

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUGUST 20, 1926.

I.—GENERAL; PLANT; MACHINERY.

Equipment for high-pressure reactions. F. A. ERNST (Ind. Eng. Chem., 1926, 18, 664—669).—The chief constructional materials used for making large-scale apparatus for carrying out reactions at high temperatures under pressure are chromium, nickel-chromium, and chromium-vanadium steels for work up to 500°, and nichrome containing 80—85% Ni and 20—15% Cr for higher temperatures. A steel containing 2.5% Cr, 0.2% V, and 0.37% C is suitable for ammonia converters working at 450—500°. The tensile strengths of the various high-temperature steels decrease slightly up to about 250°, then increase to a maximum at 300°, and finally decrease rapidly with further rise of temperature. Diagrams are given showing the construction of gas compression cylinders for laboratory work, hydraulic pumps, heads for pressure containers, high-pressure valves and connexions, high-pressure compressors, a magnetic regulating valve for high pressures, a relief valve, and an insulated high-pressure joint for an electric lead, together with details of their working and sphere of usefulness. The original must be consulted for full details.

A. R. POWELL.

Measurement of surface temperatures. I. Portable thermocouple device compensated for heat losses. M. W. BOYER and J. BUSS (Ind. Eng. Chem., 1926, 18, 728—729).—In order to compensate for heat losses when measuring the temperature of a surface up to 150°, the two wires of the thermocouple are each soldered to a 1-in. silver disc, on diametrically opposite sides, in such a way that the disc, acting as the hot junction, may be laid flat on the surface the temperature of which is to be measured. To prevent errors due to radiation an exactly similar couple is arranged just above the measuring couple, but insulated from it by a thin sheet of mica, and is heated on the outer side by means of a resistance grid to such a temperature that when both couples are connected to a galvanometer no deflexion occurs showing that both couples are at the same temperature, *i.e.*, the temperature of the surface. The whole device is mounted in a flat wooden block, 3 in. square and $\frac{1}{2}$ in. thick, so as to be easily portable.

A. R. POWELL.

See also A., July, 678, Structure of precipitates (ODÉN). 706, Preparation of nickel membranes for ultrafiltration (MANNING). 707, Apparatus for continuous percolation and for filtration in neutral atmospheres (EVANS); Non-inflammable liquids for cryostats (KANOLT).

Turbidity readings. SCOTT.—See XXIII.

PATENTS.

Regulation of evaporating, concentrating, and distilling apparatus. SOC. DES CONDENSEURS DELAS (E.P. 229,679, 18.2.25. Conv., 23.2.24).—In apparatus of the type described in E.P. 201,123 (B., 1923, 1114 A) an automatic feed regulator is provided, preferably worked by the differential expansion of two arms of a bent tube, the upper arm being filled with steam from the vapour space and the lower arm containing water condensed from the arm above, at a level varying in equilibrium with the varying level of the liquor in the evaporator.

B. M. VENABLES.

Vertical film type evaporators. GRISCOM-RUSSELL Co., Assees. of L. E. SEBALD (E.P. 232,925, 11.2.25. Conv., 25.4.24).—In a vertical evaporator of the falling film type, short lengths of smaller tubes extend into the upper parts of the heating tubes, the liquor being fed through the annular spaces and the vapour passing out through the interiors of the smaller tubes. The heating steam surrounds the outside of the heating tubes. A spiral vane may be placed in each annular space to impart a whirling motion to the falling film of liquor and the operation may be governed by maintaining a constant depth of feed liquor above the upper tube plate, surrounding the smaller tubes. The rate of evaporation may be regulated by controlling the rate of egress of concentrated liquor, most of which is returned by a circulating pump for re-evaporation. (Reference is directed under Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 154,355, 131,365, 28,537 of 1913, 22,670 of 1911, and 3006 of 1888.)

B. M. VENABLES.

Evaporating apparatus. BLAIR, CAMPBELL & McLEAN, LTD., and J. R. PHILLIPS (E.P. 252,505, 4.4.25).—In an evaporator comprising a number of superposed effects, the combined liquor and vapour passes from any calandria to the corresponding separator through a passage surmounted by a dome-like deflector, which can be raised or lowered so as to vary the size of the annular opening to suit the density of the liquor and rate of evaporation.

B. M. VENABLES.

Apparatus for boiling ["bumping"] liquids. SIEMENS-SCHUCKERTWERKE GES.M.B.H. (E.P. 235,192, 2.6.25. Conv., 3.6.24).—A device for concentrating liquids, *e.g.*, the sulphuric acid used in an absorption refrigerator, which have a tendency to boil unsteadily, comprises a tube surrounded by a source of heat. The liquid and vapour rise in the tube, which is so arranged that it is never

completely free from vapour. This may be effected by forming bulges in the tube or making it in a sinuous or helical form, so inclined that though the general slope is upwards some portions of the tube have a downward slope. B. M. VENABLES.

Pulverising mills. RAYMOND BROS. IMPACT PULVERIZER Co., Asses. of J. CRITES and W. H. VOGEL (E.P. 248,357, 16.2.26. Conv., 2.3.25).—In a pulveriser comprising an impact chamber, and preferably also a fan chamber, the outlet from the pulverising chamber is regulated by means of a ring member with a conical opening which when rotated by external means also moves longitudinally by the agency of cam-shaped slots and alters the size of the exit opening. B. M. VENABLES.

Grinding discs for use in grinding mills. A. BLUME (E.P. 251,778, 15.5.25).—In a grinding mill where two toothed discs rotate in opposite directions, the discs are arranged to have several concentric grinding zones with teeth that decrease in size from the centre outwards. Venting grooves, extending through all the zones, are arranged tangentially to a small circle at the centre of the disc, and the teeth between any two venting grooves are arranged parallel to that groove which is in the front of the teeth in the direction of rotation of the disc. B. M. VENABLES.

Crushing or grinding apparatus. W. CHAMBERS (E.P. 253,055, 16.3.26).—The apparatus comprises a number of beaters or pestles rotating within a grinding ring or mortar which is supported on rollers so that it can rotate under the influence of the beaters. The frame carrying the rollers and ring, also the main shaft of the beaters, are spring-mounted and the arms of the beaters are made of resilient material. B. M. VENABLES.

Mixing, disintegrating, or homogenising machine. H. E. AUSTIN, Assr. to AMER. CREOSOTING Co. (U.S.P. 1,587,063, 1.6.26. Appl., 18.5.25).—Two elements rotating relatively to one another are formed from segments of the surface of a paraboloid of revolution. The space between the two surfaces is not uniform and can be increased, the increase at the feed being proportionally greater than at the discharge end. In consequence discharge is hindered and economical grinding is effected. E. S. KREIS.

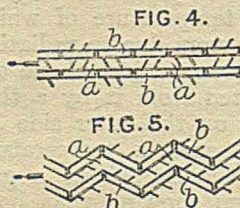
Disintegrating apparatus. J. E. KENNEDY and L. A. HUNT (U.S.P. 1,587,653, 8.6.26. Appl., 26.3.25).—A revolving drum is divided into grinding and collecting chambers by means of a perforated partition, and screening of the material is effected partly by the partition and partly by means of screens situated in another chamber surrounding and concentric with the drum. The oversize from the inner compartment of the screening chamber passes through passages in the circumference of the drum back to the grinding chamber. The fine material from the outer compartments passes through a transverse passage situated in the collecting chamber to the axial outlet of the mill. B. M. VENABLES.

Pulverising machine. D. T. FISHER, Assr. to A. M. READ (U.S.P. 1,587,769, 8.6.26. Appl., 17.9.20. Renewed 19.10.25).—A revolving horizontal barrel is divided into separate grinding chambers by perforated partitions, each partition having, on the face further from the feed end, a number of vanes extending inwards from the circumference and adapted to propel material longitudinally away from the partition. B. M. VENABLES.

Removing suspended matter from gases. R. W. BAILEY, and METROPOLITAN-VICKERS ELECTRICAL Co., LTD. (E.P. 251,682, 2.2.25).—Various forms of apparatus are described all of which embody the principle that collecting material, such as fibrous material, perforated metal, or metal wire fabric, is supported on both sides of easily removable walls, which alternate with fixed walls that serve only as guiding means for the gases. The cross-sectional area of the ducts between the fixed and removable walls preferably decreases gradually in the direction of flow of the gases, and arrangements may be made for cutting out any of the ducts while the collecting material is being cleaned. B. M. VENABLES.

Separating solid particles from gases. CHANCE AND HUNT, LTD., and W. A. S. CALDER (F.P. 252,271, 24.2.25).—The gas is passed through one or more orifices and impinges on a wall placed at a distance from the orifice equal to about one-quarter or one-half the diameter of the orifice. The gas preferably passes only once through an orifice, and a liquid or vapour may previously be added to it to aid the deposition of the solid particles. The wall may take the form of a rotating disc, which may be provided with scrapers for removing deposited matter both from the interior of the orifice and the space between the wall and the orifice. B. M. VENABLES.

Separating solid and liquid constituents from gases and vapours. H. SZAMATOLSKI and A. BLOHM (E.P. 229,305, 11.2.25. Conv., 13.2.24).—



A number of sheet metal elements with stamped out tongues or baffles are assembled as shown in the figures to form a continuous separating zone, *a*, through which the gases are passed, and a number of trap chambers, *b*, in which the material separated by impact against the baffles is collected. B. M. VENABLES.

Pyrometer sheath. W. C. JOHNSON (E.P. 251,750, 4.4.25).—A sheet of chromium steel is rolled to form a tube with the seam left open, say, 1/32 in., the seam is then electrically welded using a pencil of substantially the same material, the metal being allowed to flow through the seam to form a ridge both inside and outside the tube; a metallic mandrel with a longitudinal groove

coinciding with the seam may be used to produce a chill on the metal of the weld. The proportion of chromium in the steel should be at least 15%, and the welding voltage should be between 33 and 35 volts. The end of the tube is preferably formed by forging down to a small diameter and closing the resulting small end by welding. Chromium-steel tubes made in this way have withstood satisfactorily for extended periods a furnace temperature of 1260°.

B. M. VENABLES.

Centrifugal liquid purifiers. DE LAVAL CHADBURN CO., LTD., and W. R. CHADBURN (E.P. 252,421, 27.1, 3.2, 5.2, 9.2, 16.2, and 25.2.25).—The liquid, *e.g.*, a lubricating, transformer, or vegetable oil, or boiler feed-water, to be freed from absorbed oxygen, or beer or wine to be purified in presence of carbon dioxide, is sprayed from the bowl of the centrifuge in direct contact with inert or otherwise desirable gas delivered from a gas container into the stationary casing through a valved pipe connexion. Air may be preliminarily removed from the apparatus by a vacuum pump, and the gas-supply valve is then preferably regulated to maintain the pressure in the system above atmospheric. When economical use of the gas is necessary, the liquid is delivered into a closed collecting vessel from which the gas is re-circulated through the casing. The liquid is withdrawn from the collecting vessel while maintaining a constant liquid level therein to prevent escape of the gas. Separation of the gas from the liquid may be facilitated by the provision of partitions, inclined to the vertical, within the collecting vessel. When different liquids, *e.g.*, transformer oil and water, are separated into different compartments within the casing, each compartment is connected to a separate collecting vessel, and the two vessels are connected to equalise the pressure.

H. HOLMES.

Apparatus for separating liquids from solids. H. C. BEHR (U.S.P. 1,586,395, 25.5.26. Appl., 24.7.22).—A foraminous mantle surrounds a foraminous conveyer and the two are rotated at different speeds so as to effect a continuous separation of a mixture fed at a predetermined rate into the space between them by a rotary pump. The feed pump is driven through worm and belt gears from the centrifugal mechanism.

H. HOLMES.

Storing and shipping containers for acids and alkaline liquids. P. PICK (E.P. 252,772, 2.3.25).—Closed inner and outer shells of ebonite or like material are provided with a completely enclosed reinforcing skeleton of perforated metal. The method of making the containers is described in detail.

H. HOLMES.

Drying materials [*e.g.*, pulverised fuel]. O. Y. IMRAY. FROM INTERNAT. COMBUSTION ENGINEERING CORP. (E.P. 252,835, 27.3.25).—The damp material (before pulverising) is fed into a stream of hot gases and the mixture immediately passed through a cyclone separator. Part of the used gases from

the cyclone may be drawn back by the circulating fan and used again, only sufficient fresh hot gases being drawn in (and a corresponding amount of used gases exhausted) to maintain the necessary conditions. The hot gases may be drawn from several sources at different temperatures, and means are provided so that if the material happens to arrive sufficiently dry it may pass directly to the pulveriser without passing through the drying cyclone apparatus. The rate of feed of material is governed by the rate of pulverisation.

B. M. VENABLES.

Apparatus for drying material. G. A. GALLOWAY, Assr. to B. F. STURTEVANT CO. (U.S.P. 1,587,582, 8.6.26. Appl., 11.9.25).—A number of courses of ducts shaped as inverted troughs are arranged at right angles to one another, the ducts extending in one direction being used as supply ducts and those in the other direction as collecting ducts. The supply ducts are fed with air from an external source or through external passages from the collecting ducts next below, and the collecting ducts receive air from the supply ducts below through their open bottoms. The material to be dried passes downwards by gravity over the ducts.

B. M. VENABLES.

Dehydrating plant. G. W. BOOKSH, JUN. (U.S.P. 1,588,298, 8.6.26. Appl., 13.1.26).—The apparatus comprises a casing with interior walls so arranged that hot air supplied to a central space passes through lateral drying chambers to flues at the sides, then up and inwards to an outlet at the top.

B. M. VENABLES.

Absorption towers. N. WADE. FROM KOPPERS CO. (E.P. 253,057, 25.3.25).—A device for the top of an absorption tower to catch any spray that would strike the walls and run down them, consists of a number of concentric vertical rings, stepped downwards with decreasing diameter and with spaces left between the rings.

B. M. VENABLES.

Lixiviation or extraction of solid substances by means of volatile solvents. P. C. RUSHEN. FROM F. KRUPP GRUSONWERK A.-G. (E.P. 253,333, 11.5.25).—The volatile solvent moves in a continuous cycle through a collecting tank, lixiviation vessel, separator for coarse solids, filter for removal of slime, still for recovery of dissolved matter, condenser, and collecting tank. The temperature of condensation is adjusted so that the solvent is recovered hot enough to be used for lixiviation without additional heating. The solid matter moves counter-current to the solvent in the lixiviating vessel, which consists of a revolving conical drum provided with transverse baffles and lifting blades around the circumference, a continuous feed and discharge being maintained through hollow trunnions. The lixiviated solid matter collected in the separator and filter is dried by evaporation and the vapour returned to the solvent circuit.

B. M. VENABLES.

Apparatus for the continuous treatment of solids with liquids. [Recovery of sulphur from

spent gas-purifying material.] K. PFISTERER (G.P. 426,385, 17.1.25).—Solid material such as spent gas-purifying material is charged into a mixing chamber surrounding the liquid jet in apparatus similar to that described in G.P. 341,829 and 398,640 (cf. B., 1924, 899), whereby intimate contact between the solid and the solvent is effected as they pass together through the apparatus. L. A. COLES.

Granulating mill. F. L. BRYANT (U.S.P. 1,586,282, 25.5.26. Appl., 7.2.23).—Within a cylindrical casing which increases in diameter downwards in a number of steps, rotate a corresponding number of horizontal discs, the spaces between the discs and between the rims of the discs and the casing being so adjusted that granulation takes place without substantial attrition or heating.

B. M. VENABLES.

Heating method and apparatus. F. X. GOVERS (U.S.P. 1,586,987, 1.6.26. Appl., 2.4.21).—For endothermic and exothermic reactions taking place in the neighbourhood of, say, 400°, a temperature-controlling medium is used which is a solid in the neighbourhood of 50° and a stable liquid at the temperature in question, as for instance a lead-tin alloy. This medium is brought into thermal contact with the charge and is then circulated to the furnace or cooler as the case may be.

E. S. KREIS.

Heat treatment of chemical compounds. F. X. GOVERS (U.S.P. 1,587,115, 1.6.26. Appl., 8.8.23).—In a cylindrical heating vessel provided with an axial stirrer with radial arms, the arms are provided with vanes so arranged that the liquid is directed radially against the walls of the vessel as a result of the revolution of the stirrer, with the consequence that fresh liquid is always coming into contact with the heating surface and also that deposit on the heating walls is lessened.

E. S. KREIS.

Regeneration of decolorising materials. L. GURWITSCH (G.P. 427,805, 14.10.24).—Spent decolorising material, such as fuller's earth, charcoal, alumina, or silica gel, is regenerated by extraction with mixtures of benzol or benzine with alcohols or ketones.

L. A. COLES.

Method for making solid substances crystallise out of their solutions. SOC. DES CONDENSEURS DELAS (E.P. 238,215, 24.7.25. Conv., 6.8.24).—See G.P. 412,968; B., 1925, 699.

Grinding mill. K. MIDDLEBOE, ASSR. to F. L. SMITH & Co. (U.S.P. 1,589,302, 15.6.26. Appl., 6.8.25. Conv., 21.8.24).—See E.P. 238,866; B., 1925, 868.

Production of metallic catalysts. E. J. LUSH, ASSR. to TECHNICAL RESEARCH WORKS, LTD. (U.S.P. 1,590,897, 29.6.26. Appl., 11.5.25. Conv., 16.7.24).—See E.P. 241,278; B., 1925, 990.

[Analytical] crucibles. W. & J. GEORGE, LTD., and J. D. M. SMITH (E.P. 252,901, 5.6.25).—See J.S.C.I., 1925, 539 T; A., 1926, 41.

Air or gas filters. A. B. CLEWORTH & Co., LTD., and A. B. CLEWORTH (E.P. 253,606, 19.3.25).

Machines for grinding, refining and mixing cocoa beans, chocolate, chemicals, and such like. J. McINTYRE (E.P. 253,835, 27.2.26).

[Temperature-sensitive devices for] controlling chemical reactions. BRIT. THOMSON-HOUSTON Co., LTD., H. W. H. WARREN, R. NEWBOUND, and L. M. T. BELL (E.P. 253,984, 20.3.25).

Atomising liquids containing solid matter in solution or in suspension. P. MULLER (E.P. 254,020, 27.3.25).

Apparatus for cooling liquids. C. DUNKELBERG (E.P. 254,073, 26.5.25).

Electrodes for use in electrolytic systems for protecting boilers and the like. A. KIRKALDY (E.P. 254,188, 14.12.25).

Drying apparatus (E.P. 252,858).—See II.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Resins in coal. Composition of coal. W. FRANCIS and R. V. WHEELER (J.C.S., 1926, 1410—1412).—By the oxidation of the vitrain portion of bituminous coal with air or hydrogen peroxide, followed by extraction with alkali, whereby the ulmin constituents are removed, pale yellow rods about 0.02 in. long and 0.002 in. diameter are obtained. When a polished and etched surface of vitrain is examined microscopically a cellular structure is observed, and the rods of resin can be seen in the positions they would be expected to occupy in the original structures. C. J. STILL.

Origin of pyrites in coals. E. DONATH and F. VYKYPÍEL (Brennstoff-Chem., 1926, 7, 153—155).—Iron sulphides are produced if organic substances (e.g., wood, albumin) are impregnated with solutions of ferrous or ferric sulphate and heated in an air oven to 200—300°, the organic material becoming charred. The nature of the disulphides found in coals furnishes a clue as to whether they were produced during or after the formation of the coal, the presence of pyrites and marcasite, or of pyrites alone, indicating sulphide production at an elevated temperature during coal formation.

W. T. K. BRAUNHOLTZ.

Coal blending. D. BROWNLIE (Iron and Steel Inst., May, 1926. Advance proof, 42 pp.).—A review of the general principles of coal blending, as applied particularly to both high- and low-temperature carbonisation. Coal blending is often of advantage for boiler firing with ordinary or pulverised fuel and for preventing the caking of charges in gas producers. Coals which contain exactly the right amount of resinous material to give a homogeneous, coherent coke or semi-coke without swelling are rare, and in general it is advantageous to resort to

carefully controlled blending of strongly swelling bituminous coals, having an excess of resinous material, with non-swelling and non-caking coals, or other fuel deficient in this respect, *e.g.*, high-temperature coke breeze, low-temperature semi-coke (prepared from the same coal as that with which it is blended, or from any other suitable coal), oxidised or preheated coals in which the swelling properties are destroyed. High-grade coke may also be produced by carbonising a mixture of anthracite or other non-caking coal with tar, pitch, oils, or petroleum and other natural products. The principle of blending has found little application, however, in the brown coal, lignite, and shale carbonisation industries; nor have the possibilities of "chemical blending" been fully explored, *i.e.*, blending of coals with fuel products that have first been treated chemically (*e.g.*, coal bitumen) or with materials (*e.g.*, soap emulsions, barium carbonate) which have an advantageous influence on the resulting coke. The review, which contains photographic illustrations, concludes with a brief reference to a number of typical low-temperature carbonisation processes, in which fuel blending plays an important part.

W. T. K. BRAUNHOLTZ.

Correction for radiation in calorimetric determinations of calorific value. L. NIEDERSTRASSER (*Brennstoff-Chem.*, 1926, 7, 185—186).—Langbein's simplified form of the Regnault-Pfaundler correction (*J. pr. Chem.*, 1889, 39) is recommended provided the rise in temperature in the main determination is most rapid at the beginning and that the temperature of the calorimeter water is only so far below that of the water jacket that a minimum heat interchange takes place in the preliminary experiment.

W. T. K. BRAUNHOLTZ.

Gas yield of various solid fuels as a criterion of their combustibility and behaviour in the fire. M. DOLCH (*Brennstoff-Chem.*, 1926, 7, 133—139).—Not only the content of volatile matter in a fuel, but also its gas yield and the composition and calorific value of the gas should be considered in evaluating the fuel. In the case of coals, the ratio (expressed as percentage) of the heat available from the gas alone to the total heat available from the fuel is in fairly close agreement with the gas yield (expressed as percentage by weight of the fuel), but in the case of lignite, semi-coke, or coke this agreement does not hold good, so that the evaluation of the latter on the basis of their content of volatile matter or gas yield is fallacious. The calorific values of the gases yielded by a variety of solid fuels are tabulated, together with the percentage of the total heat contributed in each case by the hydrogen, carbon monoxide, and methane present.

W. T. K. BRAUNHOLTZ.

Coke testing. W. A. HAVEN (*Amer. Iron and Steel Inst.*, May, 1926; *Engineering*, 1926, 172, 13).—The relative rate of combustion of coke for blast-furnace purposes was determined by introducing 1 g. of carefully sized coke in a platinum boat into a combustion tube maintained at 980°, and determining the loss in weight at 1 min. intervals. Average

results expressed in *cg.* per min. were taken as the burning speed. High ash content was a detriment to rapid combustion and cellular structure influenced the combustibility. The factors involved are apparently developed from the original coal and variation in the method of coking is of little importance. A modified tumbler test was used also to determine the character of the coke, on the assumption that the portion which withstood disintegration would represent the portion most likely to reach the region of the tuyères of the blast furnace. This test showed advantages in coke made from coal with a low content of volatile matter.

C. A. KING.

Low-temperature carbonisation. D. BROWNLIE (*Proc. S. Wales Inst. Eng.*, 1926, 42, 273—376).—A detailed historical and general review of processes for producing free-burning, smokeless fuels, involving the mechanical compression or briquetting of the charge before or during carbonisation, whereby bituminous coal, in the form of slack or dust, together with a considerable amount of material poor in volatile matter, can be converted into a high-grade product in large pieces. The free-burning property is due, in these fuels, to the very close-grained structure of the carbonised material, irrespective of its content of volatile matter or the temperature of carbonisation. As examples of continuous coking processes in which the viscous charge is compressed during carbonisation, details are given of the Summers process, in which a hydraulic ram is used, and of the three adaptations of Fischer's principle, in which compression is effected by the combined influence of centrifugal force and a heavy loose roller, *viz.*, the Dobbelstein, Raffloer, and Meguin processes. A second general method, involving briquetting by means of pitch or other externally produced binder followed by carbonisation at low or high temperature, is exemplified by detailed accounts of the "carbocoal" process (high-temperature carbonisation of a briquette of semi-coke and pitch), the "Midland Coal Products" process (combined carbonisation and gasification of pitch briquettes of non-coking slacks, alone or blended with coking slacks), and the "Sun Fuel" process (carbonisation of briquettes containing pitch and a mixture of small anthracite and bituminous coal). The third general method, *viz.*, briquetting without pitch or other externally produced binder, followed by carbonisation, includes the "Pure Coal Briquette" process (briquetting of blended fuels by means of high pressures, followed by carbonisation at a medium temperature), and the Delkeskamp process (briquetting a mixture of coal and a colloidal solution of the same coal or any other carbonaceous material, followed by carbonisation at low or high temperature). The paper contains numerous illustrations and statistical tables.

W. T. K. BRAUNHOLTZ.

Determination of yield of low-temperature tar with the aluminium retort. E. FLEISCHMANN (*Brennstoff-Chem.*, 1926, 7, 186—187).—Tar and aqueous distillate are collected in a water-cooled, graduated and tared glass tube, which is re-weighed

and then filled with benzene, shaken, and centrifuged. The water separates out sharply and its volume is read off, the tar yield being determined by difference.

W. T. K. BRAUNHOLTZ.

Oil from coal. H. NIELSEN (Gas J., 1926, 174, 591—592, 650—653, 732—734).—Processes of low-temperature carbonisation, the Bergius process, and Fischer's "Synthol" process are compared from the point of view of the economic conversion of coal into oil. Emphasis is laid on the importance of producing a "primary" oil by low-temperature carbonisation. The oil from the "L. & N." process (Nielsen and Laing, B., 1924, 122) satisfies Fischer's criteria for a primary oil (B., 1919, 941 A), whilst that from an externally heated horizontal retort at 560° fails to do so. If a true primary product is obtained the neutral oils from all bituminous coals are identical. The apparent discrepancy between this result and the statement of Sinnatt and King (J.S.C.I., 1925, 44, 413 T) is due to the use by the latter of a retorting system yielding a mixture of primary and secondary products. Information about oils derived only from distillation curves may be misleading and should be supplemented by determinations of specific gravity. The necessary distillation conditions for obtaining a true primary oil are outlined. The principal essential condition consists in the rapid removal of the products of distillation from the retort by a current of inert gas. The "L. & N." neutral oil is apparently identical with Bone's "fraction 1" obtained on extracting coal with benzene under pressure (J.S.C.I., 1925, 44, 291 T). The high-boiling fractions of the "L. & N." oil are characterised by great thermal stability, and form good lubricating oils. The results of Heyn and Dunkel on the berginisation of a Silesian coal (Brennstoff-Chem., 1926, 7, 20, 81) are compared with those of the "L. & N." process applied to a similar coal. The percentage of lubricating oil in the Bergius product is negligible. The Bergius process appears to be essentially a "cracking" process. The Fischer and Tropsch "Synthol" process (B., 1924, 152, 1001) is discussed and it is concluded that the most efficient method at present available for the conversion of coal into oils consists in (a) low-temperature carbonisation in the "L. & N." retort, and (b) use of the coke as a source of water-gas and conversion of the latter into "Synthol."

A. B. MANNING.

Decomposition of "Synthol" at atmospheric pressure. H. TROPSCH (Abh. Kennt. Kohle, 1925, 7, 49—50; Chem. Zentr., 1926, I., 3583).—"Synthol" (5.8 g.) on heating to 400—480° at atmospheric pressure yielded 3.9 g. of carbon, 0.3 g. of carbon dioxide, 0.7 g. of carbon monoxide, 0.5 g. of hydrogen, 0.2 g. of methane, and 0.1 g. of hydrocarbons, these products being different from those obtained by heating it under pressure in autoclaves (cf. B., 1923, 1009 A).

L. A. COLES.

Rational utilisation of Rumanian lignite. I. Distillation at low temperatures. N. DANAILA

and I. BLUM (Bul. Chim., Soc., România Stiin., 1926, 28, 35—75).—The results of laboratory and small working scale tests on Rumanian lignites are described. The coke obtained by carbonisation at 500° in a revolving retort was friable and burnt easily but did not smoke. It did not contain too much ash and had a higher calorific value than the original lignite. The tar amounted to 6.8—9% on the dry basis, and the gas 50—130 cub. m. per ton of 3335—9095 cal. per cub. m. The utilisation of the coke and tar are deciding factors in the commercial feasibility or otherwise of the process, and depend upon the friability of the coke and the phenol content of the oils respectively. Up to the present low-temperature carbonisation cannot be applied with success unless the coke from inferior lignite is gasified or used to make good-quality briquettes.

R. A. A. TAYLOR.

Gas-washing. G. WEISSENBERGER (Gas- u. Wasserf., 1926, 69, 493—496, 528—531, 549—554; cf. B., 1925, 388, 697).—Tetralin has theoretical advantages over creosote or lignite tar oil as a benzol wash-oil and has now been tested on the working scale at a coke-oven installation and at a small gasworks. Although tetralin has a lower boiling point than ordinary wash-oil, with a suitable debenzolising column a very good separation between benzol and tetralin is obtained. Details given show only about 2% of the latter in the crude benzol, which is of low gravity and low in unsaturated hydrocarbons. The used tetralin tests are similarly good. The use of tetralin in an unaltered gas-washing plant designed for wash-oil gives less striking results, but if the benzol produced is to be used as motor spirit a proportion of tetralin in it is unobjectionable. It is preferable, however, to use a tall washer of small cross-section and a restricted flow of the circulated liquid when working with tetralin. Such a washer works badly with creosote wash-oil. Tetralin is less rapidly affected by tarry impurities than creosote and owing to the considerable increase in the solubility of naphthalene in tetralin with rise of temperature it is a very suitable agent for removing naphthalene from gas. Although pure tetralin has d 0.9709 this figure rises little with addition of naphthalene etc. in solution and no difficulty in separation should occur if direct cooling is used. It appears to have no tendency to form emulsions with water. The loss of tetralin through vaporisation with the gas was found over a period of 1 month to average 1.3 g. per cub. m. of gas. It is claimed that the use of tetralin protects gas mains against corrosion and removes naphthalene deposits.

C. IRWIN.

Extraction of oil shales and lignites with tetralin. E. BERL and W. SCHMID (Brennstoff-Chem., 1926, 7, 149—153, 181—185).—Tetralin under pressure (e.g., 14 atm.) and at a temperature of 200—270° extracts more from oil shales and lignites than does benzene under similar conditions. The material extracted from a sample of Cassel lignite, when further fractionally extracted with various

solvents, yielded 11.2% of plastic waxes, 40.5% of resin, 20.8% of pitch, and 12.3% of acidic compounds (acids and phenols). W. T. K. BRAUNHOLTZ.

Chemical constitution of a fraction of Peruvian petroleum boiling between 150° and 350°. W. F. SEYER and J. L. HUGGETT (Trans. Roy. Soc. Canada, 1926, [iii], 20, III, 193—199).—A sample of crude oil from the Lobitos Oilfield in Peru had d_{20}^{20} 0.834, and contained 0.10% of sulphur; 75% distilled between 65° and 370°. The hydrocarbons $C_{10}H_{20}$, $C_{11}H_{22}$, $C_{12}H_{24}$, $C_{13}H_{26}$, $C_{16}H_{32}$, $C_{17}H_{34}$, $C_{18}H_{36}$, $C_{13}H_{24}$, $C_{14}H_{26}$, $C_{15}H_{28}$, and $C_{16}H_{30}$ were identified in the fraction investigated. The hydrocarbons containing 14 or more carbon atoms were optically active. The values of d_{20}^{20} and n_D^{20} of the hydrocarbons increased somewhat irregularly with increasing molecular weight from 0.788 to 0.854, and from 1.435 to 1.469, respectively. The molecular volumes point to a ring structure with side chains. Some evidence was also obtained of the presence of two lower members of the C_nH_{2n} series, C_8H_{16} and C_9H_{18} , having a pronounced odour of turpentine. The optical activity displayed by some of these hydrocarbons, as well as the terpene odour of the two unidentified oils, favours the plant-theory of the origin of petroleum. J. S. CARTER.

Relation between fuel deposition temperature and equilibrium boiling point. W. A. WHATMOUGH (Ind. Eng. Chem., 1926, 18, 609—612).—Determination of fog points of combustible mixtures in internal-combustion engines (*i.e.*, the temperature at which visible droplets of liquid form in a flowing fuel-air mixture) gave results indicating that the equilibrium b.p. of a motor fuel is a measure of its volatility, and the principal factor in its valuation. The temperature of the first 10—20 c.c. of Engler distillate indicates the degree of ease of starting. The equilibrium b.p. determines the necessary induction pipe and mixture temperatures. In the experiments the fuel was evaporated in a flash-type balanced pressure boiler of a capacity only sufficient for a few revolutions of the engine. Fog points were determined, and the relation of these to equilibrium b.p. temperatures is shown by table and graph. The linear relationship between fog points and equilibrium b.p. agrees with Barnard and Wilson's figures for light fuels (*cf.* B., 1925, 487), but not for heavy fuels. The 85% boiling point of an Engler distillation does not represent accurately the equilibrium b.p. of all mixed motor fuels. H. MOORE.

