Peat increased the crop produced by dicalcium phosphate in physiologically alkaline nutrients. In the presence of peat silica was ineffective. With physiologically acid nutrients neither peat nor silica increased the crop yields. Peat and silica increased the efficiency of tricalcium phosphate in all cases examined, crop increases being the more marked in physiologically alkaline media. A. G. POLLARD.

Nutrient intake and dry matter production by buckwheat (Fagopyrum esc.) and the yield law of higher plants. K. MAIWALD (Z. Pflanz. Düng., 1930, 17A, 12-42).-Growth curves for buckwheat in pot cultures are recorded. The rate of dry matter production in leaves, stems, and roots and the corresponding intake of nitrogen and calcium are examined. The rate of calcium intake follows closely the total production of dry matter. The nitrogen intake is relatively more rapid and is closely paralleled by the dry matter production of leaves and roots. Uniform relationships, which are expressed in S-form curves, exist between the period of growth and the total dry matter of the plant, the total nitrogen intake, total leaf surface, dry weight of leaves and of roots, nitrogen intake of leaves and stems, and the calcium intake of stems.

A. G. POLLARD.

Influence of colloidal silica on the assimilation [by plants] of the phosphoric acid of rock phosphates. A. REIFENBERG (Z. Pflanz. Düng, 1930, 17A, 1—11).—The citric solubility and assimilability of rock phosphates depend on the degree of dispersion of the particles. The ease of formation of aggregates (probably by perikinetic coagulation) in aqueous suspensions increases with the fineness of division. The formation of aggregates is largely prevented by colloidal silica, and stable silica-phosphate sols can be obtained. The favourable influence of silica on the assimilation of phosphates by plants is attributed to this effect.

A. G. POLLARD.

Influence of nutrition on the size distribution of potato starch grains. G. BREDEMANN and O. NER-LING (Z. Pflanz. Düng., 1930, 16A, 331—341).—The average size of starch grains from potatoes fertilised with nitrogen is less than those from unfertilised crops, the difference being greatest before the dying back of the potato haulm. The effects of potash and phosphate fertilisers are smaller. Results vary with the nature of the fertiliser used and with the time of its application, the general tendency being to reduce the average grain size. A. G. POLLARD.

Tomato, a sensitive and rapid indicator of the phosphorus-poverty of soil. L. MEYER (Fortschr. Landw., 1929, 4, 684-686; Chem. Zentr., 1930, i, 887).—Lack of phosphorus is detectable 14 days after sowing; the method gives results in accord with those of Neubauer and of von Wrangel.

### A. A. ELDRIDGE.

Toxicity of sulphur to spores of Sclerotinia cinerea as affected by the presence of pentathionic and other sulphur acids. O. N. LIMING and H. C. YOUNG (J. Agric. Res., 1930, 40, 951-962).—Comparative tests of the toxicity of sulphuric, sulphurous, di-, tri-, and penta-thionic acids to spores of Sclerotinia cinerea are recorded. The last-named acid alone showed 736

any marked toxicity. Filtrates from wetted sulphur contained pentathionic acid and were toxic. Sulphur freed from pentathionic acid was not toxic, but regained its toxicity and pentathionic acid content after aeration. In field tests sulphur to which oxidising agents were added (e.g., permanganates) was more effective than ordinary sulphur in controlling apple scab.

### A. G. POLLARD.

Factors concerned in arsenical injury to foliage. S. F. POTTS (J. Econ. Entomol., 1930, 23, 469-470).-Moisture accumulating on leaf surfaces contains appreciable amounts of carbon dioxide and weakly acid matter derived from the leaves. The solubility of lead and calcium arsenates in such solutions is greater than in distilled water which is used in laboratory tests of solubility. Field trials show calcium arsenate to be more soluble in leaf moisture than the lead compound. Lime used in conjunction with lead acetate in sprays tends to form calcium carbonate and arsenate. On foliage, rain quickly removes the carbonate and some arsenate and the leaf surface again becomes acid with an increase in the proportion of soluble arsenic. Some cases of the protective action of lime used in lead arsenate sprays may be ascribed to such formation of soluble arsenate and its removal by rain. A. G. POLLARD.

Pyrethrum and soap, a chemically incompatible mixture. R. C. ROARK (J. Econ. Entomol., 1930, 23, 460—462).—The use of soap in pyrethrum sprays leads to partial saponification of the pyrethrins I and II and consequent loss of insecticidal value. This is minimised by the use of the least necessary amount of neutral soap and preparation in the cold. Saponin and other spreaders are recommended as substitutes for soap.

A. G. POLLARD.

Petroleum products. CARPENTER.—See II. Solubility of the phosphoric acid of monocalcium phosphate. Stollenwerk.—See VII. Sugar-beet structure and sugar content. Artschwager.—See XVII. Ash of cows' milk. SCHNECK and GÖRGEL.— See XIX.

### PATENTS.

Production of mixed fertilisers. F. Jost (B.P. 312,169, 18.5.29. Ger., 21.5.28. Addn. to B.P. 306,046; B., 1930, 611).—In a modification of the prior process, a mixture of nitric acid with an excess of calcium phosphate and one or more alkali chlorides is distilled in a current of air and, if desired, under reduced pressure; the vapours evolved are absorbed by ammonia, sodium hydroxide, milk of lime, etc. to yield non-hygroscopic, nitrogenous fertilisers; the residue, consisting of calcium phosphates and alkali nitrates and chlorides, is used, after removal of insoluble constituents, for the production of mixed fertilisers. L. A. COLES.

Manufacture of salt mixtures suitable for use as fertilisers containing carbamates or conversion products of carbamates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,326, 15.2.29).—The carbamates obtained by the process described in B.P. 328,005 (B., 1930, 612) are not separated from the solution, but are worked up, with or without conversion into other products, together with the salts formed during their production; e.g., a mixture of potassium carbamate and

ammonium nitrate is obtained by passing carbon dioxide into a solution of potassium nitrate in liquid ammonia and subsequently removing excess ammonia by evaporation. L. A. COLES.

