

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DEC. 19 and 26, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Formation and properties of boiler scale. E. P. PARTRIDGE (Dept. Eng. Res., Univ. Michigan, 1930, Bull. No. 15, 170 pp.).—Chemical analysis and crystallographic examination show that the chief constituents of boiler scale are anhydrite, calcite, brucite, and magnesium and calcium silicates, whilst aragonite and calcium hydroxide are occasionally present. The solubilities of calcium sulphate, magnesium hydroxide, and calcium hydroxide, and probably also of calcium carbonate, decrease with rise of temperature. Calcium and magnesium silicates are also believed to have solubility curves with negative slopes; hence it appears that all the substances found in boiler scale are characterised by negative solubility curves. The maximum value of the heat conductivity coefficient of boiler scales is about 2.0 B.Th.U./ft.²-hr.-ft.-1° F. Dense, compact scales such as those formed by calcium sulphate have a coefficient of 1.3, whilst porous scales, owing to the pores becoming filled with a film of poorly-conducting steam, have coefficients as low as 0.5. The actual heat loss due to scale is only about 2% for scale $\frac{1}{8}$ in. thick, but such a scale has a serious effect on the raising of the metal temperature when the heating surface is exposed to direct radiation; with a rate of heat transfer by radiation of 75,000 B.Th.U./ft.²-hr. non-porous scales 0.05–0.1 in. thick soon produce failure of the tubes by overheating. The present knowledge of the mechanism of scale formation is reviewed, and it is shown that the rate of scale growth may be expressed by the equation $dD/dt = -K \times (dS/dT)^m \cdot (dQ/dt)^n$, where S is the solubility of the scaling substance, T the temperature of the boiler, Q the quantity of heat transferred in time t to deposit an amount of scale D , K a coefficient which is constant for each scale-forming substance, and m and n are constants, both approximately equal to unity. The prevention of scale formation depends either on complete softening of the feed water or careful control of the chemical equilibria in the boiler water to precipitate solids which will form scale only slowly if at all. Any one of the methods in use for external softening at the present time is satisfactory in the first case, but probably only the phosphate method is reliable in the second. The rate of formation of calcium sulphate scales has been measured in an experimental boiler unit; at pressures up to 25 lb. hemihydrate is found in the scale, but at pressures above 50 lb. only anhydrite is formed. Equilibria in the system calcium sulphate–water have been investigated by X-rays; only three crystal forms of the salt exist, viz., gypsum, hemihydrate, and anhydrite, the transformation point for gypsum to hemihydrate being 98° and for gypsum to anhydrite 37°. In contact with water at 11–15° anhydrite is converted into gypsum

with setting similar to that of plaster of Paris. The so-called "soluble anhydrite" is identical in crystal structure with hemihydrate, and should be called, therefore, dehydrated hemihydrate, as this substance apparently belongs to the zeolitic type which loses and regains water of hydration without change in crystal structure. The solubilities of hemihydrate and of anhydrite have been determined at 100–200°, a tentative equilibrium diagram for the system calcium sulphate–water has been prepared, and a new theory of scale formation on a boiler heating surface, based on photomicrographic examination of early stages of calcium sulphate scale formation, is presented.

A. R. POWELL.

Pebble-mill linings. R. TWELLS (J. Amer. Ceram. Soc., 1930, 13, 669–674).—The relative advantages and disadvantages of using flint, fine-grained quartzite, porcelain, and rubber for pebble-mill linings are discussed. Cost data for the various types of linings are given.

R. J. CARLIDGE.

Nature of flow. E. C. BINGHAM and B. LOWE (Coll. Symp. Ann., 1929, 7, 205–212).

Forensic investigations of firearms, maliciously damaged trees, and forged coins. O. MEZGER (Chem.-Ztg., 1930, 54, 753–755, 774–776, 830–831, 851–853).

Smelting furnace. COOKE.—See VIII.

See also A., Nov., 1396, **Standardisation of a modified Ostwald viscosimeter** (CHADWELL and ASNES).

PATENTS.

Reverberatory furnace. F. F. LINES (U.S.P. 1,763,471, 10.6.30. Appl., 11.7.21).—The gas uptake of the furnace is outside the air uptake and a slidable water-cooled sleeve carries the gas across the air uptake to a throat, leading into the furnace, where air and gas mix. The result of sliding the hollow damper is that the annular space for passage of air from the uptake to throat is varied while the gas passage remains constant. When, however, the sleeve is fully retracted, the gas uptake is shut off altogether and an uptake fully open; this position may then be used at the outlet end of the furnace for the time being, only the downtake (which was the air uptake) leading to regenerators.

B. M. VENABLES.

Tunnel kiln. T. LARSSON, Assr. to NORTON CO. (U.S.P. 1,763,624, 10.6.30. Appl., 26.3.28).—The kiln is of the muffle type, combustion takes place in narrow spaces between the main and muffle walls, these spaces being continued to the end of the kiln at which the goods enter, to effect preheating. The waste gases are then passed through a separate recuperator for the air, and the latter is further preheated in a second exchanger by a current of air drawn through passages round the goods

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

leaving the kiln. The muffle walls are formed of corrugated overlapping plates which are free to expand and contract; they may be made of, *e.g.*, crystalline alumina bonded with highly refractory clays. Provision is made for admitting cold air to the muffle spaces and heat exchangers to regulate the temperature.

B. M. VENABLES.

Rotary-hearth oven. F. BARTLING (U.S.P. 1,765,196, 17.6.30. Appl., 27.6.27. Ger., 15.1.27).—The goods to be heat-treated are spread in a thin layer on a slowly rotating annular hearth, which is heated from below, during part of the circle, by surface-combustion devices. Since the latter occupy the space ordinarily taken by the rails and carriage, the hearth is formed as an arch between carriages on inner and outer sets of rails.

B. M. VENABLES.

Continuous method of calcining pulverulent materials, chemicals, pigments, or the like. R. KRAUSSE (B.P. 335,659, 11.7.29).—The material is treated in a muffle, which is jiggged to cause the material to travel through it. The suspending links and driving means are outside the furnace.

B. M. VENABLES.

[Water-cooled] retort. W. F. RIEGER, Assr. to STOKER MATIC CORP. (U.S.P. 1,763,539, 10.6.30. Appl., 27.8.28).—A device for the pre-coking of fuel in the furnace of a boiler is described.

B. M. VENABLES.

Drying apparatus. H. A. GILL. From NICHOLS PRODUCTS CORP. (B.P. 335,942, 3.7.29).—The material is subjected in a closed vessel to steam which is under a pressure sufficient to prevent the evaporation of the "moisture" fluid and which is superheated by internal electrical resistors. After soaking, the pressure is released at such a rate that the material becomes uniformly dry throughout.

B. M. VENABLES.

Conveyor-dryers. B. J. OWEN and R. O. DAVIES (B.P. 336,009, 10.7.29).—In apparatus of the type described in B.P. 334,940 (B., 1930, 1096), the main division of the air currents is effected by providing separate fans for each zone. The flow starts at the dry end and the same air passes in turn through each zone to the wet end, but provision is made for heating by steam and/or admixture of furnace or flue gases at the beginning and intermediate points. Application in the sugar industry is indicated.

B. M. VENABLES.

Centrifugal dryer. C. W. TERWILLIGER (U.S.P. 1,764,909, 17.6.30. Appl., 28.11.27).—A resilient bearing for the shaft of a centrifuge is described.

B. M. VENABLES.

Drying of liquid materials. D. J. VAN MARLE, Assr. to BUFFALO FOUNDRY & MACHINE CO., INC. (U.S.P. 1,762,250, 10.6.30. Appl., 7.4.25).—A method of feeding the liquid to a heated roller is described. A sheet of the fluid is allowed to flow, in excess, down an inclined shoot, so that it contacts with the lower part of the roller, the excess being allowed to fall freely away.

B. M. VENABLES.

Exclusion of unfiltered air from tunnel and like drying apparatus. TOMLINSONS (ROCHDALE), LTD., and J. N. TOMLINSON (B.P. 336,049, 22.8.29).—To the outlet end of a dryer, which cannot be maintained airtight owing to the continual exit of goods, is supplied an

excess of filtered air, so that there is always an outward draught.

B. M. VENABLES.

Carrying out exothermic reactions. [Production of calcium chloride.] K. STAIB, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,764,582, 17.6.30. Appl., 12.5.27. Ger., 10.7.26).—In reactions which are exothermic, more especially those between gases and solids, the temperature is regulated by admixture of inert solid material with the charge, the size of the inert particles being different from that of the active particles, so that they may afterwards be separated by screening. *E.g.*, in the production of calcium chloride, small bricks may be formed of lime and coal with a small quantity of calcium chloride as binder; these are mixed with "retort coal" as inert material, and then treated with chlorine gas after a slight preheating.

B. M. VENABLES.

Apparatus for carrying out reactions continuously in the liquid phase, under increased pressure. IMPERIAL CHEM. INDUSTRIES, LTD., and K. H. SAUNDERS (B.P. 336,136, 14.11.29. Addn. to B.P. 329,260; B., 1930, 690).—A vessel as described in the original patent is provided with a thin, non-corrodible lining, *e.g.*, of stainless iron; a non-corrosive constituent of the reaction sweeps through a narrow space between the lining and the pressure-resisting wall on its way to the reaction zone.

B. M. VENABLES.

Heat-exchange apparatus. B. F. STURTEVANT CO., Assees. of G. C. DERRY (B.P. 315,865, 5.7.29. U.S., 21.7.28).—A method of connecting in groups the horizontal tubes of an economiser or other type of heat exchanger is described.

B. M. VENABLES.

Manufacture of heat-insulating masses. I. G. FARBENIND. A.-G. (B.P. 336,163, 15.12.29. Ger., 11.3.29).—A solution of water-glass (*d* 1.38) or other alkali silicate is heated at 130–150° in air for 1 hr., and then at 450° in carbon dioxide for 3 hrs.

H. ROYAL-DAWSON.

Heat-insulating coverings. R. T. KEMPER (B.P. 335,747, 7.10.29. Ger., 27.3.29).—A tank or other vessel is covered with overlapping slabs composed of sheet metal, netting, and insulating material which are held in place by friction only, *i.e.*, without drilling the wall of the vessel.

B. M. VENABLES.

Pulverising machine. B. A. O'NEILL, Assr. to SCHUTZ-O'NEILL CO. (U.S.P. 1,765,309, 17.6.30. Appl., 28.9.23).—A disintegrator and fan are mounted on the same overhanging portion of the driving shaft, and can be removed therefrom without disturbing the main bearings; the fan volute is attached by flanges to the disintegrator casing, and can be rotated so that its outlet points in any direction. On the first disc of the disintegrator at a less radius than that of the hammers are side projections, which in conjunction with the corrugated end of the casing serve as preliminary crushers and deflect uncrushable pieces into pockets formed in the end of the casing.

B. M. VENABLES.

Pulverising or crushing machines. F. K. WOODROFFE (B.P. 336,021, 17.7.29).—A ball mill is run at a speed rather above the critical centrifugal speed, so that the balls will be carried right to the top; they are less

in number than usual and are deflected downward by a stationary internal plate, so that they will fall through a greater distance than usual and the falling stream will be separate from the rising mass. B. M. VENABLES.

Pulverising apparatus. L. V. ANDREWS, ASSR. to RILEY STOKER CORP. (U.S.P. 1,763,496, 10.6.30. Appl., 5.7.29).—The rotating disc of the disintegrator divides the casing into coarse- and fine-grinding zones which communicate only at the circumference. A resilient liner for the upper part of the casing takes the form of a steel half-hoop which is secured only to the lower half of the casing, and is lined with wearing pieces of harder material. A pocket for uncrushable pieces is formed at the bottom of the casing, and at this point the passage from coarse to fine zones is guarded by a shelf which is of frangible material, so that in the event of a serious breakage of the disintegrator the loose parts may break through the shelf and enter the pocket.

B. M. VENABLES.

Grinding mills. A. W. McALLISTER (B.P. 336,005, 9.7.29).—A machine suitable, *e.g.*, for the ingredients of chocolate is provided with main grinding surfaces comprising a cylinder rotating within a cylindrical casing, each surface being provided with rifling, of which the section is shaped like a blunted saw tooth, and the direction of rotation being such that the action is one of wiping, not of scraping. The material is admitted at one end through a preliminary coarse grinder, comprising a disc with circumferential grooves and teeth formed on the ridges rotating against the end of the casing, which is of similar form.

B. M. VENABLES.

Apparatus for comminuting materials. C. H. BIRCH (U.S.P. 1,762,381, 10.6.30. Appl., 17.10.27).—The material is flung by disintegrator hammers into the nip between two rollers. One of the rollers is built up of annular sections which, except the end ones, have a limited radial freedom on the shaft, and attached to the lower roller is a scraper device which also forms a pocket to guide the material into the nip.

B. M. VENABLES.

Apparatus for pneumatic separation of materials. R. H. KIRKUP (B.P. 336,055, 29.8.29).—The materials stratified by upward-pulsating air currents which are at all times uniform over the whole surface; a single fan is therefore used, and the pulsations are produced by a number of louvers rotating in line and in phase across the main air stream. B. M. VENABLES.

Mixing and discharging apparatus. H. S. BEERS, ASSR. to TURBO-MIXER CORP. (U.S.P. 1,764,498, 17.6.30. Appl., 24.10.29).—In the lower part of a tank containing viscous fluid is a bladed rotor which runs between fixed deflecting blades and acts as a mixer. When mixing is complete the rotor can be lowered through the bottom of the tank, and then runs within a volute casing and acts as a centrifugal pump to discharge the fluid.

B. M. VENABLES.

Centrifugal extractor. J. M. KREIDER (U.S.P. 1,764,214, 17.6.30. Appl., 3.10.28).—A small, simple, and robust machine is described, suitable, *e.g.*, for drying small metal parts which have been plated or lacquered.

B. M. VENABLES.

[Laboratory] filtration apparatus. A. W. PETREY, ASSR. to ALUMINUM CO. OF AMERICA (U.S.P. 1,762,738, 10.6.30. Appl., 7.6.29).—A device for filtering and washing a large number of exactly similar assays is described.

B. M. VENABLES.

Method of filtering. C. D. MORTON, ASSR. to MORTON SYSTEM, INC. (U.S.P. 1,762,560, 10.6.30. Appl., 15.7.27).—The method is suitable for partially de-watering sewage or other fluid in which the proportion of solids is small. The prefilter flows endways into a horizontal cylindrical chamber, and the thickened sludge is drawn off from the lower part of the other end. A cylindrical filter having an axial outlet for filtrate is rotated within the casing, leaving only a narrow annular space so that the sludge, even when thickened, has considerable longitudinal, or rather spiral, motion over the surface of the filter. The pressure and rate of feed and the speed of rotation are so co-ordinated that the filter remains clear indefinitely.

B. M. VENABLES.

Apparatus for separating solids from liquids. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 335,975, 4.7.29).—The apparatus is of the combined centrifugal basket-and-bowl type. An inner, perforated, conical wall of the rotor retains the coarser solids, and the finer solids and liquid slide down an outer, imperforate, conical wall to an outermost, imperforate, vertical wall, where the remaining solids are retained and over the upper edge of which the liquid is continuously siphoned. The solids are discharged at intervals through suitable doors, preferably while the feed is cut off.

B. M. VENABLES.

Separation of solids from liquids [by filtration]. A. H. GODBE, ASSR. to UNIVERSAL PROCESS CO. (U.S.P. 1,763,773, 17.6.30. Appl., 1.11.26).—The filter membrane forms a cylindrical or conical division in a tank. Before use, it is thoroughly cleaned from grease or other matter tending to make air adhere to it and is then dipped in water or previously obtained filtrate; during use, air is allowed to pass freely away. Clogging of the pores is thus prevented.

B. M. VENABLES.

Apparatus for removing the separated solid matter from strainers for liquids. DORR CO. (B.P. 335,767, 24.10.29. U.S., 9.11.28).—A scraper suitable for cleaning a bar screen for sewage is caused to move in an oval path by means of a pantograph mechanism having no submerged bearings.

B. M. VENABLES.

Apparatus for emulsification. L. S. and E. M. RICHARDS, and CREMAC MARKETING CO., LTD. (B.P. 335,949, 4.4.29).—The fluids, melted if necessary, are forced by a hand-operated piston through restricted passages such as those formed by a loosely fitting crew plug; no outlet valve is necessary and the inlet is through a port in the cylinder wall, which is uncovered by the piston at the end of the suction stroke. Beyond that port is a leakage-collecting recess, so that no packing is required for the piston.

B. M. VENABLES.

Evaporators [for generating steam from brine]. D. A. QUIGGIN (B.P. 335,911, 30.4.29).—An evaporator of the marine type is provided with conical baffles below the heating coils, which deflect sludge to the

bottom and cause a circulation of brine upwards at the centre and downwards at the sides; the circulation is also aided by admitting the sea-water feed through a ring-pipe with holes on its underside and by exhausting brine through a similar pipe upwardly pierced. Scale is removed from the steam coils when the evaporator is blown down by allowing them to become hot and suddenly chilling them by sprays of cold feed.

B. M. VENABLES.

Treatment of boiler water. H. KRIEGSHEIM, ASST. to PERMUTIT Co. (U.S.P. 1,762,784, 10.6.30. Appl., 25.4.27).—The treatment is suitable for alkaline make-up water. Sulphur is burned in a deficiency of air and the raw water treated with the products of combustion; the sulphur dioxide removes both dissolved oxygen and alkalinity and forms sulphates which are useful in preventing caustic embrittlement, and the residual nitrogen is used to form a neutral atmosphere over the hot well and other places where feed-water both new and returned is stored. The amount of sulphur should be insufficient to neutralise all the alkaline salts.

B. M. VENABLES.

Gas washer. H. KREISINGER, ASST. to INTERNAT. COMBUSTION ENG. CORP. (U.S.P. 1,762,338, 10.6.30. Appl., 10.10.27).—In an air-borne pulverising system the vented air only is scrubbed by steam and water moving concurrently downwards in a tower to a tank at the bottom which acts as a separator for the sludge and as a return bend for the air which is exhausted upwards through an adjacent tower.

B. M. VENABLES.

Apparatus for washing gas. H. A. BRASSERT & Co., LTD. From H. A. BRASSERT (B.P. 335,648, 6.7.29).—The apparatus is of the disintegrator type, the bars being T-shaped with the head of the T radial.

B. M. VENABLES.

Liquid and gas contact apparatus. R. P. MASE (U.S.P. 1,765,087, 17.6.30. Appl., 2.8.28).—In a bubbling tower the trays each comprise a bottom and roof which are concave to each other. A number of annular baffles alternately depend from the roof and have notched bottom edges, whilst others dip into the liquid and allow free passage for the gas above them and liquid below them, so that the gas bubbles through the liquid several times in each tray. On account of the high back-pressure thus set up, non-return flaps are provided in the downflow pipes for liquid.

B. M. VENABLES.

Decomposition of gas mixtures. GES. F. LINDE'S EISMASCHINEN A.-G., F. POLLITZER, and H. KAHLE (B.P. 335,599, 26.6.29).—A mixture of gases, e.g., coke-oven gas, is separated, by partial condensation after cooling, in several stages, each of which comprises several heat exchangers through which the gas being cooled flows in parallel and in a downward direction; such heat exchangers provide separate paths for the returning products which effect the cooling. In the case of coke-oven gas the desired product (hydrogen) is not condensed and is kept separate throughout the return flow, but the liquefied nitrogen, carbon monoxide, methane, etc. are added to stages corresponding to their b.p. and emerge together. The withdrawal of heat to compensate for losses is effected in one of the cooler exchangers by

means of liquid nitrogen from an external source which has its partial pressure reduced by admixture of a little hydrogen from the circuit.

B. M. VENABLES.

Apparatus for compressing gases. N. GRIBOJEV (B.P. 314,801, 27.6.29. Fr., 2.7.28).—An apparatus involving both piston and centrifugal compression is described, the reciprocating action being obtained from rollers running in a sinuous groove.

B. M. VENABLES.

Pumps for obtaining high vacua. E. R. GROTE (B.P. 336,001, 9.7.29).—A disc rotates at the highest possible speed with small clearance between two plates which form a gastight casing. A circular groove is cut in each plate about $\frac{1}{4}$ in. away from the edge of the disc and each groove is stopped at one point, the inlet and outlet being on either side of the stop, which is shaped so as to guide the molecules of gas to the outlet. Though the surfaces are actually smooth, they are rough in comparison with the size of a molecule, and the rebound of the molecules will have an average direction towards the outlet. The grooves may be connected in series or parallel according to the requirements of the pump.

B. M. VENABLES.

Apparatus for determining the sp. gr. of so id or liquid substances or bodies. W. & T. AVERY, LTD., and W. A. BENTON (B.P. 336,043, 14.8. and 21.11.29).—The apparatus described in B.P. 320,416 (B., 1930, 4) is simplified and extended to determine the sp. gr. of solids lighter than water and of liquids. In each case there are only two vertical sliders and one swinging arm, no knife edges or parallelogram being required.

B. M. VENABLES.

Thermometers. G. BREWER. From Z. MATSUMOTO (B.P. 335,825, 29.11.29).—To afford easier reading, the inside or outside, back or front of the thermometer tube is roughened by painting, etching, or forming serrations.

B. M. VENABLES.

Repairing heated structures such as furnaces, retorts, etc. A. McD. DUCKHAM (U.S.P. 1,780,120, 28.10.30. Appl., 15.5.29. U.K., 24.5.28).—See B.P. 313,728; B., 1929, 664.

Tube mill. W. JAEDEL, ASST. to MASCHINENBAU-ANSTALT HUMBOLDT (U.S.P. 1,780,132, 28.10.30. Appl., 19.4.29. Ger., 23.4.28).—See B.P. 310,342; B., 1930, 798.

Grinding mills. G. F. PETTINOS (B.P. 317,710, 19.8.29. U.S., 18.8.28).—See U.S.P. 1,758,010; B., 1930, 1008.

Pulverising machine. P. A. HIRSCH (B.P. 336,776, 3.10.29. U.S., 15.11.28).—See U.S.P. 1,744,895; B., 1930, 490.

Emulsifying and analogous apparatus. V. G. WALSH (U.S.P. 1,780,853, 4.11.30. Appl., 26.12.29. U.K., 4.10.28).—See B.P. 323,534; B., 1930, 224.

Filtering device. F. W. YOUNG (U.S.P. 1,780,777, 4.11.30. Appl., 27.7.26).—See B.P. 280,170; B., 1929, 79.

Sealing rings for rotary kilns and dryers. HURON INDUSTRIES, INC., and B. E. GREEN (B.P. 336,729, 28.8.29).

[Domestic] apparatus for [electrically] heating liquids. L. F. THOMPSON (B.P. 336,011, 11.7.29).

[Domestic, electrically driven] hot air drying apparatus. A. HOFFMANN (B.P. 337,337, 20.3.30. Ger., 20.3.29).

Refrigeration apparatus. SILICA GEL CORP., Assees. of E. B. MILLER (B.P. 317,471, 16.7.29. U.S., 18.8.28).

Working a continuously-acting absorption refrigerating machine. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 337,188, 26.10.29. Ger., 26.10.28).

Production of cold. K. BERGL (B.P. 336,557, 11.6.29).

[Fixing the plates in] centrifugal mills. C. TILLOY, Assee. of M. B. DAVEZIES (B.P. 336,121, 28.10.29. Fr., 28.3.29).

[Roller machine for] moulding of plastic substances. A. W. SIZER (B.P. 336,912, 25.9.29).

[Non-leaking] atomisers for liquids. R. HADDAN. From T. J. HOLMES CO., INC. (B.P. 336,123, 29.10.29).

Spray producers. I. W. P. BUCHANAN (B.P. 337,064, 30.5.29).

[Combined charging and discharging fixture for] compressed-gas containers. C. J. JOHANN (B.P. 319,760, 15.8.29. U.S., 28.9.28).

[Cleaning of bags in] air filters. I. L. BRAMWELL, C. W. H. HOLMES, and BIRTLEY IRON CO., LTD. (B.P. 335,922, 26.3.29).

Machine for dissolving cellulose xanthate etc. (U.S.P. 1,763,379).—See V. Precipitator for gases (U.S.P. 1,760,461). Pyrometer (U.S.P. 1,762,380).—See XI. Heating apparatus for milk etc. (B.P. 335,964).—See XIX.

II.—FUEL; GAS; TAR; MINERAL OILS.

Relation between ash content and calorific value of Chikuhō coal. K. TAKEI (Fuel, 1930, 9, 393).—Examination of a large number of coal samples of widely varying ash content from the Chikuhō field shows that the calorific value of such coals can be calculated from the percentage ash content of the dry coal (*A*) with an error of not more than 5% by means of the formula: cal. value (in kg.-cal.) = 8080—92*A*. A. B. MANNING.

X-Ray stereoscopic examination of coal. A. N. WILSON (J. Inst. Fuel, 1930, 4, 64—65).—The actual extent and thickness of inclusions in coal such as pyritic masses cannot be determined with certainty from the inspection of a single radiographic view, but this information may be obtained by making successive identical exposures with slight rotation of the specimen between each. The application of this method is described and radiographs of typical mineral inclusions are given.

C. B. MARSON.

Possible improvements in gas producers. J. RUDE (Gas World, 1930, 93, 320—322).—The reactions occurring in a gas producer, using coke, and the effects of steaming are described. Theories are advanced to account for the larger amounts of carbon dioxide which are formed near the producer walls. It is suggested that greater efficiencies can be obtained by preheating the

blast of air and steam; the amount of steam which can be used is thereby increased, the reaction zone is extended, greater decomposition of steam is obtained, the quality of the gas is improved, the capacity of the plant is increased, and the amount of unburnt fuel remaining on the ash is diminished. C. B. MARSON.

Resin formation in benzols. I. Factors governing and mechanism of resin formation. W. H. HOFFERT and G. CLAXTON (Fuel, 1930, 9, 359—366).—Tests have shown that little, if any, resinification of the unsaturated constituents of benzol takes place when used in internal-combustion engines under normal conditions. The deposits in the induction system are due chiefly to the non-volatile resinous material already present in the benzol. No appreciable formation of resin occurs in benzols on storage at ordinary temperatures in the absence of oxygen. The first step in the formation of resin appears to be the production of organic peroxides, but the further mechanism of the process is still obscure. The main factors governing the resinification of benzols on storage are the nature and relative proportion of the unsaturated hydrocarbons present, the material of the containing vessel, and the presence of small quantities of substances having the power of promoting or retarding the process. Resinification proceeds more rapidly in glass than in tinned iron or copper vessels. The addition of phenols, amino-compounds, pyridine, etc. inhibits the formation of resins; the optimum concentration of cresol, one of the most effective of the inhibitors, is 0.03%. Sulphur dioxide, thiophenol, alkyl sulphates, etc. accelerate resinification. A. B. MANNING.

Resin formation in benzols. II. Prevention of resinification: technical-scale storage tests and road trials. W. H. HOFFERT and G. CLAXTON (Fuel, 1930, 9, 440—447; cf. preceding abstract).—The reactions involved in the formation of resins are extremely sensitive to traces of impurities, which may either accelerate or inhibit the process. The stabilisation of motor spirits by the addition of "inhibitors" has been tested on the technical scale, using alkali-washed gas-works' and coke-oven benzols, to which 0.03% by wt. of "tricresol" had been added. The treated benzols showed no appreciable increase in resins on storage for 34 weeks, and gave very little more deposit than normal in the induction manifold, inlet valves, etc. of the engine used in the road trials. The resin content of the gas-works' benzol with no inhibitor increased from 4—9 mg./100 c.c. after one week's storage to 36—37 mg./100 c.c. after 34 weeks. The results of a similar examination of the coke-oven benzol were not so satisfactory, owing to the difficulty of removing traces of phenols already present in the crude spirit. In using inhibitors for stabilising motor spirits there is a danger of subsequent accidental contamination of the spirit by impurities, e.g., zinc chloride used in soldering the containers, which accelerate resinification. A. B. MANNING.

Accelerated tests of asphalts. O. G. STRIETER (Bur. Stand. J. Res., 1930, 5, 247—253).—Methods are described for testing asphalt by accelerated weathering by the action of arc-lamp radiation, sudden chilling, and water sprays; the results obtained correspond

closely with those of natural weathering. Four to six types of asphalt may be recognised, according to their behaviour. The deterioration may be followed by the chemical changes which occur: in general, the oil and resin contents decrease, and the asphaltene content increases. Certain grades of asphalt contract during weathering.

H. F. GILLBE.

Vegetable oils [as lubricants] in aviation and in automobile engines. N. CHAMPSAUR (Chim. et Ind., 1930, 24, 519–525).—Compared with mineral oils, vegetable oils, *e.g.*, castor or olive oil, possess the advantages of having lower temperature coefficients of viscosity, of undergoing less carbonisation in the engine with the formation of carbon of lower abrasive action, and of producing less fouling of the parts of the machine lubricated. They have the disadvantages of more rapidly gumming, of becoming acid on storage, and of setting in cold weather. Olive oil possesses the highest "oiliness" and the lowest viscosity-temperature coefficient of the vegetable oils examined. The mean mol. volumes of both castor oil and olive oil are higher than those of most mineral oils; the vegetable oils are also relatively more homogeneous in composition. Road tests on small cars have shown a marked increase in power, combined with cooler running of the engine, when the mineral oil was replaced by olive oil. No difficulties were experienced, in a short test, due to gumming or acidity.