Life of fuller's earth used in the vapour-phase treatment of cracked distillates. M. R. MANDELBAUM and P. S. NISSON (Ind. Eng. Chem., 1926, 18, 564—566).—When clays used for decolorising oils by percolation are revived by ignition their value becomes less with each re-burning, but the value of fuller's earth for vapour-phase refining remains undiminished after burning. The efficiency of fuller's earth in vapour-phase refining is practically the same for any cracked distillate. Better yields are obtained in large-scale working than in the laboratory. To determine the efficiency of the

original and re-burned earths, colour stability was taken as a decisive test. Gummy tests were found to be uncertain and not to give reproducible results, and the methylene-blue test was erratic in its results with fuller's earth. The turpentine temperature reaction (*cf.* Venable, B., 1923, 424 A) is much affected by the presence of moisture. The clay was reburned in a gas-fired muffle furnace, being frequently stirred, and was found in some cases to be more efficient than the unused clay. Relatively low temperatures of ignition, not exceeding 550°, were found to give the best results. H. MOORE.

Method of measuring viscosity at higher temperatures. H. M. MERCER (J.S.C.I., 1926, 45, 203—205 *tr.*).—A new method suitable for oils, molten waxes, tars, etc., at temperatures above 100° in which a small quantity of the oil or other material at a definite temperature is drawn into a capillary and its weight determined is described in detail. Values obtained for representative oils and paraffin wax are represented graphically and a few values for pitch are given.

Naphthenic acids. XI. Naphthenic acids derived from petroleum before acid treatment. Y. TANAKA and S. NAGAI (J. Soc. Chem. Ind. Japan, 1926, 29, 115—121; J. Fac. Eng. Tokyo, 1926, 16, 183—188).—Kerosene distillate from Nishiyama crude petroleum before refining with acid was treated with caustic soda and a mixture of crude petroleum acids was obtained from the solution by acidification. This was distilled under 9 mm. pressure and the distillate converted into potassium soaps from which a mixture of crude naphthenic acids was obtained. When the crude naphthenic acids were distilled under 9 mm. pressure a considerable amount of phenolic compounds was obtained from the first fractions boiling below 150°. The crude naphthenic acids were purified by means of their methyl esters. The naphthenic acids derived directly from the kerosene are practically indistinguishable from those obtained from the waste lye produced in the ordinary refining of the same kerosene (*cf.* B., 1926, 37). In the usual chemical treatment of petroleum, therefore, sulphuric acid does not act chemically on the naphthenic acids but on the phenolic compounds, most of which are removed. K. KASHIMA.

See also A., July, 668, Heats of combustion of cyclic hydrocarbons (KONOVALOV). 669. Temperature of the acetylene flame (HEINRICH). 673. Adsorption of gases by activated charcoal at very low pressures (ROWE). 689. Explosive reactions in gaseous media (GARNER); Uniform movement during propagation of flame (PAYMAN and WHEELER); Ignition-point of gases (DIXON, HARWOOD, and HIGGINS). 690. Ionisation and gaseous explosions (LIND); Ignition of gases by sudden compression (TIZARD and PYE). 693. Radiation in gaseous explosions (DAVID). 706. Copper covers for calorimeter jackets (WHITE).

Ammonium sulphate manufacture. HAILSTONE.—See VII.

Pyrogenic dehydration of fusel oil. GUIA and THUMIGER.—See XVIII.

PATENTS.

Apparatus for drying or dehydrating and distilling. W. DONALD (E.P. 252,858, 2.5.25).—Material to be dried or distilled is heated in a horizontal autoclave having end tube-plates, smoke-boxes at each end, and firing chambers between the smoke-boxes and the autoclave. Fire-tubes extend longitudinally through the autoclave from each firing chamber, passing through the opposed firing chamber, and terminating in the corresponding fire-box. Gas or oil is used as fuel. The material is supported inside the autoclave, out of contact with the fire-tubes, either on bogies or in the interior of a rotating perforated drum. In one form of the apparatus the autoclave and rotating drum are inclined, and means are provided for feeding the material into the upper end and discharging it from the lower end. Spiral worms inside the fire-tubes cause the products of combustion to take a tortuous path. The worms can be rotated, and so serve also to remove carbonaceous deposits from the tubes. In drying peat the pressure is allowed to rise initially to $2\frac{1}{2}$ atm. above atmospheric pressure, then partially released and kept at $\frac{3}{4}$ atm. above atmospheric pressure for half the remaining period of heating, drying finally being completed at atmospheric pressure. A. B. MANNING.

Retort for carbonisation of shale, lignite, coal, etc. E. L. SCHULTZ (E.P. 252,422, 27.1.25).—An annular vertical retort is heated from a central flue. Between the outer and inner walls of the retort is a casing provided with louvres arranged at a slight angle so that the material to be carbonised, entering from above, is fed in a layer against the inner heated wall of the retort. The casing with the louvres is moved up and down by means of an eccentric and so keeps the layer of material in motion. The gaseous products of carbonisation pass through apertures in the casing into the cooler outer space of the retort and thence to off-take pipes. The spent material is discharged intermittently at the lower end of the retort, discharge taking place automatically on the downward movement of the louvres. A. B. MANNING.

Gas producers. H. T. WHITE, D. J. SMITH, and R. F. CLAYTON (E.P. 252,862, 5 and 7.7.25).—The producer consists of two concentric chambers, air passing up through the outer and down through the inner one. The producer-gas passes through a jacket surrounding these chambers and a cylindrical fuel hopper superposed on them. A second jacket is provided for preheating the air, which then, on its way to the producer, passes through and preheats the fuel in the hopper; and a third, outermost jacket is provided for further preheating the air by means of the engine exhaust gases. By-pass inlets for either air or steam or both may also be provided. R. A. A. TAYLOR.

[Production of gas containing] hydrogen for the hydrogenation of naphthalene etc. E. A.

PRUDHOMME (F.P. 603,639, 19.12.24).—Water-gas or a similar gas is passed over calcium hydroxide. L. A. COLES.

Gas producer for the distillation of fine-grained or dusty fuel. SIEGENER MASCHINENBAU A.-G., and A. MENZEL (G.P. 427,038, 15.2.24).—The fuel passes downwards over a series of inclined plates heated by flues connected with the flue system in the walls of the furnace, and the issuing gases from the distillation are passed through this flue system, so as to utilise their heat for distilling the fuel without bringing them into direct contact with it. A. R. POWELL.

Distillation of lignite and recovery of the volatile sulphur. F. SEIDENSCHNUR (G.P. 427,323, 31.8.20).—Lignite is treated in a continuously working retort with superheated steam and the issuing vapours are cooled to such a temperature that only the tar condenses. Part of the remaining gases is passed through a superheater and returned to the circuit, whilst the remainder is further cooled to remove most of the water, and sulphur or sulphur dioxide is recovered from the rest of the gas. The quantity of the steam used and its temperature are so regulated that a highly viscous tar containing a large proportion of undecomposed bitumen is obtained together with a gas sufficiently rich in sulphur to allow of its economic recovery and the return of sufficient steam and gas to the circuit without reducing the amount of sulphur recovered. A. R. POWELL.

Washing or scrubbing coal or like gas. T. V. BLAKE (E.P. 252,867, 12.5.25).—A horizontal chamber is divided into sections by means of vertical hollow dividing plates and is partly filled with the scrubbing liquid. The gas passes out of one section through apertures in one side of the dividing wall just above the level of the liquid and into the next section through apertures on the other side of the hollow wall at the top. The scrubbing liquid is sprayed upwards from a pipe in the centre of the section just above the level of the liquid, and it is the film or spray so formed which scrubs the gas in its passage through the section. R. A. A. TAYLOR.

Gasification of [heavy] liquid fuels, for use in internal-combustion engines. A. V. DIAZ (E.P. 244,423, 24.7.25. Conv., 9.12.24).—The fuel passes through a vaporising coil and a vessel containing successive layers of coke and iron filings, heated by exhaust gases from the engine. Air is drawn through the coke by the suction of the engine, to maintain it at red heat and burn away deposited carbon. L. A. COLES.

Production of light oils suitable for use as fuel for internal-combustion engines from [A] water-gas or similar gases, or [B] liquid and solid organic bodies. M. BRUTZKUS (E.P. 252,786-7, 5.3.25).—(A) Water-gas or similar gas, alone or mixed with hydrogen, is compressed in an apparatus resembling a Diesel engine, which may contain a catalyst, and, during the compression stroke,

the gas is cooled and the concentration of the initial gas is increased by forcing in more of it by means of a pump operating at a higher pressure than that inside the apparatus. The products pass into a cooler and liquefier, the residual gas being returned to the process. (B) Hydrocarbon oils are cracked and simultaneously hydrogenated by compression with hydrogen or gases containing it, *e.g.*, water-gas, in the presence of catalysts, in an apparatus resembling a Diesel engine, the pressure, temperature, and hydrogen concentration being varied in directions opposite to those provoked by the desired reaction. Polymerisation of the products is prevented by effecting the cracking process during the expansion stroke. Light oils can also be produced in a similar manner from sugar syrup or from finely-divided hay, wood, or coal (cf. E.P. 149,915, 155,776, and 217,747; B., 1922, 87 A, 736 A; 1924, 698).

L. A. COLES.

Purification of used lubricating oil. AKTIEBOLAGET SEPARATOR (F.P. 602,304, 20.8.25).—The oil is heated and centrifuged to remove the heavier solid particles. A flocculating agent, such as trisodium phosphate, is then added to precipitate the colloidal materials present and the precipitate is removed by centrifuging. Finally, the oil is freed from volatile impurities by distillation.

A. R. POWELL.

Emulsification of tar, bitumen, creosote, petroleum, heavy oils and the like. W. E. BILLINGHAME (E.P. 252,449, 27.11.24).—Concentrated emulsions containing relatively small quantities of the material are used as emulsifying agents in the production of the desired emulsions. For example, an emulsion containing 1 pt. of neutral resin compound obtained by treating liquid resin with alkali, 1 pt. of tar, and 2 pts. of a solution of soluble casein or other suitable protein, is used for emulsifying 10 pts. of tar with 9 pts. of water. The emulsion may be thickened and stabilised by the subsequent addition of 10–50% of china clay. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 13,586 of 1912, 13,151 of 1905, and 14,391 of 1885.)

L. A. COLES.

Preparation of Trinidad pitch lake asphalt and the like. J. A. W. PINE (E.P. 252,802, 7.3.25).—The crude asphalt is pulverised, washed if necessary to remove salt and earthy material, compressed into blocks, and air-dried.

L. A. COLES.

Treatment of oils and hydrocarbons. [Production of blown oil asphalts.] J. S. MILLER, JUN., Assr. to BARBER ASPHALT Co. (U.S.P. 1,586,376, 25.5.26. Appl., 23.9.20).—Blown oil asphalts of any desired consistency are produced without distillation by spraying heated oil into a chamber in which it is brought in contact with a gaseous oxidising and condensing agent, the temperature and the proportions of oil and oxygen being regulated to prevent ignition and to minimise vapourisation of the oil.

L. A. COLES.

Rotary distillation retort. H. NIELSEN (U.S.P. 1,589,417, 22.6.26. Appl., 15.2.21. Conv., 14.1.21).—See E.P. 178,537; B., 1922, 456 A.

Destructive distillation of wood. J. S. MORGAN, Assr. to THERMAL INDUSTRIAL AND CHEMICAL (T.I.C.) RESEARCH Co., LTD. (U.S.P. 1,590,901, 29.6.26. Appl., 1.10.21. Conv., 2.11.20).—See E.P. 176,438; B., 1922, 315 A.

Cleaning blast-furnace gases. F. HÄRING, Assr. to FREYN ENGINEERING Co. (U.S.P. 1,589,803, 22.6.26. Appl., 26.8.21. Conv., 11.9.14).—See E.P. 17,081 of 1915; B., 1916, 958.

Burner apparatus for combustion of liquid, gaseous, or pulverised fuels. A. L. MOND. From E. H. PEABODY (E.P. 253,684, 12.5.25).

[Gas] retort settings. GIBBONS BROS., LTD., and E. MASTERS (E.P. 253,778, 21.10.25).

Coke extractors for vertical retorts. F. J. and E. WEST, and WEST'S GAS IMPROVEMENT Co., LTD. (E.P. 254,106, 30.6.25).

See also pages 649, Centrifugal liquid purifiers (E.P. 252,421); Drying pulverised fuel (E.P. 252,835); Recovering sulphur from gas-purifying material (G.P. 426,385). 650, Regenerating decolorising materials (G.P. 427,805). 664, Phosphoric acid and generator gas (E.P. 242,650 and 248,332).

III.—TAR AND TAR PRODUCTS.

Tar from low-temperature carbonisation of coal. E. PARRISH and F. M. ROWE (J.S.C.I., 1926, 45, 99–106 T).—A low-temperature tar made under standard conditions was examined by extraction methods. The tar was obtained by the carbonisation of Pooley Hall coal at 600° in vertical retorts. It contained less than 1% of material insoluble in the usual organic solvents, except in the case of light petroleum, which precipitated 10.8% of a black pitch. By distillation, a number of pitches were obtained, which showed a gradation in their properties, according to the temperature to which distillation was carried. During the separation of the tar by reagents, small amounts of solids were precipitated, in particular on dilution of the oil soluble in alkali with ether. Small amounts of solid carboxylic acids were found. The phenols, consisting mainly of homologues of phenol, contained 28% of high-boiling material insoluble in light petroleum, which resembled the "rhetinols" found by Edwards (J.S.C.I., 1924, 143 T, 150 T). Like these, it was converted by oxidation into a powder similar to that previously separated from the phenols. Phenol itself was present to an amount of 0.7% of the tar, an abnormally high figure, not due, however, to overheating of the tar. Pyrocatechol was identified in small amounts. The bases were almost entirely secondary and tertiary compounds of high boiling point. The neutral oil contained 12.8% of naphthenes and paraffins, including 3.75% of solid paraffins. The neutral oil of b.p. up to 170° was much more

saturated in character than the higher fractions. Benzene was absent, but higher aromatic hydrocarbons were obtained as picrates, including naphthalene amounting to 0.1% of the tar. Sulphur compounds were present in all portions of the tar.

Measuring viscosity at higher temperatures. MERCER.—See II.

PATENT.

Emulsification of tar etc. (E.P. 252,449).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Dyes from nitrosodialkylanilines, safranine, and Meldola Blue. A. COBENZL (Chem.-Ztg., 1926, 50, 494—495).—Whilst nitrosodimethylaniline forms a good crystalline hydrochloride, nitrosodiethylaniline and the safranines derived from it are best isolated as their double zinc salts. Heated in aqueous or acetic acid solution until the last trace of yellow colour has disappeared, nitrosodialkylanilines yield fast, water-soluble, grey cotton dyes, whilst by heating them with zinc oxide, zinc chloride, and aniline salts in dilute acid solution and oxidation with sodium dichromate, violet and blue safranines are obtained. *p*-Aminodiphenylamine directly oxidised with various aniline bases in aqueous solution gives economic yields (doubled if concentrated acetic acid solution is used) of rosanilines. If *p*-aminomethylidiphenylamine is used redder safranines are obtained, whilst the colour is bluer if *p*-aminotolyldiphenylamine is substituted. In the preparation of Meldola Blue, Naphthol Blue, etc., more satisfactory results and purer products are obtained if zinc chloride is added, or if the double zinc salt of the nitrosodialkylaniline is used directly, the dyes having the composition $(C_{14}H_{16}ON_2Cl)_2 \cdot ZnCl_2$ and $(C_{20}H_{20}ON_2Cl)_2 \cdot ZnCl_2$ respectively. Treatment of these with ammonia or aniline bases yields valuable blue dyes. The sulphonation of Naphthol Blue to make it suitable for dyeing wool may be effected either by direct sulphonation, or by using naphthol-sulphonic acid in place of β -naphthol during the preparation. Details for the preparation of all these dyes are given. J. W. BAKER.

Standardisation of Agalma Black 10 B. W. D. APPEL, W. R. BRODE, and I. M. WELCH (Ind. Eng. Chem., 1926, 18, 627—629).—The desirability of standardising commercial dyestuffs is emphasised, and three methods for estimating the strength of commercial Agalma Black 10 B are compared, namely titration with titanous chloride, spectrophotometric analysis, and comparative dyeings. A sample of the dye prepared in the laboratory and containing 99.2% of anhydrous dye was used as a standard. The spectrophotometric measurements were made in a Keuffel and Esser colour analyser and the negative logarithms of the spectral transmission of a 5 cm. layer of the dye solution at selected wave-lengths relative to that of the solvent in a similar cell plotted against the wave-length. The area between the curve thus obtained and the wave-length axis (within

the limits λ 540—670) is proportional to the amount of dye in solution. The percentage purity was also calculated from the value of the curve at λ 620, the wave-length of maximum absorption; the average of the two results was taken as the spectrophotometric value. The concentration of the dye was about 3.3 mg. of pure dye per litre (on the basis of the titanous chloride titration in an aqueous buffer solution containing 0.01 g.-mol. each of sodium acetate and acetic acid; p_H 5). The comparative dyeing tests (0.4% of pure dye on white wool flock) were based on the results of the titanous chloride and spectrophotometric analyses, additional dyeings being made where necessary; the relative strengths of the dyes were judged visually, a good commercial sample proving the most satisfactory standard. In the main the titanous chloride and spectrophotometric results agreed with the dyeing tests; in some cases where reducing substances other than the dye were present the titanous chloride figures were high; abnormal results obtained with a few commercial dyes appeared to be due to the presence of colouring matter other than true Agalma Black. In general, whilst neither titration with titanous chloride nor spectrophotometric analysis is sufficient alone, a combination of the two methods provides a fairly satisfactory means of evaluating Agalma Black 10 B, since where the two methods agree, the result is that obtained by the dyeing test. A standard of 60% purity is suggested. D. J. NORMAN.

Separation of the constituents of commercial xylydine. G. T. MORGAN and W. J. HICKINBOTTOM (J.S.C.I., 1926, 45, 221—222 T).—4-*o*-Xylydine forms a picrate sparingly soluble in alcohol, and this property has been utilised in devising a method for the separation of the amine from commercial mixtures of xylydines. The crude mixture is freed from *m*-xylydine by treatment with acetic acid; *p*-xylydine is removed as hydrochloride. The residual amines when treated in alcoholic solution with picric acid, insufficient in quantity to combine with all the bases present, deposit 4-*amino-o*-xylene picrate in a crude state. Crystallisation from alcohol yields the pure picrate, from which pure 4-*amino-o*-xylene may be isolated by treatment with aqueous ammonia. From the residue 2-*amino-o*-xylene is separated by fractional crystallisation of the sulphates, and conversion of the more soluble fractions into acetyl compounds. Hydrolysis of the crude acetyl derivatives with hydrochloric acid liberates the isomeric amines, leaving unhydrolysed 2-acetamido-*m*-xylene in a pure state.

Synthesis of 2-*p*-aminophenyl-5-methylbenzthiazole etc.: isomerides of dehydrothio-*p*-toluidine and of Chloramine Yellow. BOGERT and ALLEN.—See A., July, 743.

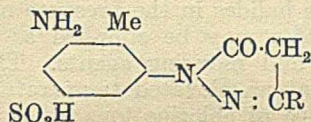
PATENTS.

Manufacture of yellow azo dyes. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 252,617, 5.11.25).—Yellow azo dyes possessing excellent affinity for wool, silk, paper, and leather,

especially chrome-tanned leather, are obtained by nitrating the monoazo-dyes obtained from naphthylaminemonosulphonic acids and diphenylamine, or the *N*-nitroso-derivatives of such dyes. Thus the monoazo-dye from 22.3 pts. of a technical mixture of β -naphthylaminemonosulphonic acids and 16.9 pts. of diphenylamine is converted into the nitrosoamine with 8.5 pts. of sodium nitrite. The nitrosoamine is nitrated at 30–35° with 46 pts. of 65% nitric acid and 37 pts. of 98% sulphuric acid. The new dyes dye silk in a slightly acid boiled-off liquor bath or in a bath containing soap and Glauber's salt.

R. BRIGHTMAN.

Manufacture of diazotisable azo-dyestuffs and intermediate products CHEM. WORKS, FORMERLY SANDOZ, and M. BÖNIGER (E.P. 252,957, 28.9.25).—By condensation of 6-nitro-2-tolylhydrazine-4-sulphonic acid with ethyl acetoacetate or ethyl oxaloacetate, followed by hydrolysis with hydrochloric acid and reduction with iron borings, new pyrazolone derivatives of the general formula,



where R=Me or CO₂H, are obtained. The symmetrical disazo dyes from a *p*-diamine and 2 mols. of these pyrazolones have no technical value. Valuable substantive dyes which can be diazotised on the fibre and developed, e.g., with β -naphthol, *m*-diamines, or phenylmethylpyrazolone, giving shades varying from orange to red and dark-brown, fast to washing, and discharged by formaldehyde-hyposulphite, are, however, obtained when these pyrazolones are combined with the intermediate compound from 1 mol. of a tetrazotised diaryl-*p*-diamine and 1 mol. of an aryl-*o*-carboxylic acid; or alternatively, 1 mol. of the diaryl tetrazo-compound is combined with 1 mol. of the new pyrazolones and the intermediate compound produced is coupled with 1 mol. of an azo component. The direct dyeings can be made fast by after-treatment with *p*-nitrodiazobenzene or formaldehyde. Thus the intermediate compound from tetrazotised benzidine and *o*-cresotic acid yields with 5'-sulpho-3'-amino-2'-methyl-1-phenyl-3-methyl-5-pyrazolone (greyish powder, which reacts with 2 mols. of sodium nitrite, one mol. of diazo-compounds, and 3 equivalents of sodium hydroxide) a bright orange dye, developed on the fibre by diazotisation and coupling with *m*-tolylenediamine to a browner shade, with β -naphthol to bright reddish-orange shades. Similarly the intermediate compound from dianisidine and salicylic acid with 5'-sulpho-3'-amino-2'-methyl-1-phenyl-5-pyrazolone-3-carboxylic acid (grey powder, reacting with 2 mols. of nitrite, 1 mol. of diazo-compounds and 2 mols. of sodium hydroxide) yields a red dye, developed on the fibre to bluish-red shades by coupling with β -naphthol. The intermediate compound from tolidine and 5'-sulpho-3'-nitro-2'-methyl-1-phenyl-5-pyrazolone-3-carboxylic acid with 2-amino-8-naphthol-6-

sulphonic acid yields a violet solution, reduced by sodium sulphide to a violet-brown dye, the nitrodisazo-dye being slightly yellow. Benzidine and 5'-sulpho-3'-amino-2'-methyl-1-phenyl-5-pyrazolone-3-carboxylic acid give an orange intermediate compound which couples with the monoazo-dye from *p*-phenylenediaminesulphonic acid and *m*-tolylenediamine to give a bright red-brown trisazo dye.

R. BRIGHTMAN.

Manufacture of monoazo dyes. W. CARPMAEL. From FARBENTFABR VORM. F. BAYER & Co. (E.P. 253,457, 29.6.25).—Monoazo dyes giving pure, even shades on wool are obtained by coupling diazotised aromatic amines or their substitution products with ω -aminoalkyl- β -naphthylamines or their derivatives (cf. U.S.P. 1,543,569 and E.P. 249,717; B., 1925, 752; 1926, 433). The new dyes give good dyeings on cellulose acetate, provided they contain only one sulphonic or carboxylic group, which must be present in the diazo-component. Thus the dye from 2:4-dinitroaniline-6-sulphonic acid and ω -aminoethyl- β -naphthylamine on treatment with acetic anhydride at 65–75° gives a product dyeing acetate silk a blue shade and wool a greenish blue. 4-Nitroaniline-2-sulphonic acid and the *monoacetyl* derivative of ω -aminoethyl- β -naphthylamine, m.p. 101–102°, give a product dyeing acetate silk a red-violet and wool a violet shade. Similarly 4-nitroaniline-2-sulphonic acid with the acetyl derivative of ω -aminoethyl-2-naphthylamine-7-sulphonic acid, or with ω -aminoethyl- β -naphthylamine-7-sulphonic acid, followed by acetylation with acetic anhydride yields a dark powder giving violet shades on wool. The acetic anhydride may be replaced by other acylating agents such as chloroacetyl chloride or toluenesulphonyl chloride, or the amino-group may be substituted in the ω -aminoethyl-2-naphthylamine-7-sulphonic acid or in the finished dye, for instance, by heating with chloroacetic acid in the presence of chalk. 2-Nitro-4-toluidine diazotised and coupled in weak mineral acid with ω -aminoethyl-2-amino-8-naphthol-6-sulphonic acid gives on acetylation a red-brown wool dye of excellent fastness to light and fulling. Similarly ω -aminoethyl-2-amino-8-naphthol-3:6-disulphonic acid when coupled in mineral acid solution with diazotised *p*-nitroaniline or *m*-nitroaniline or their homologues or derivatives followed by acetylation of the product gives red-brown wool dyes of good fastness to light and fulling. The dye from *p*-nitroaniline is a brown-black powder, that from *p*-aminoethylacetanilide gives a deep brown shade, whilst the corresponding dye from 5-nitro-2-anisidine in sodium bicarbonate solution, a black powder, gives beautiful olive-brown shades on wool. The corresponding dye from 4-nitro-2-anisidine gives a red-brown shade on wool. The 5-nitro-2-anisidine may be replaced by *o*-nitroaniline or its homologues and derivatives without markedly affecting the shade of the dye. The ω -aminoethyl-2-amino-8-naphthol-3:6-disulphonic acid may be acylated first and then coupled. 5-Nitro-2-aminobenzoic acid and ω -amino-

ethyl- β -naphthylamine give crystalline products dyeing acetate silk in bluish-pink shades.

R. BRIGHTMAN.

Blue monoazo dyestuffs for wool or silk. A. H. NEY, Assr. to T. HAEBLER (U.S.P. 1,586,693, 1.6.26. Appl., 13.11.24; cf. U.S.P. 874,967-8, B., 1908, 116, 117).—Diazotised *p*-aminodiphenylamine-*o*-sulphonic acid is coupled in alkaline solution with 1-acetamido-8-naphthol-3 : 6-disulphonic acid (acetyl-H-acid) to yield a dyestuff which dyes wool or silk in an acid bath brilliant reddish-blue shades which do not change in artificial light and are very fast to light, washing, and carbonising, and fast to fulling, milling, and alkali. The products from *p*-amino-*o*'-methyldiphenylamine-*o*-sulphonic acid and *p*-amino-*o*'-methoxydiphenylamine-*o*-sulphonic acid (formed by condensing *p*-chloronitrobenzene-*m*-sulphonic acid and *o*-anisidine and reducing the nitro-group) give purplish-blue and brilliant greenish-blue shades, respectively.

T. S. WHEELER.

Yellow monoazo dyestuffs. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of A. L. LASKA and F. WEBER (U.S.P. 1,587,005, 1.6.26. Appl., 10.9.25. Conv., 20.9.24).—Diazotised monoamines of the diphenylamine series are coupled with derivatives of 1-sulphoaryl-5-pyrazolones, such as 1-sulphoaryl-3-methyl-5-pyrazolones or 1-sulphoaryl-5-pyrazolone-3-carboxylic acids to yield dyes which dye wool yellow shades especially fast to milling. For example, *pp'*-dichloro-*o*- or -*m*-aminodiphenyl is diazotised and coupled with 1-(*o*-chloro-*p*-sulphophenyl)-3-methyl-5-pyrazolone.

T. S. WHEELER.

Bluish-green dyestuffs and colour lakes therefrom. O. STAEBLIN, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,587,435, 1.6.26. Appl., 18.12.24. Conv. 24.12.23; cf. G.P. 224,442; B., 1910, 1120).—A halogenated 1 : 5-naphthylenediaminesulphonic acid is treated with a solution of ferric chloride to give a product dyeing wool, but not cotton, greenish shades. The lakes from these dyes, e.g., the barium salts of the dyes, have a more bluish tint. Owing to their greater solubility the new dyes are more readily converted into lakes than the analogous dyes from the non-halogenated 1:2-naphthylenediaminesulphonic acids described in G.P. 224,442 (*loc. cit.*).

T. S. WHEELER.

Manufacture of anthraquinone derivatives. BRIT. DYESTUFFS CORP., LTD., W. H. PERKIN, A. W. FYFE, and M. MENDOZA (E.P. 252,922, 8.7.25).—Better yields of the *N*-methyl-*o*-sulphonic acid derivatives of 1:4- and 1:8-diaminoanthraquinone are obtained in the absence of a strong mineral acid, by the process described for diaminochryszin (E.P. 238,717, B., 1925, 840) than by the process of E.P. 23,968 of 1899 (B., 1900, 1101), although in the case of 1:5-diaminoanthraquinone good results are only obtained in the presence of strong mineral acid. Little more than the theoretical amount of formaldehyde-bisulphite is required if the base is prepared in a finely divided condition by solution in sulphuric acid and dilution with

water, the finely divided diamine being then washed free from acid with water. Thus 1 g.-mol. of 1:4-diaminoanthraquinone is dissolved in 1600 g. of sulphuric acid and after agitation for 2 hrs. at 100° is cooled and diluted with 20 litres of ice-cold water. After decanting off most of the waste acid and washing the precipitate on the filter, first with water and then with 2% soda to slight alkalinity, the paste is incorporated with a mixture of 2.5 mols. of 40% sodium bisulphite and 2.5 mols. of 40% formaldehyde. The mass is diluted with 1500 c.c. of water and stirred under a reflux for 8 hrs. at 100°, when one litre of saturated salt solution is stirred in and the *o*-sulphonate filtered off after cooling. In the case of the 1:8-diaminoanthraquinone conversion is complete in about 12 hrs.

R. BRIGHTMAN.

Manufacture of anthraquinone derivatives. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 253,386, 12.8.25).—Diazotised 1-aminoanthraquinone-2-aldehyde or its halogen derivatives, either in the solid state or in solution, are treated with cuprous halides in the presence of water. The cuprous halides may be supplied as a mixture of cupric salts and reducing agents. The reaction is carried out at an elevated temperature, preferably in the absence of chlorine ions other than those of cuprous halides. Thus 10 pts. of 1-aminoanthraquinone-2-aldehyde diazotised in sulphuric acid and diluted with ice-water are run at 90–95° into the stirred solution of cuprous chloride obtained by boiling 17 pts. of copper sulphate, 8 pts. of common salt, and 7 pts. of sodium bisulphite in 1000 pts. of water until decolorised and no more sulphur dioxide is developed. The precipitated dianthraquinonyl-dialdehyde is filtered off hot and purified by extraction, e.g., with acetone, or by crystallisation from high-boiling solvents. From 4-chloro-1-aminoanthraquinone-2-aldehyde treated in a similar manner 4:4'-dichloro-1:1'-dianthraquinonyl-2:2'-dialdehyde is obtained.

R. BRIGHTMAN.

Manufacture of benzanthrone derivatives. BRIT. ALIZARINE CO., LTD., J. ANDERSON, and W. H. DAWSON (E.P. 253,163, 10.2.25).—Benzanthrone or its derivatives such as violanthrone and isoviolanthrone are chlorinated by solution in concentrated sulphuric acid, precipitation with water, and treatment of the fine suspension thus obtained with a soluble chlorate and hydrochloric acid in the cold. Thus 20 pts. of violanthrone are dissolved in 200 pts. of 98% sulphuric acid, and the solution is poured on to 300 pts. of ice. To this suspension 15 pts. of sodium chlorate and 103.5 pts. of concentrated hydrochloric acid (*d* 1.15) are added. After keeping for 4 hrs. the insoluble chloro-derivative is filtered off and washed. It gives bluer dyeings than the dichloro-derivative obtained by the use of sulphuryl chloride in nitrobenzene.

R. BRIGHTMAN.

Manufacture of monochloronitrobenzene. J. W. LIVINGSTON and L. P. KRYDES (U.S.P. 1,586,253, 25.5.26. Appl., 8.8.25).—1175 pts. of

finely-divided sodium nitrate, 1600 pts. of 93% sulphuric acid, and 1240 pts. of chlorobenzene are added together in the course of 5 hrs. to an agitated mixture of 1000 pts. of 81% sulphuric acid and 200 pts. of chlorobenzene, at 110–130°. After keeping for a further hour at this temperature the mixture is poured into water. Chloronitrobenzene is obtained in satisfactory yield.