Manufacture of fertiliser salts. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,641, 16.1. and 2.5.29).—The salts are formed by the double decomposition, in the presence of liquid or gaseous ammonia free or practically free from water, of two or more salts of strong mineral acids which are at least slightly soluble in liquid ammonia. The products may be recovered together by evaporation of the ammonia or, where practicable, may be separated by filtration before the evaporation. In an example gypsum is stirred for about 6 hrs. at the ordinary temperature with a mixture of liquid ammonia and ammonium nitrate, and ammonium sulphate and calcium nitrate are recovered by filtration and by evaporation of ammonia from the filtrate, respectively. L. A. COLES.

Manufacture of fertiliser salts containing potash and nitrogen or potash, nitrogen, and phosphorus. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,883, 1.7.29).—Potassium carbamate, alone or mixed with other substances, is treated with acids (sulphuric, nitric) or acid salts; residual acids, if present, are neutralised, preferably with ammonia.

L. A. COLES.

Utilisation [as manure] of the final liquors in the potassium industry. KALI-FORSCHUNGS-ANSTALT G.M.B.H. (B.P. 329,495, 6.5.29. Ger., 6.12.28).—The liquors mixed with dilute phosphoric acid or phosphate solutions are treated with sufficient ammonia to neutralise the acid; magnesium ammonium phosphate is precipitated, removed, washed, and dried at about 100° to yield a fertiliser containing  $45 \cdot 7\%$  P<sub>2</sub>O<sub>5</sub>, 9%N,  $25 \cdot 9\%$  MgO, and  $11 \cdot 6\%$  H<sub>2</sub>O, which may be mixed with ammonium nitrate. The liquor is heated with lime to recover the ammonia contained in it. L. A. COLES.

[Dusting-powders for] disinfecting seeds. M. ENGELMANN, ASST. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,749,575, 4.3.30. Appl., 29.10.26).—The powders comprise mixtures of copper carbonate or other basic copper salts with acids (e.g., phosphoric, boric, benzoic, benzenesulphonic, oxalic, tartaric acid), with acid salts (e.g., sodium bisulphate, calcium hydrogen phosphate), or with salts having an acid reaction (e.g., copper or ferrous sulphate, zinc chloride). The mixtures may be prepared by grinding a copper salt, e.g., copper sulphate, with a base, preferably magnesium oxide, in quantity insufficient to convert the salt completely into the basic salt. L. A. COLES.

Adhesive compositions for catching insects. I. G. FARBENIND. A.-G. (B.P. 306,906, 26.2.29. Ger., 27.2.28).—The products obtained by exhaustive chlorination, first at room temperature and then on a waterbath, of vegetable or animal oils or fats (linseed, wood, tall, fish oils), after the remaining hydrochloric acid has been removed with a stream of air and later of a little ammonia gas, are mixed with resins, waxes (carnauba wax), and machine oil. L. A. COLES.

Immunising grain (B.P. 304,741).-See IX.

Structure of sugar beet in relation to sugar content and type. E. ARTSCHWAGER (J. Agric. Res., 1930, 40, 867—915).—Numerous data concerning the anatomical structure of sugar beet are recorded and discussed in relation to the sugar content. A. G. POLLARD.

Preliminary investigations on two cellulosic wastes as sources of xylose. W. C. HALL, C. S. SLATER, and S. F. ACREE (Bur. Stand. J. Res., 1930, 4, 329-343).-An attempt is made to extract crystalline xylose or xylose molasses from peanut hulls or cottonseed bran without materially injuring the plant fibre for use by the cellulose technologist. Both of these cellulosic wastes are rich in furfuraldehyde-forming materials, but, so far, crystalline xylose has been obtained only from cottonseed bran. The process, which has been employed on a semi-commercial scale, consists in heating the material with water under pressure to remove soluble gums which interfere with the crystallisation, followed by treatment with cold 0.12Nsulphuric acid and washing with water, whereby a large proportion of mineral matter is removed. Finally, the xylose is extracted by cooking with 0.16N-sulphuric acid under pressure, and the extract is decolorised, evaporated, neutralised with baryta, and allowed to crystallise. F. R. ENNOS.

Purification of potato and maize syrups by activated carbon ("Norit"). W. TAEGENER (Z. Spiritusind., 1930, 53, 155-157).-Bone charcoal for the purification and decolorisation of the syrups has been replaced by an activated carbon, termed "Norit" (special glucose quality), which is prepared in the form of a powder by the carbonisation of certain plant materials. It is added direct to the syrup solution at 70-75° and is stirred vigorously for 20 min. prior to being separated in the filter-press, through which it allows, relatively to bone charcoal, the syrup to pass more freely even at high concentrations. The desired standard colour can be controlled by the amount of carbon added, and as its power of adsorption is much higher than that of bone charcoal, a relatively small quantity is required. Prefiltration of the syrup is rendered unnecessary by its use, and it does not require frequent reactivation since the filter runs longer before the carbon 18 completely exhausted. Generally, 0.7-1.4% of active carbon is required, when calculated on a syrup of d 1.43. The preparation of maize syrup differs from that of potato syrup, and previous to the treatment with carbon, oil and excess proteins must be removed from the maize wort. In addition, as the carbon is more active in acid solution, the maize wort is partly neutral-1sed. Reactivation of the carbon is effected by alternate extraction with dilute hydrochloric acid and sodium hydroxide solutions followed in each case by washing with water. After drying, the carbon has 50-60% of its original activity. C. RANKEN.