A. B. MANNING.

Conversion of methane into acetylene. K. PETERS (Z. angew. Chem., 1930, 43, 855–860).—A detailed discussion of the decomposition of the methane molecule, including the change of molecular structure, the energy changes, and the physical conditions under which a maximum production of acetylene can be produced. Details are given of experimental apparatus by which the desired decomposition has been achieved and the results obtained by various workers.

J. O. CUTTER.

[Separation of graphite in] Canadian laboratory. CARNOCHAN and others.—See VII.

See also A., Nov., 1366, **Ultrafiltration of petroleum** (ZAHARIA and LUCATU). 1378, **Rates of combustion of mixtures of gases** (TAMMANN and THIELE). 1380, **Mixed catalysts** (MITTASCH). 1408, **Salts and esters of humic acid** (FUCHS and HORN). 1425, **Berginisation of anthracene** (ORLOV and LICHATSHEV). 1436, **Mellitic acid and its derivatives** (MEYER and RAUDNITZ).

PATENTS.

Drying of coal in the mill. H. KREISINGER, Assr. to INTERNAT. COMBUSTION ENG. CORP. (U.S.P. 1,760,148, 27.5.30. Appl., 11.8.28).—A fan and cyclone separator are arranged in circuit with the mill through which a constant-lifting stream of air is circulated. To dry the coal a current of hot gas is introduced into the mill in such a manner as to form a stratified layer above the entering current of relatively cooler circulating air. By this means a layer of cool air is interposed between the hot gas and those parts of the mill in which there is a collection of relatively stationary material, and ignition due to overheating of the latter is avoided. The moving

coal does not remain in the mill long enough to become overheated.

A. B. MANNING.

Hydrogenation of coal. A. MENTZEL (B.P. 334,389, 14.9.29. Ger., 12.10.28).—The material is heated in layers not more than 5 mm. thick in contact with a catalyst and in the presence of hydrogenating gases. The material is preferably carried in a loose layer, *e.g.*, by means of a current of gas or by gravity, over surfaces coated with the catalyst. [Stat ref.]

A. B. MANNING.

Apparatus for low-temperature carbonisation of bituminous and oil-bearing materials. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 334,443, 6.11.29. Addn. to B.P. 306,723; B., 1929, 347).—The internal diameter of the tubes through which the material is passed is in this modification made greater at the inlet than at the outlet end. The tubes may be uniformly conical in shape, or may be made up of two or three sections of different diameter joined together.

A. B. MANNING.

Standpipe for coke ovens. J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,747,610, 18.2.30. Appl. 13.12.23).—The gases from the coking chamber pass through a short, slightly tapering gas outlet into a vertical standpipe and thence through a lateral conduit into the collecting main. As they ascend the standpipe they are sprayed with a liquid, *e.g.*, ammonia liquor, from a nozzle at the top of the pipe. The liquid is prevented from passing into the coking chamber by an umbrella plate or baffle mounted over the end of the gas outlet. The liquid is discharged from the annular space between the gas outlet and the standpipe. By this arrangement the gases are freed from ammonium chloride and formation of graphitic material in the standpipe is prevented.

A. B. MANNING.

Carbonisation and distillation of carbonaceous materials such as lignite, coal, wood, peat, etc. W. W. ODELL, Assr. to G. W. TRAER, JUN. (U.S.P. 1,747,731, 18.2.30. Appl., 29.11.22. Renewed 24.7.25).—The material is passed continuously down a vertical retort wherein it is carbonised by the combustion within the mass of part of the distillation gases and part of the material itself. For this purpose air is supplied through tuyères at two levels in the retort, the diameter of which is constricted at the corresponding hot zones. The retort is open at the top where any excess gas evolved may be burned. Traversing the retort below the lower tuyère is a perforated gas offtake pipe through which some of the gas produced may be withdrawn. Below the retort is a zig-zag cooling chamber, provided with inlets for steam or water, and having at its lower end a revolving paddle mechanism for removing the carbonised material from the retort.

A. B. MANNING.

Production of [distillation] products from vegetable substances. E. L. RINMAN (B.P. 334,724, 24.9.29. Swed., 17.7.29).—Vegetable substances are digested under pressure at about 180° with an aqueous solution of barium and/or strontium hydroxide, and the product, preferably after the addition of a neutral substance, *e.g.*, carbon powder, is evaporated to dryness and dry-distilled. Other inorganic substances, *e.g.*, oxides of calcium, magnesium, or aluminium, may be added

before the boiling; they facilitate the digestion and also the subsequent regeneration of the barium hydroxide. The distillation products obtained include alcohols, aldehydes, ketones, hydrocarbons, and hydrogen.

A. B. MANNING.

Distillation of resinous woods. J. C. SCHAFER, Assr. to AMER. PINE PRODUCTS CORP. (U.S.P. 1,757,144, 6.5.30. Appl., 11.10.26).—The wood is distilled in a retort the upper portion of which is heated externally by the hot gases from combustion chambers while the lower portion forms the top of a steam generator heated by the same combustion chambers. The wood is stacked in the retort in the form of elongated sticks with the grains extending upwards, and is progressively heated, first to remove the highly volatile constituents, and then to expel the rosin and effect charring of the wood. The charring progresses downwards along the sticks, while the gums and rosin trickle into inclined troughs in the bottom wall of the retort and are discharged therefrom through spouts at the ends of the troughs. A controlled amount of steam from the boiler is admitted to the retort. The volatile products evolved pass from the retort to a suitable condensing system and are collected therein.

A. B. MANNING.

Manufacture of carbon electrodes and the like. ROESSLER & HASSLACHER CHEM. CO., Assees. of H. N. GILBERT (B.P. 312,940, 1.6.29. U.S., 1.6.28. Cf. B.P. 251,982; B., 1927, 805).—A mixture of graphite and a carbonaceous binder, *e.g.*, pitch, containing up to 80% of the former, is heated with agitation to a temperature at which the condensable vapours evolved from the binder displace any occluded air. The mixture is then transferred to a mould, air being excluded during the process either by dropping liquid benzol into the mould or by subjecting the material therein to a vacuum, and compressed therein at 250–450°. If desired, the baked article may be subjected to further heating to graphitise the carbon.

A. B. MANNING.

Production of mixed oil gas and water-gas. HUMPHREYS & GLASGOW, LTD., Assees. of H. G. TERZIAN (B.P. 334,485, 27.1.30. U.S., 11.7.29).—The apparatus comprises a generator, a carburettor, a superheater, a wash-box, a condenser, and a second wash-box, and the process includes the following steps: storing heat in the carburettor and superheater by burning the blow gases therein with secondary air; introducing a hydrocarbon oil alternately into the carburettor and superheater; passing the gases and vapours formed through both vessels, and in succession through the first wash-box, the condenser, the fuel bed, and the second wash-box. Water-gas may be produced in the generator by steaming the fuel bed in either direction to utilise the deposited carbon.

A. B. MANNING.

Retorts for the generation of oil gas. F. J. and E. WEST, and WEST'S GAS IMPROVEMENT CO., LTD. (B.P. 334,346, 1.8.29).—In the upper part of an externally heated vertical retort for the production of oil gas, a truncated cone is suspended and the oil is fed in at the top of the retort in such a manner that it flows down the surface of the cone in a thin film. The oil-gas outlet is near the lower end of the retort wherein an inclined grid is provided upon which any coke or solid

formed is deposited. Below the grid is an outlet for unvaporised oil, which is returned to the upper end of the retort for re-treatment. One or more such retorts may be arranged at the top of a vertical coal-carbonising plant in such a manner as to be heated by the waste gases therefrom.

A. B. MANNING.

Gas-making apparatus. A. SCHWARZ, Assr. to COAL & OIL PRODUCTS CORP. (U.S.P. 1,758,858, 13.5.30. Appl., 18.2.27).—One or more oil-gas retorts are inserted into the top of a water-gas generator, and are so placed as to lie in the path of flow of the air-blow gases and/or the water-gas. Heavy oil and superheated steam are fed into the retorts. By mixing the oil gas so produced with the water-gas in suitable proportions, a mixed gas of any desired calorific value is obtained.

A. B. MANNING.

Gas-producing apparatus. A. SEELEY (U.S.P. 1,760,782, 27.5.30. Appl., 18.7.25).—The mouthpieces of each retort in a setting can be connected either to a vertical standpipe or to a waste-heat flue lying adjacent and parallel to the standpipe. The connexion is controlled by means of a sliding cut-off movable within a casing which communicates on the one side with the mouthpiece and on the other with openings in the standpipe and flue, respectively; the cut-off closes each of the latter openings alternately. The device permits the retorts and the connexions to the standpipe to be burned out and cleared of carbonaceous deposits while the retort is being recharged.

A. B. MANNING.

Centralised control for gas producers. C. H. SMOOR (U.S.P. 1,755,567, 22.4.30. Appl., 16.4.26).—A device is provided which is controlled by the pressure of the gas produced and which varies in accordance therewith the supplies of air and steam to the producer, as well as the speed of rotation of the grate. *E.g.*, the damper in the air main, the throttle valve in the steam pipe, and the resistance in series with the motor operating the grate are controlled by levers connected to diaphragms, the movements of which are, in turn, controlled by the air pressures in a number of compartments of the central controlling device. Additional diaphragm controls serve to maintain the air and steam supplies constant as long as the pressure of the gas produced remains the same.

A. B. MANNING.

Automatic oil control for carburetted water-gas sets. M. PARSONS, Assr. to U.G.I. CONTRACTING CO. (U.S.P. 1,758,891, 13.5.30. Appl., 28.3.27).—A gas calorimeter is used in which the difference in temperature between two electrical resistance thermometers varies with the calorific value of the gas. The thermometers form two arms of a Wheatstone bridge, in which the movement of the galvanometer needle automatically controls, by means of a suitable electrical device, the valves of the oil supply to the carburettor. A gas of substantially constant calorific value is thereby obtained.

A. B. MANNING.

Gas purifier. Z. W. WICKS (U.S.P. 1,759,819, 20.5.30. Appl., 28.1.25).—An apparatus for treating a gas with a liquid purifying agent consists of a series of inclined pipes connected in zig-zag fashion by V-shaped elbows. The gas and liquid are passed together through the apparatus in the ascending direction. At

spaced intervals within the pipes are spiral plates which invert the stream of gas and liquid first in one direction and then in the other. The issuing mixture is led to suitable separating apparatus. A. B. MANNING.

Purification of gaseous mixtures. W. J. BANISTER, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,757,826, 6.5.30. Appl., 25.1.26).—Gaseous mixtures, consisting principally of oxides of carbon, are passed over iron oxide at 400–850°, and are thereby freed almost completely from sulphur compounds. The process may be carried out under pressure. The iron oxide is preferably mixed with an inert supporting material, or with steel wool. A. B. MANNING.

Recovery of ammonia from coal gas and like industrial gases containing ammonia. GAS LIGHT & COKE CO., H. HOLLINGS, S. PEXTON, and W. K. HUTCHISON (B.P. 334,619, 11.6.29).—The dilute ammoniacal liquor obtained by cooling the gas to 45–55° is treated with a gas containing carbon dioxide, *e.g.*, flue gas, in order to convert the ammonia present into ammonium carbonate; the liquor is then used to scrub the gas for the recovery of ammonia therefrom. A. B. MANNING.

Purification of sulphur-bearing gases and recovery of sulphates and sulphur. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,526, 2.3.29).—The hydrogen sulphide in the crude gas is partly or completely converted into sulphur dioxide by catalytic oxidation and the hot gases are then brought into contact with a liquid containing thiosulphates, the reaction occurring being represented by the equation: $3(\text{NH}_4)_2\text{S}_2\text{O}_8 + 6\text{NH}_4\text{HSO}_3 = 6(\text{NH}_4)_2\text{SO}_4 + 3\text{H}_2\text{O} + 6\text{S}$. The treated gases, still containing sulphur dioxide, are cooled and scrubbed with the same solution in the presence of hydrogen sulphide and ammonia in order to produce a further quantity of thiosulphate. The solution is recirculated until sufficiently concentrated, when it is drawn off and the sulphate and sulphur are recovered therefrom. A portion only of the crude gases may be subjected to the oxidation treatment, and the remainder may be scrubbed directly with the sulphate-thiosulphate liquor. [Stat. ref.] A. B. MANNING.

Recovery of cyanogen compounds from gases. J. C. HAUN, Assr. to MERRILL CO. (U.S.P. 1,742,505, 7.1.30. Appl., 23.3.27).—The gases are scrubbed with water, which absorbs the hydrogen sulphide and hydrogen cyanide. The dilute solution so produced is passed through a tower wherein the dissolved gases are boiled off under reduced pressure. The hydrogen cyanide is separated from the hydrogen sulphide, and recovered by passing the vapours from the tower through a condenser at a temperature below the b.p. of hydrogen cyanide. A. B. MANNING.

Production of pitch and like material from tar. C. F. BROADHEAD and R. S. ANDREWS (B.P. 334,380, 2.9.29. Austral., 29.11.28).—Plastic pitch or artificial bitumen is produced from tar by digesting it for 8–16 hrs. at not above 95° with an aldehyde, preferably formaldehyde, and a catalyst, *e.g.*, ammonia. Air or other oxidising gas is subsequently passed through the hot material, preferably at a higher temperature (100–200°), until it has acquired the desired degree of hardness.

The amount of aldehyde required is approx. 1.25–4 vol.-% of the tar. A. B. MANNING.

Bituminous emulsions. N.V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of L. KIRSCHBRAUN (B.P. 312,580, 27.5.29. U.S., 26.5.28).—Asphalt or bitumen emulsions of a predetermined degree of stability, and therefore of any desired setting time, are prepared by mixing unstable emulsions, *e.g.*, those in which soap is the emulsifying agent, with stable emulsions, *e.g.*, those containing gelatin or clay, in suitable proportions. By mixing such emulsions, in which water constitutes the external phase, it is also possible to produce a compound emulsion of thinner consistency than that of either of the component emulsions. It may be necessary to adjust the p_{H} of one or both of the emulsions before mixing. A. B. MANNING.

Manufacture of emulsions with the aid of humic acid or the like. I. G. FARBENIND. A.-G. (B.P. 334,426, 21.10.29. Ger., 20.10.28).—By the addition of a small proportion of a volatile organic solvent, *e.g.*, methyl alcohol, to emulsions of fatty acids, tars, bitumen, etc. prepared with the aid of humic acid, they are rendered stable to frost. A. B. MANNING.

Refining of wood-spirit oils. W. QUERFURTH, Assr. to HOLZVERKOHLUNGS-IND. A.-G. (U.S.P. 1,759,369, 20.5.30. Appl., 25.2.28. Ger., 3.3.27).—The oils are passed in the form of vapour over heavy-metal oxides, *e.g.*, nickel or manganese oxide, preferably supported on a carrier such as pumice or rusted spongy iron, at above 300°. It is sometimes advantageous to dilute the vapours with steam, hydrogen, or other gases. A. B. MANNING.

Refining of hydrocarbon oils. E. W. GARD, B. G. ALDRIDGE, H. J. MULTER, and R. T. HOWES (U.S.P. 1,759,744, 20.5.30. Appl., 12.9.27).—Dehydration of hydrocarbon emulsions is accomplished by passing them through a chamber containing spaced, solid, foraminous blocks, from between each block of which a portion of the liquid is withdrawn and allowed partly to separate. The separated oil and water are removed and the remaining emulsion is returned through the same series of spaced filter elements. The separated oil is mixed with sulphuric acid, settled to separate as much acid as possible, and the remaining acid oil is passed through a filter chamber similar to that described above. The filtered oil is treated with a clarifying and decolorising agent, then with a sweetening agent, washed with caustic soda solution, and finally washed with water. H. S. GARLICK.

Removal of ash-forming constituents from [hydrocarbon] oil. H. G. M. FISCHER, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,760,129, 27.5.30. Appl., 8.4.26).—Sodium sulphate and other ash-forming hydratable substances are removed from hydrocarbon oils (of the cylinder-stock type) by first decomposing by regulated heating the alkali soaps formed in the usual neutralisation of the oil, and then adding to the oil a 1% aqueous solution of, *e.g.*, isopropyl alcohol at 65–93°. Sufficient solution should be added to enable the sodium sulphate to form the decahydrate, which, after agitation, is allowed to separate. H. S. GARLICK.

Cracking still and process. M. J. TRUMBLE (U.S.P. 1,762,433, 10.6.30. Appl., 24.7.24).—Hydrocarbon oil is vaporised under pressure and in contact with a gaseous reagent in a two-phase oil-treating apparatus consisting of a vertical, heat-insulated still through which extends an inwardly and upwardly tapering flue, the lower part of which is connected to a furnace. Arranged in the still are a number of inverted gas or vapour cups, in the uppermost of which, out of contact with the oil in the still, is provided a suitable catalyst, *e.g.*, a platinum coil. Hydrogen or other treating gas is delivered under pressure to these cups. A helical scraper suitably supported is used to remove carbon and tarry matter from the sides and bottom of the still to an outlet. Connected to the top of the still is a vapour outlet and pressure-control valve. H. S. GARLICK.

Apparatus for cracking oil. G. EGLOFF and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,759,446, 20.5.30. Appl., 28.3.21. Renewed 18.3.29).—Cracking stock is passed initially through a heating coil situated in a furnace into an expansion chamber, where vaporisation takes place. When vapours begin to pass into the dephlegmator connected to the expansion chamber, the stream of raw oil is diverted from the heating coil and passed in a number of independent streams through nozzles, which produce a fine spray, into separate portions of the dephlegmator, valves controlling the volume and velocity of the oil being so introduced. The reflux condensate and raw oil from the dephlegmator are passed to the heating coil, while vapours are taken off from separate portions of the dephlegmator to be condensed and the resulting distillate collected. Heavy residuum is withdrawn from the expansion chamber and the whole apparatus is operated under a regulated pressure. H. S. GARLICK.

Cracking of hydrocarbon oils. E. W. ISOM, ASSR. to SINCLAIR REFINING CO. (U.S.P. 1,762,229, 10.6.30. Appl., 20.4.27).—The mixture of vapours and gases from a cracking operation is subjected to condensation and the uncondensed gases and vapours are scrubbed first with the condensate or an oil mixture containing condensate, before the condensate is redistilled, and then with raw oil to be supplied to the cracking plant. The oil mixture from the first scrubbing operation is redistilled for separation of the desired product. H. S. GARLICK.

Treatment of gasoline and the product thereof. A. P. BJERREGAARD, ASSR. to DOHERTY RES. CO. (U.S.P. 1,761,810, 3.6.30. Appl., 30.7.26).—A non-gumming, light-stable motor fuel consists of sodium plumbite-sweetened gasoline to which has been added 0.008–0.016% by wt. of an aromatic benzene homologue, preferably anthracene. H. S. GARLICK.

Manufacture of liquid fuels. I. G. FARBENIND, A.-G. (B.P. 334,181, 27.4.29. Addn. to B.P. 226,731; B., 1925, 163. Cf. B.P. 252,019; B., 1927, 357).—The fuels described in the prior patents, *i.e.*, gasolines containing at least 0.2 g. of iron carbonyl per gal., are improved by the addition of up to 15%, generally about 1%, of an aromatic amine containing at least one alkyl radical in the molecule, *e.g.*, toluidine, monomethylaniline, with or without the further addition of organic

halogen compounds or organic compounds of metals or metal-like elements, *e.g.*, alkyl or aryl halides, boric esters, lead tetraethyl, etc. A. B. MANNING.

System of lubrication. J. F. WERDER, ASSR. to E. ROGERS (U.S.P. 1,762,902, 10.6.30. Appl., 20.4.26).—A mixture of light paraffin and kerosene oils has dissolved therein carbon dioxide or other inert gas in such proportions that the resulting product exerts a pressure of 50–75 lb./in.² in a closed container fitted with a valve. H. S. GARLICK.

Production of nicotine carbolates from crude shale oil. D. T. DAY (U.S.P. 1,762,471, 10.6.30. Appl., 12.11.24).—In a cyclic process, crude shale oil is intimately mixed with alcohol (preferably a liquid monohydric alcohol), which dissolves only the oily bases (pyridine and nicotine as their carbolates) and acids. The mixture of solvent with dissolved material is separated from the insoluble oil, and the alcohol separated by distillation from the cresylic acids. The product is useful as an insecticide. H. S. GARLICK.

Treatment [cleaning] of carbonaceous materials. R. LESSING (U.S.P. 1,780,830, 4.11.30. Appl., 23.5.27. U.K., 27.5.26).—See B.P. 276,723; B., 1927, 866.

Manufacture of fuel briquettes. H. F. MAUREL, ASSR. to MAUREL INVESTMENT CORP. (U.S.P. 1,780,205, 4.11.30. Appl., 24.3.28).—See B.P. 325,869; B., 1930, 448.

Manufacture of high-purity carbon. D. GARDNER (U.S.P. 1,780,154, 4.11.30. Appl., 10.10.27. U.K., 5.3.27).—See B.P. 292,798; B., 1928, 631.

Apparatus for hot purification [desulphurisation] of gases. A. JOSEPH, ASSR. to COMP. INTERNAT. POUR LA FABR. DES ESSENCES ET PÉTROLES (U.S.P. 1,780,159, 4.11.30. Appl., 30.7.28. Fr., 6.10.27).—See B.P. 298,236; B., 1929, 842.

Splitting up of mineral oils. M. MELAMID (U.S.P. 1,779,486, 28.10.30. Appl., 12.3.25. Ger., 22.3.24).—See B.P. 231,190; B., 1926, 622.

Burners for firing boilers, furnaces, and the like with gaseous fuel. J. E. WEYMAN (B.P. 336,722, 23.8.29).

Burners for combustion of pulverised solid fuel. F. K. WOODROFFE (B.P. 336,952, 18.7.29).

Liquid and gaseous fuel burners. M. KNUFFER (B.P. 336,617, 16.5.29).

Decomposition of gas mixtures (B.P. 335,599).—See I. Urea (B.P. 335,421).—See III. Waterproofed paper (B.P. 335,559).—See V. Nitrogen-hydrogen mixtures (B.P. 335,524).—See VII. Fireproofing of wood (B.P. 334,408). **Treatment of sawdust** (U.S.P. 1,764,249).—See IX. Bituminous paint (B.P. 336,117).—See XIII.

III.—ORGANIC INTERMEDIATES.

Manufacture of synthetic borneol and synthetic camphor. J. SCHWYZER (Pharm. Ztg., 1930, 75, 1275–1276).—The conversion of α -pinene into bornyl ester by oxalic acid occurs in about 1 hr., whilst with salicylic acid several hours are necessary. The latter

acid, however, is exclusively used in the industry since the by-products (limonene, fenchene, etc.) are formed in smaller amount (30%, in place of 70%, of the weight of borneol produced). A laboratory process giving 95–100 g. of borneol from 700 g. of α -pinene (of which 500–520 g. are recovered) is described; the recovery of salicylic acid is at most 90%. For the conversion into camphor by boiling with copper powder in xylene, the borneol must be very pure and dry, the xylene dry and water-clear, and the catalyst highly active. The latter is prepared from precipitated cupric hydroxide by reduction in a quartz tube with a slow stream of hydrogen at 160–180°. The yield of camphor is 92–94 g. from 100 g. of pure borneol. On the large scale the α -pinene fraction of turpentine oil is heated with salicylic acid in a homogeneously welded or enamelled, steam-heated, iron vessel, fitted with stirrer, manometer, and condenser, but having no reflux condenser. The reaction vessel, separated by a cock from the condenser, is kept at 0.25–0.5 atm. excess pressure during the esterification. The unused pinene and by-products are then distilled off and fractionated separately, and the bornyl salicylate is hydrolysed, again under slight pressure, with dilute soda lye. The borneol is collected on a metal gauze, washed, centrifuged, and recrystallised from alcohol. For dehydrogenation an enamelled stirring vessel, with reflux condenser and a blow-off tube reaching nearly to the bottom, is used. The copper powder, after reaction, is allowed to settle, and after removal of the clear camphor solution it is ready for re-use. In the production of 100 kg. of camphor 180–200 kg. of turpentine oil are used, and the loss of salicylic acid is 10–12 kg.; the limonene by-products are sold at half the price of turpentine oil. The manufacture is profitable only when the synthetic camphor commands a price at least 4–5 times that of turpentine oil. C. HOLLINS.

Phytin from mustard. BELIAIEV.—See XIX.

See also A., Nov., 1380, **Mixed catalysts** (MITTASCH). 1409, **Volumetric determination of acetone** (MEYER and MATHEY). 1420, **Purification of acetamide** (WAGNER). 1425, **Berginisation of anthracene** (ORLOV and LICHATSCHEV). 1427, **Azo-dye intermediates** (RUGGLI and others). 1430, **Naphthalene-green** (FRISCH). 1439, **Anthraquinoneazine and anthraquinoneazhydrin** (SCHOLL and LAMPRECHT). **Synthesis of anthraquinones related to morindone and emodin** (MITTER and others).

PATENTS.

Production of methanol [methyl alcohol from carbon oxides and hydrogen]. BRIT. CELANESE, LTD., W. BADER, and E. B. THOMAS (B.P. 334,924, 12.4.29).—Zinc sulphide, alone or with chromium oxide, is used as catalyst, preferably at 360–400° and 100 atm.

C. HOLLINS.

Manufacture of primary and secondary amines. I. G. FARBENIND. A.-G. (B.P. 334,579, 5.6.29. Ger., 1.11.28. Addn. to B.P. 306,414; B., 1930, 808).—The process of the prior patent is extended to the conversion of alcohols generally into amines. *E.g.*, *n*-butyl alcohol and ammonia, led over nickel on pumice at 300°, give

mainly butyl- and dibutyl-amines; aniline and alcohol, and cyclohexylamine and alcohol diluted with hydrogen or nitrogen, at 185–200° give the secondary with only little tertiary amine. C. HOLLINS.

Manufacture of urea. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 335,421, 28.10.29. Holl., 13.11.28).—Carbon dioxide and ammonia are introduced into an inert, viscous liquid, *e.g.*, transformer oil, until a paste containing about 50% of oil is produced. This paste is pumped continuously into a high-pressure chamber maintained at 150–200° and under 100 atm. pressure. The reaction mixture is transferred to another chamber in which unconverted carbamate is distilled off, the ammonia and carbon dioxide being used again in the process, and the aqueous urea run off from the oily layer.

W. J. WRIGHT.

Manufacture of urea [carbamide] or products containing it. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,564, 2.4.29).—Ammonium carbamate is heated at 150–250° in presence of at least 3 equivalents of ammonia (calc. on the combined carbon dioxide) and at a partial pressure of ammonia which is above the critical pressure. Thus 1 pt. of ammonium carbamate and 5 pts. of liquid ammonia heated in a monel-lined autoclave ($\frac{3}{4}$ full) at 150° for 1 hr. gives urea in 95% yield. The ammonium carbamate may be produced *in situ*, *e.g.*, from other carbamates, or from ammonium hydrogen carbonate etc. C. HOLLINS.

Manufacture of acetic anhydride. IMPERIAL CHEM. INDUSTRIES, LTD., H. HEPWORTH, and F. D. LEICESTER (B.P. 334,986, 4.7.29).—The vapours containing acetic acid, anhydride, and water are passed over active charcoal maintained at 140°, whereby the acid and anhydride are absorbed, and may be recovered as 96.5% acetic acid and a mixture of acetic acid and anhydride by raising the temperature and reducing the pressure.

C. HOLLINS.

Manufacture of aliphatic [acetic] acid anhydrides. H. DREYFUS (B.P. 334,533, 1.6.29).—Acetic or other acid vapour is passed through a copper tube filled with copper turnings at 400–700°. Catalysts, *e.g.*, an alkaline-earth tungstate, may be mixed with the turnings.

C. HOLLINS.

Manufacture of glacial acetic acid from dilute acetic acid. I. G. FARBENIND. A.-G. (B.P. 315,397, 12.6.29. Ger., 14.7.28. Addn. to B.P. 312,173; B., 1930, 1015).—The 60% acetic acid obtained by freezing is further concentrated by known means other than the double-compound method described in the prior patent.

C. HOLLINS.

Manufacture of highly stable sulphonic acids or their salts. ORANIENBURGER CHEM. FABR. A.-G. (B.P. 313,453, 11.6.29. Ger., 11.6.28).—Fat acids or neutral fats are converted, *e.g.*, by means of phosphorus tri- or penta-chloride, into the corresponding acid chlorides, which are treated with concentrated sulphuric acid, oleum, or chlorosulphonic acid at about 30°. The products are probably sulphonated mixed carboxylic-sulphuric anhydrides. The sulphonation of acid chlorides from castor oil acids, acetylated castor oil acids, and olein is described. C. HOLLINS.