T. S. WHEELER.

Manufacture of benzanthrone derivatives. A. J. RANSFORD. From L. CASSELLA & Co. (E.P. 254,021, 27.3.25).—See U.S.P. 1,565,229; B., 1926, 434.

Vat [dibenzanthrone] colouring matter. BADISCHE ANILIN- & SODA-FABR., Assees. of H. NERESHEIMER (U.S.P. 1,589,303, 15.6.26. Appl., 26.4.23. Conv., 16.8.22).—See E.P. 212,145; B., 1924, 369.

Vat dyestuff. W. ECKERT and H. GREUNE (U.S.P. 1,588,451, 15.5.26. Appl., 17.7.25. Conv., 21.7.24).—See E.P. 237,294; B., 1925, 840.

Azo dyestuffs containing chromium. SOC. OF CHEM. IND. IN BASLE, Assees. of M. ISLER and L. VON MECHEL (U.S.P. 1,588,458, 15.6.26. Appl., 1.12.25. Conv., 28.12.23).—See E.P. 226,797; B., 1925, 703.

Trisazo dyestuffs. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of A. L. LASKA and F. WEBER (U.S.P. 1,590,042, 22.6.26. Appl., 23.7.25. Conv., 15.11.24).—See G.P. 423,092; B., 1926, 312.

[Azo] dyestuffs containing metals. SOC. OF CHEM. IND. IN BASLE, Assees. of F. STRAUB and H. SCHNEIDER (U.S.P. 1,590,482, 29.6.26. Appl., 7.4.25. Conv., 20.4.24).—See E.P. 232,620; B., 1925, 954.

Manufacturing perylene compounds of quinone character. A. ZINKE, Assr. to F. BENSÄ (U.S.P. 1,590,661, 29.6.26. Appl., 7.4.25. Conv., 10.4.24).—See E.P. 232,264; B., 1925, 910.

Vat sulphide dyes. F. B. F. MUTH and A. SCHMELZER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,590,718, 29.6.26. Appl., 16.1.25. Conv., 24.1.24).—See E.P. 243,557; B., 1926, 121.

Gas containing hydrogen for hydrogenation of naphthalene (F.P. 603,639).—See II.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Amidation of cotton. P. KARRER and W. WEHRLI (Helv. Chim. Acta, 1926, 9, 591–597).—Cotton fibre treated with toluenesulphonyl chloride yields a toluenesulpho-derivative which contains 1 mol. of the sulphonyl group for every 9–10 mols. of $C_6H_{10}O_5$ residue and which cannot be dyed with acid dyes, and this on treatment with aqueous ammonia yields, slowly at ordinary temperature and more rapidly at 100°, an amidated fibre which contains 1 mol. NH_2 for every 9–11 mols. of $C_6H_{10}O_5$ residue,

ammonium toluenesulphonate separating during the reaction. The amidated fibre is readily dyed with acid dyes, and shows satisfactory results with regard to fastness, the best results being obtained with dyes of the after-chrome, pyrazolone, and anthraquinone acid groups, and less satisfactory with those of the azo and triphenylmethane groups. Similar results are obtained using methyl-, dimethyl-, and ethylamines, and to a lesser extent, with benzylamine, aniline, and α -naphthylamine, but the tertiary bases trimethylamine and pyridine give better results than does ammonia. Amidated products can also be obtained from the fibre previously treated with benzenesulphonyl and disulphonyl, naphthalenesulphonyl, and ethylsulphonyl chlorides, but not from cellulose acetate, benzoylated cotton, or nitrocellulose. The action of the acid dyes is due to salt formation with the amino-group, but in fibres of albuminous origin the nitrogen is present as acid amides and has no basic properties, and it is shown that fibre treated with acetyl- or benzoyl-sulphanilic acid chloride is not dyed with acid dyes, but if it is treated with *p*-nitrobenzoyl chloride followed by the reduction of the nitro-group to the amino-group with ammonium sulphide, it is then readily dyed in this manner.

J. W. BAKER.

Action of solutions of sodium sulphide on linen fibres. P. P. VICTOROV (Textilber., 1926, 7, 61–63, 444–445).—A comparison of the purifying action of boiling solutions of sodium sulphide and caustic soda on raw linen yarns containing 83.8% of pure cellulose. Raw linen yarns digested with boiling water and a solution containing 10% (calc. on the weight of yarn) of sodium sulphide suffered losses of 3.8% and 6.4% respectively. Solutions of caustic soda effect the removal of impurities (pectic and other incrusting substances) from raw linen more rapidly than solutions of sodium sulphide of equal concentration (calc. on the weight of linen), but solutions of caustic soda produce greater deterioration of linen, especially when used for prolonged periods at high temperatures. The lustre, handle, and whiteness of bleached linen yarns previously scoured with sodium sulphide are not inferior to those of similar yarns previously scoured with caustic soda. A. J. HALL.

Action of acids on artificial silks. K. WOLFGANG (Kunstseide, 1926, 8, 175–177).—The brittleness occasionally found in artificial silks is due to the effects of acids remaining in the silk from processes of manufacture. Samples of viscose, cuprammonium and nitrocellulose silk yarn of 120 denier impregnated so that they contained their own weight of various solutions of sulphuric, formic, acetic, and lactic acids, then dried for 1 hr. at 90°, and afterwards conditioned in air for 24 hrs. suffered losses of tensile strength exceeding 5% when the concentrations of the acid solutions were greater than 0.05%, 0.3%, 0.3%, and 1.5% respectively. A. J. HALL.

Increasing the wet strength of [cellulose] artificial silks. W. BRUCKHAUS (Kunstseide, 1926, 8, 115–116).—Artificial silk yarn or fabric is impregnated with a solution containing 2 kg. of alum,

2.5 kg. of 3% lactic acid, 10—12 kg. of 4% formaldehyde, and 10—35 litres of water, then hydro-extracted, dried at 60°, soaped in a solution containing 5—7 g. of Marseilles soap per litre, washed, scoured in a 0.3% solution of acetic acid, and dried at a low temperature. The treated silk has an increased wet strength, is more resistant to alkalis, and has a greater affinity for dyes. A. J. HALL.

Effect of heat upon cellulose. J. W. BAIN and G. M. CHUTE (Trans. Roy. Soc. Canada, 1926, [iii.], 20, III., 189—191; cf. B., 1925, 349).—The presence of dextrose in cotton which has been heated at 200° is definitely established. Other hexoses, pentoses, and methylpentoses are probably absent. Some formic acid and small amounts of acetic acid are formed during the heating. J. S. CARTER.

Ripening of viscose. R. O. HERZOG (Papier-Fabr., 1926, 24, Fest- u. Ausland-Heft, 94—97; cf. B., 1925, 6).—The chemical changes which characterise the process of "ripening" of viscose are accompanied by physical changes in the colloidal condition, which undergoes a slow transition from the sol to the gel state. These may be defined as a gradual coagulation through the formation of secondary particles from the original primary particles, a process which should conform to a bimolecular reaction. The degree of ripeness is determined empirically by Hottenroth's method by titration with ammonium chloride, and the quantity of ammonium chloride may be expressed in terms of a function of the time of ripening by the formula $at = b - q$, a and b being constants. The coagulation by ammonium chloride is determined by chemical changes in the xanthate, dehydration of the colloid particles, increase in size of the colloid particles, *i.e.*, relative lowering of concentration. In addition reaction equilibria between the electrolytes already present and those added, and adsorption reactions also exert an influence. A further physical change which accompanies the ripening is the decrease in extensibility or increase in the modulus of elasticity of the regenerated thread. Thus the extensibility may also be expressed empirically as a function of the ammonium chloride titration value, the determined results for the modulus of elasticity agreeing well with those calculated by the formula $1/\sqrt{D} = H - J\rho$, H and J being constants. Lastly, the ripening process is accompanied by an increase in viscosity of a peculiar nature (cf. *loc. cit.*) which involves the gradual formation of a structure in the solution, becoming increasingly more stable towards vigorous agitation. The optimum period for spinning occurs at a ripeness degree of 7, when this incipient gelation or structure is very easily destroyed but rapidly re-forms. This alteration is due to a change in the size or shape of the particles, presumably only the secondary particles, which probably assume a rodlet formation (series of micellæ). J. F. BRIGGS.

Chemistry of wood. VIII. Further studies of sapwood and heartwood. IX. Spring wood and summer wood. G. J. RITTER and L. C. FLECK (Ind. Eng. Chem., 1926, 18, 576—577, 608—609;

cf. B., 1923, 9 A, 1123 A).—Further analyses of sapwood, heartwood, spring wood, and summer wood are given for various American woods. In soft woods the water, ether, and alkali extracts are higher and the cellulose and lignin correspondingly lower in the heartwood than in the sapwood; white spruce, however, shows practically the same cellulose and lignin content in both bands of growth. The hardwoods may be divided into two groups, those with high extractives (water, ether, and alkali extracts) and low cellulose content in the sapwood, and those with high extractives and low cellulose in the heartwood. Spring wood shows a consistently higher content of lignin together with a lower content of cellulose (except in the case of loblolly pine and red alder) than does summer wood. No other outstanding differences were noted, but there is a tendency for spring wood to contain more extractives and more pentosans both in the wood and in the cellulose isolated therefrom. D. J. NORMAN.

Fluorescence of sulphite-cellulose. C. W. LEUPOLD (Papier-Fabr., 1926, 24, 397—398).—The fluorescence of sulphite-cellulose waste lye is due to colloidal sulphur, and it is therefore possible that the fluorescence of sulphite-cellulose is due to sulphur compounds. This view is supported by the fact that the fluorescence is destroyed by treatment with chlorine or iodine, which would decompose any thio-acids or polythionic acids. A. GEAKE.

Freeness of sulphite pulp. D. S. DAVIS (Ind. Eng. Chem., 1926, 18, 631—633).—The operation of the Williams freeness tester (Pulp and Paper Mag., 1925, 23, 443) is described. Graphs and equations are given for converting freeness numbers for one orifice into freeness numbers for another of a different size; also for converting the freeness value at any known concentration between 0.3 and 0.5% into freeness at standard concentration (0.4% of bone-dry pulp). The observed freeness of mixed stock does not agree with the freeness value calculated from the proportions and freeness of the components of the mixture; a graphic method is outlined for computing the one value from the other. The mathematical treatment of the subject is of general application. D. J. NORMAN.

See also A., July, 673, **Adsorption of alkali hydroxide by cellulose** (LIEPATOV). 677, **Viscosity anomalies in cellulose sols** (VON NEUENSTEIN); **Structure of cellulose and its significance in chemical transformations** (HERZOG). 715, **Cryoscopic behaviour of crystalline cellulose acetates** (HESS and SCHULTZE).

Fermentation products from cellulose. SPEAKMAN.—See XVIII.

PATENTS.

Operating spinning machines for artificial silk. WERDOHLER PUMPENFABR. P. HILLEBRAND (E.P. 248,715, 26.1.26. Conv., 4.3.25).—In the ordinary type of artificial silk spinning machine, in which the liquid is fed to the spinnerets through a pressure-equalising chamber containing compressed

air, the liquid both enters and leaves the chamber at the bottom, with the result that the surface stratum of liquid in contact with the air remains unchanged and gradually hardens, thereby impairing the pressure-balancing functions of the device. This is obviated by introducing the spinning solution into the chamber at a point above the level of the liquid therein. D. J. NORMAN.

Nozzles for production of artificial filaments. C. L. WALKER (E.P. 253,209, 14.3 and 15.10.25).—Very fine tubes of platinum or platinum-iridium are sealed with glaze into holes in a porcelain nozzle, or are fused into a glass nozzle. Platinum rivets may be similarly sealed and then drilled with a fine hole. The tubes are formed from a rod through which a hole is bored and filled with a base metal; the rod may then be drawn out to the desired diameter and the base metal dissolved out. Tubes with bores of any desired shape may be similarly produced. A. GEAKE.

Manufacturing artificial silk and other products from nitrocellulose. E. BINDSCHEDLER and G. JUER, Assrs. to TUBIZE ARTIFICIAL SILK CO. OF AMERICA (U.S.P. 1,584,005, 11.5.26. Appl., 1.7.24).—Alcohol-ether solutions of nitrocellulose are spun into a coagulating bath comprising one or more metal halides dissolved in dilute alcohol. D. J. NORMAN.

Plastic compounds of acetylcellulose. PATHÉ CINEMA, ANC. ÉTABL. PATHÉ FRÈRES (E.P. 237,900, 22.7.25. Conv., 4.8.24).—Plastic compounds are prepared by incorporating with cellulose acetate a mixture of triphenyl phosphate and tricresyl phosphate with the assistance of a solvent. Suitable proportions are: 100 pts. of cellulose acetate, 15 of triphenyl phosphate, 12 of tricresyl phosphate. The use of such a mixture avoids the extrusion of crystals on the one hand and oily globules on the other. J. F. BRIGGS.

Reducing the viscosity of nitrocellulose. L. E. BRANCHEN, Assr. to EASTMAN KODAK CO. (U.S.P. 1,588,089, 8.6.26. Appl., 1.9.25).—Nitrocellulose is softened with a penetrant liquid and then treated with acid. A. GEAKE.

Extraction of cellulose. E. R. CLARK, Assr. to M. S. ERLANGER (U.S.P. 1,587,631, 8.6.26. Appl., 12.2.25).—Chlorine is supplied to water which is circulated by an external pump through cellulose-containing material. A. GEAKE.

Making pulp from [rice] hulls. J. F. and H. F. J. PUTTAERT (U.S.P. 1,588,335, 8.6.26. Appl., 12.2.20).—Rice hulls are boiled with dilute sodium hydroxide solution for 6–8 hrs. at a pressure of 20–80 lb. per sq. in., removed, washed, and “defiberated.” A. GEAKE.

Bleaching of paper pulp. R. B. WOLF (E.P. 251,205, 17.12.25. Conv., 28.7.25).—Air is directed on to the surface of the pulp during the bleaching operation, firstly to control the temperature of the

mass, thereby facilitating the bleaching of relatively thick stock, *i.e.*, containing more than 10% of fibre, and secondly to remove volatile decomposition products and thus accelerate the bleaching operation. A suitable apparatus consists of a vertical cylindrical tank provided with a central tube fitted internally with a screw conveyor for carrying the pulp to the top of the tube, where it is distributed over an apron and drops back into the tank. A scraper is provided at the bottom of the tank to ensure complete circulation of the pulp through the central tube. Jets of air play on the pulp as it passes in a thin layer over the apron; the amount of air introduced depends on its temperature and humidity and also on the degree of cooling it is desired to effect. For instance, in a tank with a capacity of 4 tons, with the materials entering at 30°, air should be introduced at 4000 ft. per min. through about 30 nozzles of approximately 3 in. diameter at their outer ends. D. J. NORMAN.

Sizing cellulose fibres [paper pulp]. J. A. DE CEW (U.S.P. 1,585,469, 18.5.26. Appl., 15.12.25).—The fibres are treated in the beater with aluminium sulphate, and a stream of size emulsion is added to the pulp as it is entering the Jordan engine (refiner). J. F. BRIGGS.

Manufacture of sand paper or like composite structures. MINNESOTA MINING AND MANUF. CO., Asses. of F. G. OKIE (E.P. 226,803, 9.12.24. Conv., 29.12.23; cf. E.P. 194,274 and 219,341; B., 1924, 905).—A suitable binder for flexible, waterproof sand-paper consists of turpentine, rosin, or other similar resin, and tung oil or other similar oil, *e.g.*, tung oil 53%, hardened rosin 12%, linseed oil 10%, drier 1%, and thinner 24%. The size to fix the abrasive is of similar composition to the binder, but with more thinner, and hence adheres strongly to it. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 211,203 and 209,872.) A. GEAKE.

Safety paper. A. I. CONE (U.S.P. 1,584,850, 18.5.26. Appl., 6.5.25).—The paper contains a mercurous salt. J. F. BRIGGS.

Apparatus for sizing, dyeing, and impregnating paper. B. KREMLER (G.P. 427,531, 8.11.23).—The paper passes between rollers the channels of which may be connected or disconnected with a circulating apparatus for the liquid. Uniform sizing is obtained with a minimal volume of liquid. A. GEAKE.

Making a new type of cellulose derivatives. L. LILIENFELD (U.S.P. 1,589,606, 22.6.26. Appl., 20.3.22. Conv., 2.4.21).—See E.P. 177,810; B., 1924, 11.

Making cellulose derivatives [ethers]. L. LILIENFELD (U.S.P. 1,589,607, 22.6.26. Appl., 20.3.22. Conv., 2.4.21).—See E.P. 177,809; B., 1924, 11.

Manufacture of coloured cellulose plastics and solutions thereof. BADISCHE ANILIN- & SODA-FABR., Asses. of C. IMMERHEISER, C. NEUBAUER,

and E. SCHARF (U.S.P. 1,589,700, 22.6.26. Appl., 29.11.24. Conv., 26.6.24).—See E.P. 247,288; B., 1926, 315.

Bleaching vegetable fibres (E.P. 251,014).—See VI.

Recovering calcium carbonate from caustic liquors (U.S.P. 1,587,679).—See VII.

Gelatinising nitrocellulose (E.P. 252,978).—See XXII.

Bactericide and fungicide from sulphite-cellulose waste lye (U.S.P. 1,585,792).—See XXIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

[Influence of after-treatments on the] fastness to light of dyeings obtained from Naphthol AS compounds. L. LÖSCHER (Textilber., 1926, 7, 243—244).—Samples of bleached cotton fabric dyed in the usual manner with Naphthol AS+Fast Red GL base, Naphthol AS—SW+Fast Red KB base, and Naphthol AS—RL+Fast Red RL base, and then subjected to various treatments with cold and hot water, and warm and boiling solutions of soap, were exposed for about 2½ summer months to sunlight, one series of exposures being protected by glass. The dyeings not protected by glass faded somewhat more rapidly, but both series faded similarly. Generally, fastness to light of the dyeings described above is proportional to the severity of the soaping treatment and is independent of free naphthols or bases which may be present. Severe soaping is necessary for obtaining maximum fastness of the combinations Naphthol AS+Fast Red GL base and Naphthol AS—SW+Fast Red KB base, but Naphthol AS—RL+Fast Red RL base requires but a moderate treatment with a warm soap solution. Maximum fastness to light of the combination Naphthol AS+Fast Red GL base is secured by soaping at boiling temperature for 5 min. Similarly Naphthol AS—SW+Fast Red KB base and Naphthol AS+Fast Red RL base requires treatment for ½ hr. at boiling temperature, the shade of the last-named combination being thereby rendered bluer. After washing with cold water only, Naphthol AS—RL+Fast Red RL base has the same fastness as Naphthol AS+Fast Red GL base after soaping for ½ hr. at boiling temperature. A. J. HALL.

Printing cellulose acetate silk. A. SCHNEEVOIGT (Textilber., 1926, 7, 354).—Cellulose acetate silk may be printed satisfactorily with alkaline pastes containing vat dyes (saponification of the silk thereby occurs without loss of lustre) or by the usual methods after previous superficial saponification of the silk. After immersion in water and squeezing under comparable conditions, fully, partially (as desirable for printing purposes), and non-saponified cellulose acetate silk fabrics retain 74%, 50%, and 35% of water respectively. Basic dyes are equally fast on superficially saponified cellulose acetate and cotton. Azonine, Azanine, Silkone, and Azole dyes are not satisfactory for printing purposes since in their subsequent

development by diazotisation and coupling, white effects are very easily stained. Very satisfactory effects on cellulose acetate silk are obtained by printing with a paste containing 50 g. of a suitable anthraquinone dye (e.g., Yellow 3G extra, Yellow R, Rosa R, or Violet B), 50 g. of Turkey-red oil, 50 g. of glycerol, 50 g. of Ludigol (*m*-nitrobenzenesulphonic acid), and 800 g. of a thickening containing starch and British gum, and subsequently steaming for 5 min. in a Mather-Platt (loss of lustre occurs when steaming is effected under pressure). The addition of resorcinol to discharge pastes containing Rongalite allows the production of satisfactory white discharges of basic dyes. A. J. HALL.

[Use of] Aktivin in [textile] printing. R. WEGENER (Textilber., 1926, 7, 446).—The stability of Aktivin (sodium *p*-toluenesulphochloramide) renders it more suitable than other well-known oxidising or bleaching agents for treating fabrics before or after printing. For example the chlorination of woollen fabric before printing is satisfactorily replaced by treatment for 20—30 min. in a cold solution containing per litre, 1 g. of Aktivin and 5 c.c. of hydrochloric acid of 21° B. (*d* 1.17), the fabric being subsequently treated with an "antichlor," e.g., a 1% solution of sodium thiosulphate. For clearing the whites of printed goods treatment with a solution containing per litre, 0.2 g. of Aktivin and 0.2 g. of formic acid (for fabrics printed with basic dyes and other dyes sensitive to chlorine) or 1 g. of Aktivin and 0.5 g. of formic acid or 0.6 g. of hydrochloric acid of *d* 1.17 (for fabrics printed with fast vat dyes) is satisfactory. Aktivin is suitable for the preparation of starch pastes for thickening printing pastes. A satisfactory paste is obtained by heating 2 kg. of potato starch, 15 g. of Aktivin, and 20 litres of water for 10 to 15 min. and adding to the product half of its weight of a 7% solution of gum tragacanth. Coloured effects obtained by printing with vat dyes may frequently be improved by after-treating the fabric for 10 min. with a warm solution containing per 200 litres, 100 g. of Aktivin and 100 g. of 80% formic acid. The use of standing baths containing Aktivin is possible. A. J. HALL.

Red and white discharges on dark indigo bottom. J. POKORNY (J. Soc. Dyers and Col., 1926, 42, 157—158; cf. B., 1926, 437).—Red discharges are obtained without tendering the fabric by printing cotton fabric prepared with β -naphthol or β -naphthol R (a mixture of 9 pts. of β -naphthol and 1 pt. of F-acid) with a paste containing 20 pts. of lead chromate, 40 pts. of starch-tragacanth thickening, 28 pts. of diazotised *p*-nitroaniline, and 12 pts. of water, afterwards passing the material during 3—5 sec. at 37° through hydrochloric acid of *d* 1.135 containing 30 g. of ferrous sulphate per litre, then steaming it at 35° for 35—40 sec., washing it with water, and soaping. The hydrochloric acid should not be stored in a lead-lined trough since the presence of lead acts unfavourably in the process of discharge. The printing paste requires a rather deep engraving, 22—24 lines being used instead of the usual 27—28 on the printing roller. A. J. HALL.

Amidation of cotton. KARRER and WEHRLI.—See V.

Liquefying action of various substances on starch. HALLER and HOHMANN.—See XVII.

PATENTS.

[Compositions for] scouring of textile fibres. L. L. LLOYD, A. WOMERSLEY, C. WILKINSON, and A. SCOTT (E.P. 252,811, 10.3 and 10.12.25).—Hydronaphthalene (*e.g.*, tetra- or hexa-hydronaphthalene)-sulphonic acids, or condensation products of hydronaphthalenes or hydronaphthalenesulphonic acids and fatty acids, such as oleic acid or the acids derived from castor, arachis, olive, or cottonseed oils, are satisfactory substitutes for soaps in the scouring of wool, and they may be used at higher temperatures (*e.g.*, at 60° instead of 50° for botany wool) than is possible with soap and alkalis without producing deterioration of the wool.

A. J. HALL.

Kier solution controller. A. F. TAYLOR (U.S.P. 1,584,491, 11.5.26. Appl., 7.7.25).—The vertical kier is fitted centrally at the bottom with a block; this block is provided with an axial passage and radial passages communicating therewith, and acts as a support for a perforated false bottom which divides the kier into an upper and a lower compartment. A perforated tube closed at the top and communicating at its lower end with the upper end of the axial passage in the block extends into the upper compartment.

D. J. NORMAN.

Rapid bleaching process for loose or worked-up vegetable fibres of any kind. CHEM. FABR. GRIESHEIM-ELEKTRON, C. G. SCHWALBE, and H. WENZL (E.P. 251,014, 22.1.25).—The material, optionally after a preliminary treatment with hot, very dilute acid, *e.g.*, 0.1% acetic acid, is drenched with concentrated bleaching liquor containing at least 20 g. (20–30 g.) of available chlorine per litre, and squeezed until it retains sufficient liquor to effect the desired degree of bleaching, the temperature being maintained above 30°, *e.g.*, 30–40°, by pre-heating either the material or the bleaching solution. Loose fibrous material such as paper pulp is preferably bleached in a centrifuge and washed while the centrifuge is running: where other methods are adopted the spent bleach liquor must be displaced by water as far as possible without dilution. In this way colloidal and other impurities capable of causing yellowing are not precipitated on the bleached pulp as is the case when the spent liquor is diluted before removal. When the material to be bleached is in the form of a continuous web, it may be supported on a conveyor, the speed and length of travel being so arranged that the material, after saturation with bleaching solution, is exposed for about 5 min. to a moist warm atmosphere. The use of concentrated solutions of bleaching agents under these conditions has, it is claimed, no deleterious effect on the fibre.

D. J. NORMAN.

Dyeing hanks. P. MISSY (U.S.P. 1,586,377, 25.5.26. Appl., 2.9.25).—Dye solutions are sprayed

by horizontally opposed jets on to hanks of yarn suspended on rotating rollers.

L. A. COLES.

Production of pattern and similar effects on fabrics containing cellulose. R. CLAY, LTD., H. M. SCOTT, and L. THOMPSON (E.P. 251,102, 1.5.25).—A permanent linen-like effect, not characterised by transparency, is imparted to cellulose fabrics, particularly cotton, by calendering the resist-printed fabric prior to treating it with a parchmentising agent. This process may be applied to mercerised or unmercerised fabric and any suitable reagent, *e.g.*, sulphuric acid, zinc chloride, nitric acid (d 1.46), etc., having a parchmentising action may be used. For example, heavy bleached cotton fabric is printed with a resist consisting of a 10% solution of gelatin, dried, and conditioned to contain 5% of moisture. It is then calendered at a pressure of 0.75 ton per linear in., the metal calender bowl being heated to 190°. The cooled fabric is subsequently exposed for 5 sec. at 13° to sulphuric acid (d 1.59), washed, and finished in any desired manner.

D. J. NORMAN.

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

Titration of arsenious acid with permanganate. O. CANTONI (Annali Chim. Appl., 1926, 16, 153–156).—The reaction between arsenious acid and permanganate, which proceeds slowly under ordinary conditions, becomes rapid and complete, even in the cold, in presence of a small amount of potassium iodide. Hence, if allowance is made for the slight quantity of permanganate used in liberating iodine from the iodide, titration with permanganate furnishes a convenient method of determining arsenious acid. To a little sulphuric or, in absence of iron compounds, hydrochloric acid are added a drop of dilute potassium iodide solution and then very dilute permanganate solution until the liquid turns pink. The arsenious acid solution to be examined is then added, and the liquid titrated with permanganate until the pink coloration reappears.

T. H. POPE.

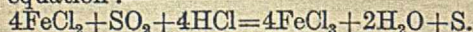
Large crystals in sulphate of ammonia manufacture. H. J. HAILSTONE (Gas J., 1926, 174, 646–648).—The production of large crystals of ammonium sulphate depends on the maintenance of correct physical conditions within the saturator for a sufficient time to allow the crystals to grow (a question of design), and the maintenance of unvarying low acidity (a matter of control). Free circulation of the liquor is desirable and a saturator of ample size free from corners and large walls is best, so that the composition of the liquor is uniform. Further refinements in design are suggested. Control is usually carried out by the hydrometer. This is affected by the difficulty of obtaining a true sample and cannot be made automatic under the conditions of practice. A better method might be worked out by means of a temperature record (a fall in acidity being accompanied by a slight fall in temperature), or by the continuous determination of acidity by measurement

of the refractive index or electrical conductivity of the liquor. The rapid change in the conductivity of sulphuric acid solutions between 0 and 5% concentrations makes the latter especially hopeful. For control of the acid feed a lead feed regulator such as is used on Gay-Lussac towers is recommended. It consists of a box with an oblique line of small holes in the side, the number of holes through which acid is flowing being a measure of the feed. C. IRWIN.

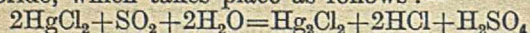
Ammonium sulphate and sodium sulphate from sodium bisulphate and ammonia. H. MOLLTOR (Chem.-Ztg., 1926, 50, 485—486).—Commercial sodium bisulphate (nitre-cake) is dissolved in water to give a solution (*d* 1.325) which is treated with ammonia or ammoniacal gases in closed lead-lined iron vessels arranged in series. When the iron present in the solution in each vessel is completely precipitated the solution is removed and filtered, and the filtrate is evaporated in steam-jacketed pans or in an evaporator until the volume is reduced to one-half. In this way 95% of the sodium sulphate is precipitated as the anhydrous salt free from iron and ammonium compounds. The precipitate is separated hot from the mother liquor on a vacuum filter, and washed with steam in a centrifuge to obtain pure anhydrous sodium sulphate in powder form. The mother liquor is further evaporated until crystals begin to form, then allowed to cool. The ammonium sulphate precipitate is treated as usual; it contains some sodium sulphate, but this does not affect its use as a fertiliser. The iron hydroxide obtained in the process yields a red pigment when heated to 700° with sodium hydroxide. A. R. POWELL.

Comparison of methods for the determination of sodium sulphide. P. P. BUDNIKOV (J. Russ. Phys. Chem. Soc., 1925, 57, 207—214).—The most satisfactory methods for the determination of sodium sulphide are: (1) the iodometric method in which excess of iodine is added and the excess titrated with thiosulphate (B., 1923, 1067A), and (2) Podreschetnikoff's method (B., 1908, 21). T. H. POPE.

Oxidising properties of sulphur dioxide. W. WARDLAW (J.S.C.I., 1926, 45, 210—214 T).—Sulphur dioxide oxidises a solution of ferrous chloride in concentrated hydrochloric acid in accordance with the equation:—



This explains why ferric chloride is incompletely reduced by sulphur dioxide in the presence of concentrated acid. Similarly, cuprous chloride is oxidised by sulphur dioxide in the presence of hydrochloric acid of concentration as low as 1.5*N*. The quantitative reduction of cupric to cuprous chloride takes place only under very special conditions. The fact that the oxidation of sulphur dioxide by mercuric chloride, which takes place as follows:—



is only quantitative when the solution contains not more than about 2 g. of sulphur dioxide per litre, is another instance of the influence of acid concentration on the properties of sulphur dioxide. In the presence of phosphoric acid, sulphur dioxide is also an oxidising

agent, for it readily oxidises ferrous phosphate to the ferric condition.

Physico-chemical and electrochemical aspects of sulphur dioxide as an oxidising agent. S. R. CARTER (J.S.C.I., 1926, 45, 207—210 T).—Determinations of oxidation-reduction potentials show that an increase in acid concentration raises the oxidising power of sulphur dioxide. On the other hand, ferrous-ferric salts become better reducing agents in hydrochloric and phosphoric acids, respectively. The potentials of the cuprous-cupric salts are less than those of the ferrous-ferric salts in solutions of low acid concentration and they give a maximum in 6*N*-hydrochloric acid. Observations on reaction velocities as well as electrolytic experiments suggest that the reduction of sulphur dioxide does not proceed to sulphur directly. It is probable that an intermediate compound X is formed reversibly and that this reaction is followed by a second in which X decomposes irreversibly giving sulphur.

See also A., July, 670, Vapour pressure of hydrogen cyanide (SINOZAKI, HARA, and MITSUKURI); Vapour tension of carbonyl chloride (GERMANN and TAYLOR). 675, Viscosity of aqueous solutions of sodium silicates (MAIN). 684, Thermal decomposition of siderite, rhodochrosite, and dolomite (HEDVALL). 685, Thermionic properties of mixtures used as catalysts in synthesis of ammonia (KUNSMAN); Heat of formation of calcium cyanamide (EHRlich); Pure calcium carbide and its heat of formation (RUFF and JOSEPHY). 691, Reduction of ferric salts with hydrogen sulphide (MOLDENHAUER and MISCHKE). 692, Reactions in the solid state (BALAREV); Thermal decomposition of carbonates (BRUZS). 695, Reactions of solid alkaline-earth oxides with sulphides, carbides, silicides, and phosphides (HEDVALL and NORSTRÖM). 696, Oxidation of ammonia (PARTINGTON). 697, Oxides of chromium (SIMON and SCHMIDT). 699, Preparation of oxygen-free nitrogen (KAUTSKY and THIELE). 701, Potentiometric titration of bromide and chloride (LANGE and SCHWARTZ). 702, Volumetric determination of hypophosphorous acid and hypophosphites (KÖSZEGI). 704, Precipitation of aluminium as hydroxide by means of ammonia (JANDER and RUPERTII).