Solvents for gum arabic. R. TAFT and L. MALM (Trans. Kansas Acad. Sci., 1929, 32, 49-50).—Gum arabic (purified by precipitation from aqueous solution with alcohol) dissolves in 1.7 pts. of water at  $25^{\circ}$ , 1.6 at  $50^{\circ}$ , or 1.5 at  $90^{\circ}$ . Gum arabic is very sparingly

soluble in solvents other than water and certain aqueous mixtures. CHEMICAL ABSTRACTS.

Determination of water. LEPPER.—See VII. Chlorine in soils [for sugar beet etc.]. ARRHENIUS.— See XVI. Lactose in milk. Gohr. Determining sugar in milk. MÜNCHBERG. Sucrose and lactose of milk-chocolate. RINCK and KAEMPF. Effect of heat on sugars in chocolate. LECOQ.—See XIX.

### PATENTS.

Production of gum from locust beans. A. MÜLLER, ASST. to S. NEUMAN (U.S.P. 1,749,833, 11.3.30. Appl., 28.11.28. Ger., 8.10.28).—A solution in cold water of a mixture of the ground beans, freed from skin and seeds, and alkylated (methyl) naphthalenesulphonic acid is boiled by direct or indirect heating with steam.

L. A. Coles.

**Digestor** (B.P. 329,600). **Refractometers** (B.P. 314,994).—See I. **Esters of carbohydrates** (B.P. 305,661).—See III.

### XVIII.—FERMENTATION INDUSTRIES.

**Research work in the yeast field.** L. H. LAMPITT (J. Inst. Brew., 1930, 36, 250–260).—The problems surrounding the life and activities of yeast are critically reviewed and a plea is made for more work on standardised lines on simple substances. C. RANKEN.

Boiling of worts. R. WORSSAM (J. Inst. Brew., 1930, 36, 260-265).-The relative advantages of boiling worts by steam-heating apparatus in the boiling vessel or by direct fire-heating beneath it are discussed. The annual depreciation of a steam-heated copper is less than that of the fired coppers, and for economy in the consumption of coal the former has an enormous advantage. Steam-jacketed vessels have the serious drawbacks that a central boil is not attained, nor does the violent upheaval of the wort occur, which is essential for circulation and aeration. In addition, the inner and outer pans are liable to leak at the joints between the two. With the modern steam heater, which is placed in the centre of the vessel, a central boil and vigorous circulation of the liquid are assured. The use of superheaters for raising the temperature of the steam supply to the coppers has no economic value unless the heat is drawn from sources which otherwise would be lost. It is essential that the steam-heating apparatus be served by a steam trap able to remove the condensed water rapidly. No material differences could be traced between the worts boiled by the two methods, and, if caramelisation did occur in a firecopper, it could only do so by the overheating of the bottom of the vessel with consequent damage.

C. RANKEN.

Titrimetric determination of carbon dioxide in beer. J. DE CLERCK (Bull. trim. Assoc. anc. Elèves Ecole Brass. Louvain, 1929, 29, 30—37; Chem. Zentr., 1930, i, 300).—A modification of Cannizzaro's method consists in the use of sodium hydroxide for absorption and the titration of the solution to the colour of phenolphthalein in a buffer solution of  $p_{\rm H} 8.4$ .

A. A. ELDRIDGE.

Detection of fruit wine in grape wine by means of the sorbitol method. G. REIF (Z. Unters. Lebensm.,

1930, 59, 99-104).-The precipitation of dibenzylidenesorbitol in Werder's test (cf. B., 1929, 619) is largely prevented by lævulose, sucrose, invert sugar, starch sugar, dextrin, etc., but not by dextrose. After fermentation for 10 days with pure wine yeast the inhibitory influence of the sugars is diminished except in the case of dextrin, in the presence of which the test is completely negative. In this case the distillate residue should be extracted with hot absolute alcohol and the extract tested after complete removal of the alcohol. In performing the test it is recommended that the viscous distillate be diluted with a few drops of water and warmed, the optimum quantities of the reagents being 0.2-0.25 c.c. of benzaldehyde and at least 1 c.c. of sulphuric acid (1:1). Experiments with grape wine containing various percentages of sweetened cider are described. H. J. DOWDEN.

## PATENTS.

Manufacture of malt. O. SLEEMAN (B.P. 329,574, 30.7.29).—The apparatus, which is rotatable on a horizontal axis, consists of a container which is divided by perforate partitions into two outer compartments constituting air spaces, and an inner compartment comprising a germination and dispersion chamber in free communication with each other. To secure a flow of air through the germination chamber, each air space is provided with an air pipe and door, so that the one air space acts as a supply and the other as an exhaust for the air and gases. The direction of flow may be reversed. Slow rotation of the container causes the grain to fall from the germination chamber to the dispersion chamber and back to the germination chamber.

C. RANKEN.

Automatic fermentation apparatus. A. P. HARRI-SON, ASST. to STANDARD BRANDS, INC. (U.S.P. 1,750,267, 11.3.30. Appl., 9.6.27).—The apparatus for the production of yeast by an addition process consists of a series of fermenters at progressively lower levels, each of which has approximately double the capacity of that preceeding. Connecting each fermenter to the next is a pipe in the form of a  $\cap$ , which controls the maximum level of the liquid. The capacities of the tubes are sufficient to empty the fermenters to which they are attached in the same period of time. Yeast nutrients may be supplied independently to each fermenter, and valves fitted to the lead-off tubes permit the removal of an aliquot portion of the liquid. C. RANKEN.

Apparatus for pasteurising beer and other liquids. R. F. GRAHAM (B.P. 329,733, 8.3.29).—Liquid containers are automatically fed on to a perforated, rotatable carrier provided with a fixed guide which spirals round the carrier. The moving containers are sprayed with pasteurising liquid from a tank with perforated bottom placed above the carrier. The tank is divided into 5 co-axial compartments each of which supplies pasteurising liquid, the temperatures of which rise gradually from the innermost compartment outwards. A similarly divided tank is placed under the carrier to collect the pasteurising liquid, which, after heating, is returned to the upper tank. C. RANKEN.

