

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 30, 1928.

I.—GENERAL; PLANT; MACHINERY.

Tube apparatus for crystallisation and evaporation. O. ZAHN (Chem. Fabr., 1928, 4—6).—The plant consists essentially of a slowly rotating iron tube, very slightly inclined to the horizontal, and provided with annular end pieces, by adjustment of which the height of the liquid passing through the tube is controlled. A current of air is drawn through in the direction opposite to that in which the liquid flows. The hot saturated solution is fed in at one end; the crystals formed are carried through with the mother-liquor and discharged continuously on to a filter or centrifugal. Brick or ebonite lining is specified for corrosive materials. When used as an evaporator, the tube is enclosed in suitable flues for heating. By connecting a heated tube with one through which cold air is drawn, evaporation and crystallisation may be effected continuously and automatically. S. I. LEVY.

Operating experiences with 1300 lb. steam pressure. J. ANDERSON (J. Inst. Fuel, 1928, 1, 131—160).—Eleven months' experience with a Stirling boiler operating at a pressure of 1300 lb. has shown that to avoid tube failures it is necessary to treat the feed water to prevent scale formation, although no difficulty arose when the same water was used in medium-pressure boilers. To prevent corrosion the oxygen content of the feed water must be reduced below 0.1 c.c./litre, and an appreciable caustic alkalinity must be maintained in the water. If, however, the concentration of alkali is too high, loss of turbine efficiency may be caused by deposition of solids on the blades. The use of trisodium phosphate in the boiler water in relatively small concentrations inhibits the formation of scale. The tensile strength of the metal of the tubes decreases with use, so that the usual method of testing when new gives too high a value for the strength after service. The general behaviour of the boiler at 1300 lb. was better than that at 300 lb. pressure, the water levels being stable and the moisture in the steam being low. A. B. MANNING.

Preheated air for boiler furnaces. P. H. N. ULANDER (J. Inst. Fuel, 1928, 1, 187—212).—The conditions are discussed under which the installation of an air preheater effects a useful economy of heat in boiler practice. The use of preheated air results in higher furnace temperatures and increased radiation, making possible a reduction in size of boiler and size of heating surface. Also a higher furnace rating can be maintained without serious loss due to incomplete combustion. Troubles such as "burning" of the links in a chain grate, uneven combustion, erosion of furnace walls, etc. may arise if preheated air is used in connexion with old type furnaces and grates, but with correctly designed

plant it is possible to operate safely with air temperatures of 250° or even higher. Examples of such installations are described and illustrated. A. B. MANNING.

Measurement of steam flow in works practice. H. C. ARMSTRONG and T. NORDENSON (J. Inst. Fuel, 1928, 1, 161—186).—Valuable economies can often be effected by control of the steam consumption in the various processes in a factory. A simple and inexpensive throttle orifice diaphragm meter for steam measurement is described and directions given for its use, together with diagrams for facilitating the necessary calculations. The theory of the instrument is developed in a second part of the paper [by M. BÄCKSTRÖM].

A. B. MANNING.

Measurement of surface temperature. D. F. OTHMER and H. B. COATS (Ind. Eng. Chem., 1928, 20, 124—128).—The study of heat transfer through a tube or other metal surface involves the measurement of the temperature of the surface. The use of thermo-couples is attended by difficulties, especially in avoiding heat conduction along the wire. The authors have obtained consistent results by plating a layer of nickel on the surface of a copper tube with nickel wires embedded in the plating. The hot junction has thus a broad interface and conducted heat is dissipated. The return wire of bright copper was simply wound round the unplated portion of copper tube, and all leads were insulated with rubber tubing. Details are given of tests with such copper-nickel thermo-couples with the tube surrounded by steam and having a current of cold water passing through it. Temperatures as much as 35° below that of the steam were read with a consistency of 0.05°.

C. IRWIN.

PATENTS.

Furnace. W. G. WISE, Assr. to WISE FURNACE Co. (U.S.P. 1,658,364, 7.2.28. Appl., 19.11.27).—A burner for gaseous or vaporising liquid fuel is situated in a fire pot from the upper portion of which radiating ducts pass out to an annular hollow radiator drum which has (near its periphery) vertical gas ducts communicating with the ducts leading from the fire pot. At the top of the drum a number of radiating ducts lead the gases inwards towards the centre, whence they pass outwards again to the vertical ducts of another similar drum. Any number of such drums may be employed, and the gases finally pass out through a centrally disposed flue. C. O. HARVEY.