Manufacture of basic products from imino-ethers of higher fatty acids. I. G. FARBERIND. A.-G. (B.P. 308,218, 18.3.29. Ger., 19.3.28).—The imino-ethyl ether from stearic or other fatty acid above C_9 is condensed with an aliphatic diamine in which at least one amino-group carries an unsubstituted hydrogen atom. Ethylenediamine reacts with stearic imino-ethyl ether with elimination of ammonia and alcohol to give a compound, m.p. 93–95° (hydrochloride, m.p. 123–125°; acetate, m.p. 70°). A similar base is obtained from β -amino- β' -diethylaminoisopropyl alcohol. The products have therapeutic properties. C. HOLLINS.

Production of unsaturated esters. RÜHM & HAAS A.-G. (B.P. 313,877, 7.6.29. Ger., 18.6.28).— β -Hydroxy- or β -alkoxy-nitriles or -amides are heated at 120–170° with an alcohol in presence of concentrated sulphuric acid. Ethylene cyanohydrin [β -hydroxypropionitrile] with methyl alcohol gives methyl acrylate; the production of methyl cinnamate from β -hydroxy- β -phenylpropionitrile, ethyl acrylate from β -hydroxypropionamide, and methyl acrylate from β -methoxypropionitrile is also described. C. HOLLINS.

Manufacture of 6-halogeno-2 : 4-di(nitrophenyl)-1 : 3 : 5-triazines. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 334,887, 10.6.29).—6-Hydroxy-2 : 4-di-*m*-nitrophenyl-1 : 3 : 5-triazine, m.p. 280–281°, heated with 10 pts. of phosphoryl chloride, gives the 6-chloro-compound, m.p. 180°, in 90% yield. C. HOLLINS.

Manufacture of naphthalene derivatives. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 334,700, 28.8.29).—A 6- or 8-arylamino- β -hydroxy-3-naphthoic acid [5- or 7-arylamino-3-hydroxy- β -naphthoic acid], obtainable from 3 : 5- or 3 : 7-dihydroxy- β -naphthoic acid by condensation with an arylamine (cf. B.P. 326,971; B., 1930, 604), is boiled with bisulphite solution and a *p*-aminophenol, whereby the 2-hydroxyl group is replaced by a *p*-hydroxyarylamino-group and the carboxyl group is lost. The products yield indophenols by oxidation with hypochlorite or chromic and acetic acids. The condensation of *p*-aminophenol with the following 2-hydroxy-3-naphthoic acids is described : 6-anilino- (product, m.p. 205°), 6-*p*-toluidino-, 6-*p*-anisidino- (product, m.p. 207°), and 8-anilino-. From 2 : 6-dichloro-*p*-aminophenol and 6-anilino-2-hydroxy-3-naphthoic acid there is obtained a product, m.p. 182–183°. C. HOLLINS.

[Catalytic] oxidation of acenaphthene, acenaphthylene, and their substitution products. SELDEN CO., ASSEES. OF A. JAEGER (B.P. 318,618, 16.8.29. U.S., 7.9.28).—Naphthalic anhydride is obtained smoothly by catalytic air-oxidation of acenaphthene etc. if steam be added to the reacting gases. If desired, sufficient steam may be used to convert the anhydride (condensed at 140–150°) into acid, which is thus obtained free from impurities volatile in steam. C. HOLLINS.

Manufacture of 4-hydroxyquinaldines. L. LIMPACH (G.P. 455,387, 17.2.26).—The yield of 4-hydroxyquinaldines from arylamines and acetoacetic esters rises from 30% to 90% when the β -arylaminoacrotic ester first formed is heated in a solvent (paraffin oil)

at 240–250° for 15–20 min. Products from aniline, *p*-phenetidine, and α -naphthylamine are described.

C. HOLLINS.

Manufacture of isatoic anhydride and derivatives thereof. I. G. FARBERIND. A.-G. (B.P. 311,336, 7.5.29. Ger., 9.5.28).—An aqueous solution of an *o*-aminocarboxylic acid, acid to Congo-red, is phosgenated below 100°. Isatoic anhydrides from anthranilic acid, 3-amino-*p*-toluic acid, benzidine-3 : 3'-dicarboxylic acid, and 2 : 3-aminonaphthoic acid are described.

C. HOLLINS.

Manufacture of [di]hydroindole-indophenols. F. BALLAUF, ASS. TO GEN. ANILINE WORKS, INC. (U.S.P. 1,780,058, 28.10.30. Appl., 25.10.26. Ger., 2.11.25).—See G.P. 443,685; B., 1928, 225.

Condensation product of the benzodiazine [quinazoline] series. W. HENTRICH, M. HARDTMANN, and R. KNOCKE, ASSS. TO GEN. ANILINE WORKS, INC. (U.S.P. 1,780,879, 4.11.30. Appl., 8.3.28. Ger., 18.3.27).—See B.P. 287,179; B., 1929, 747.

Distillation products from vegetable substances (B.P. 334,724). Refining of wood spirit oils (U.S.P. 1,759,369).—See II. Polymethine dye intermediates (B.P. 334,706). Compounds of anthraquinone series (B.P. 334,565). Intermediates of anthraquinone series (B.P. 334,872).—See IV. Treatment of sawdust (U.S.P. 1,764,249).—See IX.

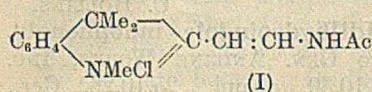
IV.—DYESTUFFS.

***ms*-Dichloroanthracene and its β -sulphonic acid as starting materials for production of alizarin.** V. I. MINAEV and B. P. FEDOROV (Rev. Gén. Mat. Col., 1930, 34, 330–332, 376–382).—Investigation of Perkin's method of making alizarin shows a 70% yield of *ms*-dichloroanthracene on chlorinating 93% anthracene in nitrobenzene (*o*-dichlorobenzene is less satisfactory); an 81.5% yield of sodium *ms*-dichloranthracene-2-sulphonate on sulphonation with 20% oleum in nitrobenzene at 10–15° (cf. G.P. 292,590; B., 1916, 921); an 82% yield of anthraquinone-2-sulphonic acid on oxidising in dilute nitric acid, or 90% with nitrogen peroxide in nitrobenzene, and a 90% yield of alizarin on fusion with sodium hydroxide and potassium chlorate. Curves are given of the rate of sulphonation; the optimum quantity of free sulphur trioxide is found to be the theoretical. The authors eliminate the isolation and purification of the *ms*-dichloroanthracene-2-sulphonic acid and increase the yield of anthraquinone-2-sulphonic acid from *ms*-dichloroanthracene to 92.2% by adding the nitric acid directly to the sulphonation melt. L. J. HOOLEY.

See also A., Nov., 1422, Plant colouring matters—lycopene and carotene (KARRER and others). 1427, Azo dyes and their intermediates (RUGGLI and others). 1430, Naphthalene-green (FRISCH). 1440, Plant colouring matters—xanthophyll from dandelion flowers and its esterification and methylation (KARRER and others). 1442, Colouring matter of wild and cultivated blackberry (KARRER and others). 1451, Dyes from 2-aminothiazole-4 : 5-dicarboxylic acid (DE). 1483, Distribution of lutein in the vegetable kingdom (KUHN and WINTERSTEIN).

PATENTS.

Manufacture of [polymethine] dyes and intermediates therefor. SOC. CHEM. IND. IN BASLE (B.P. 334,706, 2.9.29. Switz., 29.11.28).—An ammonium salt containing a reactive α -methyl group, or the corresponding ψ -base, is condensed with 1 mol. of a compound $X \cdot CH:NH$ (e.g., formimino-ethyl ether hydrochloride, formimino-chloride, formamidine hydrochloride, formiminoacetic esters, etc) in acetic anhydride. The intermediate so formed (e.g., I) or the corresponding



methylene base obtained from it by the action of alkali, may then be condensed with a second mol. of

cyclic ammonium salt etc. (the same or different) to give polymethine dyes. The intermediate (I) from 2-methylene-1:3:3-trimethylindoline (iodide, m.p. 240–247°; perchlorate, 228–229°) is condensed, e.g., with 2-methylene-1:3:3-trimethylindoline or with 2-methylene-1-methyl-1:2-dihydroquinoline. By using 2 mols. of cyclic ammonium salt or of ψ -base to 1 mol. of $X \cdot CH:NH$, the polymethine dye is formed directly.

C. HOLLINS.

Triarylmethane dyes. IMPERIAL CHEM. INDUSTRIES, LTD., E. H. RODD, and F. L. SHARP (B.P. 334,689, 19.8.29).—Violet triarylmethane dyes are obtained by condensing *m*-2-xylylene with a diaralkyldialkyldiaminobenzhydrolsulphonic acid and oxidising the leuco-compound so obtained. Preferably the hydrol is produced *in situ*, e.g., by oxidation of dibenzyl-diethyldiaminodiphenylmethanedisulphonic acid, in presence of *m*-2-xylylene, and the leuco-compound is oxidised in the same operation.

C. HOLLINS.

Vat dye enolic sulphuric esters, and their use. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 334,902 and 334,921, [A] 7.3.25, 3, and 10.5.29, [B] 7.3.29).—(A) A sulphuric ester of leuco-2-aminoanthraquinone (or its derivatives) is oxidised in presence of alkali, e.g., with hypochlorite, to form probably a tetrasulphuric ester of a leucoindanthrone, which may be isolated and purified by salting out with potassium chloride and, if desired, by washing with charcoal. The tetra-esters from 2-amino- and 3-chloro-2-aminoanthraquinones are described. (B) The tetra-esters, isolated or not, may be oxidised on the fibre for blue shades.

C. HOLLINS.

Manufacture of compounds of the anthraquinone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,565, 6.4.29).—One or both halogens in 5:8-dihalogeno-1:2-benzanthraquinone are replaced by amino-, thiol, or hydroxyl groups which may or may not carry substituents. Toluene-*p*-sulphonmethylamide gives, after hydrolysis of the primary product, 5:8-di(methylamino)-1:2-benzanthraquinone, m.p. 249–252° (greenish-blue on acetate silk). The 5:8-diamino-compound, m.p. 224–226° (reddish-blue on acetate silk), 5:8-di-*p*-toluidino-compound, m.p. 204–206° (sulphonated for a green wool dye), 5:8-di-*p*-tolylthiol compound, m.p. 306–308° (sulphonated for red wool dye), 5:8-di-(α -anthraquinonylamino)-compound, and 5:8-diphenoxo-compound, m.p. 192–194° (sulphonated for orange wool dye), are also described.

C. HOLLINS.

Production of dyes and intermediates [of the anthraquinone series]. W. SMITH, S. G. WILLMOTT, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 334,872, 5.3.29).—An aminoanthraquinone is condensed with a halogenobenzoylbenzoic acid, or a halogenoanthraquinone with an aminobenzoylbenzoic acid, the amino-group or halogen atom being *ortho* to a nitro-group. The products may be cyclised to give indanthrones. Methyl 2-*p*-chloro-*m*-nitrobenzoylbenzoate is condensed with 1-aminoanthraquinone, and the product is reduced, hydrolysed with alcoholic sodium sulphate, and cyclised to a blue vat dye.

C. HOLLINS.

Manufacture of vat dyes [of the alizarin-indigo series]. I. G. FARBENIND. A.-G. (B.P. 335,043, 4.9.29. Ger., 28.6.29. Addn. to B.P. 318,107; B., 1930, 1060).— α -Anthrol is condensed with a 5-halogeno-7-alkoxy-4-methylisatin α -chloride (etc.), e.g., 5-chloro- or 5-bromo-7-methoxy-4-methylisatin chloride, for greenish-blue vat dyes.

C. HOLLINS.

Manufacture of indigoid vat dyes. I. G. FARBENIND. A.-G. (B.P. 313,493, 7.6.29. Ger., 12.6.28).—A 5-halogenothioindoxyl is condensed with a 5:7-dichloro- or α -dibromo-isatin α -anil (etc.) for greenish-blue vat dyes. [Stat. ref.]

C. HOLLINS.

Manufacture of stable reduction compounds of [indigoid] vat dyes. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 334,878 and Addn. B.P. 334,920, 11.5.29).—(A) A 6:6'-dihalogeno-4:4'-dimethylthioindigo is reduced with hyposulphite (or other reducing agent except alkali hydrogen sulphide) in presence of less alkali than is used for vatting. A brownish product, probably ketonic, separates. (B) 6:6'-Diethoxythioindigo gives a similar product. The products are stable and insoluble in alkali.

C. HOLLINS.

Manufacture of azo dyes [acid dyes and lakes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,754, 12.10.29).—A 2:5-dialkoxyarylamine is diazotised and coupled with a 1-aroylamino-8-naphthol-4:6-disulphonic acid to give violet acid dyes for wool, which give barium lakes. Examples are: 2:5-dimethoxyaniline \rightarrow 1-benzamido-8-naphthol-4:6-disulphonic acid or the 1-(2':5'-dichlorobenzamido)-derivative; 2-amino-5-benzamidoquinol diethyl ether \rightarrow 1-benzamido-8-naphthol-4:6-disulphonic acid.

C. HOLLINS.

Manufacture of soluble compositions for dyeing and printing. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,429, 1.5.29).—A water-soluble diazo-amino-compound, prepared according to B.P. 320,324 or 324,041 (B., 1930, 233 or 364), is incorporated with an azo coupling component free from sulphonic or carboxylic acid groups, particularly a 2:3-hydroxynaphthoic arylamide. The dry mixtures are stable, and are dissolved for use in water or dilute alkali. Fifty-two examples of yellows, oranges, scarlets, reds, bordeaux, violets, blues, browns, and blacks are given, including the following: diazo-amino-compound, 2:5-dichloroaniline \rightarrow 4-sulphoanthranilic acid, with coupling component, bisacetoacetylolidine (yellow); 4-chloro-*o*-toluidine \rightarrow 4-sulphoanthranilic acid, with 2:3-hydroxynaphthoic *o*-phenetidine (scarlet); *m*-chloroaniline \rightarrow α -naphthylamine-2:4-disulphonic acid, with *o*-anisidine (red); 4:5-dichloro-*o*-toluidine \rightarrow 4-sulphoanthranilic acid, with

m-nitroanilide (Turkey-red); 6-nitrocresidine \rightarrow *N*-methylglycine, with β -naphthylamide (bordeaux); 4-amino-6-benzamidoresorcinol dimethyl ether \rightarrow *n*-butylglycine, with *o*-toluidide (violet); 3-amino-6-benzamidoquinol diethyl ether \rightarrow methylglycine, with *o*-toluidide (blue); 4-nitro-4'-amino-2':5'-dimethoxyazobenzene \rightarrow methylglycine, with *o*-toluidide (black). [Stat. ref.]

C. HOLLINS.

Preparation of sulphonated water-soluble dyes of the diaminodiphenyl series. K. THIES and B. DIECKE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,048, 28.10.30. Appl., 10.2.28. Ger., 18.2.27).—See B.P. 285,504; B., 1929, 637.

Dye [for domestic use]. W. KRITCHEVSKY, H. C. PRUTSMAN, and E. MORRILL, Assrs. to PRODUCTS CORP. (U.S.P. 1,752,184, 25.3.30. Appl., 3.12.28).—See B.P. 331,491; B., 1930, 942.

Manufacture of sulphide dyes. M. WYLER, Assr. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,779,508, 28.10.30. Appl., 23.5.29. U.K., 26.2.29).—See B.P. 329,716; B., 1930, 811.

Preparation of a reddish derivative of flavanthrone. B. WYLAM, J. E. G. HARRIS, and J. THOMAS, Assrs. to SCOTTISH DYES, LTD. (U.S.P. 1,779,791, 28.10.30. Appl., 19.4.26. U.K., 30.4.25).—See B.P. 260,638; B., 1927, 39.

Rosin soap lakes (B.P. 334,874).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Enzymic degradation of native and precipitated cellulose, of artificial silk, and of chitin. P. KARRER (Kolloid-Z., 1930, 52, 304—319).—The resistance to enzyme action offered by cellulose depends on the method of packing and orientation of the cellulose units. Resistance decreases in the order cotton, filter paper, mercerised cotton, regenerated cellulose, hydrocellulose (degraded cellulose). Stretching causes an orientation of the particles, and hence artificially-prepared threads are resistant. Cotton thread mercerised under tension is more resistant than that mercerised without tension. Bleaching causes little change in the resistance to enzyme action. Comparison of the rate of enzymic decomposition with the intensity of the X-ray diffraction ring reveals a parallelism which indicates the importance of orientation. Viscose silks show a very variable behaviour towards the action of enzymes, some being decomposed very readily and others only with difficulty. The resistance offered appears to bear no relation to chemical composition or physical properties, but depends on the shape of the cross-section of the fibres. If this is round and smooth the resistance is low, but if the fibres have a very irregular cross-section they are attacked only with difficulty. Irregular fibres are produced in acid precipitating baths and smooth fibres in concentrated salt baths, and experiments showed a direct connexion between the resistance to enzyme action and the composition of the precipitating bath. This shows again a connexion between resistance and structure and orientation of the coagulum. It is also found that the resistant viscose silks are more weakly

coloured by substantive dyes than those silks that are readily attacked, probably because the silks have the same relative adsorption capacities for the enzyme and the dye. The rate of decomposition increases with the concentration of the enzyme, as a result of the higher degree of adsorption, and agrees approximately with the course of a unimolecular reaction. Chitin from lobster shells can be hydrolysed by means of an enzyme contained in the liver and pancreas of the vineyard snail, and investigation of this reaction indicated many points of similarity to the phenomena observed with cellulose products. E. S. HEDGES.

Rapid determination of cellulose in viscose. H. HAWLIK (Chem.-Ztg., 1930, 54, 755).—About 1—1.5 g. of viscose is transferred to a glass plate, 9 \times 12 cm., and uniformly spread out by means of another superimposed plate. The plates are then separated and placed in a saturated solution of sodium or ammonium chloride, the viscose layer being uppermost. The xanthate film produced, which readily floats off the plates, is purified by immersing it two or three times in fresh salt solution, and then treated with 2% hydrochloric acid. The film of pure cellulose thus obtained is transferred to a flask with 25 c.c. of water, 15 c.c. of potassium dichromate solution (90 g. per litre) are added, followed by 25 c.c. of concentrated sulphuric acid, and the mixture is boiled for 4 min., cooled, and titrated with ferrous ammonium sulphate solution, using potassium ferricyanide as indicator. A blank test must be carried out. The determination is completed in $\frac{1}{2}$ hr.

W. J. WRIGHT.

Lilienfeld silk (Nuera silk). E. GEIGER (Helv. Chim. Acta, 1930, 13, 1114—1116).—Contrary to the statement made by Fierz-David and Brunner (B., 1930, 235), a warm spinning bath can be used in the manufacture of Lilienfeld silk. The shape of cross-sections of the fibres (various photomicrographs are given) is conditioned by the osmotic relations prevailing in the bath and the viscose sheath. When the osmotic pressure in the bath and the viscose is the same, the cross-sections are round; such silks are of the best quality and have the greatest tensile strength. The observed striation (cf. *loc. cit.*) on the fibres is due to light reflexion from the surface and not to the structure; ordinary viscose silk, spun without stretching, shows a more marked, but finer, striation. The addition of various substances to the spinning bath does not affect the stretching of the fibre; the surface tension is lowered thereby, causing more favourable coagulation and fixing conditions. The sample of silk examined by Fierz-David and Brunner has not the characteristics of the strongest Lilienfeld silk which can be produced. H. BURTON.

Test for strained viscose threads. A. J. HALL (Rayon Record, 1930, 4, 1001).—When stretched, viscose threads have an increased reactivity towards an alkaline silver nitrate solution containing silver nitrate 1 g., sodium thiosulphate 4 g., and caustic soda 4 g., per 100 c.c. When immersed in the nearly boiling solution for 1 min. stretched threads become brown and unstretched threads remain nearly colourless. The test is suitable for detecting excessively stretched viscose threads in woven and knitted materials. A. J. HALL.

[Detection of] strained viscose threads. J. M. PRESTON (Rayon Rec., 1930, 4, 1053).—Hall's test for stretched viscose threads (preceding) is also applicable to nitrocellulose silk, but not to cuprammonium silk, and it is therefore concluded that the test depends on the greater accessibility of the reducing groups present as produced by stretching. A. J. HALL.

Central Asiatic reeds as material for preparation of pulp. N. L. KARAVAEV and I. M. KRIWOZIAS (Papier-Fabr., 1930, 28, 654—656).—The stalks of *Phragmites communis* have been found to be similar to straw as papermaking material. Digestion with a 10% solution of sodium hydroxide at 160—165° and under 6—8 atm. yields pulps of quality sufficient for the manufacture of medium grades of paper. The resultant fibres are of the order of 1.5 mm. long and 17—18 μ wide. As in straw pulps, the ash contains an appreciable amount of silica, and the pentosan content is 6—8% in the best pulps obtained. T. T. POTTS.

See also A., Nov., 1415, Celluloses of some Australian plants (ARNEMAN and EARL). 1416, Dissolution of cellulose fibres in ammoniacal copper solution (SAKURADA). 1417, Kinetics of the "coppering" of cellulose fibres (SAKURADA). Copper oxide-ethylenediamine-cellulose (TRAUBE and others; TROGUS and SAKURADA). 1483, Determination of cellulose in straws (JENKINS).

PATENTS.

Degumming of silk. L. WALLERSTEIN, Assr. to WALLERSTEIN Co., Inc. (U.S.P. 1,763,112, 10.6.30. Appl., 2.2.27).—Silk is washed in a dilute soap or weak alkali solution, and then degummed by immersion at 50° in a liquor containing a proteolytic enzyme, such as papain, and an accelerator, such as potassium cyanide or hydrogen sulphide. A. J. HALL.

Manufacture of felt. G. D. BEAL and R. R. MCGREGOR (U.S.P. 1,765,046, 17.6.30. Appl., 19.12.28).—Animal fibres are subjected to the action of steam under pressure in the presence of a salt yielding an acid reaction on hydrolysis, e.g., magnesium chloride. F. R. ENNOS.

Treatment of liner [for interposition between layers of rubberised fabric]. L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,763,618, 10.6.30. Appl., 16.9.27).—The liner fabric is treated with nitrocellulose containing butyl phthalate. F. R. ENNOS.

Machine for dissolving cellulose xanthate and the like. R. THURM and E. SCHMIERER, Assrs. to BAKER PERKINS Co., Inc. (U.S.P. 1,763,379, 10.6.30. Appl., 29.11.26).—Spaced above the bottom of a dissolving bowl is a cylindrical drum open at both ends and having a number of radial bars with cutting edges fixed near the lower end; a spindle arranged co-axially with the cylinder carries a number of rotatable impeller blades adjacent to the fixed radial bars. Lumps of cellulose xanthate are continuously drawn up through the cylinder by the rotating impeller and are sheared into small pieces by the movable and fixed cutting edges, so that fresh surfaces are being continually exposed to the rapidly circulating alkaline liquor. F. R. ENNOS.

Manufacture of acetylcellulose. H. A. GARDNER (U.S.P. 1,764,204, 17.6.30. Appl., 27.4.28).—After acetylation of cellulose in the usual way, the reaction mixture is poured into ethyl ether and the precipitated acetylcellulose is washed by refluxing with ether; the ether and acetylation mixture are recovered by fractional distillation of the mother-liquor. F. R. ENNOS.

Manufacture of esters of polysaccharide ethers and of plastic or other compositions comprising the same. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,897, 11.6.29).—A polysaccharide ether is esterified by heating it with a carboxylic acid above C₆, with or without a diluent and/or a catalyst. The ether-esters are soluble in single organic solvents, relatively stable to hydrolytic agents, and suitable for use in films, plastics, lacquers, etc. Examples are: starch butyl ether esterified with linoleic acid at 110—150°; cellulose ethyl ether, with coconut oil acids at 130—150° in presence of boric acid, or with benzoic or stearic acid at 150°; cellulose benzyl or butyl ether with linoleic acid and a little boric acid at 130—150°. C. HOLLINS.

Production of artificial silk or the like from viscose solution. Twisting and reeling of artificial silk. ZELLSTOFFFABR. WALDHOF, and A. BERNSTEIN (B.P. 335,605—6, 27.6.29).—(A) In a non-continuous process, filaments which have been coagulated from viscose solution in an acid bath are led directly into hot water and simultaneously sthenosised either by addition of formalin or by tension, after which they are immediately spooled. (B) After being drawn off from the twisting spool the filament is allowed to fall directly into a liquid, whence it is guided by a guide member immersed therein to the winding reel. F. R. ENNOS.

Spinning of artificial silk. H. WADE. From COMPTOIR DES TEXTILES ARTIFIC. (B.P. 335,613, 28.6.29).—The partly coagulated thread is drawn through a narrow tube along which a current of liquid, which may or may not have the same composition as the coagulating bath, is passed in the opposite direction; by varying the length of the tube and the speed of the liquid any suitable tension may be applied to the thread. F. R. ENNOS.

Manufacture of artificial filaments and other products from organic derivatives of cellulose. BRIT. CELANESE, LTD. (B.P. 313,885, 17.6.29. U.S., 16.6.28).—Organic derivatives of cellulose are dissolved in solvent mixtures consisting of acetone with 1—30% of a glycol or other polyhydric water-soluble alcohol, under such conditions that a substantial proportion of the latter is retained in the product; yarns made in this way are self-lubricated and are readily scoured and dyed. F. R. ENNOS.

Cellulose ether, or ester, compositions. CELLULOID CORP. (B.P. 308,658 and 335,582, 25.3.29. U.S., 23.3.28).—Solutions, plastic masses, etc. are prepared from (A) cellulose ethers or (B) cellulose esters by incorporating therein non-, partly, or wholly polymerised vinyl compounds (vinyl acetate) together with solvents or plasticisers (alcohol, benzene) for the cellulose compounds and/or the polymerised vinyl compounds; poly-

merisation of the vinyl compounds is subsequently completed where necessary. F. R. ENNOS.

Production of solutions and compositions of cellulose esters and ethers and products made therefrom. H. DREYFUS (B.P. 315,766, 16.7.29. Fr., 17.7.28).—Carboxylic esters or ethers of cellulose are dissolved in aqueous, organic, or mixed aqueous and organic solutions of urea, thiourea, guanidine, or their alkyl derivatives, for the production of dopes, varnishes, plastic masses, films, artificial silk, etc. F. R. ENNOS.

Cellulosic compositions. [Plasticisers for plastics, coating compositions, films, etc. from cellulose esters or ethers.] BRIT. CELANESE, LTD. (B.P. 313,535, 13.6.29. U.S., 13.6.28. Addn. to B.P. 313,134; B., 1930, 1061).—Aromatic compounds containing a halogenated side-chain, *e.g.*, dibromides of cinnamic acid, styrene, cinnamyl alcohol, and cinnamaldehyde, are added to cellulosic compositions as plasticisers and fireproofing agents, with or without other such agents. An example is: cellulose acetate (100 pts.), xylene-sulphonmethylamides (25 pts.), tolyl phosphate (7 pts.), cinnamic acid dibromide (12 pts.), and alcohol (60 pts.). C. HOLLINS.

Production and treatment of textile and other materials made of or containing cellulose derivatives. H. DREYFUS (B.P. 334,853, 31.5.29).—Improved strength and elasticity and modifications in lustre result from the treatment of cellulose ether or ester (*e.g.*, acetate) materials, during or after spinning, with "organo-mineral" acids, *i.e.*, di- or poly-hydroxylic inorganic acids in which one or some of the hydroxyl groups have been replaced by organic radicals, excluding, however, sulphaaromatic-fatty acids. Examples are sulphaacetic acid, ethylenesulphonic acid, methane-sulphonic acid, sulphonic acids generally, phosphinic acids, etc. The acids are used in 30–45% aqueous solution. C. HOLLINS.

Waterproofing compositions [for paper containers]. DUNLOP RUBBER CO., LTD., D. F. TWISS, and W. G. GORHAM (B.P. 335,559, 25.6.29).—A protective coating for paper, cardboard, etc. consists of an emulsion of rubber and a vegetable or mineral wax containing 20–95% of wax (calc. on the mixture); vulcanisation is unnecessary, since the presence of the wax prevents tackiness, and, further, the absence of zinc accelerators enables the composition to be used on food containers. A. J. HALL.

Machine for separating solids [and wool-fat] from liquids [wool-scouring wash]. J. and C. E. THOMSON, Assrs. to J. WINSLOE (U.S.P. 1,762,899, 10.6.30. Appl., 17.5.28. N.Z., 4.7.27).—See B.P. 304,718; B., 1930, 368.

Manufacture of artificial silk and the like. W. P. DREAPEY (U.S.P. 1,780,346, 4.11.30. Appl., 3.1.27. U.K., 11.1.26).—See B.P. 273,354; B., 1927, 745.