Recovery of sulphur from waste gases from metallurgical furnaces. WILL.—See X.

PATENTS.

Manufacture of phosphoric acid and generator gas. (A) W. KYBER, Assec. of E. BRITZKE; (B) W. KYBER (E.P. [A] 242,650, 4.11.25, and [B] 248,322, 14.12.25. Conv., 7.11.24 and 25.2.25).—(A) Phosphorites are heated with silicates and carbon in a shaft furnace, the evolved gases being treated in a recuperator or Cowper stove at a temperature of 1000—1300° by addition of air, whereby the carbon dioxide formed by oxidation acts upon the phosphorus vapour in accordance

with the equation, $2P+5CO_2=P_2O_5+5CO$. The phosphorus pentoxide is separated from the mixture of gases, and the residual gases consisting chiefly of carbon monoxide can be used as a generator gas. (B) Gases containing carbon dioxide, *e.g.*, flue-gases or lime-kiln gases, may be substituted for air in the process described under (A). H. ROYAL-DAWSON.

Producing concentrated phosphoric acid. F. G. LILJENROTH (E.P. 252,953, 22.9.25).—In the method of producing phosphoric acid by oxidation of phosphorus by steam at a high temperature, with or without a catalyst, a large excess of steam is necessary to complete the reaction, and with a simple condensation of the reaction products the acid produced is dilute. Concentrated acid can, however, be obtained by fractional condensation of the vapours, for instance, by water sprays in towers packed with Raschig rings. The first tower works at 100° and condenses strong acid; the second at about 44° condenses any unoxidised phosphorus as liquid, while the last is kept as cool as possible to dry the hydrogen which is the other reaction product (cf. G.P. 406,411 and 409,344; B., 1925, 242, 448). C. IRWIN.

Recovery of hydrochloric acid from ferrous chloride liquor. F. KRAUS (G.P. 427,538, 8.7.25).—The hot liquor is poured on to hot ferric oxide, and the mixture is passed through one or more muffle furnaces, a portion of the residual ferric oxide being used again and the remainder withdrawn from the furnace. L. A. COLES.

Manufacture of hydrochloric acid from chlorine and steam. B. NEUMANN, W. STEUER, and R. DOMKE (G.P. 427,539, 4.4.25).—A mixture of chlorine and steam is passed over heated coke or carbonaceous material containing iron oxide or other iron compounds as catalysts, produced, *e.g.*, by carbonising material containing iron. L. A. COLES.

Purifying pearl essence. J. PAISSEAU (E.P. 240,858, 2.10.25. Conv., 3.10.24).—Pearl essence with its impurities in the form of an aqueous paste is agitated with a liquid immiscible with water, but capable of wetting the particles of pearl essence. Amyl acetate, petroleum spirit, aromatic hydrocarbons, and similar liquids are all suitable, but the risk of an emulsion forming renders it desirable to add a varnish or viscous substance to the liquid, *e.g.*, cellulose acetate in tetrachloroethane may be used. The purified pearl essence is decanted with the organic liquid and separated by settling or centrifuging. The impurities, which are preferentially wetted by water, remain in the aqueous layer. C. IRWIN.

Manufacture of xanthates. BRIT. DYESTUFFS CORP., C. J. T. CRONSHAW, and W. J. S. NAUNTON (E.P. 252,500, 27.3.25).—A solution of an alkali hydroxide is added slowly to a well agitated mixture containing theoretical amounts of alcohol (as methylated spirit, or, in the case of higher homologues, the

constant-boiling mixture of the alcohol and water), and carbon disulphide in a reaction vessel provided with a reflux condenser. After a few hours any unchanged alcohol and carbon disulphide are recovered by distillation, and the crude alkali xanthate is dissolved in water and treated with sufficient dilute hydrochloric acid to neutralise any free alkali and to decompose any alkali thiocarbonate that may be present. To this solution is added, while stirring, the theoretical amount of a solution of a soluble salt of a metal which gives an insoluble xanthate, the latter being precipitated in a state of division such that, after washing, it is obtained in a high state of purity. H. ROYAL-DAWSON.

Treating crude cyanide. R. E. GLOVER, JUN., and T. H. ROGERS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,586,509, 25.5.26. Appl., 12.3.21).—Solutions of crude cyanide prepared from calcium cyanamide are not suitable for some processes and are liable to decomposition because of the calcium present. Precipitation of this calcium with sodium carbonate in the ordinary way is not satisfactory as loss of cyanide always occurs. If the crude cyanide is mixed with sodium carbonate and this mixture added to water, a little at a time, the calcium is only momentarily in solution and is thrown down in a form which can be easily removed. E. S. KREIS.

Producing nitrogen compounds. E. S. MATTHEWS (U.S.P. 1,586,823, 1.6.26. Appl., 29.8.17. Renewed 23.10.25).—Air is heated under pressure and the heat abstracted from it while it is under pressure is used for compressing more air and for circulating the fluids. Water is used to cool the mixture and the water so warmed is led through the reaction products to remove the nitrogen oxides formed. E. S. KREIS.

Recovering calcium carbonate from caustic liquors. G. A. RICHTER and S. L. SWASEY, Assrs. to BROWN Co. (U.S.P. 1,587,679, 8.6.26. Appl., 17.1.24).—A solution of the sodium compounds recovered from black liquor from soda-cellulose manufacture is treated with insufficient lime to causticise it completely. After removing the precipitate the solution is again causticised with lime and the precipitate recovered. A. GEAKE.

Decomposition of insoluble thio salts [thioantimonates]. N. E. WILSON (Can. P. 252,563, 11.6.24).—Solid thioantimonates are treated with mineral acids capable of decomposing them, antimony pentasulphide being precipitated. L. A. COLES.

Manufacture of aluminium sulphate. DORR Co., Asses. of H. N. SPICER (Can. P. 253,301, 5.3.25).—Solid material containing aluminium is digested with sulphuric acid, and the residue is separated from the solution by sedimentation at a uniform temperature and under such conditions that the liquid is retained in contact with the solid only for a short time. L. A. COLES.

Production of carbon dioxide and nitrogen compounds [ammonia]. CANADIAN PRESS-AIR LTD., Asses. of H. W. JOHNSTON and E. J. POPE (Can. P. 254,386, 15.4.25).—Nitrogen obtained by passing air over red-hot coke and subsequent removal of carbon dioxide, is combined with electrolytic hydrogen. L. A. COLES.

Production of magnesium hydroxide. R. MONTERUMICI (F.P. 600,396, 7.7.25. Conv., 19.7.24).—Solutions containing magnesium salts are stirred with ammonia at the lowest practicable temperature, or, alternatively, the solid salts are treated with ammonia. L. A. COLES.

Conversion of soluble sulphides into chlorides and sulphur. A. JAHL (F.P. 603,141, 14.9.25. Conv., 3.7.25).—The sulphides are treated with hydrochloric acid and an excess of sulphur dioxide. L. A. COLES.

Production of salt from brine. A. WITTIG (G.P. 427,782, 23.1.23).—Brine is cooled and a portion of the water is simultaneously evaporated by subjecting it to reduced pressure, and heat generated by compressing the liberated vapour is used for again heating the liquid. L. A. COLES.

Decomposition of aluminium silicates. K. HEPKKE (G.P. 427,806, 26.3.24).—Aluminium silicates are heated to 400–600° with kieserite and rock salt in such quantities that the product contains alumina in addition to sodium sulphate and magnesium silicate. L. A. COLES.

Treatment of barium peroxide for obtaining hydrogen peroxide and an improved *blanc fixe*. I. E. WEBER, B. LAPORTE, LTD., and H. E. ALCOCK (E.P. 252,768, 2.3.25).—Wet hydrated barium peroxide is decomposed with phosphoric acid (*d* 1-12), and the mixture is passed through a filter-press to separate the barium phosphate, the hydrogen peroxide solution being collected. After washing the barium phosphate, it is dissolved by agitation with phosphoric acid (*d* 1-12); the impurities mostly remain undissolved. The solution is filtered, heated to 60–80°, and treated with sulphuric acid to precipitate *blanc fixe*, which is separated and mixed with barium carbonate to neutralise any free acid, before being washed and dried. The phosphoric acid is used again in the process. H. ROYAL-DAWSON.

Production of barium peroxide. A. F. MEYERHOFER (G.P. 426,735, 6.11.23. Addn. to 426,034; B., 1926, 539).—Barium silicofluoride produced by the treatment of barium phosphate with hydrofluosilicic acid and subsequent separation of phosphoric acid, is decomposed by heat into barium fluoride and silicon tetrafluoride, the latter being used for the production of more hydrofluosilicic acid. The barium fluoride is treated with nitrates of metals which form insoluble fluorides, yielding barium nitrate, which is converted successively into the oxide and peroxide by the usual methods. L. A. COLES.

Electrolytic production of hydrogen peroxide. SIEMENS & HALSKE A.-G. (F.P. 603,043, 10.9.25. Conv., 26.9.24).—Cathodes for use in the electrolytic production of hydrogen peroxide from persulphuric acid or its salts are constructed of gold or of lead, or of aluminium or its alloys coated with gold.

L. A. COLES.

Preparing highly concentrated sulphur dioxide gas. S. G. S. DICKER. From K. KUDOH (E.P. 252,928, 17.7.25).—Material containing sulphur is mixed with approximately 10–30% by weight of pyrites cinders and charged into a furnace containing an atmosphere of gaseous sulphur dioxide and at least 22% by vol. of oxygen, together with some sulphur trioxide. The gases leaving the furnace, after cooling, are washed with sulphuric acid and a portion is recirculated.

H. ROYAL-DAWSON.

Manufacture of pure sulphur. J. RILEY & SONS, LTD., and W. H. BENTLEY (E.P. 252,938, 17.8.25).—Crude sulphur is dissolved in the mother liquor from a previous operation to form a solution of polysulphide, which is treated with hydrogen sulphide at a low temperature and under pressure to precipitate elemental sulphur. H. ROYAL-DAWSON.

Refining or treating sulphur. W. P. THORNTON (U.S.P. 1,586,539, 1.6.26. Appl., 21.7.23).—Molten sulphur is refined by passing it repeatedly through hot water in a closed vessel. H. ROYAL-DAWSON.

Concentrating nitric acid in a column. W. BÜSCHING (U.S.P. 1,590,494, 29.6.26. Appl., 14.5.23. Conv., 17.8.22).—See G.P. 398,320; B., 1924, 942.

Catalyst for synthesis of ammonia. I. W. CEDERBERG, M. FJELLANGER, and V. GRUNER, ASSRS. TO NORSK HYDRO-ELEKTRISK KVAELSTOF-AKTIESELSKAB (U.S.P. 1,589,682, 22.6.26. Appl., 7.9.21. Conv., 7.9.20).—See E.P. 168,902; B., 1923, 95A.

Treating boron-containing minerals. A. KELLY, ASSR. TO BORAX CONSOLIDATED, LTD. (U.S.P. 1,590,099, 22.6.26. Appl., 2.1.24. Conv., 16.1.23).—See E.P. 208,929; B., 1924, 173.

Recovering sulphur dioxide from gases with which it may be associated. A. H. EUSTIS (U.S.P. 1,589,133, 15.6.26. Appl., 7.2.20).—See E.P. 168,627; B., 1921, 846 A.

See also pages 649, **Recovering sulphur from gas-purifying material** (G.P. 426,385). 668, **Cement from waste lime** (E.P. 253,448). 673, **Copper from ores etc.** (E.P. 253,370). 683, **Chromium from leather waste** (G.P. 427,807).

VIII.—GLASS; CERAMICS.

Index of refraction of glass at higher temperatures. C. G. PETERS (Sci. Papers U.S. Bur. Standards, 1926, 20, [521], 635–659).—Measure-

ments of the index of refraction of pyrex glass and of 5 crown and 3 flint glasses, over the range 20—700°, indicated that the index increased until the critical expansion region, near 500°, was reached, then decreased in this region, and finally increased again above the softening temperature. In every case the value was much larger than the index as computed from the density relation $(n-1)/d=C$, this increase of the index with rise of temperature being probably due to the same cause which shifts the absorption band towards the longer wavelength region. The method employed was that of placing a plate of the glass in contact with two interferometer mirrors, so that two adjacent sets of interference fringes were visible, one produced by light passing through the glass, the other by light passing through an equal space in vacuum.

A. COUSEN.

Non-actinic cobalt-blue glass. W. W. COBLENTZ and A. N. FINN (J. Amer. Ceram. Soc., 1926, 9, 423—425).—A glass of the composition, SiO₂ 69.5, CaO 6.0, CeO₂ 6.8, CoO 0.1, Na₂O 17.6, melts and fines readily at 1425° and works easily. The apparent colour is very similar to that of the corresponding soda-lime cobalt glass, but the absorption of the ultra-violet rays is greater, being practically complete with a glass 5 mm. thick.

A. COUSEN.

Attack of arsenic compounds on fireclay refractory material. W. E. S. TURNER (J. Amer. Ceram. Soc., 1926, 9, 412—417).—The conclusion of McSwiney (B., 1925, 546) that arsenious oxide employed in the glass batch was responsible for the rapid corrosion of the tank blocks, was examined in the light of analyses of several series of soda-lime glasses, made from batches with varying proportions of arsenious oxide, but with the other batch constituents, as well as the time and temperature of melting, constant in each series. Results, based on the amount of iron oxide and alumina in the glass, indicated that even when amounts of arsenious oxide 4 or 5 times those used in practice were employed, no marked corrosion took place at 1400°, and the attack at 1450° and 1500° was only marked with amounts of arsenious oxide in excess of that used in practice.

A. COUSEN.

Anomalous flocculation of clay. KERMACK and WILLIAMSON.—See A., July, 679.

PATENTS.

Manufacture of pottery or the like. J. W. MELLOR (E.P. 253,184, 9.3.25).—The composition of the body is adjusted so that the body matures at the same temperature as the glaze. The biscuit firing can then be eliminated and the whole article fired in the glost kiln.

B. W. CLARKE.

Production of refractory oxide ware. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,585,779, 25.5.26. Appl., 8.3.22).—A mixture of thoria, cryolite, phosphoric acid, and water is shaped and then fired. B. W. CLARKE.

Ceramic and refractory making process and material. A. G. BETTS (U.S.P. 1,585,826, 25.5.26. Appl., 3.3.23).—Impurities such as iron in otherwise good quality clays and refractories are removed as volatile chlorides during the burning process, if the raw material be mixed with aluminium chloride solution instead of with water.

E. S. KREIS.

Refractory and ceramic process and materials. A. G. BETTS (U.S.P. 1,585,827, 25.5.26. Appl., 12.3.23).—Ceramic and refractory materials which are resistant to acids are made up with a solution of basic aluminium chloride instead of with pure water. Besides causing the removal of impurities such as iron, as volatile chlorides, during the firing, the basic aluminium chloride solution possesses advantages as a bonding material because it is a non-crystallising solution and is equivalent to a colloidal solution of hydrated aluminium oxide. The pores of the resulting material are filled up with aluminous material which increases the density and diminishes the shrinkage on vitrifying.

E. S. KREIS.

Composition of matter. [Utilisation of scrap fused silica.] W. W. WINSHIP, Assr. to THERMAL SYND., LTD. (U.S.P. 1,587,057, 1.6.26. Appl., 8.5.25).—Fused silica scrap and quartz are ground and mixed to a stiff paste with sodium silicate solution, preferably of d 1.345. After being moulded, the articles are dried for several days and then treated with concentrated hydrochloric acid. They are then washed, or they may be treated with ammonia previous to washing.

E. S. KREIS.

Drying clays and similar materials. T. H. RHOADS, Assr. to PROCTOR & SCHWARTZ, INC. (U.S.P. 1,587,419, 1.6.26. Appl., 21.6.21).—Ceramic articles are introduced into a drying compartment provided with spraying devices; the wet bulb temperature of the compartment is raised almost to that of the article, which is then dried gradually by raising the dry bulb temperature. A flow of moisture from the interior of the article to the exterior is then induced by bringing the air in the compartment almost to the dew point, and finally by progressively raising the wet and dry bulb temperatures of the air the article is brought to the required degree of dryness.

B. W. CLARKE.

Production of fireproof and acid-proof material. DEUTS. GASGLÜHLICHT-AUER-GES. M.B.H. (F.P. 602,134, 17.8.25. Conv., 13.8.24).—The material is prepared from one or more hafnium compounds, preferably in admixture with other refractory material, such as silica or magnesia.

L. A. COLES.

Making vitreous silica. BRIT. THOMSON-HOUSTON CO., LTD., Asses. of P. K. DEVERS (E.P. 240,489, 28.9.25. Conv., 27.9.24).—See U.S.P. 1,536,821; B., 1925, 548.

Treatment of clay. W. FELDENHEIMER (U.S.P. 1,588,956, 15.6.26. Appl., 16.6.25. Conv., 7.8.24).—See E.P. 242,357; B., 1926, 55.

Tunnel kiln. A. MCD. DUCKHAM (U.S.P. 1,590,154, 22.6.26. Appl., 4.11.24. Conv., 12.11.23).—See E.P. 228,987; B., 1925, 318.

Sheet glass drawing apparatus. LIBBEY-OWENS SHEET GLASS CO. (E.P. 240,448, 18.9.25. Conv., 24.9.24).

Manufacturing plate glass. F. RIECHERS (E.P. 251,632, 28.4.26. Conv., 4.5.25).

Glass-making furnaces. J. and A. BOUCHER (E.P. 243,322, 6.10.25. Conv., 22.11.24).

Apparatus for comminuting or grinding china clay. R. G. VARCOE and H. P. GARVEY (E.P. 253,694, 18.5.25).

IX.—BUILDING MATERIALS.

Determination of soluble silica in cements, mortars, and concretes. D. FLORENTIN (Compt. rend., 1926, 183, 53—55).—The cement (1 g.) or mortar (4 g.) is added, little by little, to 50—60 c.c. of cold hydrochloric acid (d^{15} 1.12). The reaction vessel should be cooled in a current of cold water. The solution is filtered and the silica in the filtrate rendered insoluble in the usual manner. If a small quantity of the soluble silica is precipitated the residue is treated with a warm 5—6% sodium carbonate solution, this extract being added to the main filtrate. L. F. GILBERT.

Cement-lime mortars. H. V. JOHNSON (Tech. Papers U.S. Bur. Standards, 1926, 20, [308], 241—274).—The addition of lime to cement mortars increases the amount of water required to bring the mix to a given consistency, and therefore increases the shrinkage of the mortar. The addition of lime increases the strength of cement mortars leaner than 1:2 by weight, and also increases the density of the mortar. The addition of cement to a lime plaster or mortar increases the strength and shortens the time of setting without affecting the plasticity or the ease with which the mass can be worked. B. W. CLARKE.

Strengthening and indurating concrete with sulphur. W. H. KOBBE (Eng. News-Rec., 1926, 96, 940—942).—Impregnation of pre-cast concrete with sulphur results in an increase in strength and a decrease in absorption. The concrete products, preferably well cured, are immersed in a bath of molten sulphur, usually kept at a temperature of 130—150°, until the requisite degree of absorption is obtained. The treatment is applicable to all types of concrete and cement mortars including high-alumina cement and with any aggregate chemically inert toward sulphur. Concrete absorbs ordinarily from 10 to 25% of sulphur, depending upon the character of the mix and its porosity. The rate of absorption of the molten sulphur is dependent upon a number of factors, moisture content probably being the dominant one. Usual mixes, if fairly dry, may be impregnated to a depth of at least 2 in. in 8 hrs. Standard tensile briquettes of cement

mortars, which ordinarily break at about 150 lb., showed increases in strength to 1000—1700 lb. and occasionally withstood a pull of 2000 lb. per sq. in. after impregnation. Under compression concrete impregnated with sulphur showed an increase in strength over the untreated similar to that indicated in the case of the tensile tests. Water absorption is reduced to less than 2—3%. The cost of treatment is comparable with the cost of creosoting timber. Electrolytic cells made of concrete impregnated with sulphur withstand successfully the very corrosive action of a mixture of hot ferrous and ferric chloride solution. W. T. LOCKETT.

Indurating wood with sulphur. W. H. KOBBE (Chem. Met. Eng., 1926, 33, 354—356).—Soft wood immersed in molten sulphur for about 10 hrs. at 120—150° absorbs up to 70% by weight of sulphur, whereby the strength and hardness of the wood are considerably increased. The sulphur acts as a preservative owing to the complete sealing of the pores. A similar hardening effect occurs with hardwoods for use as paving blocks, sleepers, etc., on treatment with molten sulphur. B. W. CLARKE.

System calcium oxide-ferric oxide-silica. HANSEN and BOGUE. See A., July, 684.

PATENTS.

Cement and lime burning. G. E. HEYL (E.P. 252,780, 3.3.25).—A dry powdered mixture of cement- or lime-forming materials is projected into a region of a combustion chamber maintained at a vitrifying temperature by a flaming jet of oil or powdered fuel, in such proportions that the ash of the fuel is negligible in comparison with the amount of cement or lime produced. B. W. CLARKE.

Manufacturing process for cements containing iron and alumina combinations. E. MARTIN (U.S.P. 1,586,099, 25.5.26. Appl., 11.9.24).—A mixture of raw materials containing calcium carbonate, ferric oxide, and alumina in the proportion of more than 2 mols. CaCO_3 for each mol. of Fe_2O_3 and 1 mol. CaCO_3 for each mol. of Al_2O_3 is burnt at a temperature below the m.p. of any of the ingredients. B. W. CLARKE.

Manufacture of cement out of spent or waste lime. R. ILLEMANN (E.P. 253,448, 31.12.25).—Waste lime from the Leblanc soda process is heated in a revolving furnace at a temperature up to 700°, according to the properties required in the finished product, and after grinding, mixed with small quantities of alum, potash, or borax (which may be added before the heating if desired), or with ground glue. The product forms a hard cement or plaster on mixing with water. B. W. CLARKE.

Mineralising fibrous materials. NOVOCETES, LTD., and G. O. CASE (E.P. 252,906, 27.3 and 27.9.24. Addn. to 225,912; cf. B., 1925, 134).—Fibrous material, e.g., sawdust, is damped to saturation with a solution of a metal salt, e.g., a chloride, and mixed with a solution which will produce an insoluble precipitate in or on the fibrous material, without

initiating setting, thus forming a granular material suitable for use with cement, concrete, and the like.

B. W. CLARKE.

Manufacture of light forms of concrete or artificial wood composed of loose or fibrous organic materials and cement, concrete, or the like. BROADWAY TRUST CO., LTD., C. D. BURNEY, and J. E. TEMPLE (E.P. 253,007, 16.12.24).—Organic material, e.g., sawdust, is heated gradually to 120–130°, or treated with materials such as oils, resins, soaps, emulsions, etc., in order to render it water-repellent and less susceptible to change in volume with change of moisture content. It is then mineralised (cf. E.P. 244,178; B., 1926, 130) to make it suitable for combining with cement etc. Alternatively the material may be treated with a weak solution of an alkali or an acid, or with a solution which will dissolve resinous compounds, before mineralising and mixing with cement.

B. W. CLARKE.

Preserving wood by impregnation. GEBR. HIMMELSBACH A.-G. (E.P. 253,041, 23.2.26. Conv., 30.11.25).—Wood is steamed strongly at a high temperature and then dried briskly at about 150°. A large number of cracks are thereby produced in the surface of the wood, resulting in a more complete and uniform impregnation when the timber is treated in the usual way with preserving solutions. Cresol etc. may be used with the steam if desired.

B. W. CLARKE.

Emulsification of tar, bitumen, etc. (E.P. 252,449).—See II.

Preparation of Trinidad pitch lake asphalt, etc. (E.P. 252,802).—See II.

Blown oil asphalts (U.S.P. 1,586,376).—See II.

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

Phosphorus in wrought iron. H. S. RAWDON and G. EPSTEIN (Amer. Iron and Steel Inst., Mar., 1926; Engineering, 1926, 172, 13).—Comparative tests on wrought iron made from the same cast of pig iron by hand and by mechanical puddling indicated that there was no reason why the properties of mechanically puddled iron should be inferior to those of the hand puddled product. Phosphorus was eliminated more readily than by hand work, though non-uniformity of distribution persisted, and in view of this the average phosphorus content should be considerably less than 0.18%.

C. A. KING.

Malleable cast iron and the method of its graphitisation. T. KIKUTA (Sci. Rep. Tohoku, 1926, 15, 115–155).—The graphitisation of white cast iron has been studied using the dilatometer. To obtain black heart cast iron the graphitisation is best performed in two stages. For the complete graphitisation of free cementite the article must be heated at a high temperature above the Ar1 point,

and then to decompose the eutectoid cementite prolonged heating at a temperature below, or very slow cooling from the Ar1 point is essential. The annealing temperature greatly affects the graphitisation in both the first and second stages; the time required to complete them decreases logarithmically with rise of temperature. Thickness of casting increases the size of the free cementite and this consequently becomes more difficult to graphitise; moreover, the higher the tapping temperature of the melt the harder graphitisation becomes. Increase in silicon content causes a logarithmic decrease in the time of both the first and second stages. Carbon is more effective than silicon in the second stage, but it scarcely affects the first stage. Manganese lessens graphitisation, the effect being particularly great in the second stage, so that not more than 0.5% should be present in black-heart malleable iron castings. Sulphur behaves similarly, and the sulphur content should be limited to 0.6%. Phosphorus assists graphitisation in the first stage, but for the second stage the amount should not exceed 0.3%. Rapid heat treatment of a sample of white cast iron containing more than 2.5% of carbon and 1% of silicon gave a black-heart malleable iron. Black-heart malleable cast iron grows with repeated heating and cooling through the Ar1 range in a similar manner to grey cast iron. The mechanism of graphitisation of white cast iron is explained as due to the catalytic action of free carbon oxides present in the casting; a nucleus of temper carbon is formed at the boundary of the cementite and graphitisation is accelerated by the affinity between iron and the included elements.

M. CARLTON.

Diffusion of carbon in α -iron. E. ZINGG (Stahl u. Eisen, 1926, 46, 776–777).—Experiments were made to test whether carbon absorption takes place below Ac3 or Ac2. Cementation was carried out in a mixture of hydrogen, hydrocarbons, and carbon monoxide obtained by the removal of carbon dioxide, water vapour, hydrogen sulphide, ammonia and other nitrogen compounds from illuminating gas. The materials used were Swedish wrought iron 0.1 mm. and 0.05 mm. thick and a steel containing 1.2% C, 0.23% Si, and 0.27 Mn, 1 mm. thick, which were heated at 780–800° and 670–690° respectively for 20 hrs. and also for 60 hrs. The specimens were cooled in the gas and afterwards etched with sodium picrate solution. Microscopical examination showed that from 650° upwards to 800° the surface layers of the test-pieces were converted into cementite. In the α -iron region sharp boundaries were formed between the cementite layers and the ferrite grains, but the cementite also penetrated into the interior along the crystal boundaries. Inside the ferrite grains small spheres like granular cementite appeared. In the mixed-crystal region the boundaries of the cementite layers were also sharp. When sufficient time was allowed the steel was converted almost completely into cementite. Fry's distinction (cf. B., 1923, 932A) between pure and reaction diffusion was confirmed. The former only takes place up to

the content of the saturated mixed-crystals, whilst with reaction diffusion the carbon content of the highest chemical compound can be attained.

T. H. BURNHAM.

Data relating to basic open-hearth steel practice. A. N. DIEHL (Amer. Iron and Steel Inst., May, 1926; Engineering, 1926, 172, 13).—Complete records of the operation of basic steel furnaces of 50–75 tons capacity were kept. When molten pig-iron alone was charged 98% of the silicon content was eliminated within 1 hr., and manganese was oxidised almost as quickly, with, however, a marked reversion from slag to the metal in the later stages of the heat. Phosphorus was oxidised in the first stages and the duration of the heat was due to the time required for the elimination of carbon. When a carbon content of more than 0.25% was desired re-carburising was effected best by the addition of molten pig-iron, though for relatively low-carbon steels anthracite was added as the metal ran into the ladle. Manganese was added in the ladle or larger quantities in the furnaces and ferrochromium also in the furnace. Nickel was introduced as scrap or pig into the furnace, only the final adjustment being made before pouring after a preliminary analysis.

C. A. KING.

Absorption of nitrogen by iron in the basic open-hearth process. E. H. SCHULZ and R. FRERICH (Mitt. Versuchsanst. Deutsch-Luxemburg. Bergwerks u. Hütten-A.-G. Dortmund Union, 1925, 1, 251–257; Chem. Zentr., 1926, I., 3427).—The amount of nitrogen taken up by steel in the basic open-hearth process is greater the higher the temperature, the longer the time of blowing, and the larger the volume of air passed through the metal. These factors are especially pronounced during the dephosphorising period, particularly the time factor, which has a far greater influence on the nitrogen absorption than any of the others.

A. R. POWELL.

Equilibrium between austenite and the carbon oxides. G. TAKAHASHI (Sci. Rep. Tohoku, 1926, 15, 157–175).—The concentration of carbon monoxide in the gas mixture ($\text{CO} + \text{CO}_2$) must increase with rise of temperature in order to maintain austenite at the same carbon content; if, however, the carbon monoxide concentration be increased beyond a certain limit, the carbon content in austenite increases and free cementite is produced; diminishing the carbon monoxide concentration increases the percentage of pure iron. If a specimen be heated above 900° and the carbon monoxide in the gas phase be less than a certain limiting value, conversion into ferrous oxide occurs. The carbon content in austenite in equilibrium with free cementite increases with rise of temperature, whilst if austenite be in equilibrium with α -iron or ferrous oxide, the carbon content decreases with temperature rise. The carbon monoxide content in a gas mixture in equilibrium with austenite, austenite and free cementite, or austenite and ferrous oxide increases with rise in temperature but decreases for austenite

and α -iron. Cementation is not produced by a gas mixture of which the carbon monoxide concentration is less than that in equilibrium with austenite, α -iron, or ferrous oxide. Curves are given from which may be deduced the conditions under which carburisation etc. may take place. The A1 transformation point has been determined to be 726° by cementation in carbon monoxide gas.

M. CARLTON.

Cementation of steel by silicon. L. GUILLET (Compt. rend., 1926, 182, 1588–1589; cf. Fry, B., 1923, 932 A).—In presence of ammonium chloride cementation of steel by ferrosilicon (75–80% Si) takes place extremely rapidly at temperatures in the region 1100–1200°. The surface layer contains about 15% Si and is very brittle.

J. S. CARTER.

Electrolytic iron from ilmenite ores. R. H. MONK and R. J. TRAILL (Canad. Chem. Met., 1926, 10, 137–139).—Experiments were made on a Quebec ore containing 31.6% TiO_2 and 40.5% Fe. Three parts of crushed ilmenite were mixed with one part of charcoal and heated in a crucible to 900–950° for 1½ hrs. After running over a Wetherill magnetic separator the sponge product contained 40.5% of metallic iron, 46.7% of total iron, 36.9% TiO_2 , and 2.0% C. It was treated with a leaching liquor consisting of a mixture of ferrous and ferric chlorides, the charge of sponge being adjusted to cause complete reduction of all the iron to the ferrous state. A combination of thickening and settling is recommended for separating the insoluble residue. The ore tested contained small amounts of copper, lead, nickel, and cobalt which were removed by means of calcium sulphide before the electrodeposition of the iron. Nickel, copper, and cobalt below 0.02 and lead below 0.002 g. per litre had no ill effect. For the deposition of the iron a diaphragm cell of 12 litres electrolyte capacity was used, having as cathode a steel mandrel rotating at 250–400 r.p.m. The current density was 100 amp. per sq. ft. Satisfactory deposition was obtained only at 80–95°. The titanium oxide content of the ilmenite can be recovered by any of the well-known processes. The authors consider the process can be made commercially successful, starting with the production of sponge iron in a rotary furnace on the lines of the direct-fired rotary kiln described in the U.S. Bureau of Mines report No. 2656.

T. H. BURNHAM.

Hydrometallurgical treatment of iron sulphide ores for the production of electrolytic iron and the recovery of sulphur and other metals as by-products. R. J. TRAILL and W. R. McCLELLAND (Canad. Dept. Mines, 1926, [643], 92–102).—Pyrrhotite dissolves fairly readily in ferric chloride solution yielding ferrous chloride and free sulphur. Pyrites is insoluble in this reagent, but, by passing it through a furnace kept at 750–800°, it is reduced to ferrous sulphide with the liberation of half the sulphur, 50–65% of which can easily be recovered on a commercial scale, and the porous ferrous sulphide obtained dissolves readily in ferric chloride. Solution is effected at 95° with spent electrolyte containing

about 7% each of ferric and ferrous chlorides. The reduced solution is filtered and boiled with sponge iron to remove copper and the bulk of the lead. No efficient process has yet been evolved to remove the zinc, so that in the subsequent electrolysis this is deposited with the iron and can be eliminated only by melting.
A. R. POWELL.