Coal-dust furnaces. SIEMENS-SCHUCKERTWERKE G.M.B.H. (B.P. 270,295, 25.4.27. Ger., 1.5.26).—The fuel is admitted axially downwards at the top and the primary air enters tangentially close by. Over the whole, or nearly all, of the interior surface of the

combustion chamber secondary air is admitted through numerous jets at such angles that the eddying, whirling motion is gradually decreased downwards so that the solid particles are kept away from the furnace walls by centripetal action.

B. M. VENABLES.

Regenerative furnaces. W. G. BEESTON (B.P. 282,841, 14.1.27).—A furnace chamber is connected through a valve chamber at each end to a pair of regenerator chambers, each valve being common to both regenerators and to the furnace; one valve communicates with an outlet stack, the other with an air-blast chamber. The air passes through one regenerator before the furnace, and the hot gases through the other regenerator after the furnace; on reversal the direction of the gases through the furnace does not reverse. [Stat. ref. to B.P. 11,402 of 1885.]

B. M. VENABLES.

Furnaces with chain or travelling grates. A. W. BENNIS (B.P. 283,243, 7.6.27).—A number of separate cells are situated under the chain grate and air of different humidity is supplied to them.

B. M. VENABLES.

Cooling towers. K. W. BRANCIK (B.P. 282,932, 21.10.26).—A cooling tower is provided, near the mouth, with louvre-like openings and curved deflector blades, which will convert a downwardly directed wind into an updraught within the tower, assisting the natural draught.

B. M. VENABLES.

Grinding mills. VICKERS, LTD., and L. D. PARKER (B.P. 282,989, 10.1.27).—A rotary cylinder has a number of V-shaped circumferential grooves formed on its inner surface, and balls run in the grooves, the balls being preferably considerably smaller than the grooves.

B. M. VENABLES.

Introduction of materials into high-pressure apparatus. SYNTHETIC AMMONIA & NITRATES, LTD., and H. A. HUMPHREY (B.P. 282,873, 27.9.26).—Two hydraulic rams enter a common work cylinder from opposite ends. The outer ends of the rams expand to a larger diameter forming double-acting pistons sliding in power cylinders. The feed material under low pressure is introduced through a port in the upper part of one end (say the left) of the work cylinder, and a port at the other end communicates with the high-pressure vessel. At the start the two rams are near each other at the left-hand end. The right-hand ram then moves to the right and draws in material. Both rams then move together to the right, closing the left port and opening the right port; the left ram usually moves rather faster than the right ram so as to compress the material. The left ram then continues alone and pushes the material into the high-pressure vessel. At completion both rams move together to the left for a new start, or they may be moved in inverse order to above to discharge the vessel.

B. M. VENABLES.

Apparatus for separating dust from suspension in gases and vapours. H. WARING, and ASSOCIATED LEAD MANUFRS., LTD. (B.P. 282,849, 28.8.26).—A bag filter is constructed with tapering bag or bags supported at the upper smaller end by yielding means such as a counterweight which permits the bag to be shaken while in use. The dust is collected on the inside and the bags are preferably situated above a cyclone separator.

B. M. VENABLES.

Conditioning of gaseous fluids such as air. BRIT. ARCA REGULATORS, LTD., and T. LINDSAY (B.P. 282,915, 9.10.26).—In an apparatus where gases are conditioned by saturating with fluid at a lower temperature and are then re-heated to the required dry-bulb temperature, the variations in temperature of the saturating liquid are caused to alter an aperture through which a non-compressible fluid is pumped at a constant rate. By this means the pressure of the non-compressible fluid will vary according to the aperture, and the variations of pressure will operate a servo-motor which controls the supply of heat or cold. A similar device is used to control the re-heating of the gas after saturation.

B. M. VENABLES.

Apparatus for bringing liquids and gas or vapours or both gas and vapours into intimate contact, and liquid-distributing devices adapted for use therein. W. C. HOLMES & Co., LTD., C. COOPER, and D. M. HENSHAW (B.P. 283,028, 13.4.27).—In gas washers of the "brush" or other rotating type, buckets attached to the rotor are employed to lift and distribute the liquid over the effective surface without immersing much of the rotor and making use of only a small bulk of liquid. The buckets are specially shaped so as to discharge liquid at a rate proportional to the vertical depth of the effective surface below any point of discharge. The buckets are usually in pairs, one bucket of each discharging while rising 90° from horizontal to vertical and the other bucket while dropping from vertical to horizontal.

B. M. VENABLES.

Purifying separator for liquids. J. SCHAFER (B.P. 272,927, 15.6.27. Fr., 15.6.26).—A settling chamber for separating, e.g., water from petrol, is provided with a float valve and automatic siphon so that the heavier liquid is automatically drained off at intervals.