Esterification of cellulose. F. BECKER, H. HEIMANN, and A. BAYERL, Assrs. to I. G. FARBEIND. A.-G. (U.S.P. 1,780,151, 4.11.30. Appl., 22.12.28. Ger., 24.12.27).—See B.P. 303,006; B., 1930, 504.

[Mechanism for] manufacture of waterproof fibrous products. N.V. DE BATAAFSCHE PETROLEUM

MAATS., Assees. of H. L. LEVIN (B.P. 319,212, 16.9.29. U.S., 17.9.28).

Collecting of artificial wool [from the spinning machine]. M. DASSONVILLE (B.P. 336,763, 26.9.29. Fr., 19.2.29).

Sodium sulphite or bisulphite (B.P. 317,040). **Alkalisising waste pulp-liquors** (B.P. 335,371). **Sulphite waste-liquors** (U.S.P. 1,764,600—1).—See VII. **Diaphragms for electrolytic cells** (B.P. 335,151).—See XI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Naphthenic sulphonic acids in the scouring of soiled fabrics and fibrous materials. G. PETROV (Masloboino Zhir-Delo, 1929, No. 1, 34–42).—The oil-free sulphonic acids and their neutral salts have a detergent power greater than that of the soaps. The presence of sulphuric acid or sulphates has no effect on the detergent properties, which decreased with increase in the mol. wt. The application of the acids and their salts in the washing of fabrics and other materials is discussed. CHEMICAL ABSTRACTS.

Detergent value of soaps. PANTYUKOV.—See XII.

PATENTS.

Coloration of [cellulose ester and ether] textiles and other materials. BRIT. CELANESE, LTD. (B.P. 313,450—1, 11.6.29. U.S., 11.6.28. Addns. to B.P. 282,036; B., 1929, 169).—The method for applying to products containing cellulose ethers or esters fugitive colours from their solutions in organic solvents as described in the chief patent is suitable for permanently colouring (A) similar threads while they are travelling, and (B) fabrics and the like by printing, spraying, or stencilling methods. Polyhydric alcohols or their esters or ethers are particularly suitable as solvents, since they evaporate slowly; the ethylene, diethylene, and propylene glycols are among the numerous suitable solvents cited, and it is recommended that water should be added before use, equal in amount to that which would normally be absorbed by exposure to the atmosphere. A. J. HALL.

Production of coloured discharges. W. W. GROVES. From I. G. FARBEIND. A.-G. (B.P. 335,048, 7.9.29).—Goods impregnated with a 2:3-hydroxynaphthoic arylamide or other coupling component are printed with a paste containing a soluble leuco-ester of a vat dye, an insoluble chromate, and an alkali sulphite. On treatment with a diazo compound, without previous steaming, the vat dye shows as a coloured effect on coloured ground. Examples are: naphthol AS, printed with dibenzpyrenequinone leuco-ester, lead chromate, and potassium sulphite, and developed with diazotised 4-amino-4'-methoxydiphenylamine for yellow on a blue ground; naphthol AS/TR, printed with tetrabromo-indigo leuco-ester, barium chromate, and potassium sulphite, and developed with diazotised 5-chloro-*o*-toluidine for blue on red. C. HOLLINS.

Production of discharge effects on textile materials. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 334,518, 29.4.29).—The action of discharge pastes,

particularly reducing pastes, and especially when used on acetate silk etc., is facilitated by first padding the dyed material with a non-volatile acidic substance, *e.g.*, oxalic acid, benzenesulphonic acid, citric acid.

C. HOLLINS.

Production of discharge effects on materials made of or containing cellulose esters or ether. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 334,543, 29.4.29. Addn. to B.P. 312,655; B., 1929, 678).—Instead of or in addition to chlorates, chromates are used as discharging agents. For coloured discharges a nitro-diarylamine, aminoanthraquinone, or acylaminoanthraquinone is added to the discharge paste. C. HOLLINS.

Treatment of cellulose ester materials. BRIT. CELANESE, LTD. (B.P. 313,971, 20.6.29. U.S., 20.6.28).—Materials containing cellulose esters are partly or completely saponified by subjecting them at 25–30° to the action of aqueous vapours of ammonia or volatile organic bases, *e.g.*, methylamine; the products show increased resistance to ironing and also to the delustring action of hot water, and have a higher moisture regain.

F. R. ENNOS.

Manufacture and treatment of textile or other materials made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 335,583, 16.3.29. Addn. to B.P. 332,231; B., 1930, 944).—The lustre-preserving treatments described in the prior patent are utilised in the production of printed lustre patterns on cellulose ester or ether materials (*e.g.*, cellulose acetate silk fabric is printed with a paste containing a solvent or gelling agent, dried, steamed with dry steam under pressure, and subjected to delustring by boiling in soap, whereby only the printed parts retain their original lustre), or in the manufacture of partly lustred woven and knitted fabrics (*e.g.*, fabric is woven from ordinary cellulose acetate yarn and yarn which has been treated with a gelling agent, and is then delustred).

A. J. HALL.

Treatment [delustring] of textile [artificial silk] fabrics and articles. BRIT. CELANESE, LTD., W. I. TAYLOR, and C. DREYFUS (B.P. 335,204, 15.6.29).—Methods described previously for delustring threads by means of abrasives (*cf.* B.P. 328,247; B., 1930, 763) are now applied to woven and knitted fabrics, whereby pattern lustre effects are obtained. A. J. HALL.

Rendering animal fibre materials immune from attack by moth and the like. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,886, 10.6.29).—The products obtainable by condensing a phenolsulphonic acid with an aralkyl compound, or a phenol with an aralkyl compound and subsequently sulphonating, are applied as mothproofing agents. Examples are products from: 4-chlorophenol-2-sulphonic acid and tetrachlorinated benzyl chloride; 2:4-dichlorophenol and 2:6-dichlorobenzyl chloride, sulphonated; β -naphthol and 2:4-dichlorotoluene- ω -sulphonic acid; β -naphthol and trichlorobenzyl chloride (3 mols.), sulphonated.

C. HOLLINS.

Preparation of fur for shrinking and felting. J. H. MARTIN (U.S.P. 1,762,233, 10.6.30. Appl., 14.7.28).—The felting properties of animal furs are

developed by a pretreatment with solutions of hydrogen peroxide and disodium hydrogen phosphate, either separately or together. Suitable solutions for the treatment of white coney and Australian rabbit skins are given.

A. J. HALL.

Manufacture and treatment of textile [crêpe] fabrics. BRIT. CELANESE, LTD., W. A. DICKIE, and R. W. MONCRIEFF (B.P. 335,180, 17.6.29).—Fabrics are woven from cellulose acetate yarns some of which have been already fully or partly shrunk by treatment with nitric acid (*d* 1.10–1.22) at 0–60° for a few minutes, and are then treated similarly in the woven state, whereby irregular shrinkage occurs and a crêpe or semi-crêpe appearance is produced (*cf.* B.P. 274,584; B., 1927, 747).

A. J. HALL.

Impregnation of textiles and other porous materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,247, 20.6.29).—The materials are treated with solutions in organic solvents of water-insoluble cellulose derivatives (esters or ethers) and of bituminous substances, together with resins, plasticisers, colouring matter, or fillers, if desired, the quantity of solvent being sufficiently great to prevent closing of the pores.

F. R. ENNOS.

Improving the properties of artificial threads and films consisting of regenerated cellulose. W. MARSHALL (B.P. 335,176, 17.6.29).—The threads etc., and also textile materials coated or impregnated with regenerated cellulose, are treated with caustic soda solution (*d* not above 1.14, preferably 1.2–1.3) at 0–60° for a few minutes and then washed free from alkali with brine containing about 15% of sodium chloride (or other salts such as those mentioned in B.P. 323,307 (B., 1930, 281), whereby they gain improved softness, strength, elasticity, extensibility, and more uniform dyeing properties. Caustic potash is less effective than caustic soda (*cf.* B.P. 295,488; B., 1928, 745).

A. J. HALL.

Cleaning of rags by solvents. J. A. L. ROZIERES, and SOC. ANON. DES PROD. OLÉIQUES (B.P. 312,319, 17.5.29. Fr., 24.5.28).—Rags are continuously extracted in a digester by connecting in turn with a number of solvent holders containing solvent of varying grease content; after passing out of the digester the solvent is centrifuged to remove solid particles and returned to its appropriate holder. At the same time the solvent richest in grease flows continuously to a still, while the condensed solvent so obtained is returned to the holder used only for pure solvent, from which part is allowed to pass to the first container in order to maintain the grease content of the solvent contained therein at the desired value.

F. R. ENNOS.

Clearing cotton fibres out of digesters. BRIT. BEMBERG, LTD. (B.P. 335,457, 3.12.29. Ger., 15.12.28).—After running off the treating liquid, the fibres are thoroughly mixed with fresh liquid by the introduction of compressed air, which is then employed to expel the mixture of fibre and liquid.

F. R. ENNOS.

Mordanting and dyeing of materials made with or containing cellulose derivatives. H. DREYFUS (U.S.P. 1,779,464, 28.10.30. Appl., 22.11.27. U.K., 3.12.26).—See B.P. 287,204; B., 1928, 402.

Dyeing of cellulose derivatives. G. RIVAT, E. CADGÈNE, and C. DREYFUS, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,779,494, 28.10.30. Appl., 30.6.26).—See B.P. 273,692; B., 1928, 744.

Dyeing of cellulose esters and ethers. E. A. SPEIGHT, Assr. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,780,320, 4.11.30. Appl., 27.4.29. U.K., 30.5.28).—See B.P. 314,651; B., 1929, 716.

Ornamental fabric and its manufacture. C. DREYFUS and W. R. BLUME, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,780,645, 4.11.30. Appl., 26.6.28).—See B.P. 314,396; B., 1930, 1106.

Dyeing machinery. H. A. BLUNDELL, and BLUNDELL BROS. (LUTON), LTD. (B.P. 336,686, 23.7.29).

Dyeing machines for automatic fabric return. O. OBERMEYER (B.P. 336,724, 26.8.29).

Apparatus for liquid treatment of textile materials and the like. C. F. HAMMOND and W. SHACKLETON (B.P. 336,963, 22.7.29).

Drying apparatus [for clothes etc.]. C. B. COOKE (B.P. 336,679, 20.7. and 27.9.29).

Dyeing with vat dye enolic sulphuric esters (B.P. 334,921). **Soluble compositions for dyeing or printing** (B.P. 334,529).—See IV. **Treatment of textile materials** (B.P. 334,853).—See V. **Sulphite waste-liquors** (U.S.P. 1,764,600—1).—See VII. **Fire-proofing of fabrics** (B.P. 334,408).—See IX. **Colouring rubber oil substitute** (B.P. 335,912).—See XII.

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

Reaction of diphenylamine-sulphuric acid with nitrates in the presence of chlorides, with especial reference to its use in the determination of nitrates in arable soil. H. RIEHM (Z. anal. Chem., 1930, 81, 353—377).—In the reaction between nitric acid and diphenylamine in the presence of hydrochloric acid 1 mol. of nitric acid oxidises 1.5 mols. of the amine to the blue holoquinonoid salt. In the presence of excess amine two further reactions take place slowly: in the first place, the excess amine reduces the blue salt to a grey-green quinhydrone compound; and secondly, intensification of the blue colour occurs, possibly owing to the oxidation by atmospheric oxygen of the nitric oxide formed in the primary action. The stability of the blue colour is therefore at a maximum when equivalent quantities of nitrate and amine are present, and decreases with increasing excess of amine. A stable standard solution may be prepared by mixing equivalent quantities of diphenylamine and nitrate in the presence of sulphuric acid and diluting this solution as required with sulphuric acid of the same concentration. The reagent for a nitrate test is made by mixing 50 mg. of diphenylamine with 380 c.c. of water and sufficient sulphuric acid (which has previously been boiled with 5 g. of potassium chloride for 15 min.) to make 1 litre. For the analysis of a soil 5 g. are shaken with 50 c.c. of 2% potassium chloride solution for 30 min. and 2 c.c. of the filtered liquor are treated with 5 c.c. of the reagent, cooled, shaken, and set aside

for 2 hrs. The resulting colour is compared with that of solutions containing known amounts of nitrate in 2% potassium chloride after treatment with the reagent in the same way. Nitrites should be destroyed by addition of carbamide prior to making the test.

A. R. POWELL.

Deposition of vanadium pentoxide as catalyst on chamotte. I. E. ADADUROV and G. K. BORESKOV (J. Chem. Ind., Russia, 1929, 6, 208—209).—A solution of ammonium metavanadate, just acidified with hydrochloric acid, is boiled with chamotte. Precipitation is incomplete even after 1.5 hrs. CHEMICAL ABSTRACTS.

Deposition of silver metavanadate on chamotte. I. E. ADADUROV and G. K. BORESKOV (J. Chem. Ind., Russia, 1930, 6, 805—807).—Direct deposition is unsatisfactory; vanadium pentoxide is deposited first. CHEMICAL ABSTRACTS.

Determination of nitrous and nitric acids in concentrated sulphuric acid. H. A. J. PIETERS and M. J. MANNENS (Z. anal. Chem., 1930, 82, 218—224).—Nitrous acid is best determined colorimetrically with *m*-phenylenediamine and the total nitrogen by reducing the neutralised solution with Devarda's alloy and potassium hydroxide, and determining the resulting ammonia acidimetrically. A. R. POWELL.

Manufacture and use of calcium molybdate. H. C. MABEE (Canad. Dept. Mines, Ore Invest., 1928, 156—158).—Calcium molybdate for use in steel manufacture is readily prepared by roasting molybdenite at 600° until free from sulphur, and then heating the resulting molybdenum trioxide at 600° with finely powdered calcium carbonate. A. R. POWELL.

Reports of investigations: [Canadian] non-metallics laboratory. R. K. CARNOCHAN, C. S. PARSONS, and R. A. ROGERS (Canad. Dept. Mines, Ore Invest., 1928, 95—130).—Experimental work on the separation and purification of various non-metallic minerals, including asbestos, calcite, china clay, graphite, and garnet, is recorded. A. R. POWELL.

Precipitation of ferro-vanadium catalyst on chamotte. I. E. ADADUROV and G. K. BORESKOV (J. Chem. Ind., Russia, 1929, 6, 1365—1367).—A solution of ammonium metavanadate (0.1767 g. V_2O_5) is treated with ferric chloride (1.94 g.) and the precipitate is redissolved by addition of sulphuric acid (7.76 g.); the mixture is then heated with chamotte (10 g.), which, after being washed with cold water, retains 0.0635 g. V_2O_5 as $Fe_2O_3 \cdot 4V_2O_5 \cdot 4H_2O$. CHEMICAL ABSTRACTS.

Hydrogen for hydrogenation. A. MARKMAN (Masloboino-Zhir. Delo, 1929, No. 2, 17—20).—The use of electrolytic hydrogen is advocated.

CHEMICAL ABSTRACTS.

Determination of potash in dilute solutions. VON WRANGELL.—See XVI. **Sodium peroxide respirators.** STAMPE and HORN.—See XXIII.

See also A., Nov., 1337, **Preparation and analysis of chromyl chloride** (NÚÑEZ). 1369, **Preparation of hydroxide jellies** (PRAKASH and DHAR). 1380, **Mixed catalysts** (MITTASCH). **Catalytic oxidation of ammonia** (VON NAGEL). **Mixed catalyst in ammonia synthesis** (KEUNECKE and others). 1382, **Electrolytic**

preparation of silver oxide (NAYAR and MACMAHON). 1392, Determination of cyanides by copper sulphate (LORA Y TAMAYO). Determination of alkali hydroxides in the presence of alkali carbonates or of ammonia (SCHEWKET). 1393, Dermatological thallium salts (CLAVERA).

PATENTS.

Production of hydrogen cyanide. IMPERIAL CHEM. INDUSTRIES, LTD., T. S. WHEELER, J. MCAULAY, W. B. FLETCHER, and H. A. T. MILLS (B.P. 335,585 and 335,947, [A, B] 27.3., 20.4., and 24.12.29, [A] 23.4.29).—(A) A mixture of a gaseous hydrocarbon, or gas containing a hydrocarbon, and excess of ammonia is passed through an unpacked reaction chamber, the inner-wall temperature of which is at least 1150° (preferably 1350—1450°). To prevent decomposition of the ammonia and hydrocarbon into their elements with consequent separation of carbon, the mixture should be brought to the reaction temperature as rapidly as possible and exposed for a short time only. The surface of the chamber should be free from materials liable to provoke decomposition of the gases, a suitable lining being glazed silica or sillimanite. (B) The gaseous hydrocarbon, or gas containing a hydrocarbon, is mixed, not with excess, but with the stoichiometric quantity of ammonia, a substantial amount of hydrogen being present as a diluent.

W. J. WRIGHT.

Manufacture of sodium sulphite or bisulphite solutions. ZELLSTOFFFABR. WALDHOF, and O. FAUST (B.P. 317,040, 5.7.29. Ger., 9.8.28).—Waste soda lyes, such as are obtained in artificial silk manufacture, are treated with sulphur dioxide or sulphurous acid, organic substances thus being precipitated and a clear solution of sodium sulphite or bisulphite obtained.

W. J. WRIGHT.

Production of sodium thiosulphate. H. HOWARD, ASSR. to GRASSELLI CHEM. CO. (U.S.P. 1,760,137, 27.5.30. Appl., 5.12.25).—Sodium sulphide solution, *e.g.*, waste sodium sulphide liquor, and sulphur dioxide, either as an aqueous solution or in the form of burner gases, are introduced into a solution of sodium sulphite at 60° at such rates that the sulphur dioxide content of the mixture is maintained in slight excess until the proportions of the reagents indicated by the equation: $2\text{Na}_2\text{S} + 3\text{SO}_2 + \text{Na}_2\text{SO}_3 = 3\text{Na}_2\text{S}_2\text{O}_3$ have been reached. Neither hydrogen sulphide nor free sulphur is thus appreciably liberated.

W. J. WRIGHT.

Alkalisng the waste liquors from the soda- or sulphate-pulp manufacture for the purpose of dry-distilling the same. E. L. RINMAN (B.P. 335,371, 24.9.29. Swed., 15.7.29).—After evaporation to *d* 1.25, the waste liquors are treated with oxide or hydroxide of barium or strontium, or a mixture of these, in an open vessel with stirring apparatus. The thick liquor obtained is evaporated to dryness and yields a pulverulent mass which does not sinter, this mass being then dry-distilled in a continuous furnace. Oxides or hydroxides of calcium, magnesium, aluminium, zinc, or iron may be added during alkalisng; if an inert substance, such as powdered carbon, be added, the subsequent dry distillation will be the more easily controlled.

W. J. WRIGHT.

Manufacture of products from sulphite waste liquors. (A) W. E. B. BAKER, (B) W. E. B. BAKER and F. G. ROBERTS, ASSRS. (B) to W. E. B. BAKER (U.S.P. 1,764,600—1, 17.6.30. Appl., [A] 7.4.23, [B] 26.5.23).—Sulphite waste liquor, after neutralising with lime to *p*_H 9, filtering, and concentrating to a 50% solid content, is (A) treated with zeolite, filtered from the calcium aluminium silicate formed, and acidified to the desired degree with acetic acid, yielding a solution of sodium ligninsulphonate suitable for treatment of leather and as a mordant; or (B) stirred with chromic acid solution until gelatinisation occurs, the product, after washing with water and dilute acid to remove sulphates and hydrated chromic oxide, consisting of lignin in the form of a reversible gel free from ligninsulphonic acid.

F. R. ENNOS.

Production of alkali sulphates. CHEMIEVERFAHREN GES.M.B.H. (B.P. 311,226, 22.4.29. Ger., 7.5.28. Addn. to B.P. 300,630; B., 1929, 718).—In a modification of the prior process, the crude potash salt, after roasting or grinding, is heated with mother-liquor from a previous operation, substantially free from ammonia, to hydrate the kieserite, and the cooled mixture is treated with ammonia, the temperature being kept constant. The potassium sulphate which settles out is treated with water to remove sodium sulphate, and the solution is returned to the process after hydration of the kieserite. The mother-liquor, containing ammonia and chlorides of ammonium, magnesium, and sodium, is treated with carbon dioxide, whereby magnesium ammonium carbonate is precipitated, and cooled to separate ammonium chloride, the final liquor being treated again with carbon dioxide to precipitate sodium bicarbonate. From the magnesium ammonium carbonate, magnesium carbonate may be obtained. If the magnesium ammonium carbonate is not removed, its mixture with ammonium chloride may be dried and calcined, so as to yield anhydrous magnesium chloride.

W. J. WRIGHT.

Production of sodium fluoride by the dry method. E. V. BRITZKE, W. I. BREMPPEL, and M. E. JAKUBOWITZ (B.P. 335,703, 29.8.29).—Hydrofluoric acid gas at about 70° is introduced into a tubular chamber in counter-current to calcined or caustic soda, which is conveyed through the tube by means of a central shaft with stirrers. The heat evolved is sufficient to evaporate the water formed in the reaction, so that dry, friable sodium fluoride is obtained, and by regulating the amounts of the components either the acid or neutral salt may be produced.

W. J. WRIGHT.

Manufacture of diammonium phosphate. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,008, 10.7.29).—Loss of ammonia, which results when a solution of diammonium phosphate is evaporated to dryness, is avoided by mixing the solution with solid diammonium phosphate and drying this mixture. In the preparation of diammonium phosphate by treating crude phosphate with sulphuric acid, the solution, after separation of calcium sulphate, is concentrated to *d* 1.4—1.5, cooled, and filtered. The phosphoric acid, free from calcium phosphate, thus obtained is neutralised with ammonia, the diammonium phosphate solution being treated as described.

W. J. WRIGHT.

Production of (A) alkali phosphates, (B) acid disodium pyrophosphate. METALLGES. A.-G. (B.P. 335,492 and 335,501, [A] 25.1.30, [B] 14.2.30. Ger., [A] 17.10.29, [B], 17.5.29).—(A) The production of alkali phosphates by fusing alkali sulphates with ferrophosphorus is effected without explosive reaction, such as may occur when a rotary furnace is employed, by heating the finely-ground components in an electric furnace. (B) A solution of phosphoric acid, mixed with sodium chloride, is evaporated *in vacuo* at 180–200°, higher temperatures being avoided to prevent the formation of insoluble metaphosphates. W. J. WRIGHT.

Production of alkaline-earth titanates. R. H. MONK and L. FIRING; the latter Assr. to J. IRWIN (U.S.P. 1,760,513, 27.5.30. Appl., 10.2.27).—An aqueous paste of freshly-precipitated titanium hydroxide is treated with an acetate of an alkali metal or ammonium to react with any sulphate ions, which are detrimental to the formation of titanates. After washing with water to remove the sulphate, an alkaline-earth carbonate, *e.g.*, barium carbonate, is added in the form of paste to produce a compound such as BaTiO₃. Hydrochloric acid or barium chloride is added to accelerate the reaction and give the product a pure, white colour, and the mixture is finally heated to 750–880°.

W. J. WRIGHT.

Purification of zinc sulphate liquors. O. A. FISCHER, Assr. to R. H. CHANNING, JUN. (U.S.P. 1,761,782, 3.6.30. Appl., 10.2.27).—Iron salts are removed from zinc sulphate liquors by treating the latter with a mixture of minute bubbles of sulphur dioxide and air, produced by passing the mixed gases through a porous medium. In order to ensure complete oxidation of the iron salts, the sulphur dioxide should be caused to bubble through at least 2 ft. of the liquor. A precipitating agent is then added to the solution or, if desired, this may be added before the sulphur dioxide is introduced.

W. J. WRIGHT.

Production of aluminium salts. COLLOID-CHEM. FORSCHUNGS A.-G., Assees. of E. HERZFELD and H. WALKER (B.P. 336,181, 7.1.30. Switz., 7.1.29).—In the acid process for extracting aluminium, the difficulty in recovering iron-free aluminium salts from the mother-liquor is overcome by adding 96% alcohol. The almost pure aluminium sulphate, which is precipitated, is dissolved in water and reprecipitated with alcohol.

W. J. WRIGHT.

Mouldable product and its preparation. B. W. NORDLANDER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,761,740, 3.6.30. Appl., 14.5.29).—A thermoplastic material is obtained by fusing selenium sulphide with a suitable filler, such as asbestine, iron oxide, etc., at about 125° and then cooling; after pressing, the product may be hardened by "curing" at 80–90° for ½ hr. An acidproof coating is produced on materials such as iron by suspending the selenium sulphide and a filler in a volatile liquid and spraying this on the iron. After evaporation of the liquid, the iron is heated at 105–115° and "cured."

W. J. WRIGHT.

Treatment of mixtures containing carbon dioxide and hydrogen for reducing or eliminating the hydrogen content, and the formation of carbon

monoxide therefrom. BRIT. CELANESE, LTD., W. BADER, and E. E. STIMSON (B.P. 335,632, 2.7.29).—The mixture of carbon dioxide and hydrogen is passed through a tube containing chromite, heated to 700–900°, by which means the hydrogen is largely eliminated; the treated mixture is then passed over heated carbon to yield carbon monoxide. W. J. WRIGHT.

Manufacture of mixtures of nitrogen and hydrogen. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 335,524, 24.4.29).—The conversion of a solid, liquid, or gaseous carbonaceous mixture into a mixture of nitrogen and hydrogen by treatment with an electric arc together with air, hydrogen, or carbon dioxide has the disadvantage that the resultant mixture may contain undesirable amounts of methane or carbon dioxide. To avoid this, baffle plates are arranged at the end of the electric arc, thus promoting the intimate mixing of the gas by forming eddies, and preventing the formation of low-temperature zones, a temperature of at least 1400° being attained.

W. J. WRIGHT.

Manufacture of sulphur trioxide. CALCO CHEM. Co., Assees. of N. A. LAURY (B.P. 317,396, 9.7.29. U.S., 15.8.28).—A carrier for a vanadium catalyst used in the contact process is prepared from particles of a natural diatomaceous earth of a size larger than 6-mesh, these being fired at about 1000° and thereby acquiring great strength and becoming non-friable.

W. J. WRIGHT.

Production of phosphorus oxychloride. J. S. DUNN, F. BRIERS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 336,065, 6.9.29).—Rock phosphate is treated with phosgene or with a mixture of carbon monoxide and chlorine at 350–600°, the calcium chloride formed being prevented from covering the phosphate, so that fresh surfaces are constantly exposed. This is effected by continuous grinding and stirring or by introducing coke, previously heated to 350°, or the process may be conducted in a rotary furnace in presence of pebbles. The mass may be intermittently washed with water, and the residual solid, after drying, again phosgenated.

W. J. WRIGHT.

Treatment of bromine and iodine. GOVETT, LTD., Assees. of E. GOVETT (B.P. 308,281, 20.3.29. U.S., 20.3.28).—A colloidal form of iodine ("iodine alpha") is prepared by causing iodine to react with an aqueous solution of tannic or gallic acid, and treating the colloidal hydrogen iodide produced with a nitrate or acetate of mercury or other suitable metal. The insoluble metallic iodide is decomposed with colloidal bromine, so as to liberate iodine, either free or combined with hydrogen, in solution, and this on evaporation yields iodine alpha. Colloidal bromine is preferably prepared by dissolving potassium bromate in tannic acid, removing the insoluble potassium tannate, and evaporating the hydrogen bromide solution to dryness. Alkali bromides and iodides are obtained in a colloidal form by treating the halogens with tannic or gallic acid, and causing the hydrogen halide to react with an alkali hydroxide; after removal of the potassium tannate, the hydrogen iodide is neutralised with sodium hydroxide. Both the colloidal halogens and the colloidal halides have therapeutic properties.

W. J. WRIGHT.

Stabilisation of hydrocyanic acid. H. LEHRECKE, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,780,198, 4.11.30. Appl., 23.6.26. Ger., 3.7.25).—See B.P. 254,747; B., 1927, 813.

Calcium chloride (U.S.P. 1,764,582).—See I. **Distillation products from vegetable substances** (B.P. 334,724). **Ammonia from coal gas etc.** (B.P. 334,619). **Sulphates and sulphur from gases** (B.P. 334,526). **Cyanogen compounds from gases** (U.S.P. 1,742,505).—See II. **Urea etc.** (B.P. 335,421 and 334,564).—See III. **Diaphragms for electrolytic cells** (B.P. 335,151). **Electrolysis of fused electrolytes** (B.P. 316,969). **Electrolytic production of oxygen and hydrogen** (B.P. 335,987).—See XI. **Fertiliser salts** (B.P. 335,175). **Fertiliser for seeds** (U.S.P. 1,762,294). **Weed-killer** (B.P. 335,203).—See XVI.

VIII.—GLASS; CERAMICS.