Apparatus for obtaining yeast. O. LÜHRS, Assr. to Zellstoff-FABR. Waldhof (U.S.P. 1,760,706, 27.5.30. Appl., 29.2.28. Ger., 12.3.27).—See B.P. 287,052; B., 1928, 797.

Manufacture of yeast. A. P. HARRISON, ASST. to STANDARD BRANDS, INC. (U.S.P. 1,761,789, 3.6.30. Appl., 6.10.25).—See B.P. 259,572; B., 1927, 666.

Electrophoretic deposition of solids (B.P. 305,630 and 307,912).—See XIV.

## XIX.—FOODS.

Detection of hard wheat grits. E. BERLINER and R. RÜTER (Z. Getreidew., 1929, 16, 212-213; Chem. Zentr., 1930, i, 606).—A criticism of Kühl's test. A. A. ELDRIDGE.

Detection of hard wheat grits. H. KÜHL (Z. Getreidew., 1929, 16, 213-216; Chem. Zentr., 1930, i, 606).—A reply to Berliner and Rüter (preceding abstract) and statement of exact conditions.

A. A. ELDRIDGE.

Xylene value [of adulterated butter]. E. HANKE and F. DEUTSCHMANN (Milchwirt. Forsch., 1929, 8, 525-532; Chem. Zentr., 1930, i, 607).—The minimum permissible value in van Raalte's xylene test is 19:0. A. A. ELDRIDGE.

**Freshness of milk.** K. L. PESCH and U. SIMMERT (Milchwirt. Forsch., 1929, 8, 551–575; Chem. Zentr., 1930, i, 607).—A simple indicator method for the determination of the  $p_{\rm H}$  of milk is described. Resazurin is added to the milk, which is then kept at 20°, the resistance to reduction to a red shade being a measure of the freedom from bacteria. A. A. ELDRIDGE.

Viscosity of milk and milk products. I. Skim milk, full-cream milk, and cream. II. Fresh and treated milk and cream. W. MOHR and F. OLDENBURG (Milchwirt. Forsch., 1929, 8, 429-461, 576-592; Chem. Zentr., 1930, i, 765-766).—I. Considerable differences were apparent in the results obtained with different forms of apparatus. The Lawaczeck viscosimeter is preferred.

II. Determinations were made with naturally or artificially acidified milk. Ageing affects the state of aggregation of the fat. A. A. ELDRIDGE.

Electrical conductivity of milk. II. Results of practical measurements on the milk of cows from different herds. J. KRENN (Z. Unters. Lebensm., 1930, 59, 32-62; cf. B., 1929, 574).-Observations on herds of several hundred cows and on individual animals have afforded further evidence that a high value for the electrical conductivity is indicative of abnormal secretion due to diseased udders. Suspected cases were almost invariably confirmed by bacteriological and clinical examinations. Although considerable variations occur between different animals, the value of the electrical conductivity for normal milk should not exceed 46  $\times$ 10<sup>-4</sup>. As the value of  $\kappa$  rises so does the sodium chloride content, but the values for d, solids-not-fat, and refraction all decrease. The limits within which normally secreted milk may vary are given as  $d \cdot 1.0310 - 1.0360$ , fat-free solids 8.80—over 10%, and refraction 39.0 over 41.0. The lower values frequently quoted are ascribed to milk from sick cows. On the other hand, the lowering of f.p. remains practically constant, even

when the composition of the milk is so abnormally low as to suggest that water has been added.

H. J. DOWDEN. "Alkali number" of the ash of cows' milk, with special reference to fodder and the lactation period. A. SCHNECK and B. GÖRGEL (Milch. Zentr., 1930, 59, 49-53, 65-70, 81-85, 97-101).-The results hitherto recorded for the potash, soda, and lime contents of milk ash have shown considerable variation, especially in the sodium value, e.g., 3.85-13.02%, resulting in wide differences in the "alkali number" (ratio K<sub>2</sub>O/Na<sub>2</sub>O). The earlier values for this ratio range from 2 to 4, but the more recent work of Nottbohm (cf. Milch. Forsch., 1927, 4, 336) gives an average value of 6 (max. 10). Further investigation has been made on the milk of stall-fed cows which received special diets, normal, rich, and poor in mineral matter. The perchlorate method of analysis was used throughout, and was found to give results in good agreement with those obtained by the platinichloride method. Mixed milk from groups of 10 cows in full milk gave alkali numbers of  $2 \cdot 2 - 2 \cdot 4$ , whereas colostral milk gave values of  $1 \cdot 2 - 1 \cdot 5$ , and was characterised by a low potassium content. The results obtained with different animals showed considerable variation : thus the calcium value in one case was 25-30% higher than the average. Observations were made on two animals over a period of 230 days in order to study the changes during lactation. After 4 days the composition was normal, and thereafter the potassium value slowly decreased, the sodium value remaining more or less unchanged. The influence of fodder and of supplementing the diet with added mineral matter by dosing the cows with carbonates of potassium, sodium, or calcium was inconclusive, and appeared to depend on the physiological requirements of the animals, since during the period of feeding with fodder of low mineral content the highest individual value for the alkali number, viz., 4.7, and the highest sodium content, viz., 7.4, were recorded. No significant change in the calcium content was observed during the lactation period.