B. M. VENABLES.

Filters and strainers for liquids. D. A. COATES (B.P. 282,846, 29.4.27).—For a filter of the type in which a helical or conical coiled spring is used as the filtering medium, several methods of expanding or contracting the spaces between the coils are described.

B. M. VENABLES.

Degasification of liquids. SOC. ITAL. PIRELLI (B.P. 276,987, 25.8.27. Italy, 31.8.26).—The liquid is supplied to near the centre of the uppermost of several rotating saucers or shallow cones, which alternate with other fixed collecting cones with inturned edges; the latter return the liquid to the centre of the next rotating saucer below and so on. While under treatment the liquid is subjected to a vacuum, and if necessary to heat, and it is stored in vessels either under vacuum or which have expansible walls and are completely filled.

B. M. VENABLES.

Apparatus for purifying and cooling gases. A. L. MOND. From METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 282,939, 3.11.26).—The gas is caused to pass through a number of convergent and divergent passages with sharp changes of direction at the narrowest parts. The passages may be mounted on a drum or other rotary support which dips into liquid, and the depth of submergence in the liquid may be used to vary the available area of the passages according to the amount of gas under treatment.

B. M. VENABLES.

Grinding mill. J. B. SEDBERRY (U.S.P. 1,659,499, 14.2.28. Appl., 4.4.24).—See B.P. 231,386; B., 1925, 385.

Centrifugal treatment of liquids. K. J. SVENSSON and K. A. P. NORLING (U.S.P. 1,659,222, 14.2.28. Appl., 14.12.26. Swed., 11.1.26).—See B.P. 264,130; B., 1927, 287.

Superheater boilers. T. E. ROBERTSON. From POWER SPECIALTY CO. (B.P. 283,031, 20.4.27).

Joint-making packing for high-pressure vessels etc. A.-G. BROWN, BOVERI, & CIE. (B.P. 264,524, 14.1.27. Ger., 16.1.26).

Hollow grinding bodies for ball-, drum-, and tubular mills. O. KORDT (B.P. 284,580, 12.12.27).

Drying of viscid liquids (B.P. 282,480).—See V. Catalysis (B.P. 281,218).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Differential index of the coking power of coal. A. W. COFFMAN and T. E. LAYNG (Ind. Eng. Chem., 1928, 20, 165—170; cf. Layng and Hathorne, B., 1925, 195).—By use of a softening and solidification point test a graphical means of determining an index number called the agglutinating index for any coal may be arrived at. An initial softening temperature for each coal is indicated by an increase of pressure which assumes a maximum value and then rapidly decreases to normal. Study of that portion of the temperature-pressure curve falling between the initial softening point and the point of maximum pressure gives a true measure of the range of plasticity of a coal. The values d_p/d_t calculated at regular intervals and plotted against the time in minutes show an increase to a maximum value which is taken as the differential index or agglutinating index of a coal. To be comparable all data must be obtained at the same rate of heating. From the appearance of the curves under such test conditions, for a coal to coke the differential index must be above 20 and the total energy must be used in a comparatively short time giving rise to a peak in the curve. These two conditions may be fulfilled in the case of the more poorly coking coals by varying four factors which govern the agglutinating value, viz., the rate of heating at which the coal is carbonised, the previous conditioning of the coal, the amount of weathering to which the coal has been subjected, and the chemical interaction taking place between the coal constituents.

H. S. GARLICK.

Low-temperature coke from coking coals. M. DOLCH (Oesterr. Chem. Ztg., 1928, 31, 11—15).—An essential condition for the economic development of low-temperature carbonisation is that the income derived from the coke produced should cover the cost of the raw coal. Only the use of coking coals or mixtures containing coking coals can fulfil this condition, but even so the value of the coke will depend largely on the choice of a suitable type of carbonisation plant. In evaluating low-temperature cokes the calorific value of the volatile matter evolved on heating to 1150° is of more importance than its actual amount (cf. B., 1926, 651).

A. B. MANNING.

Use of the bomb for the determination of carbon and hydrogen [in combustible material]. J. W.

WHITAKER (Fuel, 1928, 7, 63—65).—Carbon and hydrogen are determined by a modification of Goutal's method (B., 1923, 1162 A). After combustion in the bomb, in the bottom of which is placed a crucible containing anhydrous calcium chloride, the gases are gradually discharged through (1) a weighed U-tube containing pumice soaked in sulphuric acid, (2) a gas sampling tube, (3) a wash-bottle containing water saturated with carbon dioxide, and (4) a meter. The carbon content of the fuel is then calculated from the total volume of gas in the bomb and the percentage of carbon dioxide therein. The hydrogen is determined from the increase in weight of the crucible containing calcium chloride and the U-tube through which the gas is passed. The presence of sulphur does not seriously interfere with the hydrogen determination.