Devitrification of soda-lime-silica glasses. G. W. MOREY (J. Amer. Ceram. Soc., 1930, 13, 683—713).—A special study is made of that part of the ternary system $\text{Na}_2\text{O}, \text{SiO}_2\text{--CaO}, \text{SiO}_2\text{--SiO}_2$ which is of importance to the glass industry, viz., the range including 62—78% SiO_2 , 0—20% CaO , this region being represented by 87 glasses, instead of by 20 as in the previous work (cf. Morey and Bowen, B., 1925, 921). In a discussion of devitrification it is shown that the liquidus temperature is the only datum point which is solely a function of glass composition; at the liquidus the first trace of crystalline phase is in equilibrium with the glass; above it, the glass will dissolve any crystals which may have been formed, whilst crystals will tend to separate only below the liquidus. This and related phenomena are discussed at length. F. SALT.

Effect of alumina on the devitrification of a soda-lime-silica glass. G. W. MOREY (J. Amer. Ceram. Soc., 1930, 13, 718—724).—Starting with an initial glass of approximately the composition $\text{Na}_2\text{O}, \text{CaO}, 6\text{SiO}_2$, two series of experiments were carried out; in the first CaO was partly replaced by Al_2O_3 ; in the second alumina was added to the glass. In the first series 2% of alumina lowered the m.p. by about 80°, and the glass rapidly crossed the tridymite field and entered the field of $\text{Na}_2\text{O}, 3\text{CaO}, 6\text{SiO}_2$. On addition of further alumina, the liquid crossed this field and the CaO, SiO_2 field, until, on further replacement of CaO by Al_2O_3 , the mineral albite ($\text{Na}_2\text{O}, \text{Al}_2\text{O}_3, 6\text{SiO}_2$) finally became the primary phase, and the curve ended at the m.p. of albite, 1100°. In the second series of experiments tridymite was the initial phase; the glass containing 4% Al_2O_3 formed $\text{Na}_2\text{O}, 3\text{CaO}, 6\text{SiO}_2$ when crystallised at 1000°, but this decomposed into CaO, SiO_2 before the liquidus was reached. Wollastonite remained the primary phase as far as the curve was followed, to 9.1% Al_2O_3 and 1156°, at which point the temperature was rising rapidly. F. SALT.

Effect of magnesia on the devitrification of soda-lime-silica glass. G. W. MOREY (J. Amer. Ceram. Soc., 1930, 13, 714—717).—A series of experiments was carried out, beginning with a glass of the composition $1.15\text{Na}_2\text{O}, 0.84\text{CaO}, 6\text{SiO}_2$, in which tridymite separated as the primary phase. The addition of 2%

of magnesia lowered the m.p. considerably and the glass passed to the $\text{Na}_2\text{O}, 3\text{CaO}, 6\text{SiO}_2$ field, but with more than 3% of magnesia diopside became the primary phase and the m.p. rose rapidly. Complete replacement of CaO by MgO lowered the m.p., but tridymite remained the primary phase. F. SALT.

Dimensional changes caused in glass by heating cycles. A. Q. TOOL, D. B. LLOYD, and G. E. MERRITT (J. Amer. Ceram. Soc., 1930, 13, 632—654).—Nine lots of small samples of a medium flint glass were treated over the range 360—620°, the heating periods being varied from a few minutes at 620° to more than 1½ months at 360°. The dimensional changes produced by subjecting these glasses to heating cycles approaching the annealing range were measured, and the results are shown graphically. The total “permanent” change in length produced in this glass at 370—470° was found to be 17—18 μ per cm. R. J. CARTLIDGE.

Mechanical properties of some rolled and polished glass. E. M. GUYER (J. Amer. Ceram. Soc., 1930, 13, 624—631).—Young's modulus of elasticity, modulus of rupture, impact modulus of rupture, and wind-resistance were determined for various types of glass. The apparatus used in the four series of tests is described, and the results are tabulated. R. J. CARTLIDGE.

Use of polariscopes in the glass industry. F. W. PRESTON (J. Amer. Ceram. Soc., 1930, 13, 595—623).—The theory and construction of various types of polariscopes are discussed, and the application of such instruments in the detection of strain in glass is described. R. J. CARTLIDGE.

Design for enamel smelting furnace. R. D. COOKE (J. Amer. Ceram. Soc., 1930, 13, 655—657).—For corrosive enamels, which seriously reduce the life of the furnace lining, an open-hearth furnace is described, having the walls and the entire outside of the hearth exposed to the air. By using good-quality smelter brick in place of an insulating lining the life of the hearth was doubled and the cost reduced. R. J. CARTLIDGE.

Soluble salts in enamels. R. D. COOKE (J. Amer. Ceram. Soc., 1930, 13, 658—661).—Increasing amounts of borax, sodium carbonate, and sodium sulphate were added to portions of an enamel which had been washed free from soluble salts, and the effects noted. The cause of salts “setting-up” the enamel to proper consistency and the prevention or promotion of rusting of the steel during drying was also studied. It is assumed that these properties depend entirely on the nature and amounts of salts in solution in the enamel water. R. J. CARTLIDGE.

Determination of the mullite content of porcelains by chemical means and the relation between such mullite content and the properties of the porcelain. R. RIEKE and W. SCHADE (Ber. Deut. Keram. Ges., 1930, 11, 427—442).—A serviceable method of separating the mullite content from porcelain, giving comparable and sufficiently accurate values, is presented. The influence of the temperature and concentration of the hydrofluoric acid, and the effect of varying the time of the reaction were studied in further detail. The effect of the mineralogical composition and

the temperature and duration of firing on the mullite content was determined on a number of trial porcelains containing 40–60% of clay substance, 15–35% of quartz, and 15–35% of felspar. A study of the relation between mullite content and the physical properties of porcelain led to no definite result. Technical porcelains of different origins were tested for mullite content, from which conclusions were drawn regarding firing temperature and composition. The influence of the kaolin was determined on a number of porcelains of the same "rational" composition, but made with different kinds of kaolin. F. SALT.

Consistencies of raw terra cotta glazes. L. D. FETTEROLF (J. Amer. Ceram. Soc., 1930, 13, 675–682).—The glaze examined was composed of felspar 64.3%, flint 2.1%, whiting 8.9%, calcined zinc oxide 15.0%, and clay 9.7%. Tests were made on consistency changes of the glaze with ageing, and the effect of using an aged clay slip, a heat-treated glaze, and of the dissolution of salts from glaze materials on the causes of spontaneous thinning were noted. Additions of gum arabic in suitable amount served to stabilise glaze consistency satisfactorily. The results of adding acid, alkali, and water to such glazes are shown graphically. R. J. CARTLIDGE.

Effect of temperature on cracking of plastic clay; temperature gradients inside the clay during steady heating in a saturated atmosphere and on drying at 100°. S. R. HIND and F. WHEELER (Trans. Ceram. Soc., 1930, 29, 314–325).—Test pieces of a standard purified Stourbridge clay containing 30.7–10.3% of moisture were subjected to steady heating in a saturated atmosphere, and the critical cracking temperatures were noted. Data obtained show that the critical temperature at which cracking commences is a function of the moisture content of the piece, and that with this clay no cracking occurs in a saturated atmosphere at any temperature up to the b.p. of water, when the moisture content of the piece is below 21.5% (on dry weight). R. J. CARTLIDGE.

Effects of firing temperature, kind of grog, and grading on the properties of firebrick material. A. J. DALE (Trans. Ceram. Soc., 1930, 29, 326–338).—Twenty-four sets of full-sized bricks were made of a single-bond clay admixed with special grogs, namely, "aloxite," "baddeleyite," silica glass, quartzite, and diaspore, which had previously been divided into coarse and fine grades. All the mixtures except diaspore contained 63% of grog and 37% of bond clay; with diaspore 25% of clay was used. Each set of bricks was divided into two parts, one portion going through the low-temperature building-brick fire, and the other to the firebrick kiln. The density, porosity, ordinary refractoriness, and refractoriness under a load of 50 lb./in.² of the mixtures were determined, and from data obtained it is concluded that when the amount of clay is not excessive, the value of a clay-bonded refractory is apparently governed by the nature of the grog, not of the bond-clay itself. R. J. CARTLIDGE.

[Separation of china clay and of garnet in] Canadian laboratory. CARNOCHAN and others.—See VII.

See also A., Nov., 1374, System $K_2O-CaO-SiO_2$ (MOREY and others).

PATENTS.

Tunnel kiln. A. McD. DUCKHAM (U.S.P. 1,780,119, 28.10.30. Appl., 16.7.28. U.K., 12.8.27).—See B.P. 285,323; B., 1928, 711.

Manufacture of refractory material. J. I. BRONN (U.S.P. 1,780,114, 28.10.30. Appl., 28.2.28. Ger., 15.7.26).—See B.P. 304,492; B., 1929, 247.

Glass-furnace forehearth. J. MONCRIEFF, LTD., and A. F. McNISH (B.P. 336,883, 6.1.30. U.S., 9.8.29).

Annealing furnaces [for glass]. W. A. MORTON (B.P. 336,596, 15.4.29).

Manufacture of cast glass plates and sheets. Y. BRANCART (B.P. 336,921 and Addn. B.P. 336,924, [A] 10.2.30, [B] 17.2.30. Belg., [A] 14.2.29, [B] 27.4.29).

[Frame for] manufacture of safety or unsplinterable glass. T. W. HOLT and J. F. W. STUART (B.P. 337,158, 2.10.29).

Manufacture of bonded abrasive articles. CARBORUNDUM Co., Assees. of H. C. MARTIN (B.P. 316,676, 31.7.29. U.S., 2.8.28).

Grading of abrasives for use in continuous glass-grinding machines, and apparatus therefor. PILKINGTON BROS., LTD., F. B. WALDRON, and J. H. GRIFFIN (B.P. 336,587–8, 12.7.29).

Tunnel kiln (U.S.P. 1,763,624).—See I. **Enamelling furnace** (B.P. 336,168). **Insulating material** (B.P. 315,265).—See XI.

IX.—BUILDING MATERIALS.

Application of the filtration method in volumetric analysis, especially in the analysis of Portland cement. H. T. BUCHERER and F. W. MEIER (Z. anal. Chem., 1930, 82, 1–44).—The method consists in adding the standard reagent to the test solution until no further precipitation can be observed, then filtering a small portion and testing one part of the filtrate with the reagent and another part with a standardised solution of the substance being determined. In this way a close approximation to the true result is obtained; a second trial is then made, adding a small deficiency of the precipitant without making a filtration and subsequently making tests after the addition of each succeeding drop until the filtrate ceases to give a precipitate. For the determination of calcium by the method, the neutral solution is treated with sodium acetate equivalent to 1–1½ times the amount of calcium present and titrated at 80° with oxalic acid. Magnesium may be determined with 8-hydroxyquinoline in a solution (100 c.c.) containing 20 c.c. of 2N-ammonium chloride and 10 c.c. of ammonia (d 0.88) at 80°; in this case the filtrate is coloured yellow when precipitation is complete. Both metals may be determined in the same solution, the calcium being first titrated with oxalic acid and then the magnesium with 8-hydroxyquinoline after addition of ammonia and ammonium chloride without filtering the calcium oxalate. Zinc and copper may be determined by titration of the ammoniacal solution with sodium sulphide at 20°; equally good results for zinc may be obtained

in acetic acid solution and for copper in sulphuric acid, and by working in these acid solutions copper and zinc may be determined successively in the same trial. Manganese may be titrated with sodium phosphate at 75° in a feebly ammoniacal 10% ammonium chloride solution, and iron and aluminium may be determined by precipitation of the cold solution containing 4 g. of sodium acetate per 100 c.c. with 8-hydroxyquinoline and titration of the excess of precipitant with bromine after filtration and acidification of the filtrate with hydrochloric acid. For the analysis of Portland cement, 1 g. is evaporated with 1:1 nitric acid to dryness and the residue is heated at 115°, cooled, and extracted with a 1:1 mixture of ether and absolute alcohol. The insoluble material is collected, washed with the extraction mixture, and digested with hydrochloric acid to obtain the silica in the usual way. The filtrate from the silica and the alcoholic solution are united and the iron and alumina removed with ammonia; calcium and magnesium are determined in an aliquot part of the filtrate by the methods described above, and the sum of the iron and alumina by the hydroxyquinoline titration. Iron alone is determined iodometrically in a portion of the solution obtained by dissolving the ammonia precipitate in hydrochloric acid. A. R. POWELL.

Petrographic investigation of aluminous cements. K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 352–358 B).—Fusion of a mixture containing 47% CaO, 44% Al_2O_3 , and 9% SiO_2 gives a clear glass which devitrifies on heating at 1200° for 24 hrs. The devitrified material resembles porcelain in appearance and contains numerous spheruliths exhibiting needle-shaped double-refracting crystals radiating from the centre; under crossed nicols thin layers show either blue or yellow interference colours. The optical properties of devitrified calcium aluminates ranging from $3\text{CaO}, 5\text{Al}_2\text{O}_3$ to $3\text{CaO}, \text{Al}_2\text{O}_3$ have been investigated: $3\text{CaO}, 5\text{Al}_2\text{O}_3$ forms prismatic doubly-refracting crystals, and $\text{CaO}, \text{Al}_2\text{O}_3$ a confused mass of twinned crystals exhibiting blue interference colours; $3\text{CaO}, \text{Al}_2\text{O}_3$ forms isotropic octahedral crystals containing free calcium oxide; the stable modification of $5\text{CaO}, 3\text{Al}_2\text{O}_3$ forms anisotropic hexagonal crystals which exhibit only feeble double refraction, whereas the crystals of the labile modification resemble in appearance spheruliths. None of these substances forms true spheruliths such as are found in the cement. $2\text{CaO}, \text{Al}_2\text{O}_3$ on fusion and devitrification gives a mixture of $3\text{CaO}, \text{Al}_2\text{O}_3$ and $5\text{CaO}, 3\text{Al}_2\text{O}_3$, and a third substance of which the isotropic crystals radiate from nuclei; $3\text{CaO}, 2\text{Al}_2\text{O}_3$ crystallises in well-developed spheruliths which show the blue and yellow interference, and are apparently identical with those of the cement; the sintering temperature of this compound is about 1250° and the temperature of complete fusion 1300°.

H. F. GILLBE.

Injury to plaster due to osmosis. F. L. BRADY (Nature, 1930, 126, 684).—When lime plaster is applied to bricks containing magnesium sulphate, a semi-permeable membrane is precipitated in the brick and, as the wall dries out, magnesium sulphate concentrates behind the membrane, crystallises, and forces the plaster, together with a thin shell of brick, away from the wall.

Membranes of magnesium hydroxide capable of supporting osmotic pressures equal to 100 cm. of water have been precipitated in the walls of porous pots.

L. S. THEOBALD.

Asphalt tests. STRIETER.—See II.

PATENTS.

Treatment [fireproofing] of combustible substances [e.g., wood, fabrics]. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 334,408, 28.9.29).—The materials are impregnated with a solution containing a combustion-retarding agent, e.g., ammonium phosphate, and a urea-formaldehyde condensation product. Other substances preventing crystallisation of the salt, e.g., starch or glue, may be added also, if desired.

A. B. MANNING.

Treatment of sawdust. E. ERLBACH (U.S.P. 1,764,249, 17.6.30. Appl., 12.8.26. Ger., 18.8.25).—Sawdust, made into a pulp with water containing caustic alkali, is pumped for about 2 hrs. through a pressure-proof system heated at about 300° at superatmospheric pressure, and the products of decomposition (acetic acid, methyl alcohol, acetone, and tar) are drawn off.

F. R. ENNOS.

Manufacture of [stringed] bricks. T. KOIDL and F. GORLICH (B.P. 337,231, 20.11.29. Austr., 20.11.28).

Moulding of artificial stone and imparting a finished surface thereto. F. T. WALKER (B.P. 337,074, 30.7.29).

[Compressed-air apparatus for] impregnation of timber. C. GOODALL (B.P. 337,133, 18.9.29).

Bituminous emulsions (B.P. 312,580). Emulsions with humic acid (B.P. 334,426).—See II. **Casein-glue film (B.P. 314,344).**—See XV. **Immunising wood (B.P. 335,527).**—See XVI.

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

Reduction of iron ores with coal in coke ovens. E. V. BRITZKE and I. V. SHMANENKOV (Min. Suir., 1930, 5, 86–91).—Phosphorus, arsenic, vanadium, and titanium are practically completely removed. When more than 50% of ore is used the coke becomes pulverulent. Reduction is due to carbon, hydrogen, and methane. About 80–95% of the iron is reduced to the metal and 5–15% to ferrous oxide, whilst 1–5% remains unreduced.

CHEMICAL ABSTRACTS.

Reports of investigations; [Canadian] ore-dressing and metallurgical laboratories. [Beneficiation and low-temperature reduction of high-sulphur iron ore and the metallisation of the iron content of ilmenite.] R. J. TRAILL, W. R. McCLELLAND, and J. D. JOHNSTON (Canad. Dept. Mines, Ore Invest., 1928, 82–94).—The iron ores tested contained 53.35% Fe, 2.95% S, 9% SiO_2 , and 5% CaO; roasting in a Herreshoff or rotary kiln furnace reduced the sulphur to a minimum of 0.8%, and subsequent reduction to sponge iron and magnetic separation gave a product containing a minimum of 0.6% S. Metallisation tests on an ilmenite containing 39% Fe, 31.75% TiO_2 , 7.7% Al_2O_3 , 7% SiO_2 , and 2.6% MgO showed that 94% of the iron

could be reduced to metal at 990–1050° by means of coal in a rotary kiln type of furnace. Leaching of the product with 10% sulphuric acid gave a residue containing 75.8% TiO_2 , 6.27% Fe, and 0.8% C.

A. R. POWELL.

Loss in burning during steel heating. N. Y. TAITZ (J. Russ. Met. Soc., 1929, Mem., 288–326).—Data concerning surface oxidation during heating, rolling, and cooling are compared. A reducing atmosphere in the heating furnace does not appreciably decrease the loss. The metal should remain for a minimal time in the hottest zone; a magnesite bottom is recommended.

CHEMICAL ABSTRACTS.

Oxygen content of basic mild open-hearth steel before the addition of deoxidisers. A. K. SILIN (J. Russ. Met. Soc., 1929, Mem., 102–112).—Metal in a furnace heated by wood generator gas contained less oxygen than that in a furnace heated by crude oil. With a deeper hearth the oxygen content of the metal was lower. The oxygen content does not depend on the temperature, provided that the percentage of carbon is constant.

CHEMICAL ABSTRACTS.

Physical chemistry of steel manufacture. De-oxidation with silicon in the basic open-hearth process. C. H. HERTY, JUN., C. F. CHRISTOPHER, and R. W. STEWART (Min. Met. Invest. U.S. Bur. Mines etc. Co-op. Bull., 1930, No. 38, 1–172).—The amounts and types of silicates formed on deoxidation with silicon, the time required for their elimination, and their effect on the cleanliness of steel in the ladle have been investigated. There is formed at the slag-metal interface a layer of silicates which do not enter the slag unless a very long time is allowed for the re-boil. For the reaction $\text{Si} + 2\text{FeO} \rightleftharpoons \text{SiO}_2 + 2\text{Fe}$ the equilibrium constant $k = 1.65 \times 10^{-4}$. A number of general conclusions have been reached.

CHEMICAL ABSTRACTS.

Mechanical properties of Thomas steel and open-hearth steel. K. DAEVES (Stahl u. Eisen, 1930, 50, 1353–1356).—Average values of the mechanical properties (taken from a large number of tests) are compared for steels produced in the Thomas converter and in the Siemens-Martin furnace. For equal values of tensile strength Thomas steels have an elongation about 2% and a reduction in area about 4% greater than that of open-hearth steels; they have, however, a higher phosphorus and a lower carbon content. Thomas steel hardens more rapidly on drawing and cold-rolling and is therefore preferable for the manufacture of wires and drawn bars of all shapes, whereas open-hearth steel is to be preferred for sheet and deep drawing. The latter steel is less sensitive to ageing and its properties are rarely deleteriously affected by heat-treatment after cold work, whereas Thomas steel becomes brittle during normalising, and should be used, therefore, only when no subsequent heat treatment is to be given after cold work.

A. R. POWELL.

Prolonged loading tests on constructional steels at high temperatures. E. POHL, H. SCHOLZ, and H. JURETZKE (Arch. Eisenhüttenw., 1930–1, 4, 105–110; Stahl u. Eisen, 1930, 50, 1330–1331).—The minimum load required to cause creep at 300–350°

has been determined for a number of steels by subjecting them to a stress equal to about one third the yield-point load at the temperature of test and then gradually increasing this load until a slow, permanent, plastic flow is produced. Under these conditions a mild steel having a tensile strength of 35–44 kg./mm.² at 20° commenced to creep with a load of 15.2 kg./mm.² at 300°, 8.45 kg./mm.² at 400°, and < 3.2 kg./mm.² at 500°. The values for a 2% nickel steel (60 kg./mm.² at 20°) were > 20 kg./mm.² at 350°, 9.85 kg./mm.² at 450°, and 5.38 kg./mm.² at 500°. A molybdenum steel having the same tensile strength as the above carbon steel had a permanent strength of 11.2 kg./mm.² at 500°. By applying a load just below the permanent strength at the testing temperature for some time, the metal becomes appreciably stronger.

A. R. POWELL.

Effect of small quantities of copper on the resistance to corrosion of structural steel. O. BAUER, O. VOGEL, and C. HOLTHAUS (Mitt. Deut. Materialprüf., 1930, Spec. No. XI, 3–25; Chem. Zentr., 1930, i, 2963).—At the ordinary temperature the tendency to rust was only slightly decreased with increase in the copper content; at 50° no difference was usually observed. The solubility of steel in acid is markedly affected by the presence of 0.2–0.3% Cu; simultaneous presence of phosphorus diminishes resistance towards sulphuric acid.

A. A. ELDRIDGE.

Tensile properties and structure of drawn-steel wires in relation to the previous heat treatment. A. POMP and A. LINDENBERG (Stahl u. Eisen, 1930, 50, 1462–1467).—Steel wire with 0.6% C, 0.24% Si, 0.5% Mn, 0.019% P, and 0.022% S was heat-treated at various temperatures and quenched in air, in a lead bath, and in oil with a subsequent anneal, then drawn in 11 stages to a 92% reduction in area; at all stages of this procedure the tensile, bending, and fatigue strengths were determined and the structure was examined. The numerical data are reproduced graphically and briefly discussed. The results show that the effect of quenching temperature on the tensile strength is very small, provided that it is above 900°. The bending properties of air-cooled wire are best after a high-temperature anneal, and those of wire quenched in a lead bath when the latter is held at 490° and the metal is quenched from 935°. The resistance to fracture on repeated bending varies considerably with the nature of the heat treatment and with the degree of reduction in the subsequent drawing operation.

A. R. POWELL.

Presumed heterogeneity of martensite. E. MAURER and G. RIEDRICH (Arch. Eisenhüttenw., 1930–1, 4, 95–98; Stahl u. Eisen, 1930, 50, 1431).—The change in structure of hardened hyper- and hypo-eutectoidal steels during subsequent annealing to cause the disappearance of the martensite has been followed by micrographical examination after etching with alkaline sodium picrate. At the lower annealing temperatures, the precipitated cementite is in all cases regularly distributed throughout the specimen. At high temperatures the distribution of the cementite becomes irregular and only a few spheres of cementite are visible in the positions previously occupied by martensite needles; this phenomenon is due to the segregation of

the precipitated cementite along the edges of the needles forming a scum of gradually increasing thickness, which subsequently agglomerates into a few spheroidal aggregates of cementite. On etching with alcoholic nitric acid, hardened hypo-eutectoidal steels reveal a structure consisting of dark needles and flakes in a light ground-mass of fine needles; the dark constituent, which increases in quantity with decreasing carbon content, is darkened by etching with alkaline sodium picrate, whereas the light-coloured constituent remains unchanged in this reagent. The former, therefore, behaves similarly to the martensite needles in hyper-eutectoidal steels which have been annealed at 100–120°, and the latter similarly to unannealed martensite. Rapid quenching and a high manganese content prevent the formation of the dark constituent. In hardened steels with 0.2% C or less, the structure consists of ferrite and "annealed martensite" only; this accounts for the absence of a contraction in volume when the steel is annealed at 100°.

A. R. POWELL.

Iodometric determination of vanadium in special steels and in ferrovanadium. W. WERZ (Z. anal. Chem., 1930, 81, 448–450).—The steel (1–3 g.) is dissolved in 50 c.c. of phosphoric acid (d 1.7), 5 c.c. of nitric acid (d 1.2), and 175 c.c. of water, and the solution is boiled with 1 g. of ammonium persulphate for 15 min. After cooling to 70°, the permanganic acid formed is destroyed by adding 5–10 c.c. of 1% oxalic acid. The solution is then cooled to room temperature and treated with potassium iodide; the liberated iodine is titrated with 0.05*N*-sodium thiosulphate (1 c.c. \equiv 0.00255 g. V). Chromium does not interfere.

A. R. POWELL.

Determination of rust-resistance of special steels. V. DUFFEK (Korrosion u. Metallschutz, 1929, 5, Beih., 32–34; Chem. Zentr., 1930, i, 2308).—Measurements of the potential of nickel- and chromium-steels in *M*-ferrous sulphate solution gave results differing from those of the copper sulphate test. The latter gives results comparable with those of the former only when 0.1*M*-copper sulphate and 0.1*N*-sulphuric acid are used.

A. A. ELDRIDGE.

Protection against corrosion by treatment with phosphate. E. RACKWITZ (Korrosion u. Metallschutz, 1929, 5, 29–32; Chem. Zentr., 1930, i, 2307).—Iron alloyed with more than 6% of another metal cannot be parkerised. The process is described. The protective layer of insoluble phosphates is markedly adsorbent towards oils etc.

A. A. ELDRIDGE.

Hydrometallurgical treatment of high-grade iron-copper sulphide concentrates. R. J. TRAILL, W. R. McCLELLAND, and J. D. JOHNSTON (Canad. Dept. Mines, Ore Invest., 1928, 132–153).—Ferric chloride leaching tests were made on a sulphide concentrate containing 26.5% Fe, 20% Cu, 9.1% Zn, 0.67% Pb, 30% S, 11.22 oz./ton Ag, and 0.8 oz./ton Au. Direct leaching gave almost as good extractions as leaching after a preliminary reducing roast with sulphur; over 90% of the zinc, copper, and silver were extracted and the residue contained practically all the gold in a form readily amenable to cyanidation. A good grade of copper precipitate was obtained, using scrap electrolytic iron as the precipitant. A preliminary leach of the

concentrate with saturated brine slightly acidified with hydrochloric acid removed over 96% of the lead with about 11% of the zinc, but no copper nor precious metals.

A. R. POWELL.

Treatment of mixed concentrates from base-metal sulphide ore. W. E. HARRIS (Canad. Dept. Mines, Ore Invest., 1928, 154–155).—In the treatment of roasted zinc-copper ores with sulphuric acid followed by electrolysis to remove the copper from the leach liquors, the copper content can be reduced to 0.8% with 6% of free acid before "burning" is observed in the deposit. Treatment of the insoluble residue by the Waelz process results in the elimination of 97–98% of the zinc in the form of an oxide containing over 56% Zn and only 0.5% Cu, leaving the greater part of the copper and all the gold and silver in the residue.

A. R. POWELL.

Reports of investigations: [Canadian] ore-dressing and metallurgical laboratories. [Flotation and cyanide tests.] C. S. PARSONS, A. K. ANDERSON, and J. S. GODARD (Canad. Dept. Mines, Ore Invest., 1928, 5–82).—A detailed account with experimental results is given of work done on the flotation of various simple and complex lead, copper, zinc, and molybdenite ores and on the cyanidation of five complex gold ores.

A. R. POWELL.

Effect of deoxidation and mould conditions on the tensile properties of carbon-steel castings. J. V. McCRAE and R. L. DOWDELL (Bur. Stand. J. Res., 1930, 5, 265–277).—Steel castings having elongation 24% and reduction of area 35% may be obtained by deoxidising with 1 lb. of aluminium per ton of steel shortly before casting; the tendency towards low ductility is avoided by using a steel containing 0.10–0.17% C, 0.60–0.70% Mn, and 0.30–0.40% Si. Moderate additions of vanadium and nickel, and heat treatment, have but little influence on the low ductility, and ferro-alloys of boron, zirconium, silicon, aluminium, or titanium are inferior to aluminium as deoxidants, and also reduce the ductility. Green-sand moulding is, in general, desirable, but tends to cause porosity, which, however, can be largely eliminated by control of the moisture content and permeability of the sand.

H. F. GILLBE.

Resistance of steels to abrasion by sand. S. J. ROSENBERG (Bur. Stand. J. Res., 1930, 5, 553–574).—The ball mill, the sand blast, and the Brinell machine have been compared as to their suitability for the testing of the resistance of metals to abrasion. The Brinell machine proved to be generally satisfactory. The resistance to wear of normalised, annealed, hardened, and tempered carbon steels increases with their hardness. Increasing the temperature of annealing resulted in a slight increase in resistance to wear, the increase being more marked in a low-carbon than in a high-carbon steel. Abrasion in this type of test is caused by the gradual breaking up of the individual grains, and not by the removal of separate grains in their entirety, and this action is accompanied by local strain-hardening.