### H. J. DOWDEN.

Titrimetric determination of lactose in milk. H. GOHR (Z. Unters. Lebensm., 1930, 59, 90-94).-The method is based on the reduction of potassium ferricyanide in the presence of sodium carbonate, followed by iodometric determination of residual ferricyanide. Fat and albumin are removed from 5 c.c. of milk by adding 1 c.c. each of potassium ferrocyanide solution (150 g./litre) and zinc sulphate solution (300 g./ litre). The mixture is made slightly alkaline with caustic soda, diluted to 100 c.c., and, after 15 min., the precipitate is filtered off. A portion of the clear filtrate (2-6 c.c.) is diluted to 20 c.c. and treated with 10 c.c. of potassium ferricyanide solution (16.46 g., together with 70 g. of anhydrous sodium carbonate in 1 litre). After heating on a water-bath for 20 min., and then cooling, 10 c.c. of potassium iodide-zinc sulphate solution (50 g. of zinc sulphate, 25 g. of potassium iodide, and 250 g. of sodium chloride in 1 litre) and 10 c.c. of 9% acetic acid are added, iodine being then titrated with 0.05N-sodium thiosulphate. After deducting the value for a blank test, the lactose content is read from tables derived from a solution of pure lactose, using the modification of the Hagedorn-Jensen method (cf. A., 1923, ii, 265) described by Issekutz and Both (cf. A., 1927, 600). H. J. DOWDEN.

Determination of chlorine and sugar in small quantities of milk. F. MÜNCHBERG (Milch. Zentr., 1930, 59, 161-163).-In determining chlorine in milk, 2 c.c. of halogen-free nitric acid (d 1.40), 1 c.c. of 0.1N-(or 5 c.c. of 0.02N-) silver nitrate, and 1 c.c. of the sample are heated together to boiling. Then 10% potassium permanganate solution is added drop by drop to the boiling liquid till it becomes clear. The liquid is cooled by dilution with 30 c.c. of distilled water and, after addition of 0.5 c.c. of saturated iron ammonium alum solution, the excess of silver is titrated with 0.02Nammonium thiocyanate. (1 c.c. of 0.02N-silver nitrate  $\equiv 0.0007092$  g. Cl.) In determining the sugar content, 10 c.c. of the sample are shaken well with 1 c.c. of 20% trichloracetic acid for several minutes and filtered clear through a dry filter. The rotation observed in the Zeiss pocket polarimeter multiplied by  $2 \cdot 2$  gives the percentage sugar content. The results are slightly lower than those obtained by the iodometric and gravimetric methods of determining sugar. W. J. BOYD.

Formulæ for the direct calculation of the sucrose and lactose content of milk-chocolate. A. RINCK and E. KAEMPF (Z. Unters. Lebensm., 1930, 59, 81-90). -The method previously employed for the determination of starch syrup and sucrose in marmalade (cf. B., 1922, 191A) has been applied to the determination of sugars in milk-chocolate. A sample (10 g.) is freed from fat with chloroform-ether, and, after drying, the residue is extracted with water containing ammonia and lead acetate to precipitate albumin. The polarisation of the extract is measured before and after Clerget inversion, and for the purpose of determining the total sugar content by the method of Welmans and Steinmann the polarisation of 10 g. of chocolate, subsequently freed from fat, dried, and dissolved in 100 c.c. of water, is also measured. The following formulæ have been derived, in which polarisation value = P, provisional total sugar = S. [Owing to the difference between the polarisation units for sucrose and lactose (1.33 and 1.051, respectively), a table of factors has been calculated for use in deducing the total sugar content.] Thus, (i) P before inversion/factor = S; (ii) S/1.59 = volume of total sugar; (iii) [S/(100 - volume)] 1000 = actual total sugar (%); (iv)  $[(S \times 0.43) + P \text{ after inver-}$  $sion]/0.1481 = provisional lactose (%); (v) [(S \times 1.051) - P after inversion] 0.1481 = provisional$ sucrose (%); (vi) 10S: actual total sugar (%) = provisional lactose (%): actual lactose (%). The percentage sucrose is given by the difference between total sugar and the percentage of lactose. Values for milk solids and cacao can also be derived. H. J. DOWDEN.

Effect of heat on the sugars entering into the composition of chocolate pastes. R. LECOQ (J. Pharm. Chim., 1930, 11, [viii], 522-529).—Heating of chocolate pastes at temperatures ranging from 50° to 120° causes a distinct caramelisation of the sugar, which is expressed by an increase in the proportion of reducing sugars at the expense of the initial sucrose. This caramelisation

depends on the duration of treatment and the temperature reached. It is produced to a small extent in cooking chocolate, but it is in overheated chocolate fondants that it is mostly shown, when the inverted sugar may amount to 25% of the total sucrose.

B. W. Town.

Soya-bean extract residues from different processes. P. MALKOMESIUS and W. SCHRAMM (Landw. Versuchs-Stat., 1930, 110, 33-47).—Examination of various samples of soya-bean cakes shows that the method of production does not affect the digestibility of the product. Differences in the digestible protein contents of various samples are traceable in part to differences in the original material and in part to the extraction method. The fat and lecithin contents are entirely controlled by the process used. A. G. POLLARD.

Pollen analysis of honey. I. C. GRIEBEL (Z. Unters. Lebensm., 1930, 59, 63—79).—To assist in the detection of imported honey the forms and structures of a large number of different types of pollen, both native and foreign, are described and illustrated by photomicrographs. H. J. DOWDEN.

Deterioration and spoilage of honey in storage. G. E. MARVIN (J. Econ. Entomol., 1930, 23, 431–438).— Granulation of honey in storage favours fermentation by sugar-tolerant yeasts with the production of carbon dioxide, alcohol (rarely > 5%), and a non-volatile acid. Such fermentation may be prevented by heating to 71°, sealing, and cooling rapidly to avoid discoloration.

A. G. POLLARD.

Methods used to detect heated honeys. G. H. VANSELL and S. B. FREEBORN (J. Econ. Entomol., 1930, 23, 428—431).—The Fiehe test for heated honey is unreliable since small amounts of hydroxymethylfurfuraldehyde may be produced without heat, especially in solidified samples. The diastatic activity of honey depends on the amount of pollen present, and, unless a simultaneous pollen count is made, is insufficient as a test of heating. A. G. POLLARD.