A. B. MANNING.

Unsaturated hydrocarbons in the gases from the carbonisation of coal. A. B. MANNING, J. G. KING, and F. S. SINNATT (Dept. Sci. Ind. Res., Fuel Res. Tech. Paper No. 19, 1928, 19 pp.).—Ethylene, propylene, and butylene are determined in a coal gas by passing a known volume (about 10 cub. ft.) of the gas through bromine, after a preliminary oil scrubbing to remove the vapours of volatile liquids. The bromine compounds of the unsaturated gases are distilled under reduced pressure and the olefines are regenerated by the action of a zinc-copper couple on a weighed quantity (about 2 g.) of the distillate in solution in 95% alcohol. The olefines in the gas so obtained are determined by (1) absorption of the propylene and butylene in 87% sulphuric acid, making a correction for the small concurrent ethylene absorption, followed by absorption of the ethylene in fuming sulphuric acid, (2) combustion of a second portion of the gas over copper oxide and measurement of the carbon dioxide produced. It is then possible to calculate the ethylene, propylene, and butylene content of the original gas. By extraction of the residue from the distillation of the bromine compounds with alcohol the butadiene tetrabromide present can be isolated and thence an approximate value of the butadiene content of the gas obtained. A typical low-temperature coal gas contained ethylene 1.18%, propylene 1.16%, butylene 0.68%, butadiene 0.15%; a high-temperature, horizontal-retort gas contained ethylene 1.90%, propylene 0.28%, butylene 0.08%, butadiene 0.02%. Results for other types of gases, including a series produced by the carbonisation of coal at temperatures from 400° to 700° (cf. Sinnatt, King, and Linnell, J.S.C.I., 1926, 45, 385 T), are given and briefly discussed.

A. B. MANNING.

Gas analysis. H. M. STANLEY and A. W. NASH (Gas J., 1928, 181, 391).—Determination of paraffin hydrocarbons in gases of unknown composition is often difficult by the explosion method, and a modified procedure of oxidation by copper oxide at 900° is available. Although this oxidation takes place somewhat slowly, and generally produces less carbon dioxide than an explosion, it is considered preferable, especially with gases rich in nitrogen.

R. H. GRIFFITH.

Method of fractionating natural gasoline. D. B. KEYES (Chem. Met. Eng., 1928, 35, 92).—The preheated oil is passed into a stripping column (steam being used

as the heating medium throughout) from the bottom of which pure absorbing oil and water are withdrawn; the oil after being cooled is separated from the water and returned to the system. The vapours from this column, consisting of all the gases, all of the product, and a little of the absorbing oil, pass to the first condenser, which is run warm. Part of this condensate is returned as reflux and part sent on to the next fractionation column. The second condenser is a cold or total condenser. The condensate from this enters a third fractionating column. The product is cooled, separated, and collected in a storage tank, whilst the uncondensed gases are sent to the compressors, coolers, and water-separators along with the uncondensed gas from the condensers on the stripping column. A second absorption in an absorbing column then takes place, an absorbing oil of lower b.p. being used. The gas-oil solution is stripped in a stripping column, and the pure absorbing oil returned to the absorber. The crude product after refining in a fractionating column passes on to the storage tank, the product in which is again fractionated to give the final pure product. H. S. GARLICK.

Cracking experiments by the Dubbs process with distillates from Hanoverian petroleum. R. HEINZE (*Petroleum*, 1928, 24, 237—241).—The yield and nature of cracked spirit obtained with Hanoverian petroleum are compared with those given by American oils. A gas-oil fraction, b.p. about 200—360°, was cracked under 14 kg./cm.² pressure, and gave 36% of motor spirit, 20% of gas oil, and 23% of fuel oil, whilst under 19 kg./cm.² the yield of gas oil decreased to 16%, and under 21 kg. pressure was still less, but no regularities appear in the proportions of the other fractions. The motor spirit produced under 21 kg. pressure contains 23% of aromatic, 27% of naphthene, and 40% of paraffin hydrocarbons, and has pronounced anti-knock properties. A heavier petroleum fraction, b.p. about 240—400°, was also cracked under 5.25 kg. to two different end-points, and gave a yield of about 30% by wt. of motor spirit, with a high aromatic and low naphthene content, and with a Ricardo compression ratio of 5.8. Refining of the cracked distillates was in all cases carried out with plumbite solution and with sulphuric acid, but the quantities necessary varied with the different products.