Combined pyro- and hydro-metallurgical process for the treatment of nickeliferous pyrrhotite ores with the subsequent recovery of iron, sulphur, nickel, and copper. H. C. MABEE and A. E. SMALL (Canad. Dept. Mines, 1926, [643], 105—108).—The present processes for recovering nickel and copper from Sudbury pyrrhotites waste the whole of the iron and sulphur content. If the ore is smelted in a reverberatory or electric furnace with limestone to flux all the gangue a low-grade nickel-copper matte is obtained which contains practically the whole of the nickel, copper, iron, and sulphur originally present. By roasting the matte at 450—500° 85% of the sulphur is recovered as sulphur dioxide. The roasted material is then mixed with 10% of sodium chloride and 5—10% of water and introduced into a rotating furnace held at 450—500° in such a way that the charge is gradually heated as it passes through the furnace. Leaching of the product results in solution of 98% of the copper and 75% of the nickel, leaving an iron oxide containing 3—4% Ni and about 0.4% S which could be used for the direct production of nickel steel. A. R. POWELL.

Specific heat of iron-nickel alloys. M. KAWAKAMI (Sci. Rept. Tohoku, 1926, 15, 251—262).—Alloys of iron and nickel of varying composition were subjected to different heat treatments, *i.e.*, annealed, annealed and then cooled in liquid air, annealed and quenched in water, and their mean specific heat between 30° and 300° determined by the method of mixtures. The specific heat varies somewhat with different structures, *e.g.*, the specific heat of martensitic alloys is less than that of nickel-ferrite and this in turn is greater than that of austenitic alloys. The maximum specific heat of nickel-iron alloys is connected with the minimum expansibility in accordance with Grüneisen's law (Ann. Physik, 1908, [iv.], 26, 211). M. CARLTON.

Determination of small amounts of bismuth in copper. C. O. JONES and E. C. FROST (Ind. Eng. Chem., 1926, 18, 596).—Minute proportions of bismuth in copper (about 0.002%) were most accurately determined colorimetrically by precipitating with sodium phosphate and ammonium carbonate from a nitric acid solution of the copper after adding a crystal of ferric sulphate and making the solution ammoniacal. The precipitate is dissolved in dilute sulphuric acid, hydrogen sulphide passed through, and the antimony and arsenic sulphides are separated from the bismuth sulphide by potassium hydroxide or ammonium sulphide. The remaining copper is separated by precipitating the bismuth as sulphide in ammoniacal potassium cyanide solution, and the bismuth sulphide is then dissolved in nitric acid, the solution evaporated with sulphuric acid,

and any lead sulphate removed. After addition of potassium iodide and sulphurous acid the colour is compared with that of standard solutions similarly treated.
D. G. HEWER.

Equilibrium diagram of copper-tin system. T. ISIHARA (Sci. Rep. Tohoku, 1926, 15, 225—246).—The equilibrium diagram of the copper-tin system previously described (A., 1925, ii., 122) is confirmed by results of dilatometric and hardness measurements. Dilatometric measurements show that the change $\alpha + \delta \rightarrow \beta$ appears as a contraction while $\eta + \delta \rightarrow \gamma$ produces an expansion. The hardness of specimens measured by the Shore and Brinell methods and the Honda-Sato dynamic method give results in fair agreement. The eutectoid transformation $\alpha + \delta \rightarrow \beta$ is accompanied by an increase and the change $\delta + \eta \rightarrow \gamma$ by a decrease in hardness. The relation between hardness and composition in specimens quenched at different temperatures agrees with the equilibrium diagram, as also do microscopical observations.
M. CARLTON.

Formation of carbon tetrafluoride in the technical recovery of aluminium. W. D. TREADWELL and A. KÖHL (Helv. Chim. Acta., 1926, 9, 681—691).—No trace of carbon tetrafluoride is produced in the electrolytic production of aluminium from alumina-cryolite baths although from theoretical considerations it should be formed at the anode as readily as carbon dioxide. This is probably due to the homopolar nature of carbon tetrafluoride so that in the presence of aluminium oxide the oxygen is preferably discharged at the anode. In support of this is the fact that formation of carbon tetrafluoride during the electrolysis of cryolite in a magnesia crucible ceases as soon as a trace of oxide has dissolved from the surface of the crucible. Carbon tetrafluoride dissolves in water to the extent of 7.45 c.c. per 100 c.c. at 16° and 6.5 c.c. per 100 c.c. at 20°. The solution is stable and is not hydrolysed by boiling with aqueous alkalis, although alcoholic potassium hydroxide causes slow hydrolysis. On heating to redness carbon tetrafluoride is decomposed with the formation of a white solid containing carbon and fluorine and with the liberation of hydrogen fluoride, the etching properties of which serve as a test for the presence of the tetrafluoride. A. R. POWELL.

Corrosion of aluminium by water. I. L. W. HAASE (Z. Elektrochem., 1926, 32, 286—289).—Experiments on the corrosion of aluminium by water have been made by placing samples of surface, drinking, and sewage water in corked aluminium flasks and following the changes in the hydrogen-ion concentration and the conductivity and determining the aluminium hydroxide formed. All the samples of water attacked the aluminium more or less, sometimes with formation of holes. The corrosion was greatest at the bottom of the flasks and at the liquid-air boundary. This may be explained both electrolytically and electrochemically. The aluminium hydroxide was in some cases partly colloidal. Both acid and alkaline waters attack aluminium owing to its amphoteric nature and

suffer a corresponding change in their p_H values, but the final stage is never neutral since a small part of the hydroxide is always dissociated in a way depending on whether hydrogen or hydroxyl ions preponderate. N. H. HARTSHORNE.

Equilibrium diagram of the aluminium-zinc system. T. ISIHARA (Sci. Rep. Tohoku, 1926, 15, 209—224; cf. B., 1925, 246).—Eutectoid transformation in aluminium-zinc alloys has been studied using the dilatometer. Alloys containing 1—60% of aluminium show a discontinuous expansion at 280°, the magnitude of which increases up to 21% Al and is directly proportional to the amount of the eutectoid present. The eutectoid change $\alpha + \gamma \rightarrow \beta$ is accompanied by a discontinuous increase in the scleroscope (Shore) hardness. The hardness of quenched specimens reaches a maximum in less than 1 hr. after quenching and then decreases rapidly, finally becoming asymptotic. This ageing effect is assumed to be chiefly due to an intermediate β^1 form: $\beta + \beta^1 \rightarrow \alpha + \gamma$, but no difference between β and β^1 can be found either microscopically or by X-ray analysis. M. CARLTON.

Boron in aluminium and its alloys. P. HAENNI (Rev. Mét., 1926, 23, 342—352).—A fuller description and amplification of earlier work (cf. B., 1926, 58). A small quantity of boron improves the structure and properties of copper-aluminium and zinc-aluminium alloys; it also improves slightly the resistance of the alloys to corrosion. A. R. POWELL.

Influence of the thermal zone of work on the selection of metals for aviation motors. Application to exhaust valves. GRARD (Rev. Mét., 1926, 23, 317—330).—Fuller details are given of work the results of which have already been published (cf. B., 1926, 92). A. R. POWELL.

Hardening of printing type alloys. A. TRAVERS and HOVOT (Compt. rend., 1926, 182, 1627—1628).—The alloys as cast show a considerable contraction during heating for dilatometric examination; the magnitude of the contraction under comparable conditions increases with the tin content. The contraction also varies with the casting temperature, increasing 20% for example when the casting temperature is raised from 300° to 360°. It is diminished by previous annealing of the alloy. Annealing occurs even at the ordinary temperature, but is not complete even after 6 weeks; it is completed in 24 hrs. at about 200°. The alloys become exceedingly brittle after ageing for several weeks at the ordinary temperature. J. S. CARTER.

Determination of fluorine [in ores]. F. G. HAWLEY (Ind. Eng. Chem., 1926, 18, 573—576).—The fluorine is determined indirectly from the chlorine contained in precipitated lead chlorofluoride. The sample is adjusted to contain at least four times as much silica as fluorine, and fused with sodium and potassium carbonates. After adding just enough sodium peroxide for oxidation of any sulphur present (which is liable to cause loss of fluorine), the fused

mass is heated with water until disintegrated, the residue boiled again with water with addition of sodium carbonate, filtered, concentrated hydrochloric acid added to the filtrate, the liquid warmed, neutralisation completed with nitric acid, and glacial acetic acid and 10% lead acetate solution containing 1% of acetic acid added to the slightly acid solution. The precipitated lead chlorofluoride is filtered off, washed, redissolved in nitric acid, chlorine precipitated by silver nitrate, and determined. The presence of lead and zinc have little effect on the results. The method gives the best results for low- to medium-grade ores. D. G. HEWER.

Reports of investigations. Ore dressing and metallurgical laboratory. W. B. TIMMS, C. S. PARSONS, R. K. CARNOCHAN, and J. S. GODARD (Canad. Dept. Mines, 1926, [643], 8—91).—Details with flow-sheets and analyses of the products are given of numerous concentration tests on zinc-lead, gold-copper, molybdenite, silver-lead, garnet, and titanite ores, of cyanide tests on gold ores, and of cleaning and trimming experiments on the product of a mica mine. A. R. POWELL.

Concentration of Lake George antimony ores. C. S. PARSONS (Canad. Dept. Mines, 1926, [643], 110—115).—The ore consists of stibnite with a small amount of mispickel disseminated in fissures in slate and quartzite. It averages 11.6% Sb and 0.4% As. Flotation tests on the ore passing through 65-mesh gave a concentrate containing 64% Sb and 0.28% As with a recovery of 86% of the antimony; the oil used consisted of a mixture of 40% of coal tar and 60% of coal tar creosote with a small addition of pine oil, and the pulp was neutral. An acid pulp gave an extraction of 95% in a concentrate assaying 63.4% Sb and 0.31% As, whereas an alkaline pulp gave a poorer extraction and over 1% As was present in the concentrate. A. R. POWELL.

Concentration of lead-zinc ores of Eastern Canada. C. S. PARSONS (Canad. Dept. Mines, 1926, [643], 109—110).—An historical summary. A. R. POWELL.

Recovery of sulphur from waste gases from metallurgical furnaces. E. WILL (Mitt. Versuchsinst. Deutsch-Luxemburg. Bergwerks- u. Hütten-A.-G. Dortmunder Union, 1925, 1, 237—242; Chem. Zentr., 1926, I., 3426).—Attempts to recover the sulphur dioxide from waste gases from furnaces by passing the gases at 450—800° over ferric oxide, copper chromate, or chromic oxide and alumina contact masses failed owing to the large proportion of moisture present causing the formation of sulphates. By cooling the gases to 10°, however, the mist formed by the condensation of this moisture resulted in a precipitation of 50% of the sulphur dioxide content of the gases. A. R. POWELL.

A2 line in the equilibrium diagram of the iron-carbon system. K. HONDA (Sci. Rep. Tohoku, 1926, 15, 247—250).—See B., 1926, 194.

See also A., July, 665, **Magnetic properties of single crystals of iron** (HONDA, KAYA, and

MASUYAMA). 666, Behaviour of single crystals of aluminium under static and repeated stress (GOUGH, HANSON, and WRIGHT); Influence of mean principal stress on flow of iron, copper, and nickel (LODE); Properties of gold-silver-copper alloys (STERNER-RAINER). 669, Dilatometric investigation of A3 and A4 transformations in pure iron (SATO). 671, Hardness of copper-tin alloys (MALLOCK). 673, Absorption of hydrogen and carbon dioxide by pyrophoric iron, nickel, and cobalt (NIKITIN). 683, Ternary diagram of system iron-carbon-copper (ISHIWARA, YONEKURA, and ISHIGAKI). 684, Equilibrium between carbon monoxide, carbon, and carbon dioxide, and reactions between ferrous oxide and carbon, and between carbon monoxide and iron (FALCKE and FISCHER). 692, Tarnishing of copper in sulphur vapour (FISCHBECK); High-temperature oxidation of metals; Low-temperature oxidation of metals (DUNN). 699, Preparation of pure zirconium (DE BOER and FAST). 705, Detection of tin in minerals, using the blowpipe (BRALY). 707, Oberhoffer's etching mixture (HEINRICH and VOIGT).

Equipment for high-pressure reactions. ERNST.—See I.

Coke testing. HAVEN.—See II.

Electrodes of V2A-steel in electro-analysis. SCHLEICHER and TOUSSAINT.—See XI.

Prevention of corrosion. BAYLIS.—See XXIII.

PATENTS.

Kiln for production of iron sponge. S. E. SIEURIN (E.P. 252,899, 4.6.25).—In a kiln for the production of iron sponge the containers for the charge are in the form of vertical polygonal retorts, built up of shaped bricks so arranged that horizontal heating ducts are formed between the retorts. In one form of construction the bricks are made with flanges to space the retorts and so form ducts. Certain of the vertical retorts may be used as combustion chambers, having side outlets into the heating ducts. C. A. KING.

Production of metals [iron, chromium, and manganese] and their alloys [from ores]. S. HEULAND (F.P. 602,448, 22.8.25).—Iron, chromium, or manganese ores or mixtures of these are melted in an electric furnace with a reducing agent sufficient to produce only a small amount of metal which will contain all the deleterious impurities in the ores, *e.g.*, phosphorus, carbon, or iron. The remainder of the metal is then reduced from the fused slag by addition of calcium silicide. A. R. POWELL.

Recovery of metals, especially iron and steel [from ores and scrap]. L. BLANCHET (F.P. 602,703, 10.12.24).—Iron ores or scrap iron are heated to 400–1200°, preferably 600–1200°, and a stream of hydrocarbon vapours is passed over them whereby finely divided carbon is deposited on the

ore or scrap and lighter oils are recovered from the issuing gases together with inflammable gas which is used for heating the furnace. The carbon-coated ore or scrap is subsequently smelted in a separate furnace. A. R. POWELL.

[Non-oxidising iron-nickel] alloys. R. L. SPITZLEY and A. M. THOMPSON, Assrs. to ALLOYS FOUNDRY Co. (U.S.P. 1,587,992-4, 8.6.26. Appl., [A] 22.5, [B] 25.5, and [C] 2.6.25).—Non-oxidising alloys suitable for fusing with cast-in steel parts contain (A) 56% Ni, 8% Cr, 7% Mn, 26% Fe, 2% Si, and 1% W, (B) 64% Ni, 7% Mn, 14% Fe, 2% Si, 1% W, and 12% Cu, and (C) 58% Ni, 8% Cr, 7% Mn, 14% Fe, 1% W, and 12% Cu. A. R. POWELL.

Casting having silicon-alloy surface. C. B. JACOBS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,588,111, 8.6.26. Appl., 30.4.21).—A casting of iron which has a surface highly resistant to the action of acids is produced by coating the surface of the mould with a mixture of ferrosilicon particles (15- to 40-mesh) and a suitable binder, hardening the coating, and then casting molten iron at such a temperature that the ferrosilicon is dissolved in the surface layer of metal. C. A. KING.

Treatment of solid materials with liquids. [Precipitation of copper with scrap iron.] A. RAMÉN (E.P. 235,902, 18.6.25. Conv., 20.6.24).—In the precipitation of copper from copper lyes by means of scrap iron, atmospheric oxidation of the copper is prevented by the use of an elongated container rotating or rocking about its inclined longitudinal axis, and maintaining a permanent liquid seal at each end during the continuous passage of the liquid through the container. The outlet is provided with a sieve to retain the scrap iron. The copper is collected in a filter-press, and the liquid leaving the press is, if not exhausted, re-circulated through the container. H. HOLMES.

Extraction of copper from ores, concentrates, and residues. H. S. MACKAY (E.P. 253,370, 2.7.25).—The ore is roasted in such a way that the whole of the copper and a small part of the iron are converted into sulphates. The product is then leached with dilute sulphuric acid, previously used to finish the leaching of a similar charge, so as to obtain a neutral solution which is then purified by treatment with calcium carbonate (*cf.* E.P. 238,962, B., 1925, 854). The filtrate is evaporated to obtain crystals of copper sulphate. A. R. POWELL.

Electrolytic precipitation of copper. F. F. FRICK and C. E. CARSTENS, Assrs. to ANACONDA COPPER MINING Co. (U.S.P. 1,586,035, 25.5.26. Appl., 23.10.25).—Waste solutions containing copper obtained during the leaching of copper ores are treated with a basic substance to precipitate the copper, and the resulting sludge is leached with acid to give a copper solution from which the metal is deposited electrolytically. A. R. POWELL.

Obtaining electrolytic deposits of chromium. G. LE BRIS (E.P. 243,667, 25.5.25.

Conv., 1.12.24).—A bath for the electrodeposition of chromium comprises a colloidal solution obtained by boiling chromic hydroxide with chromic acid in the molecular ratio of 1:4 and adding a small amount of sodium perborate or other oxidising salt. Deposition is effected at 4–6 volts with a current density of 7–8 amp./dm.² using lead anodes.

A. R. POWELL.

Apparatus for roasting or sintering ores and the like. METALLBANK & METALLURGISCHE GES. A.-G. (E.P. 244,126, 7.12.25. Conv., 6.12.24).—In a roasting furnace of the circular Dwight and Lloyd type ore is introduced into a number of separate compartments resting on the periphery of the rotating table. Each compartment is connected by a pipe with a segment of a central chamber which rotates over a stationary chamber also divided into segments. Suction in the compartments of the fixed chamber may be regulated by valves as desired, e.g., as the ignited ore passes over the different segments in turn, the draught may be increased gradually.

C. A. KING.

Ore-reducing furnace. W. WINKELMAN (U.S.P. 1,588,217, 8.6.26. Appl., 27.6.23).—In a furnace for reducing ores, inverted troughs are supported on shelves projecting from the side walls of the furnace. Means are provided for producing carbon dioxide, for reducing it to a mixture containing carbon monoxide, and for circulating this mixture through the transverse passages formed by the inverted troughs, in contact with a subdivided ore.

C. A. KING.

Manufacture of metals and alloys. D. CROESE (E.P. 252,455, 26.2.25).—Titaniferous iron sand is caused to fall by gravity through a vertical electric furnace so arranged as to allow an unobstructed path for the falling material. The furnace is heated by a vertical series of arcs, the electrodes of any arc being set at an angle to the electrodes of the adjoining arc. Molten metal (titanium-steel) and slag are collected in a crucible at the bottom of the furnace.

C. A. KING.

Melting and re-melting of metals and metallic waste. K. SCHMIDT (E.P. 253,342, 24.3.25).—The walls of the furnace are directly heated and are then caused to come under the metal to be melted. For example, a reverberatory furnace rotating continuously about an axis may be used, each of the furnace walls being successively heated, brought under the metal to be melted, removed from that position, and reheated. A protective covering is maintained over the metal bath.

C. A. KING.

Electrolytic production of metals. E. DUHME, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,567,791, 29.12.25. Appl., 1.11.24).—A small auxiliary cathode is placed near the anode and supplied with alternating current and with direct current at a lower voltage than that supplied to the more remote main cathode. The impurities are deposited on the subsidiary cathode with only a small percentage of the metal, which is mainly deposited on the cathode proper.

E. S. KREIS.

Treatment of minerals. W. O. BORCHERT, Assr. to NEW JERSEY ZINC Co. (U.S.P. 1,585,756, 25.5.26. Appl., 11.12.22).—The mineral pulp is first subjected to a froth-flotation process in which a substantial amount of the colloidal constituent is removed, and is then treated by a non-flotation method of separation.

C. A. KING.

Flotation of ores. H. W. MORSE, Assr. to COMP. DU BOLES (U.S.P. 1,587,789, 8.6.26. Appl., 29.7.25).—A flotation agent consisting of a mixture of an alkali xanthate and an alkali chloride is used.

C. A. KING.

Aluminium alloy and method of manufacture. H. K. RICHARDSON, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,585,786, 25.5.26. Appl., 5.11.23).—Ductile alloys of aluminium and zirconium are made by electrolysis a mixture of unpurified bauxite and zirconium oxide, the quantity of the latter being insufficient to give more than 10% Zr in the alloy.

C. A. KING.

Process of making acid-proof alloys. J. P. KEGG (U.S.P. 1,586,368, 25.5.26. Appl., 28.2.22).—Copper and lead are melted together, the latter being present in such quantity as to segregate. An alkali metal is added to the melt to hold the lead in suspension.

C. A. KING.

Alloy. C. W. HEPPENSTALL, Assr. to HEPPENSTALL FORGE & KNIFE Co. (U.S.P. 1,587,231, 1.6.26. Appl., 7.3.25).—The alloy contains C 1.5–2.25%, Cr 3.5–6%, W 0.5–2%, the remainder being the usual components of commercial steel.

C. A. KING.

Combining molten metal with a gas. H. A. POPPENHUSEN (U.S.P. 1,586,328, 25.5.26. Appl., 26.3.24).—The gas is blown across the open end of a pipe communicating with a supply of molten metal, whereby the metal is drawn up into the pipe by the action of the blast and just sufficient metal is carried forward from the open end of the pipe to react with the blast. The blast is preheated by leading the supply pipes through the main mass of molten metal.

E. S. KREIS.

Refining metals. S. WESTBERG (U.S.P. 1,586,543, 1.6.26. Appl., 21.10.24).—Impurities in ferrous material are removed by heating the material in a reducing atmosphere at normal pressure, in the presence of an alkaline-earth compound, to a welding temperature but below the melting point.

H. ROYAL-DAWSON.

Method of introducing additional agents into metallurgical baths. O. NIELSEN (U.S.P. 1,587,600, 8.6.26. Appl., 4.12.24. Conv., 4.12.23).—Inert gas is introduced into a metallurgical bath with the object of removing air from the pipe line to the bath, and the refining agent is then introduced through the same pipe line, in gaseous, liquid, or solid form.

C. A. KING.

Purification of zinc solutions. T. P. CAMPBELL (U.S.P. 1,587,695, 8.6.26. Appl., 8.9.25).—Impure

acid or alkaline zinc solutions are passed upwards through a narrow pipe in the middle of a closed cylindrical vessel, overflow from the top into a compartment made by standing a vessel in the shape of a truncated cone on a screen disposed just above the bottom of the cylinder, and then pass upwards again between the walls of the cylinder and the cone. All the three compartments are packed with zinc shot or scrap, and by this arrangement the rate of flow of the solution is gradually diminished in its passage through the apparatus.

A. R. POWELL.

Recovery of zinc and lead from zinc-lead ores. SOC. GÉN. MÉTALLURGIQUE DE HOBOKEN (F.P. 602,385, 24.11.24).—Zinc-lead ores are heated in rotating cylindrical furnaces to volatilise lead and zinc oxides, which are subsequently separated by leaching with ammonium carbonate solution. If the ore contains sulphur, sufficient copper material is added to retain the whole of the sulphur in the speiss. If both copper and sulphur are present the proportion of each is so regulated, either by addition of more copper or sulphur, or by roasting, that the whole of the copper and sulphur remain in the residue in the furnace.

A. R. POWELL.

Recovery of nickel from ores. J. J. HISSINK (F.P. 595,275, 18.3.25. Conv., 31.3.24).—Finely-divided nickel ores are extracted with gas liquor in the presence of a current of air. When the ore has previously been heated under reducing conditions, it must be allowed to cool in the presence of air so that it is re-oxidised and the nickel thus rendered soluble in the gas liquor.

L. A. COLES.

Electroplating with cadmium. UDYLITE PROCESS Co., Assees. of M. E. LOUTH and A. W. YOUNG (E.P. 235,159, 30.4.25. Conv., 5.6.24).—See U.S.P. 1,537,047; B., 1925, 554. A concentration of 2.5–10 oz. of free sodium cyanide per gall. of plating solution is specified.

Brass alloy. F. HEUSLER, Assr. to ISABELLENHÜTTE GES.M.B.H. (U.S.P. 1,590,092, 22.6.26. Appl., 29.4.21. Conv., 2.2.17).—See G.P. 303,864; B., 1921, 265 A. The proportions specified are Cu 37, Si 13, Fe 20, Mn 30%.

Iron casting. J. E. FLETCHER and H. J. YOUNG (U.S.P. 1,589,062, 15.6.26. Appl., 18.3.26. Conv., 1.10.24).—See E.P. 245,196; B., 1926, 195.

Desulphurising iron. G. S. EVANS, Assr. to MATHIESON ALKALI WORKS (U.S.P. 1,590,730, 29.6.26. Appl., 23.4.24).—See E.P. 232,963; B., 1925, 962.

Manufacture of metal, more especially magnesium, from the corresponding chloride. C. ARNOLD. From DOW CHEMICAL Co. (E.P. 254,050, 7.5.25).—See U.S.P. 1,567,318; B., 1926, 196.

Silver alloy. F. HEUSLER, Assr. to ISABELLENHÜTTE GES.M.B.H. (U.S.P. 1,590,091, 22.6.26. Appl., 29.3.21. Conv., 13.9.20).—See E.P. 169,144; B., 1922, 298 A.

Condensing apparatus in connexion with an electric furnace for the production of volatile metals. F. JURETZKA (U.S.P. 1,590,521, 29.6.26. Appl., 20.4.23. Conv., 22.4.22).—See G.P. 385,893; B., 1924, 301.

Apparatus [furnaces] for use in the cementation of iron or iron alloys. W. H. FISHER and P. CHAMBERS (E.P. 253,641, 30.3.25).

Increasing the efficiency of Cowper stoves in existing blast-furnace plants. HAUTS FOURNEAUX & ACIÉRIES DE DIFFERDANGE-ST.-INGBERT-RUMELANGE SOC. ANON., and P. RIES (E.P. 249,063, 14.5.25. Conv., 16.3.25. Addn. to 245,058).

Froth-flotation apparatus. MINERALS SEPARATION, LTD. From MINERALS SEPARATION NORTH AMERICAN CORP. (E.P. 253,618, 20.3.25).

Electromagnetic separators for the separation or concentration of minerals. M. KOIZUMI (E.P. 254,030, 3.4.25).

Electro-deposition of metals on wire or narrow strip. J. A. PARKER (E.P. 254,067, 23.5.25).

Pyrometer sheath (E.P. 251,750).—See I.

Electric furnaces (E.P. 239,510).—See XI.

XI.—ELECTROTECHNICS.

Use of electrodes of V2A-steel in electro-analysis. A. SCHLEICHER and L. TOUSSAINT (Z. angew. Chem., 1926, 39, 822–824).—An investigation of the stability of an electrode of V2A-steel towards various acid, alkaline, and saline media, and of the conditions under which the technically important metals may be quantitatively deposited in a weighable form from aqueous solutions containing their salts when the customary platinum cathode is replaced by a gauze of V2A-steel. Since the steel is attacked by hydrochloric and sulphuric acids, determinations in presence of these acids are not possible. The electrode may, however, be used in the presence of nitric acid, a mixture of nitric and sulphuric acids, acetic, oxalic, and tartaric acids, salts of organic acids, cyanides, ammonia, or free alkali. Using such a cathode, copper, bismuth, nickel, iron, antimony, and tin may be determined quantitatively under the usual conditions for the quantitative electrodeposition of these metals on platinum. Silver is deposited from solutions containing nitric acid in the form of relatively large, non-adherent crystals. Excellent results are, however, obtained when the deposition is from alkaline cyanide solutions. Zinc is deposited quantitatively in a compact form from acetic acid solutions but not from alkaline solutions. Cadmium is deposited quantitatively from acetic acid solutions, but has a tendency to form loose crystals, and consequently an electrode with a large surface is necessary. The electrode may not be used for the determination of mercury or in any process involving heating of the

deposited metal or metal oxide, *e.g.*, anodic deposition of lead, manganese, etc. Provided that it is possible to obtain a sufficiently large surface, V2A-steel is an excellent substitute for platinum. The electrode surface is prepared by washing with nitric acid, water, and, finally, methyl alcohol. J. S. CARTER.

Electric furnaces for temperatures up to 3300°. LÖWENSTEIN.—See A., July, 706.

Indurating concrete with sulphur. KOBÉ.—See IX.

Electric properties of condensation products of phenols and aldehydes.—SHONO.—See XIII.

Electrodeposition of rubber. ELLIOTT.—See XIV.

Electrolytic preparation of glycollic acid. TOJA and CEVA.—See XX.

PATENTS.

Electric discharge tubes with rarefied atmospheres. J. B. J. M. ABADIE and N. M. COURTINES (E.P. 230,467, 4.3.25. Conv., 4.3.24).—The pressure inside an electric discharge tube with a rarefied atmosphere functioning with high frequency or high tension may be maintained constant by providing a substance adapted to emit a vapour or gas corresponding to the atmosphere of the tube. The required vapour tension may be reached at ordinary temperatures or, if at higher temperatures, the heating is effected by cathodic bombardment, the substance being placed in a hollow electrode provided for the purpose. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 225,890, 210,728, 5557 of 1915, 12,256 and 1218 of 1913, and 29,839 of 1912.) M. E. NOTTAGE.

Electric discharge vessels, tubes, and the like. E. F. HUTH GES. FÜR FUNKENTELEGRAFIE M.B.H. (E.P. 230,492, 9.3.25. Conv., 8.3.24).—An electron-emitting cathode, in an electric discharge vessel of the dull emitter type, if made of an alloy of molybdenum and thorium, is capable of emitting electrons without any heat treatment being employed for rendering it active. An electrode of molybdenum is mounted in the discharge vessel and heated therein for a short time before or after being coated with thorium nitrate, the electrode being produced without the use of a binding or reducing agent. The electron emission of this electrode takes place at comparatively low temperatures, *e.g.*, about 1200°, whereby its life is prolonged; also the heating current is smaller than that required for heating a tungsten or tungsten-thorium cathode of the same specific emissivity. M. E. NOTTAGE.

Making sag-resisting bodies [filaments] of tungsten. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of S. DUSHMAN and P. A. KOBER (E.P. 244,116, 4.12.25. Conv., 8.12.24).—Change of structure and shape of coiled tungsten filaments at the high temperatures attained in gas-filled incandescence

lamps and similar devices may be prevented by heating the filaments to within the temperature range of rapid grain growth, while supported so that every part is free from stress great enough to cause deformation. M. E. NOTTAGE.

Leading-in wire for glass vessels [electric lamp bulbs etc.]. E. FRIEDERICH, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,588,179, 8.6.26. Appl., 30.8.21. Conv., 10.2.16).—The wire is composed of an alloy of iron and tungsten having a coefficient of expansion approximating to that of glass. C. O. HARVEY.

Electric furnaces. SIEMENS & HALSKE A.-G. (E.P. 239,510, 28.8.25. Conv., 2.9.24).—In an electric induction furnace for smelting metals the heating body, which forms part of the secondary winding of a transformer, is in the form of an annular hollow pipe of rectangular cross-section, and consists, on the inner side, of a fireproof material, *e.g.*, silit, and on the outer side of a metal. The silit walls are made up of separate plates which may be brought close together and insulated from one another. This hollow pipe completely encloses the primary winding which consists of copper pipes, through which water circulates constantly, wound on an annular iron core made up of plates; by this arrangement no appreciable currents are induced in the material to be treated. The primary winding is divided into sections which can be connected in series or in parallel, thus facilitating regulation of the furnace. The metal to be smelted is placed in the centre of the annulus in a crucible; or, alternatively, the inner wall of the annulus formed by the heating body may form part of the receptacle. M. E. NOTTAGE.

Electric furnaces. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of C. L. IPSEN (E.P. 241,897, 19.10.25. Conv., 23.10.24).—In an electric resistor furnace with a heat-refractory wall forming the heating chamber, the highest possible safe operating temperature, without injury to the heating resistor, may be obtained by mounting the resistor directly in the heating chamber. The resistor, *e.g.*, a nickel-chromium alloy, is bent or cast into a series of convolutions, the upper bends of which are secured, by means of metallic rings, to a bar made of insulating material fixed near the top of the furnace. The lower bends are secured in the same way, but the bar is not fixed to the furnace wall so that the resistor is free to expand or contract; also, the weight of this bar keeps the resistor straight. M. E. NOTTAGE.