Viscosity of pectin sols. II. Effects of citric acid and various sugars. A. OHN (Ind. Eng. Chem., 1930, 22, 635-639; cf. B., 1927, 91).-The effect of citric acid, dextrose, lævulose, sucrose, or lactose on the viscosity and gelation of non-heated pectin sols has been investigated. Citric acid decreases the viscosity of pectin or pectin-sucrose sols, whilst the carbohydrates increase the viscosity of pectin sols ; sucrose has the greatest effect. The pectin-acid-carbohydrate jellies containing sucrose are firmer and more stable than those containing dextrose, lævulose, or lactose. Crystalline formation appears at a lower hydrogen-ion concentration in sucrose than in dextrose or lactose jellies, whilst under similar conditions in lævulose jellies no crystalline formation occurs. L. S. THEOBALD.

Determination of water. LEPPER.-See VII.

### PATENTS.

Manufacture of bread. J. MATTI, Assr. to Aug-MENTINE HOLDING Soc. ANON. (U.S.P. 1,750,720, 18.3.30. Appl., 5.4.28. Ger., 21.4.27).—An emulsion of paraffin oil (55%), glycerin (35%), and water (10%) is added to a bread dough in the proportion of 0.3% of the flour weight. The process is claimed to improve the quality of the bread and to give an increased yield.

E. B. HUGHES.

Production of bakery goods, pastries, confectionery, etc. H. BOLLMANN and B. REWALD (B.P. 328,075, 23.2.29).—In the preparation of such products, egg yolk and the whole or part of the fat normally employed may be replaced by a mixture comprising food fat and an amount of lecithin (from soya bean) not less than 5% of the fat. E. B. HUGHES.

Self-preserving [condensed] milk product. T. O'CONNOR (U.S.P. 1,749,153, 4.3.30. Appl., 8.2.26).— The product, suitable for use in bakeries, comprises 7— 14% of sodium chloride, not less than 28% of milk solids, about 20% of dextrose, and not less than 25% of water. L. A. COLES.

Flavouring tea. L. ARONSON and A. FELDBLET (U.S.P. 1,750,768, 18.3.30. Appl., 21.12.27).—Tea is impregnated with citric acid solution and dried, then exposed for  $\frac{1}{2}$  hr. to the vapours of essential oils (lemon, orange, etc.) diss\_lved in alcohol. After redrying it is blended with an equal weight of untreated tea.

E. B. HUGHES.

Treatment of seaweed. A. CARTIER and P. GLOESS (F.P. 633,696, 8.9.26).—Seaweed is heated with a suspension of magnesia in water, whereby the unpleasant odour and taste is removed. The product is suitable for the manufacture of foodstuffs and electrical insulators. A. R. POWELL.

Drying apparatus (U.S.P. 1,749,451). Mixing machine (U.S.P. 1,745,993). Congealing of liquid substances. (B.P. 329,129).—See I. Table salt (B.P. 312,088).—See VII. Vitamin-A (B.P. 306,881). —See XX.

XX.-MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Tests for aloes. A. H. WARE (Pharm. J., 1930, 124, 596-597).—The colour reactions described by Schorn (B., 1930, 485) are unsatisfactory. Details of improvements in tests previously described by the author (A., 1925, i, 1122) are given.

H. E. F. NOTTON.

Oil from cascara sagrada. A. JERMSTAD (Pharm. Acta Helv., 1929, 4, 90—98; Chem. Zentr., 1930, i, 845).—When the residue from the alcoholic extraction of cascara sagrada is distilled with steam an oil is collected which has  $d^{15}$  0.8923,  $n_D^{20}$  1.4568, acid value 3.7, saponif. value 189.4, iodine value 86.2. It contains palmitic, oleic, linoleic, and linolenic acids; glycerol is not present. The unsaponifiable matter contains docosane and an oil, b.p. (?) 285–289° or (?) 185–189°,  $n_D^{10}$  1.4901. A. A. ELDRIDGE.

New constituent of peppermint oil. H. CARLES (Parf. mod., 1930, 22, 615–637; Chem. Zentr., 1930, i, 759).—Peppermint oil from the 1927 crop had an exceptionally high density (0.915) and  $\alpha_{\rm D}$  up to  $-10^{\circ}$ ; it contained up to 10% of a constituent having  $d^{15}$  0.965,  $\alpha_{\rm D}$  + 81°,  $n_{\rm D}^{30}$  1.4807, b.p. 95°/20 mm. or 196°/atm. pressure. A. A. ELDRIDGE.

Portuguese petitgrain oil. L. S. GLICHITCH and V. R. NAVES (Parfums de France, 1929, 7, 223-228; CL. XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Chem. Zentr., 1930, i, 604).—Sweet orange (branch) oil from Algeria,  $d^{15}$  0.8585,  $\alpha_{D}^{203} + 40^{\circ}$  55',  $n_{D}^{20}$ 1.4726, contained 2.33% of esters (as linalyl acetate), together with palmitic and geranic acids, guaiacol (?), methyl methylanthranilate and anthranilate, pyrrole, furfuraldehyde, citral, pinene, dipentene, *d*-limonene, a terpene,  $d^{15}$  0.8274,  $n_{D}^{20}$  1.4812,  $\alpha_{D}$  +5°42', *d*-linalool, *d*-terpineol, geraniol, nerol, acetic and higher fatty acids, bisabolene (?), farnesol (?), and only traces of camphene. A. A. ELDRIDGE.