W. E. DOWNEY.

Metallographic investigation of cast-iron pipe. V. N. SVECHNIKOV and K. F. STARODOUDOV (Messenger

ind. (russe) mét., 1928, No. 1, 94—108).—In defective pipes there were accumulations of fine flakes of graphite near the periphery, and fine-grained graphite was found between dendritic crystals of ferrite.

CHEMICAL ABSTRACTS.

Bearing bronzes with additions of zinc, phosphorus, nickel, and antimony. E. M. STAPLES, R. L. DOWDELL, and C. E. EGGENSWILER (Bur. Stand. J. Res., 1930, 5, 349—364).—Tests of the resistance to impact, wear, and repeated pounding, and of the Brinell hardness have been made with copper-tin-lead bearing bronzes with and without zinc, phosphorus, nickel, and antimony. Alloys containing 4% Zn or 0.05% P are superior to the simple ternary alloys and to those containing nickel or antimony; 2% Ni reduces the wear-resistance, but gives increased resistance to pounding at the ordinary temperature, whilst 1% Sb reduces the impact-resistance without appreciably affecting the other properties. Three-dimensional models have been prepared showing the variation of the mechanical properties with the content of copper, tin, and lead.

H. F. GILLBE.

Occlusion of lead and copper in non-ferritic alloys by metastannic and metantimonic acids. S. DEL MUNDO (Philippine J. Sci., 1930, 43, 403—407).—The metastannic and metantimonic acids produced when white metal containing tin and antimony in a lead base is treated with nitric acid contain 1—3% of adsorbed lead. In the procedure recommended for the rapid routine determination of lead in such alloys, the metal (0.5 g.) is heated with 10 c.c. of concentrated sulphuric acid until reaction is complete, 50 c.c. of water are added, and the precipitate of lead sulphate, after being washed first with a 5% solution of sulphuric acid and then with water, is extracted with hot acid ammonium acetate and the lead determined as chromate in the usual way. This method is not suitable for alloys rich in tin, but the metastannic and metantimonic acids formed from these alloys usually contain only 0.1—0.5% of adsorbed lead and copper. R. CUTHILL.

Determination of phosphorus in aluminium. STEINHÄUSER (Z. anal. Chem., 1930, 81, 433—438).—Blackening of silver nitrate by the gases evolved when aluminium is dissolved in acid is not a sure indication of the presence of phosphorus, as silicon hydrides produce a similar effect. Objection is raised to all of the usual methods of determining phosphorus in aluminium, and the following method is recommended: 30—50 g. of metal are dissolved in hydrochloric acid in a vessel through which a current of hydrogen is passed and the gases are burned at a small jet beneath a funnel through which the products of combustion are drawn into 0.25% sodium hydroxide solution. When all the metal has dissolved, the funnel is rinsed with 50 c.c. of 3% hydrofluoric acid to remove the silica deposit and the acid and alkaline solutions are evaporated with a few drops of sulphuric acid to expel fluorine. The phosphorus is then precipitated as phosphomolybdate in the usual way. A. R. POWELL.

Solubility of copper in aluminium in the solid state. P. Y. SALDAU and N. ANISSIMOV (Zapiski Gornovo Inst., Moscow, 1929, 7, No. 3, 139—149).—

The limit of solubility at 15° is at 2.7% Cu. The hardness of the quenched metal increases from 2.7% Cu to a minimum at 6% Cu. CHEMICAL ABSTRACTS.

Corrosion of duralumin in relation to its composition. W. KROENIG (Korrosion u. Metallschutz, 1930, 6, 25—34; Chem. Zentr., 1930, i, 3235).—The binary aluminium-copper alloys showed moderate resistance to corrosion, which resistance was diminished by alternate immersion in sea-water and drying, when intercrystalline corrosion occurred. Magnesium abolishes these properties. Iron diminishes the resistance, whilst manganese (up to 0.6%) is advantageous.

A. A. ELDRIDGE.

Diffusion in cast bismuth-antimony alloys. G. MASING and H. OVERLACH (Wiss. Veröff. Siemens-Konz., 1930, 9, 331—338).—Homogenisation of such alloys on solidification causes an increase in the external measurements of the test piece. It is concluded that this phenomenon is due to diffusion in the solid state.

W. E. DOWNEY.

Behaviour of heat-stable alloys towards sulphur, and a new sulphur-resistant alloy. H. GRUBER (Heraeus Festschr., 1930, 45—51; Chem. Zentr., 1930, i, 2305).—Ordinary heat-resistant alloys are but slightly resistant towards hydrogen sulphide. Addition of chromium increases the resistance of iron towards sulphur; nickel is readily attacked. Addition of aluminium diminishes the corrosion. A resistant chromium-nickel alloy is specified.

A. A. ELDRIDGE.

Recovery of precious metals from residues. A. LEVINŠ (Latvij. Univ. Raksti, 1929, 1, 1—12).—The dry-fusion method for the recovery of precious metals gives better results than the chloride or cyanide methods when the total content of gold and silver is high, i.e., 1—3%. A single fusion may result in the separation of 99.92% of the whole. For complete separation the following factors must be taken into consideration: (a) The viscosity of the melt should be reduced by employing a flux containing calcium and magnesium carbonates. (b) The melt must be kept a sufficient length of time at 1350—1400°. A melt of composition 10SiO₂, 0.83Al₂O₃, 1.10FeO, 9.25CaO, 0.58MgO, and 1.6Na₂O gave a satisfactory yield. (c) The higher the percentage of precious metal in the charge the more complete is its separation, and not only the relative but also the absolute content of precious metal in the slag is smaller. (d) The richer the charge the smaller is the ratio (Au + Ag) : Pb necessary to obtain slags with equal content in noble metal. (e) By repeated fusion of the slag with 10% of litharge a further 20—40% of the precious metal contained in the slag can be separated.

M. S. BURR.

Improvement of the soldering of tramway cables. J. N. ALCÁZER (Anal. Fis. Quím., 1930, 28, 1050—1054).—The hardness (Brinell) of a soldered cable joint is increased if, instead of being air-cooled, the joint be cooled in water at 25° (or, better still, at 3°): also the use of a copper-zinc-silver solder of lower m.p. than that of the usual copper-zinc solder is advantageous. The deformation on flexion is but little altered by this treatment.

H. F. GILLBE.

Electrostatics of flotation. H. B. BULL (Coll. Symp. Ann., 1929, 7, 115—118).—The *P.D.* established by galena falling through solutions of thorium, chromium, silver, lead, or cobalt nitrate of varying concentrations were determined. The "toxic" effect of thorium and chromium is not related to the charge on the galena, but that of silver, lead, and cobalt ions is attributed to an increase of that charge.

CHEMICAL ABSTRACTS.

Molybdenum and its applications. S. L. MALOWAN (Chem.-Ztg., 1930, 54, 893—894.)

Calcium molybdate. MABEE.—See VII. **Electrotyping solutions.** HALL and BLUM.—See XI.

See also A., Nov., 1354, **Change of resistance produced in metals by cold-drawing** (TAKAHASHI). 1359, **Cadmium-zinc alloys** (STOCKDALE). **Lead-antimony alloys** (SOLOMON and JONES). **System lead-tin** (HONDA and ABÉ). 1360, **Vanadium steels** (ŌYA). **Resistance and hardness of lead-thallium alloys** (TAMMANN and RÜDIGER). 1381, **Electrocrystallisation of copper** (KISTIakovSKI and others). 1382, **Preparation of pure electrolytic nickel** (FINK and ROHRMAN). **Electrodeposition of metals from liquid ammonia solutions of their salts** (AUDRIETH and YNTEMA). 1392, **Determination of silver by titration with thiocyanate** (HOLWECH). 1394, **Electrodetermination of nickel, cobalt, and zinc** (TOUGARINOV). 1395, **Electromagnetic separator for mineral powders** (HALLIMOND).

PATENTS.

Ore[roasting] furnace. C. T. KNIGHT (U.S.P. 1,764,313, 17.6.30. Appl., 29.10.28).—The furnace comprises a horizontal cylinder provided with an axial shaft carrying a spaced series of band wheels bound together with a number of spiral strips provided with a series of metal brushes at regular intervals; by rotating the axis of the cylinder the charge is caused to travel through the furnace and the walls are kept clean by the scouring action of the brushes. A. R. POWELL.

Furnaces and/or apparatus for annealing or heat-treating metal etc. A. SMALLWOOD and J. FALLON (B.P. 334,965, 25.4.29).—The furnace comprises a long, horizontal heating chamber containing a series of parallel tubes in which the goods are heated out of contact with the heating flames. Each tube is connected to a similar tube outside the furnace, which serves for cooling the charge, and finally means are provided for ejecting the charge into a quenching bath without its coming into contact with the air. An additional tube is provided within the heating chamber for preheating inert or reducing gases to be passed into the annealing tubes. A. R. POWELL.

Manufacture of wrought iron. A. M. BYERS Co., Assees. of A. H. BEALE and (A, C, D) J. ASTON (B.P. 315,826—9, 20.6.29. U.S., 19.7.28).—Molten steel is (A) passed through a slag bath, (B) poured over a slag bath, (C) poured into a bath of puddle slag, or (D) bessemerised to 0.1% C and poured through a ferrous silicate slag. In all cases the metal is worked up in the usual way to obtain an ingot of wrought iron. [Stat. ref. in (A—D).] A. R. POWELL.

Manufacture of malleable iron. BRIT. THOMSON-HOUSTON Co., LTD.; Assees. of I. R. VALENTINE (B.P. 315,724, 16.7.29. U.S., 16.7.28).—White cast iron is heated at 1000° for 4 hrs. to cause the maximum amount of carbon to enter into solid solution, cooled to below 700°, reheated at 800—850°, cooled abruptly to 700—750°, and maintained within this range for 4 hrs. with occasional abrupt changes of temperature of about 10°. This treatment causes complete graphitisation of the carbon. A. R. POWELL.

Stainless steel and the melting thereof. W. E. MARTIN and J. A. BERLYN (B.P. 336,024, 22.7.29).—The charge containing the requisite amount of nickel and chromium is heated in a crucible at such a rate that the temperature increases at the rate of 400° in 15 min. and the charge is poured at above 1800° after deoxidation with aluminium, with or without a small amount of chromium hydroxide to compensate for the loss of chromium during melting. A. R. POWELL.

Iron or steel alloys [manganese steel]. H. WADE. From TAYLOR-WHARTON IRON & STEEL Co. (B.P. 336,091, 4.10.29).—A steel containing 0.3—0.85% C, up to 4% Ni, < 0.4% Si, 10—15% Mn, and up to 8% Cr is claimed. After shaping, the article is annealed at 800—1150° and air-cooled. [Stat. ref.] A. R. POWELL.

Stable-surface alloy steel. R. P. DE VRIES, Assr. to LUDLUM STEEL Co. (U.S.P. 1,759,605—6, 20.5.30. Appl., [A] 20.1.26, [B] 22.1.26).—(A) The alloy consists of iron with 0.5—5% Cu, 0.5—6% Si, 0.1—6% Al, and 0.1—5% Ti, the sum of the silicon and aluminium being 2—6.1%. (B) The steel contains 1.5—6% Al, 0.5—6% Cu, and 0.5—1% C. These steels resist scaling up to 1000°. A. R. POWELL.

Heat-resistant metal [galvanised iron or steel] sheet. E. R. WEHR and C. C. MAHLIE, Assrs. to AMER. ROLLING MILL Co. (U.S.P. 1,764,132, 17.6.30. Appl., 13.9.26).—The galvanised coating contains more than 2% Al (preferably about 8%); it remains untarnished when heated at 350° in air and is free from spangle and brittleness. A. R. POWELL.

Mineral flotation apparatus. R. LORD, Assr. to SOUTHWESTERN ENG. Co. (U.S.P. 1,761,136, 3.6.30. Appl., 1.9.26).—The apparatus comprises a hollow, frusto-conical aerating rotor the smaller end of which dips into the pulp container; the upper end carries an apertured head connecting with the driving shaft. The aerator is provided with lower and upper slots and has, internally, a series of broken spiral riffles and, externally, vertical riffles. By rotating the apparatus at a high speed, air is drawn into the aerator through the head slots and thoroughly intermingled with the pulp by the action of the riffles. Ejection of froth from the pulp container is prevented by a series of vertical baffles regularly disposed around, but some distance away from, the aerator. A. R. POWELL.

Production of acid-proof etching grounds on printing plates. A. AXTHELM (B.P. 334,629, 13.6.29).—The plate is covered with powdered asphalt or resin and then placed in a chamber in which it is exposed to the vapour of alcohol or other solvent, which is distributed over its surface by means of a small fan in such a

way that a small amount of the solvent condenses on the surface of the plate, thus converting the powder thereon into a smooth, acid-resisting film which rapidly dries when the plate is removed from the chamber.

A. R. POWELL.

Production of finely-divided metals from metal carbonyls. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 336,007, 10.7.29).—A mixture of carbonyl vapour and inert gas is introduced into a vessel with heated walls and provided with a mechanism to impart a gentle whirling motion to the gases; simultaneously a stream of hot carbon monoxide is passed into the vessel so as to cause decomposition of the carbonyl out of contact with the walls of the vessel. A. R. POWELL.

Production of metals [chromium] and alloys. T. R. HAGLUND (U.S.P. 1,764,511, 17.6.30. Appl., 28.1.28. Swed., 1.2.27).—Briquettes of chromium oxide and carbon are smelted below a layer of slag in an electric arc furnace, the amount of carbon being so regulated that the slag contains at least 35% Cr_2O_3 and the current density being kept below 2.5 amp./cm.^2 of cross-section of electrode. After the charge is tranquil a further part of the chromic oxide is reduced from the slag by adding silicon or a silicon alloy.

A. R. POWELL.

Metallisation [with nickel] of electric non-conducting and/or inert base materials. J. G. A. RHODIN, and BRIT. METALLISING CO., LTD. (B.P. 334,510, 29.5. and 28.1.30).—The base material is heated at 350° and immersed in a solution of 10 vols. of nickel carbonyl in 90 vols. of trichloroethylene or tetrachloroethane, whereby it becomes coated with a film of metallic nickel containing carbon. The article is then heated to redness and subsequently electroplated with nickel, chromium, or other suitable metal.

A. R. POWELL.

Electrolytic system [for recovery of metals]. J. W. SCOTT, ASSR. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,764,650, 17.6.30. Appl., 23.12.26).—A number of pairs of tanks containing electrolyte and electrodes is electrically connected in series, and each pair is interconnected by a coupling, permitting electrolyte to pass from one tank to the other and constituting an auxiliary electrolyte cell, provided with cathode and anode, for recovery of metal within the coupling. J. S. G. THOMAS.

Electrolytic separation of copper. A. MOZER (B.P. 336,109, 18.10.29. Ger., 29.10.28).—A bath comprising a solution of cuprous iodide in potassium iodide is claimed; e.g., with a solution of 60 g. of cuprous iodide and 600 g. of potassium iodide in 1 litre of water a current density of 400 amp./m.² can be used at 0.3 volt. Addition of 5–10 g./litre of colloids, e.g., tannin or glue, improves the brightness and smoothness of the copper deposit.

A. R. POWELL.

Reduction of ores. F. L. DUFFIELD (U.S.P. 1,779,411, 28.10.30. Appl., 21.9.28. U.K., 10.10.27).—See B.P. 304,174; B., 1929, 287.

Recovery of metal values from their ores or from metalliferous materials. A. E. WHITE. FROM MEYER MINERAL SEPARATION CORP. (B.P. 336,770, 30.9.29).—See U.S.P. 1,730,584; B., 1930, 9.

Manufacture of metallurgical fuel. G. JAKOVA-MERTURI, ASSR. to CARBONISATION SOC. GÉN. D'EXPLOIT. DES CARBONES (U.S.P. 1,779,744, 28.10.30. Appl., 4.4.28. Fr., 11.4.27).—See B.P. 288,551; B., 1929, 915.

Steel alloy. L. M. BROWN (Re-issue 17,846, 28.10.30. of U.S.P. 1,643,054, 20.9.27).—See B., 1927, 847.

Extraction of tin from ores or materials containing tin. E. A. ASHCROFT (U.S.P. 1,780,106, 28.10.30. Appl., 5.5.28. U.K., 7.7.27).—See B.P. 295,805; B., 1928, 789.

[Anodes for recovery by] electrolysis of zinc from impure sulphate solutions. U. C. TAINTON (U.S.P. 1,759,493, 20.5.30. Appl., 1.4.26. Renewed 13.5.29).—See B.P. 280,103; B., 1928, 21.

Electrolytic manufacture of metal sheets and tubes. E. KELSEN (B.P. 314,444, 27.6.29. Austr., 27.6.28).

Centrifugal extractor (U.S.P. 1,764,214). Filtration apparatus (U.S.P. 1,762,738).—See I. Aluminium salts (B.P. 336,181).—See VII. Carburisation of metals (U.S.P. 1,763,248). **Resistance furnaces (B.P. 336,078). Soldering of aluminium (U.S.P. 1,763,417). Arc-welding (B.P. 307,834). Electrolysis of fused electrolytes (B.P. 316,969).**—See XI.

XI.—ELECTROTECHNICS.

Addition agents in copper electrotyping solutions. R. O. HALL and W. BLUM (Bur. Stand. J. Res., 1930, 5, 767–773).—Phenolsulphonic acid is the best addition agent. Its use permits the electrotypes to be produced in much shorter time and to be harder than those obtained in ordinary baths. A suitable bath is described and the best conditions of temperature and current density are given. W. E. DOWNEY.

Treatment of sulphide-ore concentrates. HARRIS. —See X. **Resistance of rubber and gutta-percha stored under water.** CURTIS and SCOTT.—See XIV.

See also A., Nov., 1354, **Change in resistance produced in metals by cold-drawing (TAKAHASI).** 1360, **Resistance and hardness of lead-thallium alloys (TAMMANN and RÜDIGER).** 1381, **Electrocrystallisation of copper (KISTIAKOVSKI and others).** 1382, **Pure electrolytic nickel (FINK and ROHRMAN).** **Preparation of silver oxide (NAYAR and MACMAHON).** **Electrodeposition of metals from liquid ammonia solutions of their salts (AUDRIETH and YNTEMA).** 1394, **Electrodetermination of nickel, cobalt, and zinc (TOUGARINOV).** 1395, **Electromagnetic separator for mineral powders (HALLIMOND).**

PATENTS.

Electric furnace method [for carburising metallic charges]. W. E. MOORE, ASSR. to PITTSBURGH RES. CORP. (U.S.P. 1,763,248, 10.6.30. Appl., 12.4.29).—Gas under pressure is forced up through the furnace bottom comprising a porous layer of carbonaceous material upon which the charge rests. (Cf. U.S.P. 1,674,982; B., 1928, 645.) J. S. G. THOMAS.

[Conveyor enamelling] electric furnaces. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. of C. L. IPSEN

and J. L. McFARLAND (B.P. 336,168, 18.12.29. U.S., 18.12.28).—In an electric furnace comprising a regenerative chamber and a hair-pin-shaped heating chamber, the walls and floor of which are covered with resistors, means are provided for conveying suspended articles in parallel adjacent paths through the regenerator to and from the heating chamber.

J. S. G. THOMAS.

Electric furnace. A. D. KEENE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,763,239, 10.6.30. Appl., 29.4.24).—A resistor unit supporting device is described.

J. S. G. THOMAS.

Electric resistance furnaces. W. J. MILLAR, and ELECTRIC FURNACE Co., LTD. (B.P. 336,097, 8.10.29).—A recess is formed in the furnace brickwork at each end of the resistors and holes are provided in a metallic casing, enclosing the brickwork, for the admission of air into contact with the ends of the resistors, convected air being discharged through a substantially vertical channel between the brickwork and casing.

J. S. G. THOMAS.

Heating device for attaining high temperatures by means of electric heating resistances. F. NEUMANN (B.P. 312,932, 31.5.29).—Resistance wire, wound upon a refractory ceramic material rich in alumina, is enclosed within a casing of the same material.

J. S. G. THOMAS.

[Cooling of] electrical resistance [annealing] furnaces. P. CARLBERG (B.P. 336,078, 23.9.29).—A cooling medium, *e.g.*, air and/or water, is passed through the tubular resistances of the furnace.

J. S. G. THOMAS.

[Cooling of] induction furnace. G. B. TJOFLAT, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,763,200, 10.6.30. Appl., 13.9.28).—Induction coils of solid section are interposed between, and in heat-conducting relation to, the turns of an induction coil having a longitudinal passage way.

J. S. G. THOMAS.

[Fan for] electric ovens. L. W. WILD, and WILDBARFIELD ELECTRIC FURNACES, LTD. (B.P. 334,621, 11.6.29).—Air within the oven is collected axially by a centrifugal fan and projected horizontally against the oven walls.

J. S. G. THOMAS.

Promoting chemical reactions and physical processes by the use of a high-frequency rotary electric field. W. LÜDKE (B.P. 315,367, 12.7.29. Ger., 12.7.28).—Linear electromagnetic oscillations produced by condensers, inclined to one another, are superposed and combined to form more complicated, *e.g.*, circular or elliptical, oscillations.

J. S. G. THOMAS.

Electric insulating material. TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H. (B.P. 315,354, 18.6.29. Ger., 12.7.28).—The use of a powdered piezo-electric material, *e.g.*, quartz, soaked in transformer oil is claimed.

J. S. G. THOMAS.

Manufacture of [vitreous] heat-resisting insulating material. INTERNAT. GEN. ELECTRIC Co., INC., Assees. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 315,265, 26.6.29. Ger., 10.7.28).—Kaolin, talc, or heavy spar, with or without mica or asbestos, is used in place of the siliceous materials described in B.P. 152,780 (B., 1920, 820 A). [Stat. ref.]

H. ROYAL-DAWSON.

Electrical insulating materials [containing rubber]. W. S. SMITH, H. J. GARNETT, J. N. DEAN, B. J. HABGOOD, and H. C. CHANNON (B.P. 334,587, 6.6.29. Addn. to B.P. 329,275; B., 1930, 723).—Dirt-free rubber is prepared by heating rubber under pressure at 150° in a solvent of b.p. about 120°, *e.g.*, light petroleum or benzene, whereby the viscosity of the solution is reduced sufficiently to enable non-rubber substances to be removed by filtration etc. The rubber is stabilised by addition of an antioxidant, *e.g.*, *p*-aminophenol, and, if desired, mixed with gutta-percha and/or balata. Alternatively, the rubber may be hardened by mixing with bitumen of low ash and free carbon contents and of high m.p.

J. S. G. THOMAS.

Manufacture of resistor material. J. SLEPIAN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,763,268, 10.6.30. Appl., 15.10.23).—Plates composed of a hardened mixture of lampblack, carborundum, and kaolin are saturated with an oxidising agent, *e.g.*, chromic acid, potassium nitrate, potassium permanganate, and heated at about 300°, whereby the carbonaceous material is oxidised.

J. S. G. THOMAS.

Electrode for electric welding or soldering [of aluminium or its alloys]. E. J. CLARKE, Assr. to ALLOY WELDING PROCESSES, LTD. (U.S.P. 1,763,417, 10.6.30. Appl., 15.2.28. U.K., 29.3.27).—An aluminium core is coated with a flux composed of 30–60 pts. of potassium fluoride, 30–60 pts. of potassium chloride, and 3–20 pts. of sawdust, of sufficient thickness to confine the arc to the crater formed.

J. S. G. THOMAS.

Increasing the electrical conductivity of atmospheres surrounding electric arcs used for heating or welding purposes. I. G. FARBERIND. A.-G. (B.P. 307,834, 11.3.29. Ger., 14.3.28).—An aqueous solution or paste containing salts of potassium or alkali metals of higher at. wt., together with oxygen compounds of titanium, uranium, molybdenum, tungsten, aluminium, or iron is introduced into the region surrounding the arc, *e.g.*, by means of a hollow electrode. [Stat. ref.]

J. S. G. THOMAS.

System of electrical precipitation [for gases]. C. S. WEYANDT, Assr. to SYNTRON Co. (U.S.P. 1,760,461, 27.5.30. Appl., 21.11.23).—A vibrating device, controlled by an electric valve, for periodically vibrating the vertically suspended conductor of the precipitator is claimed.

J. S. G. THOMAS.

[Electrodes for] electrostatic precipitator. R. B. RATHBUN, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,763,010, 10.6.30. Appl., 12.8.26).—Pairs of co-axial electrodes, comprising a wire surrounded by a cylindrical open-mesh electrode, are arranged in a conduit through which gas flows substantially at right angles to the axes of the electrodes.

J. S. G. THOMAS.

Heat-treatment of loaded [electrical] conductors. G. W. ELMEN, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,763,884, 17.6.30. Appl., 29.5.26).—A loaded conductor, heated at 900°, is cooled at the rate of about 50° per min. so as to increase the permeability of the loading material (nickel-iron alloy).

J. S. G. THOMAS.

Electron-emission material. H. C. RENTSCHLER, J. W. MARDEN, and C. T. ULREY, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,440, 27.5.30. Appl., 23.12.22).—A refractory cathode composed of 93% W and 7% U is claimed. J. S. G. THOMAS.

Manufacture of [activated] electron-emitting [cathode] devices etc. C. T. ULREY, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,454, 27.5.30. Appl., 24.7.22).—The refractory cathode is arranged in an evacuated envelope with another electrode composed in part of thorium which is heated by electron bombardment and sputtered on to the refractory cathode. J. S. G. THOMAS.

Electric gaseous-discharge device. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. G. FOUND (B.P. 314,811, 1.7.29. U.S., 2.7.28).—Broad thermionic electrodes, each comprising a hollow nickel body coated with, *e.g.*, barium carbonate mixed with a cellulose compound binder and then baked, and a tungsten or molybdenum heater, are arranged at opposite ends of an envelope containing gas, *e.g.*, neon, helium, or mercury vapour, at a pressure of 1–5 mm. Hg. J. S. G. THOMAS.

[Gaseous-discharge current] rectifier. H. C. RENTSCHLER and W. W. MERRYMON, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,525, 27.5.30. Appl., 15.12.26).—A substantially pure and gas-free carbon anode of relatively small area and a relatively large cathode coated with misch-metal or metal of the cerium group of rare-earth metals are arranged within an envelope containing a monatomic gas, *e.g.*, argon, neon, or helium. J. S. G. THOMAS.

Electro-discharge devices. Electrode. H. C. RENTSCHLER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,760,524 and 1,760,526, 27.5.30. Appl., [A] 2.10.25, [B] 16.2.29).—(A) A relatively large cathode and a relatively small anode, formed at least in part of misch-metal or a metal of the cerium group of the rare-earth metals, are arranged within an envelope containing a monatomic gas, whereby the starting and operating potentials are reduced. (B) A cathode composed at least in part of alkaline-earth oxide(s) and a co-operating electrode, composed of an alloy of chromium and nickel and, if desired, iron, coated with chromium oxide but free from iron and nickel oxides, are arranged in an envelope. J. S. G. THOMAS.

Coated cathodes for electron-discharge devices. WESTINGHOUSE LAMP Co., Assees. of (A) E. G. WIDELL, (B) M. N. FREUDENBURGH (B.P. 315,800 and 316,104, [A] 18.7.29, [B] 22.7.29. U.S., [A] 18.7.28, [B] 20.7.28).—Cathodes are coated with material prepared by (A) dissolving a cellulose ester, *e.g.*, cellulose acetate or nitrate, in a solvent, *e.g.*, alcohol-ether, amyl acetate, to which about 0.5% of a plasticiser, *e.g.*, ethyl phthalate, is added; or (B) suspending electron-emission material, *e.g.*, a mixture of pure alkaline-earth carbonates and a binder, *e.g.*, nitrocellulose, in an organic liquid which is completely vaporisable *in vacuo* and is inert towards the electron-emission material, and in which the binder is soluble, *e.g.*, ethyl carbonate and/or oxalate, together with, if desired, anhydrous alcohols of lower b.p. J. S. G. THOMAS.

Electric incandescence lamps. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 335,903, 1.7.29).—A singly- or doubly-helical tungsten filament is arranged in an envelope containing mercury or other substance the vapour of which is inert to tungsten, the distance between the filament and the envelope wall being not more than 0.4 of the corresponding distance in the ordinary tungsten-filament gas-filled lamp of the same wattage with spherical bulb. [Stat. ref.] J. S. G. THOMAS.

Photo-electric cells. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of H. F. MESICK (B.P. 319,734, 4.9.29. U.S., 27.9.28. Addn. to B.P. 303,476; B., 1930, 108).—By arranging within the envelope of the cell a metal member coated with a colloidal suspension of graphite in water, the process of heating *in vacuo* for the removal of excess alkali metal, described previously, is rendered unnecessary. J. S. G. THOMAS.

Photo-electric cells [of condenser type]. ARCTURUS RADIO TUBE Co., Assees. of S. RUBEN (B.P. 318,641, 7.9.29. U.S., 8.9.28).—Adjacent surfaces of co-operating electrodes, *e.g.*, of copper, coated with light-sensitive material, *e.g.*, cuprous oxide, are separated by a translucent dielectric material, preferably of high sp. inductive capacity, *e.g.*, glycerin, castor oil, or ethyl alcohol. J. S. G. THOMAS.