Flame-proof insulating material. WESTERN ELECTRIC CO., LTD. FROM WESTERN ELECTRIC CO., INC. (E.P. 252,856, 1.5.25).—The material is made by intimately mixing together 25 pts. of chlorinated naphthalene (preferably tetrachloronaphthalene), 20 pts. of rubber, 33 pts. of powdered silica, 2 pts. of sulphur or other vulcanising agent, and 10 pts. of litharge or other substance capable of accelerating vulcanisation. The resulting mixture is sufficiently plastic to be moulded or to be extruded upon an electrical conductor in strand form. After being

moulded or extruded it is subjected to heat and pressure to vulcanise the rubber. Chlorinated naphthalene will not decompose or burn unless subjected to an intense heat, and silica has a high thermal conductivity. M. E. NOTTAGE.

Electrolytic tank. L. ARNALOT (U.S.P. 1,586,435, 25.5.26. Appl., 5.12.23).—An electrolytic tank is connected with a refrigerator and a receiving tank, liquid being passed from the receiving tank, through the refrigerator, and thence to the electrolytic tank, and back from the electrolytic tank to the receiving tank by means of the gas pressure generated within the electrolytic tank. M. E. NOTTAGE.

Production of gas by electrolysis. R. L. WALSH (U.S.P. 1,588,214, 8.6.26. Appl., 29.6.21).—The current is supplied through a number of electrodes. Means are provided for depolarising the electrodes simultaneously by washing, and for regulating the current density. C. O. HARVEY.

Rendering a porous powder [used in an electric cell] impermeable to liquids. R. OPPENHEIM, Assr. to Soc. ANON. LE CARBONE (U.S.P. 1,588,607, 15.6.26. Appl., 10.4.24. Conv., 27.12.23).—See E.P. 226,769; B., 1925, 555.

Galvanic battery. R. OPPENHEIM, Assr. to Soc. ANON. LE CARBONE (U.S.P. 1,588,608, 15.6.26. Appl., 20.5.24. Conv., 10.4.24).—See E.P. 230,307; B., 1925, 364.

Portable ozonisers. E. L. R. CAILLIET and M. L. J. BOURDAIS (E.P. 246,841, 26.1.26. Conv., 29.1.25).

Electrical apparatus for generating ozone. J. McBLAIN (U.S.P. 1,588,976, 15.6.26. Appl., 12.4.24. Conv., 24.4.23).—See E.P. 219,387; B., 1924, 795.

Metallic compositions. [Lead amalgams for storage-battery plates.] A. MILLER (E.P. 254,209, 12.1.26).—See U.S.P. 1,570,763; B., 1926, 496.

Electrolytic production of hydrogen peroxide (F.P. 603,043).—See VII.

Electrolytic deposits of chromium (E.P. 243,667).—See X.

Aluminium alloy (U.S.P. 1,585,786).—See X.

Electrolytic production of metals (U.S.P. 1,567,791).—See X.

Electrometric determination of acidity or alkalinity of soils, waters, etc. (G.P. 413,043).—See XVI.

XII.—FATS; OILS; WAXES.

Oil extraction in theory and practice. L. C. WHITON (Ind. Eng. Chem., 1926, 18, 605—606).—An examination of the practical working of extraction plants shows that many features theoretically excellent are undesirable. Simplicity of construction

with absence of movable parts, units of small size, and individual extractors as opposed to a series arrangements are advocated. D. G. HEWER.

Olive oil. I. F. TRAIETTA-MOSCA (Annali Chim. Appl., 1926, 16, 169—177).—The best olive oil, as regards aroma, fluidity, and stability, is obtained from slightly immature olives, which give also a good yield. If the fruit is too unripe, the oil is bitter and the yield poor; fully ripe olives furnish good oil, but fruit left too long on the trees produces a dense, discoloured oil which readily turns rancid. Between gathering and pressing the olives should be kept spread in thin layers in a cool place, since if heaped or trenched they become heated to 35—50° and undergo such fermentative changes that the resulting oil is of bad odour and persistently turbid. A sample of oil from worm-eaten and fermented olives contained lipolytic and proteolytic enzymes and yielded three varieties of *Saccharomyces*, the morphological characters of which are described. As regards analytical results, the most important differences between oil from sound and that from altered olives lie in the total acidity and in the volatile and soluble acid value. For good oils the acid value ranges from 1.02 to 1.22, whereas with the altered oils it varies between 3.04 and 5.58; the volatile and soluble acid value is 1.10—1.15 and as high as 3.6 in the two cases. T. H. POPE.

Sensitive reaction for olive oil extracted with carbon disulphide. P. SACCARDI (Giorn. Chim. Ind. Appl., 1926, 8, 11).—In this reaction use is made of a filtered solution of 5 g. of lead soap in 100 c.c. of benzene, which keeps well, and of a solution of 30 g. of potassium hydroxide in 100 g. of 95% alcohol, this being filtered and stored in glass-stoppered bottles. If 1 c.c. of an oil which has been obtained by extraction with carbon disulphide is heated to boiling with 1 c.c. of the alcoholic potassium hydroxide solution and 1 c.c. of the lead soap solution, intense blackening, followed by precipitation of lead sulphide, takes place. This method detects about 20 g. of the so-called sulphur oil per litre of olive oil, or, if the oil is shaken with 25% of 95% alcohol and distilled, and the distillate tested, about 10 c.c. of the sulphur oil per litre. This test is not invalidated if the oil is previously either heated at 130° or treated with superheated steam for 2 hrs. Traces of carbon disulphide vapour can also be detected in this way. T. H. POPE.

Oil extracted from the head of a dolphin. H. MARCELET (Compt. rend., 1926, 182, 1416—1417).—The oils obtained from (1) the maxillary glands, (2) the nose, and (3) the head of a dolphin (*Delphinus delphis*, Lin.), although generally similar in type, exhibited well-marked differences, e.g., d_{4}^{15} (1) 0.9206, (2) 0.9308, (3) 0.9330; n_D^{17} (1) 1.4548, (2) 1.4640, (3) 1.4790; saponif. value (1) 267, (2) 259, (3) 212; iodine value (Wijs) (1) 17, (2) 56, (3) 133; soluble volatile acids expressed as Reichert value (1) 145.3, (2) 111.3, (3) 39.1; unsaponif. matter (1) 16.30%, (2) 6.07%, (3) 1.77%; solid

fatty acids (1) 30.82%, (2) 19.28%, (3) 10.08%; liquid fatty acids (1) 18.70%, (2) 43.17%, (3) 74.04%.

H. J. EVANS.

Detection of α - in presence of β -palmitodistearin. [Detection of tallow etc. in lard.] F. J. F. MUSCHTER and G. VISSER (Chem. Weekblad, 1926, 23, 250—252).—The presence of tallow and other fats in lard can be detected with more certainty by microscopical examination than by determination of the Boemer value (cf. B., 1926, 637). Staining with Romanowsky-Giemsa solution (azur-eosin in alcohol) greatly facilitates the detection of the isomeric palmitodistearins. Many photomicrographs are reproduced.

S. I. LEVY.

Determination of milk fat in mixtures of fats. J. GROSSFELD (Z. Unters. Lebensm., 1926, 51, 203—213).—The determination of the butyric acid number, from which the proportion of milk fat in a mixture can be calculated (cf. B., 1926, 447), is influenced by the solubility of the butyric acid in the non-aqueous phase, especially in liquid or molten insoluble fatty acids. Hence in the case of oils containing a considerable proportion of liquid fatty acids, it is preferable to remove these as magnesium soaps by means of magnesium sulphate and then add a corresponding quantity of fatty acids (in the form of soap) having a butyric acid number 0, before determining the butyric acid number.

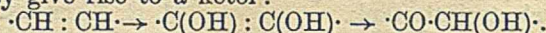
B. W. CLARKE.

Detection of linseed oil in soya bean oil. J. F. CARRIÈRE (Chem. Weekblad, 1926, 23, 274—279).—Whilst the iodine value or the hexabromide number alone may not be sufficient, a consideration of the two together will serve for the detection. The hexabromide number is best determined on the oil itself, not on the free fatty acids, and a suitable procedure is described. For linseed oils the relation between the iodine value, I , and the hexabromide number, H , is given from many determinations as $I - 126.29 = 1.574H$, whilst for soya bean oil the corresponding relation is $I - 126.19 > 12H$. Formulæ are deduced from these relations by means of which the linseed oil content of a mixture may be determined from the values I and H within moderate limits, provided marine animal oils be not present.

S. I. LEVY.

Chemistry of drying oils. III. G. W. ELLIS (J.S.C.I., 1926, 45, 193—199 T; cf. B., 1925, 768, 930).—A further study of linoxyn from linseed oil and of the oxy-acids obtained from the unsaturated acids of this oil is described. On both products a number of determinations of the hydroxyl groups and of the ketone groups were carried out by means of Grignard's reagent and phenylhydrazine respectively. The number of groups obtained were calculated to the formula $C_{57}H_{96}O_{20}$ representing, or closely approximating to, that previously assigned to linoxyn, whilst in the case of the oxy-acids they were calculated to a mixture representing 1 mol. of oxylinolenic acid to 2 mols. of oxylinoic acid. The results showed that linoxyn contained about

7 hydroxyl groups and the mixed oxy-acids about 10 hydroxyl groups. Since the reagent reacts with acidic hydroxyl groups the two series of results were in agreement. The number of ketone groups present in linoxyn and in the oxy-acids amounted to 6 or 7. Hence the 14 atoms of oxygen introduced during the autoxidation process were, for the most part, accounted for. The explanation offered for the presence of these groups is that during the autoxidation process an atom of oxygen is introduced between each ethylene carbon and its accompanying hydrogen atom, with formation of an enol grouping which may give rise to a ketol:



The enol forms explain the susceptibility of linoxyn and of the oxy-acids to hydrolysis with the production of aldehydes and acids and the resinous products of the former. Other properties of linoxyn such as its behaviour towards solvents and its ready solubility in the molten phenols are given in evidence for the presence of the atomic grouping C-OH. Possible formulæ for oxylinoic and oxylinolenic acids are suggested.

Hydrogenation of cottonseed oil with platinum. Heterogeneous catalysis. III. A. S. RICHARDSON and A. O. SNODDY (Ind. Eng. Chem., 1926, 18, 570—571).—Hydrogenation of refined cottonseed oil between 40° and 240° with platinum catalyst is not so selective as with nickel catalyst (B., 1924, 564; 1925, 137), although the proportion of stearic acid formed is substantially less than would be expected if hydrogenation of linoleic to oleic and of oleic to stearic acid proceeded continuously at rates proportional to the concentration of the reacting acids. Partial hydrogenation with platinum to a given point would produce a more readily oxidisable product than with nickel. Rise of temperature was found to favour selective hydrogenation.

D. G. HEWER.

Detection of traces of nickel in hardened fats. M. WAGENAAR (Pharm. Weekblad, 1926, 63, 570—575).—The fat is melted in a small porcelain basin, and a strip of (analytical) filter paper, rolled into a cylinder round a thin platinum wire, is inserted vertically to serve as a wick, at the top of which the fat is burned. When no fat is left, the paper is burned to ash in a silica or platinum crucible, and the residue damped with acid and tested for nickel with dimethylglyoxime. 0.03 mg. in 30 g. of fat can be detected with certainty.

S. L. LEVY.

Determination of concentration of liquid soaps by the immersion refractometer. L. F. HOYT and A. VERWIEBE (Ind. Eng. Chem., 1926, 18, 581—582).—The Zeiss immersion refractometer is particularly adapted for the routine control analysis of liquid soaps of known fat composition. In the case of aqueous solutions of potassium soaps made from various thoroughly saponified fats the refractive index was found to be directly proportional to the content of total solids, which may be rapidly calculated to an accuracy of 0.05%. The refractometer can be

read to 0.1°, which corresponds to about 0.025% of soap solids, and accuracy is obtained to the limit of the readings (20—22% of total solids). The slope of the curve of concentration versus the refractive index of the liquid potassium soaps of various oils (cottonseed, maize, olive, linseed) is proportional to the refractive indices of the oils whether measured at 20° or 40°, but the corresponding results with soaps prepared from fatty acids are not so consistent. The concentration of the total solids may be calculated from the equation $y = ax + k$, where y is the refractive index at 20°, x the % of soap solids, k is 1.33299 (the refractive index of distilled water at 20° on the Zeiss instrument), and a is the numerical value of the slope, ranging from 1.425×10^{-3} for coconut oil to 1.561×10^{-3} for linseed oil. D. G. HEWER.

See also A., July, 672, Relative concentrations of various electrolytes required to salt out soap solutions (MCBAIN and PITZER). 712, X-Ray identification of higher fatty acids (MORGAN and HOLMES); Stereoisomerism of ethylenic acids. Hydrogenation of stearic and behenic acids (GONZALEZ).

Measuring viscosity at higher temperatures. MERCER.—See II.

Fatty acids associated with maize starch. TAYLOR and LEHRMAN.—See XVII.

PATENT.

Centrifugal liquid purifiers (E.P. 252,421).—See I.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Analysis of Prussian blues. T. H. BOWLES and J. F. HIRST (J. Oil and Colour Chem. Assoc., 1926, 9, 153—156).—A scheme for the complete analysis of Prussian blue is recommended. It comprises determinations of ferric iron (cation), ferrocyanide radical, alkalis, total iron, hygroscopic and combined water, and water-soluble impurities. The results of analyses of 5 English, 1 German, and 2 American samples are given, and the influence of the alkalis present is discussed, the combined water content of potash and ammonia blues being considerably lower than that of the soda blues (of inferior colouring power). The determination of the iron-cyanogen complex is a more satisfactory criterion of the value of a Prussian blue than is the ash. Support is given to the view that the pigment is mainly ferric ferrocyanide rather than ferrous ferrocyanide. S. S. WOOLF.

Titanium white. C. P. VAN HOEK (Farben-Ztg., 1926, 31, 2296—2298).—A summary of the hitherto published information on the chemical and physical properties of the mixtures of titanium dioxide and barium sulphate sold as titanium white. A comprehensive bibliography is given. S. S. WOOLF.

Alteration of paint films on heating. H. WOLFF and G. ZEIDLER (Korrosion u. Metallschutz, 1926, 95; Farben-Ztg., 1926, 31, 2300).—Tensile

strength and extension determinations were carried out on detached paint films that were heated at 80° for various lengths of time. In all cases the tensile strength per gram increased and the percentage extension decreased on heating. In the case of films of white lead and lithopone paints the tensile strength first rose rapidly, then much more slowly, until the film became brittle and crumbled. Zinc white and red oxide of iron films, on the other hand, showed a steady rise to a maximum, after which a loosening of the structure took place, the tensile strength falling with eventual disintegration of the film. These phenomena are attributed to colloid-chemical changes in the film rather than to the chemical nature of the pigments used.

S. S. WOOLF.

Protective paint coatings. J. N. TERVET (J. Oil and Colour Chem. Assoc., 1926, 9, 157).—The exposure panel previously described by the author—a steel panel coated with metallic lead primer, followed by green paint (cf. J.S.C.I., 1925, 28 T)—has been re-examined after a further exposure of 18 months, making 3 years 3 months in all. Complete protection is still afforded by the coating as metallic lead is seen in close contact with the steel when the film is broken. While rust is apparent where the steel has been exposed in previous examinations, there has been no creeping of the rust under the film of primer. S. S. WOOLF.

Effect of various carbon pigments upon the rate of oxidation of linseed oil. F. H. RHODES and H. E. GOLDSMITH (Ind. Eng. Chem., 1926, 18, 566—570).—The marked inhibition of the oxidation of raw linseed oil which occurs on addition of lampblack or carbon black is due to the continuous adsorption of the intermediate oxidation product which is the true catalyst in the drying reaction. With paints prepared with cobalt drier the inhibiting effect of carbon is more marked on keeping, as the pigment adsorbs the cobalt drier; with lead drier oxidation is nearly entirely inhibited during the first hours of exposure, as the small amount of auto-catalyst originally present in the oil or produced during the initial period appears to be adsorbed, as well as a large part of the lead drier; with manganese drier the rate of oxidation is scarcely affected by carbon pigments. D. G. HEWER.

Capillary-analysis and its application to the examination of resins. E. STOCK (Farben-Ztg., 1926, 31, 1903—1904, 1959—1960, 2133—2134, 2187—2188, 2240—2243).—On drying filter-paper strips that have been partially immersed in resin solutions for periods up to 24 hours, a series of "adsorption-pictures" result, showing strata of different colour, extent, intensity, opacity, etc. When this test is carried out under specified conditions in respect of dimensions and quality of filter-paper, strength of solution, depth and time of immersion, size of vessel, temperature, relative humidity, etc., the critical examination of the zones in the adsorption-pictures obtained furnishes information as to the identity and purity of the resin, the existence of

admixed resins, whether advantageous or adulterant, etc. A series of 48 reproductions (in black and white) of the results of tests on resins and resin mixtures is given, with full descriptions in the text.

S. S. WOOLF.

Condensation products of phenols and aldehydes. III. Electrical properties. T. SHONO (J. Soc. Chem. Ind. Japan, 1926, 29, 121—126).—The surface colour of the phenol-formaldehyde condensation product prepared by using ammonia and potassium hydroxide as the condensing agents under ordinary pressure is darkened by exposure to an oxidising atmosphere, especially on heating. The mechanism of the condensation seems to be different according to the nature of the condensing agents used. When the condensation product is heated in an oxidising atmosphere, the weight and volume of the material diminish, and the interior colour fades; the capacity for acquiring an electric charge increases nearly to the same degree as the deepening of the surface colour, and the dielectric strength is increased. (Cf. B., 1926, 595.)

K. KASHIMA.

Chemistry of drying oils. ELLIS.—See XII.

PATENTS.

Titanium pigments. J. BLUMENFELD and M. MAYER (E.P. 252,262, 28.11.24).—Titanium oxide is suspended in a solution containing one of the rare earths of the titanium-thorium group (*e.g.*, the solution of titanium sulphate obtained by treating ilmenite with sulphuric acid). On heating, oxides or hydroxides are precipitated on to and seal the pores of the titanium oxide base. The titanium pigment so produced does not "chalk" on weathering. An alternative method is to mix the titanium oxide with an aqueous colloidal solution of oxide of zirconium, thorium, or titanium, and then evaporate the water. After neutralising any acids present the mixed product is calcined at red heat.

S. S. WOOLF.

Fillers for [lake] pigment colours, rubber, etc. W. EBERLEIN, and COLLOISIL COLOUR CO. (E.P. 252,416, 16.1.25).—A colloidal suspension of a colour-fixing earth, such as fuller's earth, green earth, white earth, bentonite, or any natural or artificial silicate that absorbs and fixes basic dyes, is mixed with an organic substance (excluding basic dyes), *e.g.*, amino- or hydroxy-compounds, oils, fats, waxes. After drying a non-oily, fine, soft powder is obtained which still retains the property of fixing basic dyes and may be used as a filler for lake pigments, rubber, etc.

S. S. WOOLF.

Treatment of zinc white or similar metallic oxides, lithopone, and white lead for use in the preparation of paint. A. VAN LERBERGHE (E.P. 252,756, 2.1.25).—The pigment in the form of an aqueous paste is ground with a drying oil containing free fatty acid, whereby the excess of water separates and saponification occurs with formation of metal soaps. For example, 220 lb. of zinc oxide are ground

in 155 lb. of water for $\frac{1}{2}$ hr., 30 lb. of drying oil containing 6—7% of admixed linoleic acid are added, and grinding is continued for a further $\frac{1}{4}$ hr. The resulting zinc linoleate paste is crushed to a smooth non-setting paste capable of being readily diluted for use in paint.

S. S. WOOLF.

Self-disintegrating metallic compounds for use in anti-fouling paints. W. P. HESKETT and H. B. MOLESWORTH (E.P. 252,766, 26.2.25).—One or more of the metals specified in E.P. 158,740 (B., 1921, 224A) is fused with one or more of the compounds of these metals, to give a self-disintegrating material which may be incorporated in a suitable medium to form an anti-fouling paint, since it is decomposed by water to yield a poisonous gas. A typical mixture is iron oxide, iron sulphide, and aluminium, which will evolve hydrogen sulphide. On introducing antimony or arsenic or arsenical pyrites into the mixture, stibine or arsine is produced together with the hydrogen sulphide. The material may be pulverised mechanically and converted into paint by means of one of the carriers described in E.P. 226,286 (B., 1925, 107).

S. S. WOOLF.

Composite [green] pigment. H. DOURIF (U.S.P. 1,587,704, 8.6.26. Appl., 1.4.24).—Solutions of a chromate and of a metal salt capable of reacting therewith to form an insoluble chromate are added to a non-settling suspension of ultramarine blue, whereupon a composite pigment is precipitated.

S. S. WOOLF.

[Rubber] paint. G. A. SHINE (U.S.P. 1,588,150, 8.6.26. Appl., 3.6.25).—Devulcanised old rubber is fused with fossil resin, with or without the addition of hydrated lime, and the whole dissolved in a rubber solvent. 10% by weight of zinc sulphate may be added to the thinned mixture.

S. S. WOOLF.

Polymerising vinyl esters. CONSORTIUM FÜR ELEKTROCHEM. IND., Assees. of W. O. HERMANN and E. BAUM (U.S.P. 1,586,803, 1.6.26. Appl., 8.8.25. Conv., 13.8.24. Cf. G.P. 281,687—8; B., 1915, 623).—Vinyl esters are readily polymerised to clear hard resins by heating under pressure with water and a catalyst consisting of an organic or inorganic peroxide, a per-salt, a per-acid, or a mixture of such compounds. The action of hydrogen peroxide and of organic peroxides is increased by addition of alkaline substances, and the properties of the product may be modified by performing the reaction in presence of an organic solvent such as glycol, glycerol, paraldehyde, or ethylidene diacetate. Polymerisation may also be effected by heating the vinyl ester with water alone under pressure or by treating it with water in presence of radiant energy.

T. S. WHEELER.

Process of treating resins. C. F. WILLARD (U.S.P. 1,587,622, 8.6.26. Appl., 21.5.24).—Resin is heated with glycerol, rubber, and a suitable catalyst until the resin has melted and combined with the rubber and glycerol to form a resin gum ester.

D. F. TWISS.

[Paracoumarone] varnish. W. W. KING, Assr. to BARRETT Co. (U.S.P. 1,587,333, 1.6.26. Appl., 9.2.21).—A drying oil is heated to about 240°, paracoumarone resin is added, and the temperature is raised to a higher degree and maintained thereat until the mixture will set when cooled; further paracoumarone is then added. S. S. WOOLF.

Manufacture of titanin and zinc compounds [pigments]. P. PIPEREAUT and A. HELBRONNER (U.S.P. 1,590,697, 29.6.26. Appl., 21.11.23. Conv., 25.11.22).—See E.P. 207,555; B., 1925, 107.

Sulphur phenol resins. SOC. OF CHEM. IND. IN BASLE, Asses. of A. BLUMFELDT (U.S.P. 1,588,439, 15.6.26. Appl., 31.7.23. Conv., 1.9.22).—See E.P. 203,310; B., 1924, 566.

[Control of stirrer in] apparatus employed in synthetic resin condensation processes. BRIT. THOMSON-HOUSTON Co., LTD., H. W. H. WARREN, R. NEWBOUND, and L. M. T. BELL (E.P. 253,614, 20.3.25).

Apparatus for testing the viscosity of fusible materials [shellac]. METROPOLITAN-VICKERS ELECTRICAL Co., LTD. FROM WESTINGHOUSE ELECTRIC AND MANUF. Co. (E.P. 254,239, 26.3.26).

Colour lakes (U.S.P. 1,587,435).—See IV.

Obtaining hydrogen peroxide and *blanc fixe* (E.P. 252,768).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Application of disinfectants used in the cultivation of rubber. A. STEINMANN and J. J. B. DEUSS (Comm. Centr. Rubber, Buitenzorg, 1926, [3], 159—197; Arch. Rubbercultuur, 1926, [5]).—Protection of rubber trees against infectious diseases is commonly effected by the application of certain forms of tar or preparations of tar. Various types of these disinfectants are described together with methods for their examination in the laboratory and on the tree.

D. F. TWISS.

Specific gravity of *Hevea latex*. V. O. DE VRIES (Comm. Centr. Rubber Stat. Buitenzorg, 1926, [53], 1—23; Arch. Rubbercultuur, 1926, [1]).—It is probable that all earlier statements as to *Hevea latex* with a specific gravity exceeding that of water are in error. Undiluted latex, the rubber content of which, because of heavy tapping or other physiological factors, decreases below 17%, may actually show a specific gravity exceeding 1. For example, latex from one tree contained 14% of rubber and less, and its specific gravity reached 1.004 and higher. With small deviations the specific gravity accorded with that expected for a mixture of serum and rubber with specific gravity 1.020 and 0.914 respectively.

D. F. TWISS.

Preservation of [rubber] latex with ammonia combined with other means. O. DE VRIES (Comm. Proefstat. Rubber, Buitenzorg, 1926, [2],

149—157; Arch. Rubbercultuur, 1926, [4]).—It is not possible to effect an economy by using a reduced proportion of ammonia to neutralise natural acidity together with some additional means of checking the growth of micro-organisms. Examination was made of the effect of heat, formalin, and potassium cyanide respectively on latex containing less ammonia than customary. D. F. TWISS.

Native rubber. W. SPOON (Comm. Centr. Rubber Stat. Buitenzorg, 1926, [54], 29—106; Arch. Rubbercultuur, 1926, [2]; cf. B., 1925, 891).—A summarised report on the examination of 253 samples of "native" rubber from various parts of the Outer Provinces of the Dutch East Indies. D. F. TWISS.

Molecular magnitude of caoutchouc and gutta-percha. E. OTT (Naturwiss., 1926, 14, 320; Chem. Zentr., 1926, I, 3400).—The extensibility of rubber is not necessarily evidence of the simultaneous presence of an amorphous and a crystalline phase. By X-ray investigation the maximal formula for caoutchouc is found to be $(C_5H_8)_6$. Gutta-percha is also found to be crystalline and to have a maximal formula $(C_5H_8)_{12}$. D. F. TWISS.

Constitution of high polymerides. E. GEIGER (Diss., Zurich, 1926; Gummi-Ztg., 1926, 40, 2143—2144).—In the formation of the colloidal condensation products of dimethylketen with aryl-carbimides, the primary complexes, which are probably of open-chain structure, combine to form groups containing 5—9 of the initial complexes. Rubber when heated in ether at 250° yield an amorphous polycyclo-compound (*d* 0.992) containing one ethylene linkage to 5 isoprene nuclei; the product from gutta-percha is identical with this. In the pyrogenic decomposition of rubber (or gutta-percha) the more volatile products such as isoprene and dipentene arise from the decomposition of the caoutchouc molecules, whereas the less volatile products obtained at a somewhat higher temperature, result from the decomposition of polycyclocaoutchouc (or polycyclogutta) which has been formed previously. On hydrogenation at 270° rubber gives a mixture of two saturated compounds derived from caoutchouc and polyhydrocaoutchouc respectively; gutta-percha and balata hydrocarbons appear to yield identical products. Similarly metastyrene is reducible to hydrometastyrene without resolution into styrene. These results indicate that chemical valencies must be involved in linking the primary complexes to produce the colloid. Caoutchouc tetrabromide reacts with triphenylphosphine forming amorphous products which are strongly ionised in solution and undergo double decomposition with an alkali picrate. The oxidation products of rubber and of hydrocaoutchouc also support the view that the colloidal structure of these substances is dependent on chemical valency. The earlier observation (Klein and Stamberger, B., 1925, 462) that rubber, after being milled, gives solutions containing particles visible in the ultra-microscope, is incorrect. D. F. TWISS.

Electrodeposition of rubber. F. A. ELLIOTT (Chem. Met. Eng., 1926, 33, 358—359).—With electrodes 4 in. apart, voltage 110, and current density at anode 0.4 amp./sq. in., a bath mixture of rubber (from latex) 8.0 grms., sulphur 0.3, zinc oxide 1.5, whiting, 4.5, carbon black 0.3, paraffin wax 0.3, tetraethylthiuram disulphide 0.03, gum arabic 0.075, and ammonia 0.16 g. per 100 c.c., gave in one minute a deposit 0.03 in. thick, of practically the same composition as the total solids of the bath. When dried and vulcanised the product was at least as tough and strong as an ordinary product of similar composition. The possibilities of the Sheppard and Eberlin process (cf. B., 1925, 681; also U.S.P., 1,580,795; B., 1926, 639) are reviewed.

D. F. TWISS.

Reaction between caoutchouc and sulphur. G. S. WHITBY and R. S. JANE (Trans. Roy. Soc. Canada, 1926, [iii], 20, III, 121—131).—Experiments carried out at various temperatures, in various solvents, and in the presence and in the absence of accelerators, show that, however prolonged be the period of heating, and however great the excess of sulphur used, the reaction product, after being freed from uncombined sulphur by the usual method of exhaustive acetone extraction always contains about 32% S, corresponding with $(C_5H_8S)_n$. Accelerators have a much smaller effect on the rate of vulcanisation in solution than when used in a dry rubber-sulphur mixture. Removal of protein and resin from rubber reduces the rate of combination with sulphur but is without effect on the 32% limit. Prolonged extraction of the caoutchouc-sulphur reaction product with alcoholic alkali gradually removes sulphur, the rate of removal being different for products obtained in different solvents; products obtained in halogenated aromatic hydrocarbons are more resistant than those obtained in aromatic hydrocarbons. After exhaustive extraction with fairly concentrated alcoholic alkali the products still contain about 19% S. The caoutchouc-sulphur reaction product cannot bring about vulcanisation, Ostromislenski's theory (B., 1916, 370) being untenable.

J. S. CARTER.

Latex viscosimeter. R. DITMAR (Chem.-Ztg., 1926, 50, 497—498).—A viscosimeter for rapid measurements of the viscosity of rubber latex consists of a wide glass tube, graduated at 100 and 200 c.c., slightly expanded at the top to hold a ring of cloth (which serves to filter off any coagulated rubber), and tapering at the bottom to a narrower tube which is attached by means of a rubber tube, fitted with a pinchcock, to a tube drawn out to a fine jet, the diameter of which varies according to the type of latex under examination (1 mm. for natural 38% latex). The time of flow of the first or second 100 c.c. is compared with the corresponding time for water in the usual manner.

J. W. BAKER.

PATENTS.

Vulcanisation of rubber. BRIT. DYESTUFFS CORP., C. J. T. CRONSHAW, and W. J. S. NAUNTON (E.P. 253,197, 12.3.25).—The dixylylguanidines,

particularly the three di-*m*-xylyl compounds, are more active accelerators of vulcanisation than the lower homologues hitherto used. D. F. TWISS.

Removing sulphur from vulcanised rubber. L. L. ODOM (U.S.P. 1,587,408, 1.6.26. Appl., 7.1.25).—Material containing vulcanised rubber is distilled through a heated substance having a stronger affinity for sulphur than rubber.

D. F. TWISS.

Rubber-vulcanisation accelerator. W. SCOTT, Assr. to RUBBER SERVICE LABORATORIES CO. (U.S.P. 1,586,121, 25.5.26. Appl., 9.9.25).—The compounds obtained by the condensation of 2 mols. of a secondary amine such as piperidine, diethylamine, tetrahydro-2-methylquinoline, or piperazine with 1 mol. of an aldehyde, e.g., formaldehyde, acetaldehyde, acraldehyde, or aldol, may be further condensed with 1 mol. of carbon disulphide to yield products which are of value as accelerators for either hot or cold vulcanisation of rubber. The substance $CH_2(C_5H_{10}N)_2$ formed by the condensation of piperidine and formaldehyde forms an oil, b.p. 237°; it reacts with 1 mol. of carbon disulphide to yield a crystalline compound. Dibenzylamine and formaldehyde yield a substance, m.p. 99°, which condenses with carbon disulphide to give a compound, m.p. 64°. 2 mols. of diethylamine and 1 mol. of formaldehyde give bisdiethylaminomethane, which yields a liquid product on treatment with carbon disulphide.

T. S. WHEELER.

Fillers for rubber (E.P. 252,416).—See XIII.

Rubber paint (U.S.P. 1,588,150).—See XIII.

XV.—LEATHER; GLUE.