Philippine eucalyptus oil. A. P. WEST and H. TAGUIBAO (Philippine J. Sci., 1930, 42, 1—11).— The oil from several species of eucalyptus grown in the Philippines has been examined. The highest yields of oil  $(3\frac{1}{2}-5\%)$ , which were greater than those from the same species grown in Australia) were obtained from *E. globulus, tereticornis, polyanthemos,* and *citriodora*. For reliable comparison the yields of oil (which is almost entirely located in the leaves) should be calculated on a moisture-free basis. The constants and cineol content of the oil of *E. globulus* compared favourably with the corresponding figures for Australian oil from the same species. E. LEWKOWITSCH.

Composition of the oil of Dracocephalum Moldavica, L. B. N. RUTOVSKI and I. V. VINOGRADOVA (Riechstoffind., 1929, 4, 137–138; Chem. Zentr., 1930, i, 758).—The oil (yield 0.01-0.17%) has  $d^{20}$  0.9038-0.9491,  $\alpha_D - 5.92\%$  to 0%,  $n_{25}^{25}$  1.4764–1.4874, acid value 2.32-18.64, ester value 34.64-170.33, ester value after acetylation 204.0-288.11, aldehyde content 25-68%. Citral, thymol, geraniol, nerol, an aldehyde, m.p. 60-62%, a terpene (? limonene), and a monocyclic sesquiterpene are present.

A. A. ELDRIDGE.

Determination of allylthiocarbamide in black mustard. R. MEESEMAECKER and J. BOIVIN (J. Pharm. Chim., 1930, [viii], 11, 478-784).-5 G. of finely-powdered mustard (Brassica nigra) and 100 c.c. of water are heated in a closed flash on a water-bath at 68° for 1 hr.; 100 c.c. of ammonia solution are then added and the mixture is kept for 15 min. The solution is cooled, made up to 200 c.c. with distilled water, 20 c.c. of Courtonne's reagent are added, and, after shaking, the liquid is filtered. 100 C.c. of the filtrate are neutralised, then acidified with 5 c.c. of 0.5N-sulphuric acid, 10 c.c. of 0.1N-iodine are added, and the mixture is set aside for 15 min. After the addition of 10 c.c. of benzene, the excess of iodine is titrated against 0.05N-thiosulphate. The process is based on the fixation of iodine by thiosinamine. (1 C.c. of 0.1N-iodine is equivalent to 0.0058 g. of thiosinamine and 0.00485 g. of allylthiocarbamide.) The maximum of allylthiocarbamide is obtained by maceration at  $68^{\circ}$  and the above method allows the production to be followed under varying conditions. This has been done with different varieties of mustard. For an "oil-free" meal the optimum temperature for maceration to obtain the highest yield of essence is 40°. E. H. SHARPLES.

## PATENTS.

Manufacture of stable emulsions of paraffin. E. GLÜCKSMANN, Assee. of A. MÜLLER (G.P. 446,598, 15.7.25. Cf. B.P. 255,456; B., 1927, 892).—Emulsions having the consistency of a soft fat and stable up to 40° are obtained by the process disclosed in the B.P.; they may be flavoured by addition of essences to the water in which the paraffin is emulsified.

A. R. POWELL.

Preparing vitamin-A in free or combined condition. T. SHIMIZU (B.P. 306,881, 18.2.29).—The sterol-free unsaponifiable matter of substances rich in vitamin-A is treated with suitable bile acids, which form a crystalline vitamin-A-choleic acid complex  $(C_{24}H_{40}O_4)_5, C_{27}H_{44}O_2, H_2O$ , m.p. 179°. This compound is stable towards oxygen and insensitive to light; on treatment by the usual methods, it is decomposed into choleic acid and crystalline vitamin-A,  $C_{27}H_{44}O_2, H_2O$ , m.p. 187°. The above operations are preferably carried out in absence of oxygen.

E. H. SHARPLES.

Manufacture of the hormone of the anterior lobe of the hypophysis. SCHERING-KAHLBAUM A.-G. (B.P. 329,605, 14.10.29. Ger., 13.10.28).—Lime is removed from the urine of pregnant warm-blooded animals by treatment with a soluble oxalate and, after removal of the precipitate, the hormone is precipitated from the solution in the usual manner. E. H. SHARPLES.

Preparation of compounds of alkaline-earth nitrites with methylxanthines. H. P. KAUFFMANN (B.P. 311,231, 2.5.29. Ger., 7.5.28).—Compounds useful as vascular agents are obtained by evaporating a solution of an alkaline-earth nitrite to which caffeine, or an alkaline-earth salt of theobromine or theophylline, or a mixture of alkaline-earth hydroxide and theobromine or theophylline, has been added. C. HOLLINS.

Manufacture of double compounds of the acridine series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,212, 18.1.29).—The double salts containing both 3:7-diaminoacridine salt (N = 5) and 3:7-diamino-N-alkylacridinium salt are much more soluble in water than is either component. Hot aqueous or alcoholic solutions of 3:7-diamino-N-methylacridinium chloride and 3:7-diaminoacridine hydrochloride are mixed and the double salt is salted out or recovered by evaporation; or the acridine base is added to the acridinium hydrogen sulphate in aqueous suspension at 60°. C. HOLLINS.

Manufacture of pharmaceutical products. W. SCHULEMANN, F. SCHÖNHÖFER, and A. WINGLER, ASSIS. to WINTHROP CHEM. Co., INC. (U.S.P. 1,760,781, 27.5.30. Appl., 16.12.27. Ger., 20.12.26).—See B.P. 282,453; B., 1929, 958.

Purified ethylene (U.S.P. 1,741,559).—See III. Lyophile products (B.P. 329,305). Emulsions (B.P. 329,266).—See XII.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Developers and development. I. Physicochemical basis of a rational composition for developer solutions. K. TSCHIBISSOV. II. Metolquinol and metoquinone developers. III. Effect of dilution of developer on photographic properties. K. TSCHIBISSOV and V. TSCHELZOV (Kinotechnik, 1929, 11, 227-231, 315-321, 373-376; Chem. Zentr., 1930, i, 473-474).—I. The relations between chemical constitution and developing power are considered, together with concentrations applicable to practice and the use of alkali hydroxide.