Light-sensitive discharge apparatus. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of L. R. KOLLER (B.P. 314,096, 22.6.29. U.S., 22.6.28).—A photo-electric device comprising a highly evacuated, elongated envelope containing a cathode and anode, and a light-sensitive discharge controller composed of a band of monatomic photo-sensitive material, *e.g.*, caesium or rubidium, deposited on the inner wall of the envelope remote from the cathode, is claimed. J. S. G. THOMAS.

Weston standard electric cell. W. W. TRIGGS. From WESTON ELECTRICAL INSTRUMENT CORP. (B.P. 335,452, 28.11.29).—An apertured disc covered with fine-mesh cloth prevents access of the mercurous sulphate, used as depolariser, to the neighbourhood of the cell terminal. J. S. G. THOMAS.

Dry cell. W. D. STALEY, Assr. to BURGESS BATTERY Co. (U.S.P. 1,760,090, 27.5.30. Appl., 6.7.28).—A dry-cell cathode core is coated first with agar-agar and then with a mixture composed of ungelatinised potato starch, 10–40% of ammonium chloride, and 1–4% of a binder of gelatinised starch. The bottom of the cell is insulated by means of wax. J. S. G. THOMAS.

Production of plates for electric storage batteries and accumulators. G. F. JUDD and J. CARLIER (B.P. 344,711, 9.9.29).—A mixture containing flowers of sulphur 1 pt., caustic soda 1 pt., and water 8 pts. is heated, allowed to cool, and then mixed with lead oxides to form a paste which is pressed into a frame and baked hard. After cooling, the plate is coated with lead. J. S. G. THOMAS.

[Electrolyte for] electrical storage batteries or accumulators. T. STANDLEY (B.P. 336,113, 23.10.29).—Warm solutions containing, respectively, 2 pts. of sulphuric acid and 2 pts. of sodium silicate each in

5 pts. of water, are mixed at about the same temperature. J. S. G. THOMAS.

[Grids, frames, etc. for] electric accumulators.

I. G. FARBERIND A.-G. (B.P. 334,628, 12.6.29. Addn. to B.P. 311,401; B., 1930, 824).—The grids etc. are swaged or stamped from a hard-lead band, containing about 3% Sb, worked through an extrusion press, and, if desired, further treated by hammering or pressing.

J. S. G. THOMAS.

Manufacture of [diaphragms for] electrolytic cells. J. Y. JOHNSON. From I. G. FARBERIND A.-G. (B.P. 335,151, 22.3.29).—A woven fabric containing wires and spun threads, preferably of asbestos, is claimed.

J. S. G. THOMAS.

Electrolysis of fused electrolytes. HIRSCH, KUPFER-U. MESSING-WERKE A.-G. (B.P. 316,969, 10.7.29. Ger., 7.8.28).—The electrolyte is heated by eddy currents induced in electrically conducting, heat-transferring material, e.g., the electrodes, the electrolyte container, or loose material arranged in the electrolyte. The method is applicable to the electrolytic production of aluminium, potassium, sodium, etc.

J. S. G. THOMAS.

Electrolytic apparatus [for production of oxygen and hydrogen]. A. E. KNOWLES (B.P. 335,987, 6.7.29. Addn. to B.P. 320,388; B., 1930, 21).—An automatic feed-water and gas-washing arrangement is fitted to the cells described previously.

J. S. G. THOMAS.

Production of magnetic bodies [cores etc.].

F. J. GIVEN, R. M. C. GREENIDGE, and J. R. WEEKS, JUN., ASSRS. to BELL TELEPHONE LABS., INC. (U.S.P. 1,759,612, 20.5.30. Appl., 6.6.29).—The magnetic stability of finely-divided nickel-iron alloys is controlled by suitable pre-annealing heat-treatment of the particles, the time and temperature of treatment being determined by the relative proportions of the constituents present in the alloy.

J. S. G. THOMAS.

Measuring the intensity of light. F. W. GILLARD (B.P. 335,179, 17.6.29).—A graduated light filter is used to maintain substantially constant the intensity of light falling upon a photo-electric cell connected with or inserted in an electric circuit including a valve amplifier.

J. S. G. THOMAS.

Optical pyrometer. A. BESTELMEYER, ASSR. to HARTMANN & BRAUN A.-G. (U.S.P. 1,762,380, 10.6.30. Appl., 16.7.26).—The apparatus comprises an ammeter reading the current (but graduated in degrees of temperature) taken by a lamp which is fed by small dry battery or transformer through an adjustable resistance; a telescope passing through the ammeter case and having slits for viewing the lamp and radiant body simultaneously; a removable red screen to allow the test to be made with rays of one wave-length only; a removable smoked screen for use at high temperatures, the ammeter being doubly graduated.

B. M. VENABLES.

Heat-treatment of magnetic materials. L. W. MCKEEHAN, ASSR. to BELL TELEPHONE LABS., INC. (U.S.P. 1,762,730, 10.6.30. Appl., 30.10.26).—See B.P. 303,928; B., 1929, 217.

[Positive-column gaseous] discharge tubes. A. E. WHITE. From CLAUDE NEON LIGHTS, INC. (B.P. 337,002, 18.7.29).

[Gas-filled] electric-discharge devices. H. J. SPANNER and U. DOERING (B.P. 310,895, 2.5.29. Ger., 2.5.28).

[Electromagnetic] testing of magnetisable bodies. E. POTTER. From C. KINSLEY (B.P. 336,987, 19.6.29).

[Enclosed-type] hydrometers [for accumulators]. J. H. COLLIE (B.P. 335,698, 29.8.29).

Drying apparatus (B.P. 335,942). **High-vacuum pumps** (B.P. 336,001).—See I. **Carbon electrodes** (B.P. 312,940). **Control of water-gas sets** (U.S.P. 1,758,891).—See II. **Nitrogen-hydrogen mixtures** (B.P. 335,524).—See VII. **Chromium and alloys** (U.S.P. 1,764,511). **Metallisation of non-conducting materials** (B.P. 334,510). **Electrolytic system for metals** (U.S.P. 1,764,650). **Metal sheets and tubes** (B.P. 314,444). **Copper** (B.P. 336,109).—See X. **White lead** (B.P. 314,987).—See XIII.

XII.—FATS; OILS; WAXES.

Determination of the detergent value of soaps. N. PANTYUKHOV (Masloboino-Zhir. Delo, 1929, No. 2, 20—25).—The relative interfacial tension between benzene and 0.2% solutions of commercial soaps was determined by Traube's drop method. The highest value was given by neutral soaps containing 75% of fatty acid, whilst alkaline soaps (alkali 0.21%, fatty acid 46.7%) gave the lowest values.

CHEMICAL ABSTRACTS.

Olive oil. II. Acidity. F. TRAETTA-MOSCA and M. VENEZIA (Annali Chim. Appl., 1930, 20, 452—456).—The development of acidity depends on the temperature treatment and other conditions to which the olives are subjected.

T. H. POPE.

Detection of adulteration of "Abrasinöl" (wood oil from Indo-China) with castor and arachis oils. E. STOCK (Farben-Ztg., 1930, 36, 173).—Examination of laboratory-prepared mixtures of "Abrasinöl" (a recently introduced varnish raw material) with various proportions of castor and arachis oils shows that whilst acid value and saponif. value determinations will not reveal adulteration, the refractive index of Abrasinöl (1.512 at 19°) falls with increasing proportion of adulterant. Alcohol extraction serves to indicate which of the above two adulterants has been used.

S. S. WOOLF.

Brazil-nut oil. H. A. SCHUETTE, R. W. THOMAS, and M. DUTHEY (J. Amer. Chem. Soc., 1930, 52, 4114—4117).—The oil expressed from the nuts and that extracted from the residue by light petroleum have the following constants (the figures in parentheses are for the extracted residual oil): d_{25}^{25} 0.9150 (0.9143), n_{20}^{20} 1.4678 (1.4683), iodine value (Wijs) 99.92 (95.21), saponif. value 194 (198), ester value 193.9 (193.8), Reichert-Meissl value 0 (0.31), Polenske value 0 (0.32), free fatty acids (as % oleic) 0.006 (0.02), acetyl value 12.3 (12.3), unsaponifiable matter 0.64% (0.68%).

The residual oil contains myristin (1.79%), palmitin (13.55%), stearin (2.58%), olein (55.64%), and linolein (21.65%).
H. BURTON.

Oil in the juice of berries. V. RUCHKIN (Masloboino-Zhir. Delo, 1929, No. 2, 47—48).—Oils of the bark (3.06%), berries (2.00%), and seeds (12.13%), respectively, of *Hippophae rhamnifolia*, L. (Siberia), afford the following values: acid 9.60, 7.35, 4.42; saponif. 189.2, 190.0, 192.5; ester 179.6, 182.7, 188.1; iodine (Hübl) —, 76.5, 138.2; iodine (Margosches) 56.3, 76.8, 138.4; Reichert-Meissl —, 0.79, 0.58; acetyl (Normann) —, 16.4, 10.4; unsaponif. —, 3.66, 1.78; hydroxy-acids —, 0.79, 0.61; hexabromides —, 0, 19.58; *d* —, 0.92435, 0.92783; f.p. 12.0°, 6.4°, —20.0°; *n* 1.4639, 1.4642, 1.4739; f.p. of fatty acids 39.5°, 34°, 1.3°; mol. wt. of fatty acids —, 276.5, 278.9; iodine 61.2, 78.2, 140.3; linolenic acid 0, 0, 14.63; linoleic acid 0, 10.5, 12.31; isolinoleic acid 0, 0, 20.69; oleic acid 62.6, 63.4, 41.47; palmitic and stearic acids 37.4, 10.4, 10.9%.

CHEMICAL ABSTRACTS.

Determination of oil in vegetable materials. B. MASLENIKOV (Masloboino-Zhir. Delo, 1930, No. 2, 28—30).—The finely-powdered material is mixed with a soluble salt, and a smooth paste is prepared by the addition of water, affording on drying a stable, porous mass. The mixture is repeatedly granulated during the extraction.
CHEMICAL ABSTRACTS.

Coriander as a source of fatty oil. J. ORLOV (Masloboino-Zhir. Delo, 1929, No. 3, 26—28).—The greenish fatty oil (17—21%) has iodine value 89.7, saponif. value 214.6, and acid value 4.3.

CHEMICAL ABSTRACTS.

Oil from Kuban *Perilla ocumoides* seeds. M. ZHDAN-PUSHKIN (Masloboino-Zhir. Delo, 1929, No. 2, 44—47).—The seeds contain moisture 6.30, protein 23.12, oil 45.07, nitrogen-free extractive matter 10.28, crude fibre 10.28, ash 4.64, essential oil 0.29%. The essential oil, *d* 0.9304, has saponif. value 24.8, acid value 2.6, ester value 22.2; it polymerises, with thickening, when heated. The fatty oil has acid value 1.0, saponif. value 190.06, ester value 189.06, Reichert-Meissl value 1.43, Polenske value 1.62, acetyl value 0.71, iodine value 203.05, hexabromide value 63.65, unsaponifiable matter 0.3%, Hehner value 95.8. The fatty acids were examined.
CHEMICAL ABSTRACTS.

Solid vegetable oils. S. IVANOV (Masloboino-Zhir. Delo, 1929, No. 1, 32—34).—The seeds of *Simmondsia californica* (Mexico) contain 6.85% of moisture and 44.49% of a solid oil having acidity 5.4, saponif. value 165.7, iodine value 79.3—80.2, *d*₂₅ 0.8990. Oil of the seeds of *Pentaclethra macrophylla* (W. Africa) has *d*₁₅ 0.916, m.p. 20.1°, acid value 6.02, saponif. value 189, iodine value 76.57, titre 51.5. Oleic acid and acids similar to linolenic acid are present. Oil (36%) of the seeds of *Butyrospermum parkii* (Sudan) has *d*₁₅¹⁰⁰ 0.860, m.p. 27.2°, acid value 106, saponif. value 244.7, iodine value 52—61, and contains stearic (34—37%) and oleic (60%) acids.
CHEMICAL ABSTRACTS.

Chemistry of the sulphation of oils. Report of a Committee of the Society of Leather Trades'

Chemists. D. BURTON and F. G. A. ENNA (J. Soc. Leather Trades' Chem., 1930, 14, 459—477).—The possible reactions which may occur when an oil is sulphonated are discussed. Since commercial sulphonated oils may contain neutral oil, free fatty acids, sulphuric acid compounds, various polymerides, hydroxy-compounds, oxidised products, inorganic salts, soaps, and water, the insoluble bromide value is found to yield more information about the behaviour of marine animal oils on sulphonation than does the iodine value. A high figure indicates the presence of sufficient acids of the clupanodonic type to render sulphonation difficult. Three cod oils of normal iodine value were found by the present authors to be too violent on sulphonation. By determination of the acetyl value of the sulphonated oil the nature of the sulphonation, whether at the double linking or hydroxyl group, can be ascertained.

D. WOODROFFE.

Vegetable oils as lubricants. CHAMPSAUR.—See II. "Water of vegetation" of olives. TRAETTA-MOSCA and VENEZIA.—See XIX.

See also A., Nov., 1368, Separation of sodium soaps of higher fatty acids (OSTWALD and ERBRING). 1483, Oil from seeds of *Cassia Tora* (JOIS and MANJUNATH). Oil from *Cocos nucifera* (CHANDRASENA). 1483, Calculation of lipins as fat (FINCKE).

PATENTS.

Manufacture of soaps. A. IMHAUSEN (B.P. 335,954, 6.5.29).—Soap of high lathering power is made by mixing coconut (or palm-kernel) oil, oil paste soap, or coconut oil and the equivalent alkaline lye (after saponification), into a (liquid) boiled and grained soap, after separation of the latter from the foots.

E. LEWKOWITSCH.

Colouring rubber oil substitute. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 335,912, 29.5.29).—Emulsions or suspensions of rubber oil substitute (factice from linseed or rape oils etc.) are mixed with aqueous pastes of inorganic or organic, water-insoluble dyes, pigments, lakes, leuco-vat dyes, etc.; or such colouring matters are precipitated in the emulsions, which are afterwards broken by the addition of acid agents, salts, etc.

E. LEWKOWITSCH.

Preparation of water-soluble product derived from the fatty acids occurring in wool fat. A. THAUSS and G. MAUTHE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,252, 4.11.30. Appl., 16.12.27. Ger., 17.12.26).—See B.P. 307,776; B., 1929, 442.

Preparation of a product obtainable by treating wool fat with a sulphonating agent. G. MAUTHE and A. THAUSS, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,027, 28.10.30. Appl., 24.2.28. Ger., 1.3.27).—See B.P. 286,252; B., 1929, 608.

Oxidation of fats, [oils], waxes, and resins. W. PUNGS, Assr. to I. G. FARBERIND. A.-G. (U.S.P. 1,780,632, 4.11.30. Appl., 19.10.25. Ger., 22.10.24).—See B.P. 258,099; B., 1926, 987.

Stable sulphonic acids etc. (B.P. 313,453).—See III. Waterproofed paper (B.P. 335,559).—See V. Artificial masses (B.P. 334,572).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

[Paint] **siccative.** A. ZINOVIEV (Masloboino-Zhir. Delo, 1929, No. 2, 39—43; No. 3, 51—56).—Lead manganese linoleate is preferred; its preparation is described. **CHEMICAL ABSTRACTS.**

Preparation of varnish oil by dehydration. A. ZINOVIEV (Masloboino-Zhir. Delo, 1929, No. 1, 12—16).—The colloidal substances remain in solution at all temperatures if the oil is previously dehydrated, preferably by passing a current of dry carbon dioxide through it at 140—150°. **CHEMICAL ABSTRACTS.**

Synthetic camphor etc. SCHWYZER.—See III.

PATENTS.

Preparation of oil paints. L. BLUMER (L. BLUMER CHEM. FABR.) (B.P. 314,992, 28.6.29. Ger., 7.7.28).—Phthalic acid, salts thereof, or salts of similar polybasic carboxylic acids are introduced as "swelling bodies" into oil paints. The partly gelatinised product does not sink into porous surfaces and several coats may be applied without intervening drying periods.

S. S. WOOLF.

Manufacture of [weather-resistant] paints. A. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 335,626, 2.5.29).—Precipitated zinc oxide dried at not much above 500° is introduced into paints in quantity at least sufficient to convert into soaps all free or combined fatty acids present in the vehicle, the remaining quantity of pigment being of the usual character. **S. S. WOOLF.**

Production of bituminous paint or varnish. CRAIGBANK CHEM. CO., LTD., and J. H. TAYLOR (B.P. 336,117, 26.10.29).—Tannic acid (5—15 vol.-%) is compounded with the bituminous paint or varnish, or its constituents, to ensure drying with a plastic skin and to give the product anti-corrosive and anti-fouling properties.

H. ROYAL-DAWSON.

Electrolytic production of white lead. R. J. FROST (B.P. 314,987, 24.6.29. Austral., 6.7.28).—Metallic lead electrodes immersed in a dilute aqueous solution of commercial sodium bicarbonate are subjected to the action of a direct current flowing in alternate directions for 15 min. in each direction, the electrolyte being regenerated by the addition of carbon dioxide. The flotation of the white lead thus formed and its removal to hydro-extractors and filters are assisted by circulation of the electrolyte and by the evolution of gases from the electrodes. **S. S. WOOLF.**

Rosin soap lakes of azo compounds. (SIR) G. C. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 334,874, 8.3.29).—A heavy-metal salt is added to a mixture of acid azo dye and rosin soap in aqueous medium, whereby the metal lake and resinate are co-precipitated. The azo dye may be substantially insoluble in water, and the rosin soap added prior to or during coupling of the azo-dye components. The resinate lakes are insoluble in oil and in water, and are suitable for printing inks. Examples are: β -naphthylamine-1-sulphonic acid \rightarrow β -naphthol, rosin soap, barium or calcium chloride; *p*-toluidine-*o*-sulphonic acid \rightarrow 2:3-hydroxynaphthoic acid, rosin soap, calcium chloride; β -naphthylamine-1-sulphonic acid \rightarrow 2:3-hydroxynaphthoic *m*-nitroanilide, rosin soap, barium

chloride; α -naphthylamine \rightarrow Laurent acid, rosin soap, calcium chloride. **C. HOLLINS.**

Manufacture of artificial masses as bases for coatings, impregnating agents, binders, etc. H. HÖNEL (B.P. 334,572, 8.5.29).—An ester, particularly a polyhydric alcohol ester of a mono- or poly-basic acid, is heated, in absence of solvent or dispersing agent, with a low-molecular product obtained by alkaline condensation of formaldehyde with a phenol in which at least one of the positions 2, 4, and 6 is substituted; the low-molecular product should be one which is capable of thermo-hardening. Amongst the examples given are: beeswax with the liquid product from *p*-tert.-amylphenol and formaldehyde; castor oil with *p*-cresol-formaldehyde; "honey oil" (moderately boiled wood oil and linseed oil) with thymol-formaldehyde; glyceryl succinate-lactate with *p*-chlorophenol-formaldehyde; glyceryl citrate-ricinoleate-salicylate with *p*-cresol-formaldehyde; glyceryl phthalate-stearate with the formaldehyde condensation product from the reaction product of *o*-cresol and methyl ethyl ketone; etc. **C. HOLLINS.**

Solution of cellulose derivatives [lacquer]. A. KNORR, Assf. to I. G. FARBEIND. A.-G. (U.S.P. 1,780,883, 4.11.30. Appl., 16.3.28. Ger., 23.12.24).—See B.P. 275,653; B., 1927, 905.

Applying nitrocellulose varnishes. H. F. SCHULZ (U.S.P. 1,780,844, 4.11.30. Appl., 30.11.26. Ger., 4.12.25).—See B.P. 262,440; B., 1927, 916.

Manufacture of polybasic acid-polyhydric alcohol resins. H. WADE. From BAKELITE CORP (B.P. 336,645, 10.7.29).—See U.S.P. 1,739,771; B., 1930, 677.

Manufacture of inlaid linoleum. ARMSTRONG CORK CO., Assees. of J. C. MCCARTHY (B.P. 317,003, 15.7.29. U.S., 8.8.28).

(A) **Decoration, (B) manufacture, of surface coverings [inlaid linoleum].** ARMSTRONG CORK CO., Assees. of [A] H. W. PRENTIS, [B] S. H. HARTMAN and C. F. HUMPHREYS (B.P. 316,549 and 316,970, 15.7.29. U.S., [A] 15.7.29, [B] 7.8.28).

Floor-covering materials [linoleum] and their decoration. ARMSTRONG CORK CO., Assees. of J. C. MCCARTHY (B.P. 319,655, 20.7.29. U.S., 25.9.28).

Calcination of pigments (B.P. 335,659).—See I. **Distillation of resinous woods** (U.S.P. 1,757,144).—See II. **Azo lakes** (B.P. 334,754).—See IV. **Poly-saccharide ether compositions** (B.P. 334,897). **Cellulose ether or ester compositions** (B.P. 308,658 and 335,582). **Products from cellulose esters or ethers** (B.P. 315,766). **Cellulosic compositions** (B.P. 313,535).—See V. **Titanates** (U.S.P. 1,760,513). **Mouldable product** (U.S.P. 1,761,740).—See VII. **Coated cathodes** (B.P. 315,800).—See XI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Change of electrical properties of rubber and gutta-percha during storage under water. H. L. CURTIS and A. H. SCOTT (Bur. Stand. J. Res., 1930, 5, 539—552).—The effect of storage under water for periods up to seven years on the resistivity, dielectric constant, and power factor of samples of rubber and

gutta-percha has been investigated. The resistance-time curves showed a break or marked change in direction several months before actual failure of the rubber occurred. In the case of power factor and *D.-C.* dielectric constant curves, breaks occurred at a later time. The decrease in resistivity is ascribed to the formation of fine holes through the material. Microscopical examination showed fern-like figures projecting into the rubber. The catalytic action of copper salts from electrodes inside the tube specimens accelerated the ageing of the rubber and probably changed its character.

W. E. DOWNEY.

PATENTS.

Rubber compositions [anti-agers] and their manufacture. H. A. MORTON (B.P. 314,756, 1.7.29. U.S., 30.6.28).—Ethylenediamines in which one or more of the 8 hydrogen atoms is replaced by a hydrocarbon residue, especially an aryl group, excluding *N*-naphthyl derivatives, are incorporated with a rubber mix as anti-agers. The *N*-aryl groups may be monosubstituted, e.g., by hydroxyl, amino-, chloro-, or nitro-groups. Amongst the anti-agers mentioned are: *NN'*-diphenylethylenediamine, *NN'*-diphenyl-*NN'*-diethylethylenediamine, *NN'*-diphenyl- $\alpha\beta$ -propylenediamine, *NN'*-diphenyl- $\beta\gamma$ -butylenediamine, *NN'*-diphenyl- $\beta\gamma$ -diaminoisopentane [$\beta\gamma$ -dianilinoisopentane], and *NN'*-diphenyl- $\alpha\alpha\beta\beta$ -tetramethylethylenediamine. The substituted ethylenediamines have practically no accelerating effect.

C. HOLLINS.

Manufacture of rubber vulcanisation accelerators. W. SCOTT, ASSR. to RUBBER SERVICE LABS. CO. (U.S.P. 1,779,715, 28.10.30. Appl., 2.3.27).—See B.P. 286,749; B., 1928, 377.

Rubberised fabric (U.S.P. 1,763,618). Water-proofed paper (B.P. 335,559).—See V. **Insulating material (B.P. 334,587).**—See XI. **Colouring rubber-oil substitute (B.P. 335,912).**—See XII.

XV.—LEATHER; GLUE.

Standardisation of hide powder. IV. Interpretation of the results of the relative specific surface test. H. G. BENNETT (J. Soc. Leather Trades' Chem., 1930, 14, 481—485; cf. B., 1927, 534).—There is a definite relationship between the relative specific surface results and the soluble matter in a hide powder. The coefficient of correlation for the successful powders (B3—B6) was -0.86 , and for another series of powders was -0.95 . As the soluble matters decrease and the purity of the powders increases the degree of correlation will decrease. When the soluble matter is reduced to zero the correlation coefficient will be zero also. The coefficient if regularly determined will indicate how near this ideal of zero water-soluble matter has been approached.

D. WOODROFFE.

Influence of temperature in single-bath chrome-tanning. C. OTIN and G. ALEXA (J. Soc. Leather Trades' Chem., 1930, 14, 450—459).—Samples of hide powder were treated at 25°, 32°, and 42°, respectively, with one-bath chrome-tanning liquors having basicities varying from 0 to 53%, and after 48 hrs. the powder was washed and analysed. The amount of chromic oxide fixed by the powder was found to be a function of

the basicity (the higher the basicity the greater was the quantity fixed), and to increase with rise in temperature. The effect of temperature was most marked for the liquor of 32% basicity; when such was used 42% and 91% more chromic oxide was fixed at 32° and 42°, respectively, than at 25°. The amount of SO_4 fixed by the powder decreased as the basicity of the solutions increased. It was shown by time tests that the amounts of chromic oxide and sulphuric acid fixed by the powder were a function of the time at all temperatures, and the greatest increase in the fixation of chromium was caused by increasing the temperature of the tanning liquors in the early stages of tanning. The basicity of the liquors was diminished by a rise in temperature. The effect of temperature on chrome-tanning liquors is attributed to the expulsion of carbon dioxide, increased hydrolysis and polymerisation, and greater degree of "olation" with increased colloidal character.

D. WOODROFFE.

Wilson-Kern method [of tannin analysis] in laboratory practice. A. T. HOUGH and R. DRU (J. Soc. Leather Trades' Chem., 1930, 14, 478—481).—The tanned powder is transferred to a 500-c.c. filter bell 10 cm. in diam., the mouth of which is covered with the usual filter cloth; the filter bell is immersed to a depth of 3 cm. in a 500-c.c. porcelain basin (carrying a stirrer) full of distilled water and rests on a Y-shaped stand. Means are provided for the inflow of distilled water and for the restricted exit of the washings. The filter bell is gently agitated during the first hour of washing and then set aside overnight. To complete the process 6—10 litres of water and 12—20 hrs.' washing are necessary.

D. WOODROFFE.

See also A., Nov., 1458, **Change in gelatin on storage** (THIMANN and PAGE).

PATENTS.

Production of a lather-forming depilating preparation. E. HADANK (B.P. 317,707, 19.8.29. Ger., 18.8.28).—Alkali or alkaline-earth sulphides are mixed with a peroxide, an inert diluent, and a mucilage.

D. WOODROFFE.

Unhairing agent [for hides]. A. C. ORTHMANN, ASSR. to PFISTER & VOGEL LEATHER CO. (U.S.P. 1,763,319, 10.6.30. Appl., 5.3.28).—Beet-sugar residues (8—20 pts.) are mixed with caustic soda (3—3.5 pts.), lime, or other alkaline medium, and water (150 pts.), and the hides are immersed therein for 5 days.

D. WOODROFFE.

Tanning animal hides with iron salts. K. STÜRMER (U.S.P. 1,763,596, 10.6.30. Appl., 12.2.29. Ger., 18.2.28).—Hides are treated with solutions of ferrous salts, which may or may not have been pre-treated with nitrogen monoxide. Pure nitrogen monoxide is then introduced into the solutions, or the hides are subjected to the action of air or oxygen under pressure.

D. WOODROFFE.

Acceleration and improvement of tanning processes. F. POSPIECH, ASSR. to CHEM. FABR. POTT & CO. (U.S.P. 1,763,363, 10.6.30. Appl., 26.8.27. Ger., 21.8.26).—Condensation products of naphthalenesulphonic acids and aliphatic alcohols above C_2 , e.g.,

naphthalenesulphonic acid and isopropyl alcohol, are added to the tan liquors. D. WOODROFFE.

Manufacture of a casein-glue film. P. M. F. BEHNKE, N.V. HANDELMAATS. "CUBA," and N.V. HOUTIND. "PICUS" (B.P. 314,344, 21.6.29. Ger., 25.6.28).—Heated liquid casein glue or a hot solution of casein glue is brought on to a cold or cooled metal surface, dried if necessary, removed, and used in the preparation of ply-wood. D. WOODROFFE.

Felt (U.S.P. 1,765,046).—See V. **Preparation of fur** (U.S.P. 1,762,233).—See VI. **Sulphite waste-liquors** (U.S.P. 1,764,600—1).—See VII. **Immunising glue** (B.P. 335,527).—See XVI.

XVI.—AGRICULTURE.