New and old theories of the bating process. R. H. MARRIOTT (J. Soc. Leather Trades Chem., 1926, 10, 132—142; cf. Wilson, B., 1921, 92 A).—Experiments have shown that skins can be limed until only traces of elastin remain and the skin can be fallen, but yet not properly bated, the grain not being silky nor the skin flaccid. Ammonia tends to give flaccidity, by causing the separation of fibres into fibrils. The mere production of minimum swelling and the separation of fibres into fibrils, however, does not suffice to produce flaccidity and it is probable that it is necessary to remove degraded collagen also. The porosity of a bated skin seems to be connected with the cleansing of the hair follicles rather than with the mere splitting of the fibres. The elastin theory of bating does not account for the supple nature of the tanned skin, and the latter is only partially dependent on the degree of plumpness of the fibres during the tanning process. Excellent leathers can be obtained which contain all their elastin. There appears to be a connexion between the flaccidity and resilience of the bated skin and the softness of the resulting leather. Ammonia does not, but trypsin does, digest elastin, produce a silky grain, give porosity, and a more flaccid skin and rapidly hydrolyses.

degraded collagen. Experiments show that during the liming process the collagen of the fibres is altered so that they begin to stain similarly to gelatin. During the bating this property is destroyed. The degraded form of collagen does not dissolve entirely in warm water. The presence of a degraded form of collagen in the skin tends to stiffness in the leather. The hydrolysis of collagen to peptone takes place in at least four steps, α -collagen \rightarrow β -collagen \rightarrow gelatin \rightarrow proteose \rightarrow peptone. Pepsin can readily change α -collagen into β -collagen, but it cannot hydrolyse β -collagen so rapidly. Trypsin cannot hydrolyse α -collagen, but rapidly dissolves the β -form. β -Collagen is probably produced during the liming process and although it may be dissolved by the lime liquor, more is produced than is dissolved and therefore it accumulates. Bating consists of the reduction of plumping, separation of the fibres into fibrils, removal of degraded collagen, cleansing of the grain, production of silky grain, and a minimum hydrolysis of the collagen itself.

D. WOODROFFE.

Tannin content of British Columbian *Alnus rubra*. R. H. CLARK and H. R. OFFORD (Trans. Roy. Soc. Canada, 1926, [iii.], 20, III., 149—152).—The variation of the tannin content of the dried bark of red-alder from the Vancouver district with the season of the year has been investigated. During October and November the tannin content is of the order 5—6%, falling to below 3% in January, and rising again to 6—7% in March. The tannin content does not warrant commercial extraction.

J. S. CARTER.

Electrometric determination of hydrogen-ion concentration in colloidal solutions, particularly tan liquors. V. KUBELKA and J. WAGNER (Collegium, 1926, 266—269).—The errors obtained in determinations of hydrogen-ion concentration owing to the foaming caused by organic colloids are overcome by the Kubelka-Belavsky form of electrode (Collegium, 1925, 247). The ordinary electrode becomes coated with an impermeable membrane of coagulated colloidal particles which cannot be removed by washing. The repeated platinising of the electrode is inconvenient and interferes with the measurements. In the Kubelka-Belavsky electrode the platinum wire is adjustable and the amount dipping into the liquor can be regulated, starting with a small portion and increasing the depth of immersion so as to expose a fresh surface after each measurement. Comparative results are given to show the differences obtained with the new electrode compared with the old fixed type. One electrode of the new type will serve for 20—30 determinations before it needs to be re-platinised.

D. WOODROFFE.

Determination of chromium in used chrome [tanning] liquors. V. KUBELKA and J. WAGNER (Collegium, 1926, 257—266).—The chrome liquor is filtered and diluted to a content of 1% Cr. 50 c.c. of the diluted liquor are pipetted into a 500-c.c. graduated flask, potassium hydroxide is added

until the chromium hydroxide precipitated at first re-dissolves, and then a further 1 c.c. of 25% potassium hydroxide added. The clear chromite solution thus obtained is heated for about 10 min. on the water bath or over a small flame. As soon as the solution becomes bright yellow, *N*-potassium permanganate is run in drop by drop until a brown flocculent precipitate of manganese dioxide is produced, the mixture is well shaken until effervescence ceases, made up to the mark after 5 min., and the chromium determined in 100 c.c. of this solution iodometrically.

D. WOODROFFE.

Examination of leather. G. POVARNIN and J. SCHICHIREV (Collegium, 1926, 269—275).—In the determination of soluble matter extracted from leather by alcohol and water, more tannin is extracted if the treatment with alcohol precedes that with water. The higher the "true tanning figure" (cf. B., 1923, 1141 A; 1926, 556) the greater is the wearing power of the dried leather. Wearing tests with sand are done better by rotating the samples of leather on sand than by means of a falling stream of sand. The wearing properties change inversely with the tensile strength. In estimating the effect of free sulphuric acid on leather, the amount of soluble salts in the leather must also be taken into account. A fresh variable is suggested for judging the regularity of the tannage. The leather is cut into three layers of equal thickness. The nitrogen content of each layer is determined and also that of the original unsplit leather. If N_u , N_m , and N_l are the nitrogen contents of the upper, middle, and lower layers respectively, and A that of the original leather, then $100 [1 - (N_m - N_u)/A]$ and $100 [1 - (N_m - N_l)/A]$ are the "coefficients of regularity of tannage" for the grain and flesh sides respectively of the leather. In spite of the higher regularity of tannage, the "true tanning figure" of drum-tanned leather is lower than that of pit-tanned. The wearing properties of the drum-tanned leather are greater than those of pit-tanned in spite of a higher content of free sulphuric acid. The pit-tanned leather is submitted to a swelling before tannage and from previous researches the authors have shown that such swelling is detrimental to the wearing properties.

D. WOODROFFE.

PATENTS.

Bleaching sole leather. J. RAISOR (U.S.P. 1,588,686, 15.6.26. Appl., 25.1.22).—Leather is dipped successively into (1) water at approximately 49°, (2) an alkali solution, (3) an aqueous solution, *d* 1.383, containing equal pts. by wt. of sulphuric acid, sodium chloride, and alum, (4) dilute sulphuric acid, *d* 1.162, (5) water.

L. A. COLES.

Recovery of chromium [from leather waste]. ELLENBERGER & SCHRECKER (G.P. 427,807, 29.3.24).—Leather waste from which glue has been extracted is treated hot with chlorine to obtain chromic chloride.

A. R. POWELL.

Dressing skins. A. J. CLERMONT (E.P. 250,178, 15.1.26. Conv., 31.3.25).

XVI.—AGRICULTURE.

Some relations between climate and soils in Europe. A. MEYER (Chem. Erde, 1926, 2, 209—347).—An exhaustive account of investigations on the influence of climatic factors, particularly temperature and abundance or shortage of water, on the origin and characters of European soils. A bibliography of 160 titles is included. C. T. GIMINGHAM.

Crust soils, crust formation, and red soils, with special reference to the soil formations of Palestine. E. BLANCK, S. PASSARGE, and A. RIESER (Chem. Erde, 1926, 2, 348—395; cf. B., 1925, 20).—A discussion of the occurrence and formation of crusts and crust soils, particularly chalk crusts and red soils, is followed by an account of the distribution of soil types in the hill country of Palestine and of the investigation of some characteristic soil profiles. C. T. GIMINGHAM.

Soil microbiology. II. Nitrogen-fixing bacteria. S. WINOGRADSKY (Ann. Inst. Pasteur, 1926, 40, 455—520; cf. B., 1925, 328, 465; 1926, 415, 505).—The author's methods for classifying soils according to their nitrogen-fixing powers are described and a detailed account is given of the experiments on which the procedures are based. C. T. GIMINGHAM.

Nature and availability of the plant-food constituents of Philippine guano. M. TIRONA (Philippine J. Sci., 1926, 30, 69—78).—The phosphorus in Philippine guanos is generally of rather low availability and exists mainly as phosphates of iron and aluminium. The total nitrogen present ranges from 0.7 to 4.7%, and a high proportion (16—60%) is soluble in water. About one quarter of the soluble nitrogen is in the form of nitrates, a feature which distinguishes Philippine guanos from those found elsewhere. Potassium is always present but the amount seldom exceeds 1.5% K_2O . C. T. GIMINGHAM.

Determination of ammonia nitrogen in fertilisers by visual conductometric titrations. G. JANDER and O. PFUNDT (Z. angew. Chem., 1926, 39, 856—858).—Ammonia nitrogen may be determined by titration of the aqueous extract of fertilisers with sodium hydroxide, the course of the reaction being determined by a conductometric method in which the usual telephone is replaced by a crossed thermocouple combined with a galvanometer or millivoltmeter. The conductivity of the solution is proportional to the square root of the throw of the galvanometer, so that by plotting this against the c.c. of alkali added a series of intersecting straight lines is obtained. The first point of intersection corresponds with the alkali necessary to neutralise the free acidity, the second with that required to convert the primary phosphate into secondary, and the third with the liberation of the ammonia. The last-named result must be corrected for the precipitation of calcium phosphate by determining the calcium in a separate portion of

the solution by precipitation as oxalate after addition of acetic acid. A. R. POWELL.

Determination of calcium carbide in calcium cyanamide. G. FLUSIN and H. GIRAN (Compt. rend., 1926, 182, 1628—1629).—The acetylene disengaged by treatment with boiling water is aspirated into ammoniacal silver nitrate solution. Treatment of the mixed precipitate of silver acetylide and silver sulphide with hydrochloric acid gives silver chloride and unchanged sulphide. The chloride is dissolved out with ammonia and determined by titration with potassium cyanide. M. CLARK.

Action of farmyard manure alone and in combination with mineral fertilisers. KLEBERGER (Z. Pflanz. Düng., 1926, B 5, 241—271).—The results of field experiments, extending over seven successive crops—potatoes, wheat, barley, green crop, sugar-beet, wheat, oats—are recorded, the use of dung with and without various combinations of mineral fertilisers being compared. In addition to the total yields of produce, the average amounts of nitrogen, potassium, and phosphorus taken up by the crops are given and the effects of the various treatments on the utilisation of plant nutrients are discussed. The results are also considered from the economic point of view. A combination of dung and complete mineral fertilisers gave in most cases the highest yields. C. T. GIMINGHAM.

Asahi-Promoloid. F. MÜNTER (Z. Pflanz. Düng., 1926, B 5, 272—274).—No increases of yield of lupins, carrots, and lettuce were obtained by the use of Asahi-Promoloid in vegetation experiments with a loamy soil. C. T. GIMINGHAM.

See also A., July, 673, Adsorption and mobilisation of the potassium ion in colloidal clays (DEMOLON). 679, Anomalous flocculation of clay (KERMACK and WILLIAMSON). 762, Phosphates in vegetation experiments (DOMONTOVICH).

Coli-aerogenes group [of bacteria] in soil. KOSER.—See XXIII.

Silicofluorides as insecticides. MARCOVITCH.—See XXIII.

PATENTS.

Fertilisers. W. R. FIELDING (E.P. 252,039, 16.12.24).—An organic material, such as straw, bracken, peat, sawdust, wood pulp, sewage sludge, or the like, is treated with a solution of sodium carbonate or sulphate or other efflorescent salt, and is then placed in a bath containing potassium carbonate, sodium nitrate, or other fertilising substances. The resulting mass, with or without additions of other materials to supply soil deficiencies, is dried and graded for use. It is claimed that the treatment with sodium carbonate enables the organic base to keep a fertiliser, in which deliquescent substances may be present, in a dry condition. C. T. GIMINGHAM.

Manufacture of fertilisers. E. L. PEASE (E.P. 253,291, 16.4.25).—A mineral or organic calcium phosphate is mixed with a porous material, such as peat, coarse coke dust, or sewage powder, and is treated with crude phosphoric acid. The mass is then used as an absorbent for ammonia, whereby a dry porous product containing a high proportion of readily available phosphorus and nitrogen is obtained. C. T. GIMINGHAM.

Electrometric determination of the acidity or alkalinity of soils, waters, solutions, and the like. M. TRÉNEL (G.P. 413,043, 16.7.24. Addn. to 399,410; B., 1926, 208).—The apparatus described in the chief patent is modified in that one electrode is placed in the comparison solution contained in a porous porcelain cell, the pores of which are sufficiently fine to prevent diffusion, and the other is placed directly in the vessel containing the solution under investigation to which the quinhydrone has been added. The cells used are preferably cylindrical and may consist entirely of porous porcelain or only the bottom or the lower portion may consist of this material.

A. R. POWELL.

Fungicide. W. MOORE and C. D. VREELAND (U.S.P. 1,587,977, 8.6.26. Appl., 30.3.25).—A fungicide, comprising an insoluble copper compound having an adsorbed positively charged ion, is prepared by precipitating the copper from a solution of copper sulphate with lime, and adding about 5% of calcium acetate to the precipitate.

C. T. GIMINGHAM.

Producing fertiliser and cattle feed (U.S.P. 1,587,101).—See XIX.

Bactericide and fungicide (U.S.P. 1,585,792).—See XXIII.

Fumigant (U.S.P. 1,586,175).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Determination of the ash content of raw sugar by the electrical conductivity method. L. KAYSER (Z. Ver. deuts. Zucker-Ind., 1926, 369—380).—Comparison of the electrical conductivity method with the incineration (sulphate) process shows the agreement to be satisfactory in most of the samples of raw beet sugars examined, but in other cases, particularly with after-product sugars, the differences between the two processes were much beyond the limit of error of the latter method. Where they cannot be accounted for by the presence of insoluble matter in the sugars examined, these differences are due to variations in the composition of the mineral matter. J. P. OGILVIE.

Complete analysis of mixtures of starch sugars and cane sugar products. D. R. NANJI and R. G. L. BEAZELEY (J.S.C.I., 1926, 45, 220—221 T).—A method is described for the determination of the chief constituents—dextrose, lævulose, sucrose, maltose, β -disaccharides and dextrin—

which are present in mixtures of starch sugars and cane sugar products. Adulteration of a cane sugar product such as invert sugar with starch sugars may be detected qualitatively by the decrease in specific rotatory power after hydrolysis with 8% (by weight) sulphuric acid for 3 hrs. in a boiling water-bath, and by the presence of a considerable quantity of dextrorotatory matter unfermentable by Froberg yeast. The quantitative method consists of the following operations:—(1) The reducing power of the sample is determined with Fehling's solution, by the volumetric method, and calculated in terms of apparent dextrose and apparent invert sugar. This reducing power represents the dextrose, lævulose, maltose together with the other reducing disaccharides, and dextrans present in the product. (2) The reducing power is then determined with Fehling's solution after inversion with 10% citric acid. The reducing power after inversion is calculated in terms of apparent invert sugar. It represents dextrose, lævulose, maltose, dextrin, and the invert sugar formed from sucrose present in the sample. (3) The reducing power of the sample is also determined iodometrically, and calculated in terms of apparent dextrose. Since iodine does not act on lævulose, the reducing power by this method represents all the reducing sugars in the sample except lævulose. In practice a slight correction has always to be applied for the very small absorption of iodine by lævulose. (4) The rotation of a 10% solution of the sample is determined, before and after fermentation with Froberg yeast. (5) The specific gravity of a 10% solution is determined before and after fermentation with Froberg yeast. In the latter case the alcohol is, of course, removed before taking the gravity. (6) Lastly, ash and nitrogen in the sample are determined in the usual way. From these data it is possible to determine the amounts of the various constituents present. An example is given showing the method of calculation.

Hydrolysis of starch by acids. D. R. NANJI and R. G. L. BEAZELEY (J.S.C.I., 1926, 45, 215—219 T).—A fraction resembling isomaltose is present among the products of the hydrolysis of starch by acids only after 78% of the starch has been hydrolysed to maltose and dextrose. This fraction is unfermentable by low attenuating yeasts, and its specific rotatory power varies ($[\alpha]_D$ 55—81°) according to the stage of hydrolysis. It yields a crystalline osazone, m.p. 155—160°, resembling isomaltosazone. The specific rotatory power of different osazone preparations varies considerably, some being dextrorotatory ($[\alpha]_D$ +15—20°), whilst others are lævorotatory ($[\alpha]_D$ —11°). It is probable, therefore, that the fraction is not a homogeneous substance, but a mixture of gentiobiose formed by reversion, and true isomaltose formed directly from the starch. The hydrolysis of starch by acids appears to be the resultant of over a dozen reactions taking place simultaneously. The liquefaction observed during the earliest stages of hydrolysis is shown to be due to the conversion of the calcium salts of the esters of

amylose and amylopectin into the free phosphoric esters. The whole of the phosphorus in starches is present in organic combination and liquefaction of starch takes place before the hydrolysis of the phosphoric esters. A method is described for studying quantitatively the products obtained at different stages of hydrolysis. The percentages of dextrin and the isomaltose fraction are determined by fermentation with Saaz and Froberg yeasts, both being unfermented by the former, and dextrin only by the latter. The optical rotation of a 10% solution is taken before and after fermentation with Saaz yeast. The reading of the solution before fermentation is due to dextrin, isomaltose, maltose, and dextrose. The reading of the same solution after fermentation is the rotation due to dextrin and isomaltose. By subtracting the percentages of dextrin and isomaltose, together with the percentages of ash and proteins from 100, the percentage of maltose and dextrose together is obtained. If the specific rotation of the mixture of these two sugars then be calculated, the relative proportions of the two sugars can be determined by the equation $52.5x + 138(1-x) = [\alpha]_D$ of the mixture of dextrose and maltose, where x = the amount of dextrose in 1 g. of the mixture.

Comparison of the hydrolytic [liquefying] action of various substances on starch. R. HALLER and A. HOHMANN (Textilber., 1926, 7, 239—242).—The effect on starch of various oxidising agents and enzymes was determined by exposing gelatinous solutions of potato starch to the action of the various agents at 65° under specified conditions, subsequently terminating the action of the agents by heating for 10 min. at the boiling point, and then comparing the viscosities of the products at 70° in an Ostwald viscosimeter. The liquefied starch products obtained by the action of enzymes are less viscous than those obtained by means of oxidising agents. The liquefying actions of oxidising agents and enzymes are largely dependent on their concentrations. The liquefying action of Aktivin on starch in glass vessels is assisted by the presence of traces of copper sulphate and nickel sulphate, but not by palladium chloride. The viscosities of the products obtained by the action of concentrations of Aktivin, bleaching powder, and sodium perborate proportional to their oxidising powers are in the ratio 2.70 : 3.58 : 6.50. Less viscous starch products are obtained by the action of Aktivin in the presence of sodium carbonate. The liquefied products obtained by means of Aktivin, bleaching powder, sodium perborate, Novo-Fermasol, Diastafor extra, Diastaphor double conc., Biolase, and Degomma D gave blue, blue, blue, bluish-red, red, red, yellow, violet, and blue colours respectively, with an alcoholic solution of iodine, and contained (as determined by a method of dialysis under similar conditions) 0%, 0%, 0%, 4.83%, 8.15%, 15.76%, 16.64%, 11.88% and 31.50% of reducing sugars (calc. as dextrose on the weight of starch used), respectively. The products obtained by means of Aktivin in the presence of copper sulphate (as catalyst) are whiter than the products

similarly obtained by means of Aktivin in the presence of sodium carbonate, or by sodium perborate or bleaching powder, and are therefore more satisfactory for the dressing of textile materials. A. J. HALL.

Unsaturated fatty acids associated with corn [maize] starch. T. C. TAYLOR and L. LEHRMANN (J. Amer. Chem. Soc., 1926, 48, 1739—1743).—The fatty acids liberated on hydrolysis of maize starch (α -amylose) have been identified as palmitic acid (24%), oleic acid (40%), and linoleic acid (36%). Nitrogen and phosphorus are absent.

F. G. WILLSON.

See also A., July, 714, *iso*Maltose (ISAJEV). 715, Starch, Amylobiose (PRINGSHEIM and STEINGROEVER).

Beet molasses as raw material for yeast production. CLAASSEN.—See XVIII.

PATENTS.

Process of treating sugar-beet diffusion juice. H. S. THATCHER and S. E. JOSI, Assrs. to CELITE CO. (U.S.P. 1,586,486, 25.5.26. Appl., 22.8.23).—Beet diffusion juice is heated and passed through a coating of diatomaceous earth previously formed on a filtering surface by passing through the filtering apparatus a suspension of the earth in purified beet juice.

J. P. OGILVIE.

Calandria for evaporators of sugar mills. A. P. LEONARD (U.S.P. 1,586,814, 1.6.26. Appl., 17.3.24).—A calandria has a closed heating chamber with downtake and steam inlet, a baffle forming a steam passage leading from the inlet, and an outlet for the passage at the end opposite the inlet. A number of circulation pipes are provided in the chamber between the inlet and outlet, these being so arranged as to form an unobstructed passage-way from the inlet along the baffle. J. P. OGILVIE.

XVIII.—FERMENTATION INDUSTRIES.

Beet molasses as raw material for the production of yeast by the aeration process. H. CLAASSEN (Z. Ver. deuts. Zucker-Ind., 1926, 349—368; Z. angew. Chem., 1926, 39, 880).—Using final molasses from German beet factories and refineries containing 1.2—1.9% N, white, vigorous bakers' yeast, conforming to standard fermentation tests, and of good keeping quality, was produced in small-scale tests. Phosphoric acid was the only nutrient added. Yields of yeast (25% of dry substance) equal to 41—59% of the molasses were obtained, and the amount of nitrogen assimilated by the yeast varied from 40 to 60% of that originally present.

J. P. OGILVIE.

Buffer substances in wort and beer. II. P. KOLBACH (Woch. Brau., 1926, 43, 277—280, 289—294).—This is mainly a survey of the subject. The advantages of a plentiful supply of buffer substances in malt liquors outweigh any disadvantages. There is conclusive evidence that

acid flavour is not determined solely by the concentration of hydrogen ions. Beer has a much less pronounced acid flavour than would be anticipated from its p_H value, probably owing to the mollifying influence of proteins (cf. Bermann, B., 1925, 1006). For practical purposes the buffering characters of malt extracts and beers can be satisfactorily estimated by titration in presence of different indicators, *e.g.*, neutral-red and γ -dinitrophenol (see Windisch and others, B., 1925, 951). Windisch has shown that the effect of phosphates can be approximately estimated by titration in presence of two indicators, before and after precipitation with barium hydroxide. The only other inorganic buffer substance in malt is silicic acid, but it is too small in amount to influence the reaction of wort or beer. It deserves investigation, however, in relation to turbidity in beer, for it appears to be present in an unstable condition, since it constitutes about 25% of the total mineral matter present in the coagulum formed when beer is heated.

J. H. LANE.

Influence of hop constituents on head-formation in beer. W. WINDISCH, P. KOLBACH, and W. BANHOIZER (Woch. Brau., 1926, 43, 207—209, 217—223, 229—235, 241—246, 253—258).—Of the constituents of beer, those derived from the hops contribute most to head-formation and head-retention. The length of time during which wort is boiled with hops has little influence on the head-forming power of the fermented beer; the authors found no difference after boiling for 1 and 2 hrs., respectively, but boiling for 3 hrs. produced a slight decrease in head-forming power. Within the practical range of hydrogen-ion concentration in beer head-formation is impaired by an increase of acid reaction, owing doubtless to lessened solubility of the foam-forming hop constituents. In beer made from unhopped wort, change of reaction does not influence head-formation. Ether will extract from hops the whole of the foam-forming constituents. The most important of these is the α -bitter acid, humulone, which has also been shown to be the most important in respect of flavouring and antiseptic properties. It is about 2.5—5 times as active as the β -bitter acid, lupulone, in promoting head-formation, and as it is present in hops in larger quantity than the latter, the foam-forming properties of different samples of hops show a much closer correspondence with their contents of humulone than with their contents of total resins. The soft resins into which humulone is partially converted during wort-boiling also have an important head-promoting influence on the fermented beer. Humulinic acid, a product of hydrolysis of humulone, is even more active than humulone itself in this respect. The method used for measuring head-forming and head-retaining power was that recently described (B., 1925, 1007). J. H. LANE.

Fermentation products from cellulose. H. B. SPEAKMAN (Pulp and Paper Mag., 1926, 24, 731—736).—The fermentation of sugars such as galactose and xylose by the butyl alcohol-acetone fermentation

organisms depends on their initial concentration, and upon the proportion of fermentable sugars with which they are mixed. Thus, a 2% solution of xylose is completely fermented, whereas a 5% solution remains unchanged; galactose alone is fermented to the extent of 10%, but this is increased to 50% in the presence of an equal amount of dextrose. The yield of acetone by the fermentation of sulphite-cellulose waste liquor is increased if this is neutralised, and if the maize mash culture is allowed to develop for 12—24 hrs. before the addition of the sulphite-cellulose waste liquor. A. GEAKE.

Determination of citric acid and the results of experiments with wine. O. REICHARD (Z. Unters. Lebensm., 1926, 51, 274—289).—Methods for the determination of citric acid are reviewed. The question as to how far citric acid can be described as a natural constituent of wine, and the possibility of distinguishing natural citric acid and that artificially added are discussed. Denigès' method for determining citric acid (cf. B., 1898, 802) is not satisfactory since other substances, *e.g.*, malic acid, produce a turbidity with the mercuric sulphate solution. Stahre's reaction as modified by Kunz (B., 1915, 974; cf. Von der Heide and Straube, B., 1922, 912 A) gives a trustworthy method for the detection and the determination of citric acid in wines.

A. G. POLLARD.

Pyrogenic dehydration of fusel oil. M. GIUA and L. THUMIGER (Atti R. Accad. Sci. Torino, 1926, 61, 199—208; Chem. Zentr., 1926, I., 3508).—Fusel oil obtained by fermentation by the Fernbach process contains about 65% of *n*-butyl alcohol, whereas ordinary fusel oil contains about 24% of *isobutyl* alcohol, 68% of amyl alcohol, 7% of propyl alcohol, and small quantities of esters, furfuraldehyde, and other compounds. On passing fusel oil vapour mixed with ethyl alcohol over heated catalysts, mixtures of ethyl esters of high boiling point, suitable, *e.g.*, for denaturing alcohol-benzine mixtures, are obtained. Olefin hydrocarbons of high boiling point are obtained by passing fusel oil vapour over heated mixtures of alumina and chromic oxide.

L. A. COLES.

See also A., July, 715, **Specific action of amylases (PRINGSHEIM and LEIBOWITZ); Fermentative degradation of starch by "biolase" (PRINGSHEIM and SCHAPIRO), 757, Methods for measuring rate of hydrolysis of starch and dextrin by taka-diastrase. Effect of p_H on starch-liquefying and dextrin-liquefying powers of taka-diastrase (MASLOW and DAVISON); Enzymes and light. Diastrase (PINOUSSEN). 758, Action of drugs and irradiation on yeast (ZELLER); Nitrogenous equilibrium in the yeast cell and augmentation of the invertase action (VON EULER, JOSEPHSON, and FINK). 759, Yeast amylase and fermentation of polysaccharides (GOTTSCHALK); Proteases of yeast (WILLSTÄTTER and GRASSMANN); Forms of lactic acid produced by pure and mixed cultures of bacteria (PEDERSON, PETERSON, and FRED). 764, Determination of volatile fatty acids in bacterial cultures (VIRTANEN).**

Dry yeast. ULEX.—See XIX.

PATENTS.

Absolute alcohol process. E. I. CLAPP, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,586,732, 1.6.26. Appl., 18.11.24).—In the manufacture of absolute alcohol by fractional distillation of a mixture of alcohol, water, and low-boiling hydrocarbon in a column still, the still is supplied with a mixture of the three substances in which the ratio of the low-boiling hydrocarbon to water is between 10.5:1 and 17:1.

T. H. POPE.

Enzymes and process of isolating them from their solutions. H. ALTGELT and O. HOCHMUTH, Assrs. to KALLE & Co. A.-G. (U.S.P. 1,590,663, 29.6.26. Appl., 17.3.25. Conv., 17.3.24).—See E.P. 251,405; B., 1926, 604.

Centrifugal liquid purifiers (E.P. 252,421).—See I.

Producing fertiliser and cattle feed (U.S.P. 1,587,101).—See XIX.

XIX.—FOODS.

Gluten. B. d'ARBOUET (Rev. gén. Colloid., 1926, 4, 97—107).—The colloidal nature of gluten and its importance in bread-making is discussed. Differences in the baking values of flours are largely explained by the physical condition of the gluten. The presence of a continuous network of colloidal gluten in the dough is important for good baking properties. Inherent differences in the grain, methods of grinding, and the reaction of the dough are the main factors affecting the colloidal condition and "cohesiveness" of the gluten. The poor baking value of rye flour and the effect of small additions of pea flour to a weak wheat flour are explained on similar lines.

A. G. POLLARD.

Preparation and examination of cheese fat. O. BAUMANN (Z. Unters. Lebensm., 1926, 51, 267—272).—For the isolation of cheese fat the process of Grossfeld (extraction with trichloroethylene after boiling with hydrochloric acid) gave the highest yield. Fats obtained from a number of cheese samples by neutral, acid, and alkaline processes of extraction were examined. Excepting in the case of the Reichert-Meissl values, no definite relationship was found between the process of extraction and the standard analytical values determined in fat analysis. Reichert-Meissl values were highest where the fats were extracted by the neutral process and lowest where the acid process was used. In no case, however, was the value lower than that for butter-fat.

A. G. POLLARD.

Determination of starch in pectin and apple juices by a sedimentation process. H. ECKART and A. DIEM (Z. Unters. Lebensm., 1926, 51, 272—275).—Ten c.c. of the sample are heated in a water bath for 10 min. with 80 c.c. of calcium chloride solution (1 pt. of calcium chloride to 2 pts. of water). The latter should be made neutral to phenolphthalein

before use. The mixture is transferred to a graduated flask and diluted to 100 c.c. with calcium chloride solution. A standard starch solution is prepared by heating on a water bath 0.1 g. of pure potato starch with a few drops of calcium chloride solution and adding gradually 50 c.c. of water. The solution is then diluted to 100 c.c.; 10 c.c. (or suitable dilutions) of the two solutions are treated with excess of iodine in potassium iodide solution in graduated centrifuge tubes. After 30 min., when the precipitates have flocculated, the solutions are centrifuged and the starch contents compared by means of the volumes of the precipitates.

A. G. POLLARD.

Determination of shell in cocoa and chocolate. J. GROSSFELD (Z. Unters. Lebensm., 1926, 51, 249—262).—Moderate quantities of shell in cocoa powders may be detected by determining the crude fibre and the ratio of nitrogenous substances to crude fibre. This ratio is greater than 4 for cocoa and approximately 1 for shell. The silica content also gives an indication of the presence of shell. The Filsinger sedimentation process (Z. öffentl. Chem., 1899, 5, 27) is useless for modern cocoa powders. It is shown that during the treatment with water the proportion of crude fibre in the residues increases and the nitrogen content decreases. The difference in the crude fibre content of cocoa and shell is lessened by leaching with water, but the actual values tend to become more constant. A process is described whereby the proportion of shell in cocoa powders may be calculated from the percentage residues after sedimentation and the crude fibre content of these residues.

A. G. POLLARD.

Incineration [of foodstuffs]. P. FORTNER (Z. Unters. Lebensm., 1926, 51, 300—301).—In the incineration of residues containing much sugar preliminary heating at low temperatures hastens the process. After evaporation on a water bath the residue is transferred to a platinum crucible which is placed on an asbestos plate heated by a burner placed 3—4 cm. from the crucible until the smell of burnt sugar ceases, when the flame is brought steadily nearer and increased. Finally the crucible is heated over the naked flame. To avoid loss of material by spitting during the ashing of foodstuffs containing much water, fat, or common salt, the residue after evaporation is covered with a circle of filter paper, cut to fit the crucible. If this is pressed down on the material it absorbs the fat and the subsequent ignition can be carried out without loss.

A. G. POLLARD.

Can corrosion and blackening in certain marine products. D. B. DILL and P. B. CLARK (Ind. Eng. Chem., 1926, 18, 560—563).—Free oxygen and volatile bases were not found to be significant factors in can corrosion by canned marine products. The content of sulphide sulphur is related to the blackening which is increased by rise of temperature. Of all the marine products examined only *Crustacea* contained organic sulphur metastable at ordinary temperatures and only *Crustacea* blackened to any great extent. The conversion of

organic sulphur into sulphide sulphur is accelerated by the metal of the container, and the proportion of sulphide sulphur increases during storage. All products which blacken and corrode the container were found to be on the alkaline side of p_H 6.5. Products more acid than p_H 6.5 do not blacken and for the most part do not corrode the container. D. G. HEWER.

"Blown" tins. An exceptional case. T. B. SHAW and R. C. FREDERICK (J. Roy. Naval Med. Service, April, 1926, Reprint).—The occurrence of "blown" tins is an indication of the unwholesomeness of a tinned food, but this rule does not apply to tinned coffee; "blowing" in the latter case may be due to a normal reaction between the freshly ground coffee and atmospheric oxygen. C. O. HARVEY.