II. Substitution of sodium hydroxide for carbonate increases the developing power of quinol, but diminishes that of metol.

III. Variable results were obtained owing to hydrolysis, oxidation, inadequate diffusion, etc. Solutions of different concentrations do not behave regularly towards changes of temperature. A. A. ELDRIDGE.

Test for thiosulphate. BAINES.-See VII.

### PATENTS.

Preparation of light-sensitive layers. M. P. SCHMIDT and W. KRIEGER, Assrs. to KALLE & Co. A.-G. (U.S.P. 1,760,780, 27.5.30. Appl., 7.8.28. Ger., 6.9.27). --See B.P. 296,725; B., 1930, 303.

Desensitisation of silver bromide emulsions and plates or films covered with such emulsions. B. HOMOLKA, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,760,756, 27.5.30. Appl., 20.12.27. Ger., 15.6.27).— See B.P. 292,140; B., 1928, 875.

[Lenticular] films for colour photography. KODAK, LTD., Assees. of H. E. HASTINGS (B.P. 330,151, 21.6.29. U.S., 2.11.28).

Printing of kinematograph films. FILM OZA-PHANE Soc. ANON. (B.P. 330,024, 14.3.29. Fr., 24.1.29).

[Preparations of records for] coloured talkingpicture photography. BRIT. TALKING PICTURES, LTD., Assees. of L. DE FOREST (B.P. 319,779, 26.9.29. U.S., 28.9.28).

Cellulose ester waste (B.P. 311,368).—See V.

### XXII.-EXPLOSIVES: MATCHES.

#### PATENTS.

Explosive compositions. A. STETTBACHER (B.P. 312,316, 14.5.29. Switz., 24.5.28).—Plastic explosive mixtures for use especially for filling mines, torpedoes, and detonators comprise 70—90 pts. of pentaerythritol tetranitrate precipitated in a finely-crystalline form, and 30—10 pts. of nitroglycerin and/or glycol dinitrate (cf. B., 1929, 152, 998). L. A. COLES.

Composition rods igniting by friction [match substitutes]. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 329,796, 25.4.29).—The rods, which can be extinguished and re-ignited repeatedly, comprise an organic foundation yielding practically no ash, having a speed of combustion less than that of weakly nitrated cellulose, and containing, e.g., cellulose nitrate or acetate mixed with ammonium oxalate, camphor, naphthalene, etc., and an ignition mixture free from phosphorus, e.g. potassium chlorate and sulphur; binders, materials capable of regulating combustion, fillers, pigments, etc. may also be added. L. A. COLES.

Production of nitric acid (B.P. 329,427).-See VII.

# XXIII.—SANITATION; WATER PURIFICATION.

Red-water trouble and the remedy at West Palm L. O. WILLIAMS (J. Amer. Water Works' Beach. Assoc., 1930, 22, 791-795).-The presence in the finished water of 9 p.p.m. of dissolved oxygen and 5 p.p.m. of carbon dioxide caused severe corrosion in the iron mains which, owing to lack of systematic flushing, became coated with a soft mass containing ferrous carbonate, ferric hydroxide, and ferric oxide. Small portions of this material dislodged by the normal variations in the rate of flow were the cause of frequent complaints of red water. By the addition of lime, a protective coating of calcium carbonate has been deposited in the mains and, as the portions of rust scale which are not thus coated have proved to be much harder than formerly, the complaints have ceased. C. JEPSON.

Identification and determination of nitrates in water. G. BINI (Atti R. Accad. Lincei, 1930, [vi], 11, 593—596).—An examination of reagents suggested for the identification of nitrates in the presence of nitrites shows that quinolsulphonic acid, for which details of preparation are given, is the most suitable. This reagent gives with nitrates, according to concentration, colours varying from pale green to brown, and will detect 1 pt. of nitrate in  $10^5$  pts. of water. The presence of 20 pts. per  $10^5$  of nitrite is without influence. In this respect this reagent is to be preferred to pyrogallolsulphonic acid. F. G. TRYHORN.

### PATENTS.

Utilising [for water sterilisation] the oligodynamic action of metals and metal compounds. G. A. KRAUSE (B.P. 306,547, 20.2.29. Ger., 24.2.28).— Oligodynamically active metals, alloys, and compounds are deposited by heat upon carriers, containers, etc. for use in sterilising water ; *e.g.*, ceramic material, silica gel, clay, charcoal, etc. impregnated with solutions containing compounds of silver, gold, thallium, copper, etc. is heated to decompose the compounds, or alternatively, the metals are applied by spraying or electrical atomisation processes. [Stat. ref.] L. A. COLES.

Production of a balanced environment for fish life. J. T. and M. E. TRAVERS, ASSTS. to OHIO SANITARY ENG. CORP. (U.S.P. 1,747,804, 18.2.30. Appl., 18.5.28). —The water contains travertine, composed of calcium carbonate and clay in the process of decay, preferably having a porosity not less than 100% and/or  $d 2 \cdot 3 - 2 \cdot 5$ , and/or a calcium carbonate content of 90—96%. The travertine is capable of adsorbing the noxious odourproducing compounds in the excrement of fish.

S. K. TWEEDY.

Disinfecting or sterilising devices for use in connexion with bottle and like washing apparatus. R. HAIMES (B.P. 330,249, 2.3.29).

Water-softening apparatus. UNITED WATER SOFTENERS, LTD., and H. S. LAWRENCE (B.P. 330,120, 17.5.29. Addn. to B.P. 302,744).

Air filters etc. (B.P. 328,948, 329,099, and U.S.P. 1,747,694).—See I. Water-softening agent (U.S.P. 1,750,847).—See VII.