Magnesium and calcium in zeolitic soils. J. F. BREAZEALE (Arizona Agric. Exp. Sta., Tech. Bull., 1929, No. 26).—Alkali soils were percolated with various simple and mixed salt solutions and the nature of the exchanged bases was examined. The presence of a common ion in the percolating solution depressed the ionisation of the soil zeolites and retarded the exchange of bases. Percolation of a calcareous magnesium-zeolite soil with equivalent solutions of either calcium or magnesium salts led to the same final equilibrium in the percolate. Replacement of bases by solutions of calcium or magnesium salts was similar in both calcareous magnesium- and calcium-zeolite soils. Calcium carbonate in zeolitic soils took part in base-exchange reactions as readily as the more soluble calcium salts, but magnesium carbonate in the form of magnesite or dolomite was practically inactive in this respect, except in the presence of carbon dioxide. Treatment of soil with dilute hydrochloric acid resulted in some decomposition of the zeolite as well as replacement of base by H⁺. This action was reversed by subsequent addition of solutions containing exchangeable base, the re-formation of zeolite being largely controlled by the reaction of the solution used. A. G. POLLARD.

I. Effect [on crops] of yard manure and "huminit" in comparison with mineral fertilisers. II. Composition of some green fodders and green manuring plants. III. Effect of chromium on plant growth. IV. Effect of arsenic on plant growth. E. HASELHOFF, F. HAUN, and W. ELBERT (Landw. Versuchs-Stat., 1930, 110, 247—268, 268—283, 283—286, 287—289).—I. "Huminit" used in conjunction with artificial fertilisers tends to increase crop yields, but is inferior in this respect to farmyard manure.

II. Analyses are recorded of the food values of the tops and the content of fertiliser constituents in both tops and roots of a number of crops in varying stages of growth.

III. In sand cultures and in soils chromium compounds depressed the growth of barley and mustard. Chromium hydroxide was less injurious than chromium trioxide, and the effects of both were more pronounced in a sandy soil than in loam. Small quantities of chromium were absorbed by the plants.

IV. Addition of calcium arsenate to soil (up to 0.003% As) prevented the growth of barley in a sandy soil, but was without influence on the crop yield on a loam.

A. G. POLLARD.

Interrelation of nutrients and soil reaction on growth and inoculation of lucerne. M. C. SEWELL and P. L. GAINES (Soil Sci., 1930, 30, 297—305).—In sand cultures lucerne grew well in media of p_H 4.5 if well supplied with nutrients, including calcium. Nodulation was scanty in media of p_H 6.0, but well-developed at p_H 7.5. In soils the supply of phosphate to the plant may be more important than the reaction in controlling the amount of nodulation. A. G. POLLARD.

Determination of potash in very dilute solutions and in soil liquors. M. VON WRANGELL (Z. anal. Chem., 1930, 82, 224—226).—The aqueous extract of the soil is evaporated to dryness, the residue heated to expel ammonium salts and dissolved in 2 c.c. of 3% acetic acid, and 1 c.c. of the solution treated with 1 c.c. of sodium cobaltinitrite solution (cf. Kramer and Tisdall, A., 1921, ii, 412). Next day the precipitate is collected, washed by centrifuging, and dissolved in 5 c.c. of 0.1N-sodium hydroxide; the solution is diluted, treated with 1 c.c. of a 0.15% solution of indole in 10% alcohol and with 1 c.c. of 1:1 sulphuric acid, and diluted to 100 c.c. The resulting colour is compared with that produced by a standard sodium nitrite solution under the same conditions. A. R. POWELL.

Use of preservatives to prevent loss of nitrogen from cow excreta during the day of collection. R. B. FRENCH (J. Agric. Res., 1930, 41, 503—506).—Cow faeces and urine placed together, but unmixed, lose 1—2.6% of their total nitrogen, and, when mixed, 6—30% during 18 hrs. These losses may be prevented by the addition of 2 c.c. of N-copper sulphate, -zinc sulphate, or -formaldehyde per 100 g. of mixture. E. HOLMES.

See also A., Nov., 1482, **Germination of seeds** (SASAKI). **Energy-storage of plants and their carbon and nitrogen contents** (HÖNL).

PATENTS.

Manufacture of fertiliser salts capable of being stored. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,175, 15.6.29).—The caking on storage of granular, non-hygroscopic fertilisers is prevented by coating them with 1—5% of water-insoluble non-volatile oils. The oils may contain solid, water-repellent substances such as waxes, paraffins, or fats. W. J. WRIGHT.

Fertilising solution for seed treatment. W. F. GERIQUE (U.S.P. 1,762,294, 10.6.30. Appl., 11.8.25).—Seeds are immersed for 24 hrs. in an aqueous solution containing 4 mols. of monopotassium phosphate and 1 mol. of caustic potash per litre, and, while still wet, are coated with plaster of Paris. A. G. POLLARD.

Disinfection of seeds. W. P. RALEIGH, Assr. to PITTSBURGH PLATE GLASS CO. (U.S.P. 1,764,888, 17.6.30. Appl., 26.4.28).—Seeds are dusted with an insoluble additive compound of a mercuric salt (e.g., mercuric chloride) and hexamethylenetetramine mixed with an inert, finely-divided carrier such as graphite. A. G. POLLARD.

Fungicide. E. B. ALVORD, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,762,709, 10.6.30. Appl., 7.9.26).—The use of colloidal copper sulphide, either dry or in a

liquid vehicle containing a dispersing agent, is claimed. The product does not "burn" delicate foliage when used as a fungicide.

A. G. POLLARD.

Non-alkaline chlorate weed-killer. R. N. CHIPMAN (B.P. 335,203, 14.6.29. U.S., 22.12.28).—The herbicidal efficiency of chlorate weed-killers is increased by addition of acid substances in sufficient amount to bring the p_H below 7, whilst avoiding liberation of chlorine. Thus by adding 1, 2, and 5% of zinc chloride, the p_H was lowered to 7.1, 6.6, and 6.4, respectively; by the same percentages of copper chloride, to 5.2, 5.1, and 5.1; and by boric acid, to 7.0, 6.6, and 6.2.

W. J. WRIGHT.

Nicotine carbolates (U.S.P. 1,762,471).—See II.

XVII.—SUGARS; STARCHES; GUMS.

PATENTS.

Manufacture of adhesives. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 334,876, 1.5.29).—Adhesives made from gum arabic, dextrin, etc. are protected from bacteria, moulds, etc. by incorporation of hydroxylated di- or tri-arylmethanes containing nuclear halogen, e.g., the compounds of B.P. 316,900 and 330,893—4 (B., 1929, 888; 1930, 896), such as 5:5'-dichloro-2:2'-dihydroxydiphenylmethane, 3:3':5:5'-tetrachloro-2:2'-dihydroxydiphenylmethane, 4:5'-dichloro-2-hydroxydiphenylmethane, 3':3''':5':5''-tetrabromo-4':4''-dihydroxy-1:1-diphenylcyclohexane, 4-chloro-2:6-bis-(5-chloro-2-hydroxybenzyl)phenol, etc.

C. HOLLINS.

Conveyor-dryers (B.P. 336,009).—See I. Un-hairing hides (U.S.P. 1,763,319).—See XV.

XVIII.—FERMENTATION INDUSTRIES.

Pectin from lemon residues. LEONE.—See XIX.

See also A., Nov., 1409, **Volumetric determination of acetone** (MEYER and MATHEY). 1431, **Sterols from yeast** (WIELAND and GOUGH). 1473, **Catalase of milk and its determination** (ZAYKOVSKY and ALEXEEV). 1476, **Uricase from ox-kidney: its preparation and properties** (TRUSZKOWSKI).

XIX.—FOODS.

Direct iodising of milk is possible. E. D. DEVEREUX (Mich. Agric. Exp. Sta. Quart. Bull., 1929, 193—194).—Colloidal iodine may be added to milk (up to 100 p.p.m.) without alteration of colour or taste, even after ageing or souring.

A. G. POLLARD.

Determination of lactic acid in milk and milk products. L. H. LAMPITT and M. BOGOD (Compt. rend. 9me. Congrès de Chim. Ind., 1930, 6 pp.).—The method is based on the oxidation of lactic acid with potassium permanganate, the resultant acetaldehyde being distilled into a known volume of bisulphite solution, and the excess titrated with iodine. Full experimental details are given. Experiments showed that losses occur during the distillation unless the receiver is fitted with a U-trap containing 1 c.c. of 0.01N-iodine diluted with 10 c.c. of water. The optimum concentration for the permanganate solution was found to be 0.004N, the addition of 100 c.c. occupying 1 hr. The distillate

(80—100 c.c.) and contents of the U-tube are titrated with 0.01N-iodine, allowance being made for that contained in the trap. The iodine equivalent of the bisulphite solution having been determined, the difference between the titrations gives the acetaldehyde value (1 c.c. of 0.01N-iodine \equiv 0.45 mg. of lactic acid). When tested with zinc lactate and milk powder containing known amounts of zinc lactate it was found that a correction factor of 10/9 must be applied to obtain the true results. It has thus been shown that the lactic acid content of normal milk is lower than the acidity as indicated by titration, but if alkali has been added during manufacture the difference diminishes and may even change its sign.

H. J. DOWDEN.

Refractometer in milk analysis. G. D. ELSDON and J. R. STUBBS (Analyst, 1930, 55, 618—625).—The copper sulphate method (B., 1927, 375) of producing milk serum for determining the refraction gives good and concordant results if care be taken to prevent evaporation. Sera may also be prepared by the use of 7% phosphotungstic acid solution containing 2.5 c.c. of hydrochloric acid per 100 c.c., but this reagent also precipitates the albumin, and the refractions obtained are, on the average, 3.25 lower than those obtained with copper sulphate. The sour-serum method is satisfactory, provided that the milk is kept at a uniform temperature; 21° is suggested as a standard. In general, the refraction of milk decreases slightly with increasing age whatever method is used in the test.

A. R. POWELL.

Variation in the analytical data of butter as a result of alteration and ageing. F. ARNAUDO and A. SOLDI (Annali Chim. Appl., 1930, 20, 405—409).—Results obtained with a number of samples show that ageing and alteration of butter under ordinary conditions are accompanied by diminution of the Reichert-Meissl and Zeiss refractometer values, by slight variations in either direction of the Polenske value, and by marked increase in the total free acid. As the last-named increases in amount from 8 to 80 (c.c. of N-alkali per 100 g.), the refractive index falls progressively by 0.2—2.2 and the Reichert-Meissl value by 0.4—7.2. In those exceptional cases when the samples have an odour of butyric esters, the decrease in the Reichert-Meissl value may amount to 16.8 and that in the Zeiss value to 4.2, the total acidity being 138—145.

T. H. POPE.

Composition of some rabbit carcasses. W. K. WILSON (J. Min. Agric., 1930, 36, 1203—1206).—The chemical composition of rabbit flesh (Angora) closely resembles that of chicken, the average protein content differing by less than 1%. The composition of the flesh from male and female rabbits was noticeably different, especially in the fat content, females containing 4—6% more fat than the males.

B. W. TOWN.

Substance contained in the "water of vegetation" of olives. F. TRAETTA-MOSCA and M. VENEZIA (Annali Chim. Appl., 1930, 20, 449—451).—Application of high pressure to comminuted olives yields, together with the oil, water of vegetation, which contains a methylpentitol, $C_6H_{14}O_5 \cdot H_2O$, m.p. 164°, this giving a penta-acetyl derivative, m.p. 121°. On oxidation with

bromine the polyhydric alcohol forms a compound which gives a *phenylhydrazone*, $C_6H_5O_4(N_2HPh)_2$, m.p. 195° .

T. H. POPE.

Extraction of pectins from lemon residues (pastaccio). P. LEONE (Annali Chim. Appl., 1930, 20, 433–444).—Owing to the threat directed against the citrus industry by the competition of fermentation citric acid, the economical production of pectin from pastaccio becomes of importance. The most satisfactory method of precipitating solid pectin is by means of alcohol, and to replace the inevitable losses of alcohol the author proposes to produce alcohol by fermenting the carbohydrates of the lemon residues. Pastaccio contains 80% of water, the dry matter having the percentage composition: ash 4–4.2, fats, resins, and waxes 24–35, cellulose 19–22, lignin 8.4–9.2, groups forming furfuraldehyde 8–9.5, reducing sugars 1.2–1.8, hemicellulose, gum, etc. 9.2–15.7, pectins 15–19, nitrogenous substances not determined. The treatment suggested is briefly as follows: the pastaccio is heated with twice its weight of 0.05% hydrochloric acid at 103 – 105° and filtered, the filtrate being concentrated to one half of its volume in a triple-effect apparatus and fermented, best with a selected yeast. The fermented liquid is cleared by centrifuging and is then treated with an equal volume of 96% alcohol to precipitate the pectins, which are filtered off. The dilute alcoholic filtrate is concentrated in a rectifying column, the amount retained by the pectin being recovered by means of a vacuum desiccator followed by a column. Appreciable loss of pectin occurs only if the fermented liquid is kept for some time.

T. H. POPE.

Sulphuring of apricots. A. V. LYON (J. Coun. Sci. Ind. Res. Australia, 1930, 3, 161–166).—The sulphuring is best performed in hoods with small vents to ensure the slow burning of the sulphur (the open system). With the vents shut (the closed system) burning ceases before an adequate supply of sulphur dioxide is produced. The period of sulphuring was 4–5 hrs., and the mean sulphur dioxide content was 11 grains per lb. Properly sulphured apricots possess the bright colour desired by the trade; a dark or dull colour indicates under-sulphuring.

B. W. TOWN.

Methylene-blue in tinned peas. D. HENVILLE (Analyst, 1930, 55, 629).—A sample of tinned peas became green when the liquid was poured away and the peas were exposed to the air. A similar effect was obtained by boiling raw peas with 0.01–0.02 grain of methylene-blue per lb. and subsequently exposing them to the air.

A. R. POWELL.

Phytin from refuse of mustard manufacture. N. BELIAIEV (Masloboino-Zhir. Delo, 1929, No. 3, 28–30).—The ground press-cake is washed with water and treated with dilute hydrochloric acid; the extract is heated to coagulate the proteins and the phytin is precipitated by neutralising the filtered solution with 10% ammonia solution. The extraction and precipitation are repeated twice; the yields were 5.73 and 3.87% from the press-cake and bran, respectively.

CHEMICAL ABSTRACTS.

PATENTS.

Heating of apparatus for condensing milk and the like. N. J. NIELSEN (B.P. 335,964, 5.6.29).—The use of hot water instead of steam as the heating medium for the evaporation of milk etc. is found to reduce the formation of crusts on the tubes.

B. M. VENABLES.

Production of egg-yolk substitutes. B. REWALD (U.S.P. 1,762,077, 3.6.30. Appl., 12.3.29. Ger., 7.9.27).—A substitute for egg yolk in bakery goods consists of an emulsion of butter or margarine with 5–20% of soya-bean lecithin.

E. B. HUGHES.

Machines for kneading butter, margarine, and other foodstuffs. N. V. GRASSO'S MACHINEFABRIEKEN, and H. A. M. GRASSO (B.P. 336,837, 13.11.29).

Manufacture of [withered] tea. J. A. MAIN (B.P. 336,619, 14.6. and 7.11.29 and 3.3.30).

Grinding mills (B.P. 336,005).—See I. **Water-proofed paper** (B.P. 335,559).—See V.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Reactions of antifebrin and phenacetin. L. EKKERT (Pharm. Zentr., 1930, 71, 626–628).—Full details are given of the hydrolysis of the drugs to aniline and *p*-phenetidine, respectively, and the identification of the latter by well-known colour reactions.

H. E. F. NOTTON.

Acidimetric determination of theobromine in diuretin, calcium-diuretin, and in other theobromine preparations and mixtures. H. BOIE (Pharm. Ztg., 1930, 75, 968–969).—The sample of diuretin (0.5 g.) dissolved in 100 c.c. of water is made weakly acid by adding 15 c.c. of 0.1N-sulphuric acid, and to the mixture, after boiling to expel carbon dioxide and cooling, are added 1.5 c.c. of phenol-red solution (0.1 g. of phenol-red, 5.7 c.c. of 0.05N-caustic soda in 500 c.c. of water) and 2–3 c.c. of 0.1N-caustic soda (slight excess). The solution is then titrated until distinctly acid with 0.1N-sulphuric acid and by deducting from the total acid used the volume of alkali added the alkalinity of the sample is found (*a*). To the solution are now added 20 c.c. of 0.1N-silver nitrate, and the nitric acid liberated by the formation of the theobromine-silver complex is titrated to completion with 0.1N-caustic soda (*b*) (1 c.c. of 0.1N-caustic soda \equiv 0.01801 g. of theobromine). In diuretin, if equimolecular proportions of caustic soda and theobromine be present, the values of *a* and *b* are identical. The method is applicable to other theobromine preparations, provided a sufficient excess of silver nitrate solution be added to remove any iodides or thiocyanates. The results are in good agreement with those calculated from the nitrogen content and those determined iodometrically. The values obtained by the official method are about 15% too low.

H. J. DOWDEN.

Determination of colchicine in the seeds of *Colchicum autumnale*. L. P. R. COLL and P. J. PREIONI (Rev. farm., 1930, 72, 187–189).—The seeds (20 g.) are digested with water (200 c.c.) at 55 – 60° for 3 hrs. After 6 hrs. 140 c.c. are decanted, shaken with

lead acetate solution, and filtered. After addition of disodium hydrogen phosphate and shaking the mixture is filtered, 30 g. of sodium chloride then being dissolved in 110 c.c. The liquid is shaken with 50 c.c. of chloroform, 40 c.c. being evaporated and the residue weighed. Heraill's method is not trustworthy.

CHEMICAL ABSTRACTS.

Oil from *Perilla ocumoides* seeds. ZHDAN-PUSHKIN.—See XII.

See also A., Nov., 1393, **Dermatological thallium salts** (CLAVERA). 1418, **Local anæsthetics from amino-alcohols** (FOURNEAU and others). 1421, **Organo-antimony compounds** (DYKE and JONES). 1430, **Synthesis of an isomeride and a homologue of ephedrine** (FOURNEAU and others). 1431, **Cannabinol, the active principle of hashish** (BERGEL and others). 1432, **Organo-antimony compounds** (NIYOGI). 1451, **Synthesis of pharmacological thiazole amines** (HINEGARDNER and JOHNSON). 1454, **Alkaloid of *Skimmia repens*** (ASAHINA and others). **Skimmianine** (ASAHINA and INUBUSE). 1455, **Strychnos alkaloids** (LEUCHS and WEGENER). 1456, **Strychnine; sensitivity of chemical and physiological tests** (WARD and MUNCH). **Sulphonated aromatic arsenic compounds** (BARBER). 1458, **Mercurated phthaleins** (GREENBAUM). 1460, **Colour reaction of ephedrine** (SIVADJIAN). **Reactions of thiophen** (EKKERT). **Micro-detection of barbituric acid derivatives** (VAN ITALLIE and STEENHAUER). 1461, **Reactions of homatropine and novatropine** (EKKERT). **Titrimetric determination of primary arsinic acids** (KING and RUTHERFORD). **Toxicological determination of morphine** (PEREIRA). 1471, **Physiological assay of belladonna extracts etc.** (JENDRASSIK and WILL). **Methods of ergot assay** (SMITH and STOHLMANN). 1480, **Preparation of crystalline follicular hormone** (BUTENANDT). 1484, **Degradation of nicotine in tobacco** (FAITLOWITZ).

PATENTS.

Manufacture of 1-phenyl-2-methyl-3:4-trimethylene-5-pyrazolone. F. HOFFMANN-LA ROCHE & Co. A.-G. (Swiss P. 122,466, 12.5.26. Cf. B.P. 260,577; B., 1927, 869).—Ethyl cyclopentanonecarboxylate, b.p. 108°/12 mm., is condensed with α -phenyl- β -methylhydrazine in presence of sodium ethoxide at 140°.

C. HOLLINS.

Manufacture of compounds of 1-aryl-2-alkyl (or -2-aralkyl)-3:4-trimethylene-5-pyrazolones with dialkyl- and aralkyl-barbituric acids. C. MANNICH (G.P. 454,697, 17.1.26).—Analgesics are obtained by mixing the named ingredients in molecular proportions, or by mixing their solutions (using excess of the more soluble) and allowing to crystallise. Products from 1-phenyl-2-methyl-3:4-trimethylene-5-pyrazolone (cf. B.P. 260,577; B., 1927, 869) with allylisopropylbarbituric acid (m.p. 141°), with phenylethylbarbituric acid (m.p. 125°); from 1-*p*-tolyl-2-ethyl-3:4-trimethylene-5-pyrazolone with allylisopropylbarbituric acid (m.p. 127°); from 1-phenyl-2-benzyl-3:4-trimethylene-5-pyrazolone with phenylethylbarbituric acid (m.p. 64°), are described.

C. HOLLINS.

Preparation of remedies for cardiac diseases.

W. W. GROVES. From I. G. FARBERIND. A.-G. (B.P. 335,202, 10.6.29).—Fresh or dried muscles of warm-blooded animals are extracted at the natural p_H of the material or in presence of added alkali, with a water-soluble organic solvent in presence of more than 10% of water. The solvent-free extract is freed from lipins and albumin by treatment with a water-insoluble organic solvent and the final aqueous extract is saponified, and the resulting solution, if necessary, freed from by-products by treatment with a water-insoluble organic solvent or by evaporation and treatment of the residue with a water-soluble solvent. The initial extraction of the muscles may be carried out with a water-insoluble solvent, in presence of water if dried muscles are used, and the residue extracted with a water-soluble solvent in presence of water; after removal of the solids the aqueous extract is treated as above. E. H. SHARPLES.

Manufacture of preparations possessing intense antirachitic properties. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 335,277, 25.6.29).—Ergosterol, either solid or dissolved in an organic solvent such as ethyl alcohol, acetone, or ethyl acetate, is irradiated by means of ultra-violet rays, irradiation preferably being stopped before the maximum of antirachitic activity is reached. Unchanged ergosterol is then separated from the irradiated product by crystallisation from the solvent, this separation being continued until the product has $[\alpha]_{D}^{25}$ greater than +25° in alcohol. It is better to irradiate the hot solution. Substances having activities greater than 50 million units are claimed. E. H. SHARPLES.

Betaine thiocyanate. R. BERENDES and L. SCHÜTZ, Assrs. to WINTHROP CHEM. Co., Inc. (U.S.P. 1,780,860, 4.11.30. Appl., 18.6.28. Ger., 28.6.27).—See B.P. 316,693; B., 1929, 835.

[Production of] alkaline-earth salts of arsenobenzene derivatives. R. WERNER and A. ROTHMANN, Assrs. to WINTHROP CHEM. Co., Inc. (U.S.P. 1,779,649, 28.10.30. Appl., 21.9.29. Ger., 5.9.28).—See B.P. 318,556; B., 1930, 1046.

Basic imino-ethers (B.P. 308,218).—See III. **Bromine and iodine** (B.P. 308,281).—See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Action of ammonium salts on photographic developers. M. MIYATA and Y. SASAKI (Rep. Imp. Ind. Res. Inst., Osaka, 1930, 10, No. 17).—The activation is physical in character. CHEMICAL ABSTRACTS.

See also A., Nov., 1329, **Actinic power of magnesium light** (ARENS and EGGERT). 1384, **Relationship between particle size and sensitivity of photographic emulsions towards X-rays** (EGGERT). **Influence of the developer on the properties of a photographic plate** (PERRAKIS). 1385, **Action of mercuric chloride on the photographic plate** (REYCHLER). **Herschel effect** (NARBUTT).

PATENTS.

Development of photographic reversal films. E. MANKENBERG, Asst. to AGFA ANSCO CORP. (U.S.P.

1,780,025, 28.10.30. Appl., 15.3.28. Ger., 25.3.27).—See B.P. 287,542; B., 1929, 378.

[Tri-pack for] colour photography. T. T. BAKER, A. B. KLEIN, and COLOUR SNAPSHOTS (1928), LTD. (B.P. 337,057, 27.7.29).

[Die-cast cellulosic] photographic film spools. I. G. FARBENIND. A.-G. (B.P. 316,302, 10.7.29. Ger., 27.7.28).

Making of X-ray photographs. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 336,918, 7.2.30. Holl., 20.2.28).

[Production of prints in natural colour in] colour photography. T. T. BAKER (B.P. 337,040—1, 25.7.29).

Measuring the intensity of light (B.P. 335,179).—See XI.

XXII.—EXPLOSIVES; MATCHES.

Sensitiveness of gelatinised [smokeless] powders. M. TARLÉ (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 398—400).—The influence of the temperature of drying on the sensitiveness to shock and the ignition temperature of strip and flaked powders was investigated, the fall-hammer results being quoted as percentages of those obtained with undried powder. Loss in weight on drying at various temperatures for various periods was also determined. Up to 100° sensitiveness varied approximately with loss in weight; it may be inferred that at low temperatures, at which the stability is only slowly affected, a physical change occurs in the powder. At temperatures above 100° decomposition is a factor, since heating for 2 hrs. at 120° causes a loss in weight of only 2%, but gives a fall-hammer test of 81—82%. Between 60 and 80° a definite surface alteration takes place in gelatinised powders, and a study of this alteration, as well as of the relation between loss in weight and sensitiveness, may be the means of determining the age of a powder and its suitability for use. No appreciable change in the temperature of ignition was observed with various temperatures or times of heating. This does not necessarily imply that the powder underwent no change, since small changes in the physical structure do not affect the test.

W. J. WRIGHT.

Properties of nitroglycerin isomerides. A. A. DSERSCHKOVITSCH and K. K. ANDREEV (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 353—356, 400—403).—By Hess' method the liquid labile and stable isomerides of nitroglycerin gave total compressions of 26.5 and 27.5 mm., respectively. The solid stable form gave an average compression of the lower cylinder of 6.3, and the solid labile form of 4.8 mm.; in the former case the upper cylinder was completely demolished. Tested by Kast's method, liquid nitroglycerin gave an average compression of 1.84, the solid stable isomeride gave 3.07, and the solid labile one 0.70 mm. The velocities of detonation, determined by Dautriche's method with a No. 8 detonator, were 1165 m./sec. for nitroglycerin and 9150 m./sec. for the solid stable isomeride. The labile isomeride failed to detonate completely with a No. 8 detonator, but when initiated by a 20-g. tetryl primer it gave the value 9100 m./sec. Liquid nitroglycerin initiated by 10—15 g. of the stable isomeride also attained a velocity of 9100 m./sec. The labile isomeride gave the highest lead block results, 518—562 c.c.; the stable form gave 392—397, and liquid nitroglycerin 486—518 c.c.

Investigations were made of the linear velocity of crystallisation; for the labile form it is only half that of the stable form. The velocity of isomerisation was also determined. The chemical nature of the two isomerides is discussed, and an explanation advanced of the explosion abnormalities of liquid nitroglycerin and nitroglycerin explosives.

W. J. WRIGHT.

See also A., Nov., 1379, Detonation of solid explosives (GARNER).

XXIII.—SANITATION; WATER PURIFICATION.

Effectiveness of sodium peroxide respirators. G. STAMPE and E. HORN (Z. ges. Schiess- u. Sprengstoffw., 1930, 25, 419—422; cf. B., 1929, 798).—The evolution of oxygen from hydrated "pyroxylyt" (water 6%) is continuous; in 200 days 1 kg. evolved 3800 c.c. of oxygen, and there was no evidence of cessation at the end of that period. It is considered that during hydration superficial formation of sodium peroxide dihydrate occurs, and this readily decomposes, the liberated hydrogen serving to hydrate other molecules of the peroxide. The decomposition of sodium peroxide cartridges, when used in respirators, has been investigated, and is discussed.

W. J. WRIGHT.

Determination of small quantities of active chlorine in purified water. S. ANSELM and A. CALÒ (Annali Chim. Appl., 1930, 20, 410—424).—Examination of the various methods proposed for the determination of small proportions of active chlorine in water purified by means of hypochlorites, chloramine, or gaseous chlorine shows that the most satisfactory is the o-tolidine colorimetric method (cf. Ellms and Hauser, B., 1914, 804), which is sensitive to 0.02 mg. Cl per litre and may be used for amounts up to 0.2 mg. per litre. The coloration is best compared with a scale obtained by mixing copper sulphate and potassium dichromate solutions in various proportions. For higher concentrations of chlorine, the potassium iodide-starch paste method is recommended, the iodine liberated being titrated with 0.01N-sodium thiosulphate; the proportion of chlorine found should be increased by 0.1 mg. per litre, which represents the limit of sensitivity of the reaction.

T. H. POPE.

ERRATUM. B., 1930, 796.—In the abstract of the paper on removal and determination of nitrates in sewage effluents and waters (by J. W. H. JOHNSON), in line 13 and subsequent lines the words "by suggesting . . . distillation" should be replaced by the following: "by suggesting that the samples were not boiled down as indicated, but distilled. The examination of these distillates would then disclose only half the original amount of nitrates, the other half having been found to disappear on distillation; this would fully account for the Commission's results of only 50% nitrate removal by boiling."

[Fatal] poisoning by amyl acetate. CRECELIUS (Klin. Woch., 1930, 9, 452—454; Chem. Zentr., 1930, 1930, 1170).

PATENTS.

Method of filtering (U.S.P. 1,762,560). Removing solids from strainers (B.P. 335,767). Treatment of boiler water (U.S.P. 1,762,748).—See I.

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