Dry yeast. H. ULEX (Chem.-Ztg., 1926, 50, 475—476).—Used as fodder, dry yeast is put on the market in unextracted and extracted forms, which vary in appearance, taste, fat and protein contents, and in the amount of aqueous extractives. The unextracted form, which is more expensive, produces more rapid increase of weight and improved growth in the animal. Its superiority depends upon the content of the water-soluble substances, which are absent from the extracted variety, having been removed during the process of manufacture. In this process of preparation, no fat is lost from the yeast and owing to the removal of the soluble substances, the fat content of the extracted variety is correspondingly higher. On an average, unextracted yeast contains 48.8% of protein, 4.1% of fat, and yields approximately 36% of extractives. The corresponding values for the extracted form are 34.9%, 6.6%, and 16%. The contents of phosphoric acid, sodium chloride, and the alcoholic extract are almost identical in the two varieties. C. RANKEN.

See also A., July, 760, Concentration of growth-promoting principle (vitamin-B) obtained from yeast (LEVENE and VAN DER HOEVEN). 761, Mineral composition of sunflowers grown for silage (NEIDIG); Proteins of green-forage plants (DAVIES). 762, Nutritive value of various layers of the wheat and maize kernel (KLEIN, HARROW, PINE, and FUNK); Biological value of bread protein (HINDHEDE).

Detection of tallow in lard. MUSCHTER and VISSER.—See XII.

Determining milk fat in mixtures of fats. GROSSFELD.—See XII.

Crude fibre determinations in spices. OTTE and WEISS.—See XX.

PATENTS.

Food preparations made from meat and like edible animal matter. D. THOMSON and F. N. PICKETT (E.P. 252,754, 8.12.24).—The minced meat, mixed with water, is passed through two high-speed disintegrating machines until a practically colloidal suspension of the meat in water is produced. The acid liquid is neutralized by the addition of sodium

hydroxide and the liquids remaining are removed by treatment in a centrifuge or by filtering. The product is readily assimilable and may be administered as a medicinal food in admixture with alcohol. E. H. SHARPLES.

Producing fertiliser and cattle feed. A. WOOLNER, JUN. (U.S.P. 1,587,101, 1.6.26. Appl., 26.12.24).—The density of thin butyl alcohol distillation slop is raised by the addition of material of relatively high density containing sugar, so that the valuable constituents of the slop can be economically utilised in the production of fertilisers and feeding-stuffs. L. A. COLES.

Manufacture of bread. H. HEWITT, Assr. to BRIT. ARKADY Co., LTD. (U.S.P. 1,589,311, 15.6.26. Appl., 19.8.24. Conv., 12.12.23).—See E.P. 232,284; B., 1925, 564.

Preparation of cheese having an increased or supplemented vitamin content. H. LIEBERS, Assr. to HAMBURGER & Co. GES.m.b.H. (U.S.P. 1,590,837, 29.6.26. Appl., 9.4.24. Conv., 21.12.23).—See E.P. 226,549; B., 1926, 460.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Alkaloidal content of British Columbian *Datura stramonium* and *Conium maculatum*. R. H. CLARK and H. R. OFFORD (Trans. Roy. Soc. Canada, 1926, [iii.], 20, III., 153—155).—The dried seeds and stems of *Datura stramonium* contain about 0.28% and 0.06% respectively, of alkaloids, mainly atropine, hyoscyamine, and hyoscyne. The dried leaves contain about 0.3% of alkaloids, the amount rising to about 0.4% during August, when the plant is in flower. *Conium maculatum* (spotted hemlock) grows to much greater heights in British Columbia than in Great Britain and the conine content is considerably above the average found elsewhere. The dried seeds contain about 0.92% of alkaloids, and the dried stems about 0.025%. J. S. CARTER.

Piperonal in vanilla extract. C. B. GNADINGER (Ind. Eng. Chem., 1926, 18, 588—589).—Piperonal could not be detected in any variety of vanilla beans, except Tahiti beans and vanillons, which appear to contain very small quantities. In using the gallic acid test for piperonal it is necessary to separate the aldehydes from any anisyl alcohol present (cf. B., 1925, 376), as the deep red colour produced by the latter will mask the blue colour of the reaction. D. G. HEWER.

Manufacture of phenacetin from *p*-chloronitrobenzene. D. H. RICHARDSON (J.S.C.I., 1926, 45, 200—203 T).—By heating *p*-chloronitrobenzene at 60° for 140 hrs. in a 0.5 *N*-solution of caustic soda in 95% alcohol free from acetaldehyde, 92% of the theoretical yield of *p*-nitrophenetole may be obtained, the only by-product being *p*-nitrophenol. *p*-Nitrophenetole may be converted directly into phenacetin (96% yield) by refluxing with iron filings and five

times its weight of glacial acetic acid, but even when allowance is made for the value of the acetic acid recovered this method is more costly than the reduction of nitrophenetole to phenetidine and the acetylation of the base. In material cost it is cheaper to reduce nitrophenetole with iron and hydrochloric acid than with sodium sulphide, but the latter process has the advantage in ease of working and isolation of the product. Various methods of isolating the phenetidine are compared, but none yielded more than 75% of the theoretical amount. Both phenetidine base and hydrochloride can be satisfactorily acetylated by distilling with a solution of acetic acid in toluene, the distillate being dehydrated by anhydrous sodium acetate and returned to the reaction vessel.

Acidimetric titration and composition of commercial lactic acid. R. EDER and F. KUTTER (Helv. Chim. Acta, 1926, 9, 557—578; cf. A, 1926, 499).—A critical survey of the literature on the formation and determination of lactic acid and its dehydration products is given. In the absence of lactide the following method may be used for the determination of free lactic acid and lactyl-lactic acid in commercial lactic acid. About 0.4 g. of concentrated lactic acid (p) is diluted with 20 c.c. of water, and titrated directly with 0.1 N -sodium hydroxide (a) using neutral-red as an indicator. Sufficient 0.1 N -alkali (b) is then added to hydrolyse the lactyl-lactic acid, the solution heated for 10 min. on the water bath, and titrated with 0.1 N -acid to about 1—2 c.c. excess (c). After again warming, the excess of acid is titrated with standard alkali (d). Since 1 c.c. of N -sodium hydroxide solution = 0.09005 g. of lactic acid, free acid present = $0.9(a+c-b-d)/p\%$, and the lactyl-lactic acid = $1.8(b+d-c)/p\%$. Direct titration of lactide with N -alkali solution determines 50% of the lactide in accordance with the equation $C_6H_8O_4 + NaOH = CHMe(OH) \cdot CO \cdot O \cdot CHMe \cdot CO_2Na$ but it is not attacked by an alcoholic solution; and hence the following method is evolved for the determination of free lactic acid ($X\%$), lactyl-lactic acid ($Y\%$), lactide ($Z\%$), and water ($W\%$) in the presence of each other. Direct titration with N -sodium hydroxide solution in aqueous solution, using phenolphthalein as an indicator (a), gives the value of $X+Y/2+Z/2$, and subsequent hydrolysis with N -alkali in aqueous solution as described above (b) represents the value of $Y/2+Z/2$. Direct titration of the sample dissolved in absolute alcohol with N -alcoholic sodium hydroxide, using thymol-blue as an indicator, represents $X+Y/2$, and subsequent hydrolysis for 20 min. on the water bath after the addition of 25 c.c. of water represents $Y/2+Z$. The water content is found by difference. Analysis of samples of 90% and 100% commercial lactic acid showed that the quantity of lactide present is less than the value of the experimental error, and hence commercial lactic acid contains only free lactic acid, lactyl-lactic acid, and water.

J. W. BAKER.

Electrolytic preparation of glycollic acid. V. TOJA and U. CEVA (Giorn. Chim. Ind. Appl., 1926,

8, 3—7).—The most economical method of preparing glycollic acid consists in the electrolytic reduction of oxalic acid in presence of sulphuric acid, using pure lead cathodes and comparatively low current densities. The sulphuric acid is removed by treating the liquor with lime, and the solution thus purified is concentrated in a vacuum and the resulting syrup allowed to cool. The acid then deposited is of good quality. An outline is given of an industrial plant and the view is expressed that the manufacture of the acid must be conducted in conjunction with that of oxalic acid. At present, however, glycollic acid can scarcely compete seriously with tartaric acid for industrial purposes.

T. H. POPE.

Industrial extraction of citric acid from lemon juice. B. MELIS (Annali Chim. Appl., 1926, 16, 135—141).—To extract pure calcium citrate from lemon juice, the juice is filtered after being mixed with one or more of the following: kieselguhr, tufa, kaolin, talc, or natural amorphous or precipitated gypsum, particularly that obtained by the decomposition of calcium citrate by sulphuric acid. The ratio of filtering material to juice should not exceed 1:5 if filter-presses, or 1:10 if continuous, revolving, vacuum filters are used. The filtered juice is neutralised, when boiling, with dense homogeneous milk of lime, free from magnesia; this addition is made in small amounts and with vigorous stirring. The final tenth of the total acidity may be neutralised by English chalk or by powdered marble.

T. H. POPE.

Comparative crude fibre determinations in spices. W. OTTE and H. WEISS (Pharm. Zentr., 1926, 67, 401—404).—The method of Huggenberg (Mitt. Lebensm. Hyg., 1916, 7, 297) for the determination of crude fibre in spices is described and is recommended as a rapid "sorting" test, especially for pepper. Results given by 20 samples of pepper were, on an average, 0.78% lower than those given by the Henneberg and Stohmann method (Weender process), which must, however, always be used as a standard in doubtful cases. For pepper husks and scraps the results are considerably lower and for cardamom fruits, husks, and seeds higher, than those obtained by the standard method. The causes for these deviations are discussed.

E. H. SHARPLES.

Essential oil of the needles of *Pseudotsuga Douglasii*. Carr. E. ALINARI (Annali Chim. Appl., 1926, 16, 185—190).—From an American sample of this essential oil Brandel and Sweet (Pharm. Rev., 1908, 26, 326) isolated fractions with the odours of pinene and limonene and identified camphene and borneol. Schorger (B., 1914, 1170) showed that the terpene fraction of a Californian oil consisted mainly of α - and β -pinenes, together with a small proportion of limonene, camphene being absent; the heavier fractions contained borneol. In an oil prepared from needles of English growth Bennett (Perf. Ess. Oil Rec., 1920, 11, 218) found limonene, dipentene, and a small amount of pinene, the principal component of the oil being geraniol. In view of the discordance between these results, the author has analysed an oil

prepared from the needles of this tree grown in the forest of Vallombrosa. The greenish-yellow oil of pleasant aromatic odour has d^{15} 0.8712, $[\alpha]_D^{20}$ -25.43° , n_D^{20} 1.4736, ester value 18.87 (6.60% of bornyl acetate), ester value of acetylated oil 52.29 (about 9.94% of free alcohols in original oil); it consists mainly of β -pinene and of geraniol and nerol, which are mostly free but partly esterified by acetic and capric acids.

T. H. POPE.

Mentha pulegium [pennyroyal] oil. P. LIOTTA (Riv. Ital. Ess. Profumi, 1926, 8, 44—45; Chem. Zentr., 1926, I., 3507).—Steam distillation of 100 kg. of *Mentha pulegium* yielded 350—500 g. of a dirty straw-yellow oil having d 0.8400, α^{15} $26^\circ 30'$, and acid value 0.31. The oil is soluble in 2 pts. of 70% alcohol and contains 0.85% of esters and 62% of pulegone and piperitone.

L. A. COLES.

Essential oil from Medang Lesoh [*Cinnamomum Parthenoxylon*]. B. J. EATON and G. L. TEIK (Malay. Agric. J., 1926, 14, 81—83).—The oil is very similar to that formerly described (B., 1926, 215), consisting principally of safrole and having $d_{15.5}^{15.5}$ 1.101, n_D^{20} 1.538, ester value 2.5, and ester value after acetylation 8.4. It is optically inactive and is soluble in 3.3 vols. of 90% alcohol. E. H. SHARPLES.

See also A., July, 671, Azeotropic properties of formic and acetic esters of saturated aliphatic alcohols (HANNOTTE). 690, Rate of reaction between ethylene and chlorine (STEWART and FOWLER). 702, Is Kjeldahl's method for determination of nitrogen in organic substances generally applicable? (KÜRSCHNER). 722, Synthesis of adrenaline. Preparation of pyrocatechol chloroacetates (OTT); Reaction of some polyhydric phenols with sodium antimonyl tartrate (CHRISTIANSEN). 724, Thyroxin. Constitution and synthesis of deiodothyroxin (HARRINGTON). 739, Compound of veronal with pyramidone (PFEIFFER and ANGERN). 744, Alipine hydrochloride (SEVILLA); Alkaloid of *Julocroton montevidiense* (ANASTASI). 746, Alkali xanthates as reagents for alkaloids (NAVARRO); Trypanocidal action and chemical constitution. Arylamides of aminohydroxyphenylarsinic acids (HEWITT and KING). 749, Refractometric determination of alcohols and esters in aqueous and in cottonseed oil solutions (MUNCH). 750, Electrometric determination of alkaloids without the use of the hydrogen electrode (POPOV and McHENRY). 755, Preparation and chemical composition of active substances of *Digitalis* leaves (CLOETTA).

Determination of citric acid. REICHARD.—See XVIII.

PATENTS.

Manufacture of colloidal organic mercury compounds soluble in water. W. CARPMAEL. From FARBENFABR. VORM. F. BAYER & Co. (E.P. 243,361, 18.11.25).—Soluble colloidal organic mercury compounds, particularly suitable for immunising grain, are obtained by precipitating a solution of an organic mercury compound containing a protective

colloid with an organic solvent which is miscible with water. The colloid may be added during the manufacture of the mercury compound, or the latter may be dissolved in an acid or alkali, and the colloid added before precipitation with the organic solvent. The precipitation is preferably effected from a neutral solution. Thus a solution of 10 pts. of the mercury compound of *o*-nitrophenol in 100 pts. of water and 2 pts. of sodium hydroxide is stirred into a solution of 100 pts. of albumose in 800 pts. of water. After neutralisation with acetic acid, precipitation is effected with acetone. The product contains 15.5% Hg and gives a neutral solution in water.

R. BRIGHTMAN.

Manufacture of aluminium ethylate [ethoxide]. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 245,473, 4.1.26. Conv., 3.1.25).—Alcohol containing a catalyst such as mercuric chloride, stannic chloride, or iodine in solution, is added slowly to a suspension of aluminium in aluminium ethoxide or in an inert solvent, such as xylene, the aluminium being in excess during the reaction. For example, 440 c.c. of alcohol containing in solution small quantities of mercuric chloride and iodine, are added slowly to a suspension of 100 g. of aluminium grit in 650 c.c. of boiling xylene. During the addition of about the first 320 c.c. of alcohol, the xylene is maintained at the boil by the heat of reaction, but external heat is applied during the addition of the remainder and for a further $\frac{1}{4}$ hr. after it has all been added. The mixture is filtered and distilled to remove mercury and xylene, yielding aluminium ethoxide readily soluble in the usual solvents, and containing no alcohol of crystallisation.

L. A. COLES.

Isolating alcohols or phenols from mixtures. A. DEPPE SÖHNE, and F. O. ZEITSCHEL (E.P. 252,570, 21.7.25).—Mixtures containing alcohols or phenols are treated with quantities of the mixed anhydride of boric and acetic acids corresponding to the alcohol or phenol present. The volatile non-alcoholic constituents and the acetic acid formed are distilled off under reduced pressure, and the triborates of the phenols or alcohols which remain in the residue are hydrolysed by the addition of water or aqueous alkali. If solid the borates can be purified by recrystallisation prior to hydrolysis. In place of the boric-acetic anhydride may be used the corresponding double anhydrides of arsenious acid, antimonious acid, or phosphorous acid, and the acetic anhydride may similarly be replaced by any other organic acid anhydride. The reaction can also be effected with boric, arsenious, antimonious, or phosphorous acid or their anhydrides alone. The process may be applied to the preparation of pure menthol from Japanese peppermint oil, geraniol from Ceylon citronella oil, and linalool from shiu oil. Trial on a mixture of benzyl acetate and benzyl alcohol indicated that the method is quantitative. The method may also be applied to the approximate separation of primary, secondary, and tertiary alcohols, the reactivity of the three classes of alcohols being in the order named. Thus from a mixture of geraniol, borneol, and terpineol, almost

pure terpeneol is readily obtained by heating with boric acid at 80–100° and hydrolysis of the borate formed. The arsenious acid anhydride method requires a somewhat higher temperature, e.g., 150°.

R. BRIGHTMAN.

Process for catalytically preparing methanol [methyl alcohol] or higher alcohols or other oxygenated organic compounds or mixtures. L. CASALE (E.P. 252,573, 27.7.25).—Methyl alcohol or higher alcohols or other oxygenated compounds or mixtures of alcohols and other oxygenated compounds are obtained by catalytic reactions between carbon monoxide, carbon dioxide, hydrogen, and a gaseous hydrocarbon in a closed circuit of apparatus containing no moving parts. Circulation is obtained by admitting the mixture to the circuit at a higher pressure than that obtaining in the circuit, part of the potential energy of the mixture thereby being transformed into kinetic energy. In this way poisoning of the catalyst by the lubricating oil from the pistons and stuffing-boxes of circulating pumps is avoided.

R. BRIGHTMAN.

Preparation of ethylidene diacetate. SOC. CHIM. USINES DU RHÔNE (E.P. [A] 252,632, 30.11.25, and [B] 252,640, 6.1.26. Conv., 13.6. and 18.9.25).—(A) Ethylidene diacetate free from vinyl acetate is obtained in excellent yield by absorbing acetylene in acetic acid in the presence of the mercuric salt of a strong acid, such as sulphuric acid, sulphoacetic acid, benzene- or naphthalene-sulphonic acids, provided that a strong acid and an excess of acetic anhydride are present throughout. The free acid may be the same as or different from that giving the mercuric salt. The reaction is carried out at 80–90° until about 80% of the theoretical amount of acetylene has been absorbed by the acetic acid. The formation of tar is practically negligible and only 1% of mercury (on the acetic acid) is required as against 4% by known processes. (B) In carrying out the process described under (A) the mercuric salt may be replaced by metallic mercury in the presence of ferric sulphate. At the end of the reaction the ethylidene diacetate, carrying the ferrous sulphate formed, may be drawn off and the mercury left in the apparatus, a feature which makes a continuous process possible. From 50 pts. of metallic mercury, 1000 pts. of glacial acetic acid, 20 pts. of anhydrous ferric sulphate, 65 pts. of acetic anhydride, and a mixture of 50 pts. of sulphoacetic acid and 125 pts. of glacial acetic acid, 1500 pts. of crude, or 1200 pts. of purified ethylidene diacetate are obtained.

R. BRIGHTMAN.

Manufacture of *n*-butyl esters of amino-benzoic acids. E. C. R. MARKS. FROM ABBOTT LABORATORIES (E.P. 252,870, 12.5.25).—*n*-Butyl esters of aminobenzoic acids are obtained by the action of 2 mols. of *n*-butyl halides (preferably *n*-butyl bromide) on a metal salt of the aminobenzoic acid in the presence of a catalyst, such as diethylamine, dipropylamine, dibutylamine, etc., or copper powder. Alternatively the metal salt of a nitrobenzoic acid may be used and the *n*-butyl nitrobenzoate reduced by means of iron and hydrochloric acid.

n-Butyl *p*-nitrobenzoate has b.p. 160°/6 mm., m.p. 33–35°; *n*-butyl *p*-aminobenzoate, m.p. 57–58.5°.

R. BRIGHTMAN.

Production of pure lecithin. C. H. BOEHRINGER SOHN, Assees. of O. DENGLER (U.S.P. 1,586,145, 25.5.26. Appl., 11.5.25).—By treating 100 g. of sodium cholate dissolved in 100 c.c. of water with 20 g. of lecithin, a soluble double compound of lecithin and the alkali cholate is formed. It is precipitated by addition of alcohol and ether and is completely freed from fatty impurities and cholesterol by treatment with ether or benzene. The purified product is dissolved in water and decomposed into lecithin and cholic acid by treatment with 10% aqueous hydrochloric acid and the purified lecithin is recovered by extraction with ether. Alternatively an aqueous solution containing lecithin and sodium cholate is extracted with ether and evaporated to dryness under reduced pressure, when a portion of the lecithin can be directly recovered from the residue by extraction with ether. The remainder is recovered, after treatment of the extracted residue with dilute hydrochloric acid, by a further extraction with ether.

T. S. WHEELER.

Apparatus for generating ether. F. E. LICHTENTHAELER (U.S.P. 1,587,161, 1.6.26. Appl., 28.9.23).—A reservoir for sulphuric acid is provided with a series of tubes of acid-resisting material (lead) connected to it at the top and bottom. Each of the tubes is surrounded by a steam jacket and circulation of the acid in the reservoir through the tubes is thus induced. Alcohol is introduced into each of the tubes by means of a perforated pipe extending some distance into it.

T. S. WHEELER.

Manufacture of a pharmaceutical product. [Compound of diallylbarbituric acid and 4-dimethylamino-1-phenyl-2:3-dimethyl-5-pyrazolone.] A. THIELE, ASSR. to CHEMISCHE FABRIK AUF ACTIEN (VORM. E. SCHERING). (U.S.P. 1,588,554, 15.6.26. Appl., 18.5.23).—See E.P. 198,379; B., 1924, 114.

Antipyretic. [*o*-Acetoxy-*p*-methoxybenzoic acid.] W. SCHULEMANN and F. SCHÖNHÖFER, ASSRS. to WINTHROP CHEMICAL Co., INC. (U.S.P. 1,588,814, 15.6.26. Appl., 21.11.25. Conv., 4.4.24).—See E.P. 231,886; B., 1925, 942.

Process of making olefine oxides. Process of making propylene oxides. J. N. BURDICK, ASSR. to CARBIDE AND CARBON CHEMICALS CORP. (U.S.P. 1,589,358–9, 22.6.26. Appl., 7.8.23 and 14.11.25).—See E.P. 236,379; B., 1925, 738.

Manufacture of ethylsulphuric acid. A. A. L. J. DAMIENS, M. C. J. E. DE LOISY, and O. J. G. PIETTE (U.S.P. 1,589,372, 22.6.26. Appl., 6.12.19. Conv., 5.9.19).—See E.P. 152,495; B., 1920, 833 A.

Manufacturing acetic anhydride. L. HÖRMANN and F. KAUFER, ASSRS. to A. WACKER-GES. FÜR ELEKTROCHEM. IND. G.m.b.H. (U.S.P. 1,590,097, 22.6.26. Appl., 2.2.24. Conv., 12.2.23).—See E.P. 211,167; B., 1924, 890.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Effect of evacuation of a photographic plate on its sensitivity. O. MASAKI (Mem. Coll. Sci. Kyoto, 1926, A 9, 285—302).—Various commercial plates were exposed to light from a Mazda lamp, using a Hurter and Driffield wheel to give differences of exposure, and the density of the plates was measured by means of a photo-electric cell. In all cases the plate which had been subjected to reduced pressure showed greater density than the corresponding untreated plate, though to a greatly varying extent; for Ilford panchromatic plates the effect is small. For many plates the contrast is increased, but for Ilford panchromatic plates the opposite effect is produced. The lower the pressure the greater is the effect produced. In the case of red light, however, the opposite effect was observed, the treated plate being less sensitive than the untreated one; on the other hand, the contrast is increased; thus, treatment reduces fog. For dyed plates the sensitising action of the dye is generally enhanced. Experiments on drying the plates indicated that the main effect of evacuating the plates was the removal of moisture and the change in sensitivity and contrast was due to the dryness of the emulsion. E. B. LUDLAM.

Photographic reversal. H. BELLIOU (Compt. rend., 1926, 182, 1609—1612).—An investigation of the photographic properties of red and infra-red radiation. A previously fogged plate, on which reversal has been produced by radiation of wave-length greater than 0.7μ , recovers much of its sensitivity to visible light, whilst the same plate, solarised, is no longer sensitive. Radiation which produces reversal on a fogged plate blackens unused plates. If the portion of the spectrum between 0.5 and 0.95μ falls on a plate fogged in one portion and solarised in another the radiations of greater wave-length reverse the fogged portion and blacken the solarised portion. The radiations of shorter wave-length, however, enhance the existing solarisation.

J. S. CARTER.

Spectral distribution of sensitivity of photographic materials. JONES and SANDVIK.—See A., July, 694.

PATENTS.

Production of photographs on parchment, paper, etc. E. E. JELLEY (E.P. 253,380, 24.7.25).—Any pure paper is sensitised by coating with a solution containing a soluble silver salt, in which the silver is combined with one or more of the three higher halogens, and a soluble, non-volatile, crystallisable weak acid, e.g., citric acid, tartaric acid, etc. The best silver salt is silver chlorate, but silver perchlorate, silver bromate, and silver salts of organic acids containing substituted halogens, soluble in water to a greater extent than 0.01 pt. in 100 pts. by weight, may be used. It is preferable to coat with the silver salt first, dry, and then coat with an alcoholic solution of the acid. The paper is exposed to give a faintly visible image and is developed in a

solution of an organic reducer such as metol, quinol, metol-quinol, or pyrogallol. By suitable choice of exposure time and developer, a range of tones from warm brown to black is obtained. After development the paper is well washed and fixed in a solution containing sodium thiosulphate and sodium bicarbonate, or in an acid fixing bath. If the paper is sensitised to the extent of less than 0.0005 g. of silver salt per cm^2 , a soluble silver salt is added to the developer and shorter exposure times are used. The hydrogen-ion concentration of the developer should be kept within the limits 10^{-4} and 10^{-2} . Amidol tends to fog the paper. W. CLARK.

Photographic developer. K. BINDER (E.P. 253,732, 14.7.25).—See F.P. 600,801; B., 1926, 566.

XXII.—EXPLOSIVES; MATCHES.

Method for the determination of the velocity of detonation over short lengths of explosive. E. JONES (Safety in Mines Res. Bd., Paper 22, 1926, 21 pp.).—The apparatus is arranged so that a travelling detonation wave is caused to break two electrical circuits at known distances (e.g., 2 cm.) from each other. The first break causes a charged condenser of known capacity to discharge through a known resistance; the second break causes the discharge to cease. Measurement of the residual charge in the condenser enables the time of discharge to be calculated. The velocity of detonation obtained by this method was somewhat less than 3% lower than that obtained over 1 m. of the same TNT fuse by the Mettegang method (5th. Int. Congr. Appl. Chem., Berlin, 1903, 2, 322). S. K. TWEEDY.

PATENTS.

Gelatinising nitrocellulose in nitro-compound powders and celluloid. L. PARODI-DELFINO (E.P. 252,978, 28.10.25).—Phthalide or its homologues are claimed as gelatinisers and stabilisers in the manufacture of celluloid and of nitrocellulose powders which may or may not contain nitroglycerin. These compounds have the advantage of being non-volatile and enable cool-burning colloided nitrocellulose powders containing a reduced proportion of nitroglycerin to be obtained without the use of volatile solvents. A suitable composition comprises nitrocellulose 12 pts., nitroglycerin 4—6 pts., and phthalide or its homologues 1—4 pts.

T. S. WHEELER.

Primer composition. H. RATHSBURG (U.S.P. 1,586,380, 25.5.26. Appl., 18.3.25. Conv., 23.10.23).—The composition contains guanyl nitrosoamino-guanyltetracene. L. A. COLES.

XXIII.—SANITATION; WATER PURIFICATION.

Effect of chlorination on trickling sewage filters. M. M. COHN (Eng. News-Rec., 1926, 96, 943—948).—At Schenectady, N.Y., the continuous application of liquid chlorine in quantities equivalent to 4—30 p.p.m., to the Imhoff tank effluent, prior to filtration, did not materially affect the nitrification efficiency of healthy filters, although the tank

effluent itself was rendered sterile by the treatment. Odours were reduced proportionately to the quantity of chlorine applied. Biological growths present in the nozzles of sprays and in the distribution pipes were destroyed and removed, the nozzles subsequently remaining in a clean condition. Another effect of the chlorination was the removal of the gelatinous films from the surface of the beds; in consequence, "ponding" did not occur and nuisance from flies was substantially reduced. Bleaching powder gave results similar in character, and is a suitable reagent to use for fly control. Dichlorobenzene mixture when sprayed on the walls and sheltered spots about a filter rapidly destroys adult *Psychoda*, mosquitos, and young spiders.

W. T. LOCKETT.

Determination of p_H in natural waters. I. R. NASINI and C. PORLEZZA (Annali Chim. Appl., 1926, 16, 156—166).—The results obtained in the electrometric and colorimetric determination of the p_H value of natural waters are discussed and the special sources of error and means of avoiding them indicated. Determination of this magnitude is of particular value in following the variation in the proportion of carbon dioxide dissolved in these waters, and in tracing the alteration of sulphur waters, especially as regards the oxidation of the hydrogen sulphide.

T. H. POPE.

Prevention of corrosion and "red water." J. R. BAYLIS (J. Amer. Water Works Assoc., 1926, 15, 598—633).—The fundamental factors influencing corrosion rates of iron, zinc, lead, and cement-lined pipes are discussed and experimental results relating thereto are given. The problem of prevention of corrosion in water mains is one of protection by film formation. Films may be formed by precipitating the products of corrosion on the metal surface or by producing a supersaturation of some compound in the water, e.g., calcium carbonate, which will form an impervious coating. No ordinary compound of iron will exist in solution in most waters in quantities greater than 0.1 p.p.m. of soluble iron when the water is at calcium carbonate equilibrium or more alkaline, within the range of natural waters. When the water is at this point or more alkaline the rate of solution of iron is comparatively slow and all products of corrosion where dissolved oxygen is present are quickly precipitated. The amount of calcium carbonate which will be held in solution depends upon the alkalinity, hydrogen-ion concentration, and to a slight extent upon the amount of neutral salt present. When it is not desired to produce a coating of calcium carbonate on the pipe surface the p_H of the water should be so adjusted that very little soluble iron will exist in the water in the absence of dissolved oxygen and at the same time calcium carbonate supersaturation will be avoided. Values of p_H suitable for this purpose for waters with varying alkalinities are plotted. These vary from p_H 8.5 with water having an alkalinity of 12, to 8.25, 7.8, 7.5, 7.4, and 6.75 with alkalinities of 25, 50, 75, 100, and 350 respectively. Treatment along these lines prevents staining and reduces "red water" to an

occasional occurrence. When it is desired to form a protective coating of calcium carbonate, lime should be added to raise the p_H value to 9.5 with an alkalinity of 25, or to 8.5, 8.0, 7.8, 6.9 with alkalinities of 50, 75, 100, 350, respectively. After a coating has been formed the water may be treated so as to obtain p_H values intermediate between the two series given, at which values, with the respective alkalinities, the calcium carbonate is substantially in equilibrium.

W. T. LOCKETT.

***Coli-aerogenes* group [of bacteria] in soil.** S. A. KOSER (J. Amer. Water Works Assoc., 1926, 15, 641—646).—Members of the *coli-aerogenes* group were found to be fairly abundant in the soils of cultivated fields and pastures. The results of the methyl-red, Voges-Proskauer, and citrate tests brought out a general correlation between the proportion of the various sections of the colon group and the apparent degree of faecal pollution of the soil. The proportion of the *Bact. coli* type (MR+, VP—, citrate—) increased with the amount of faecal contamination, being highest in the soil from pastures.

W. T. LOCKETT.

Fluosilicates [silicofluorides] as insecticides. S. MARCOVITCH (Ind. Eng. Chem., 1926, 18, 572—573).—Sodium silicofluoride consisting of 70—75% of the salt and the remainder alumina and occupying a volume of 60 cub. in. per lb., and calcium silicofluoride, a by-product in the volatilisation method of treating phosphate rock for phosphoric acid, with a volume-weight of about 70 cub. in. per lb. in one form and 120 cub. in. in the other, were used in field trials and found effective against a variety of insects not controlled by arsenic. These substances are also far less toxic to man than arsenicals. Flea beetles, and blister and striped cucumber beetles were readily controlled, and the bacterial spot disease of the peach was successfully combated with sodium silicofluoride at dilutions of 1 to 4000 or 4500.

D. G. HEWER.

Danger of mercury vapour. STOCK.—See A., July, 707.

PATENTS.

Compound having bactericidal and fungicidal properties [from sulphite-cellulose waste lye]. E. SCHLUMBERGER (U.S.P. 1,585,792, 25.5.26. Appl., 26.10.23).—1000 kg. of concentrated sulphite-cellulose waste liquor are diluted with 500 litres of water and treated with chlorine. The separated yellowish-red mass is filtered off and dried. It contains 25—28% Cl, is soluble in water, and possesses strong bactericidal and fungicidal properties. The properties of the disinfectant are improved by a subsequent treatment with an oxidising agent such as potassium chlorate and hydrochloric acid.

E. S. KREIS.

Centrifugal liquid purifiers. (E.P. 252,421).—See I.

Electrometric determination of acidity or alkalinity of water (G.P. 413,043).—See XVI.

Fungicide (U.S.P. 1,587,977).—See XVI.