R. H. GRIFFITH.

Steam-temperature control in distillation practice and in paraffin manufacture. E. BELANI (*Petroleum*, 1928, 24, 199—201).—When steam is used in distillation of oils and tars, or in refining of paraffin wax, control of its temperature and pressure is very important; if the steam is raised by a Benson boiler or similar apparatus in which water is heated above its critical temperature and pressure, and is converted into steam without boiling, it will be too hot for such purposes. Any device for lowering the temperature of the steam must provide a steady exit temperature and pressure regardless of inlet variations, and a machine for effecting this has now been perfected. Steam enters at the bottom of a vertical vessel and meets a fine spray of water; this is completely vaporised, and the exit steam will have a temperature depending on the quantity of water used. Thermometers at the inlet and outlet operate a relay which opens or closes a valve controlling the amount

of this added water, so that fluctuations at the inlet are immediately compensated. R. H. GRIFFITH.

Preheated air for boiler furnaces. ULANDER.—See I. **Synthetic phenol.** HALE and BRITTON.—See III.

PATENTS.

By-product coke oven. J. V. K. BORLAND (U.S.P. 1,658,796, 14.2.28. Appl., 13.9.23).—To obtain a uniform distribution of fuel gas from a main supplying a battery of coke ovens, the gases are maintained at a substantially constant temperature throughout the entire length of the main. A. B. MANNING.

Cleaning of coal. C. R. DOWNS and C. H. STRANGE, Asss. to HIRSCH-LEDERER SYND., INC. (U.S.P. 1,656,270—1, 17.1.28. Appl., 8.5.25).—(A) Coal and its impurities are separated by gravity in a medium formed by a suspension of a finely-divided solid in a liquid, the sp. gr. of the medium being intermediate between that of the coal and that of the impurities. The finely-divided solid separated from the washed coal by screening is cleaned from fine coal and other impurities by flotation and returned to the gravity separator. In (B) the finely-divided solid used for the suspension is made up largely of finely-divided material separated from the coal itself.

A. B. MANNING.

Washing of coal, ores, and like substances. L. HOVOIS (B.P. 266,723, 22.2.27. Belg., 25.2.26. Addn. to B.P. 258,753; B., 1926, 971).—The principles of the main patent and the additional patent B.P. 265,202 (B., 1928, 114) are applied for the further separation of the products. By the use of a projecting box with several adjustable openings and an adjustable current of water the material is re-graded and subdivided; hoppers and chutes are arranged to receive the various products obtained. A. C. MONKHOUSE.

Elaborating the products arising during the liquefaction of coal by hydrogenation. DEUTS. BERGIN-A.-G. F. KOHLE U. ERDÖLCHEMIE (B.P. 262,738, 5.11.26. Ger., 11.12.25).—A small quantity, e.g., 20%, of liquid aliphatic hydrocarbons such as gas oil is added to the products of hydrogenation which have been previously freed from water and constituents boiling below 280°. Asphalt and solid insoluble substances are precipitated and the oil is separated. On keeping, a further quantity of soft asphalt is precipitated; the precipitating medium is recovered from the fractions by steam-distillation. A. C. MONKHOUSE.

Distillation of coal. B. ZWILLINGER, Assr. to TAR & PETROLEUM PROCESS Co. (U.S.P. 1,656,617, 17.1.28. Appl., 14.7.22).—A thin horizontal layer of coal is distilled in an oven chamber heated from beneath by a number of longitudinal, parallel flames which are independently regulable, and the distillates so produced are collected. A. B. MANNING.

Distillation of carbonaceous material. L. C. KARRICK (B.P. 261,362, 2.11.26. U.S., 16.11.25).—A highly reactive fuel is obtained by distilling coal etc. in continuous vertical retorts internally heated by superheated steam or gas. Waste steam is admitted to superheaters situated in the lower half of the setting between the retorts and is heated at 540—940° by

the burning of fuel gas and air, both preheated in the upper portion of the setting. The steam or gas is admitted at the mid point of the retorts, and additional steam or gas is admitted at the bottom for cooling purposes.

A. C. MONKHOUSE.

Coking of coal. URBANA COKE CORP., Assees. of S. W. PARR and T. E. LAYNG (B.P. 263,785, 13.12.26. U.S., 2.1.26; cf. Parr, B., 1926, 697).—Coal is fed from a hopper into an inclined rotating drum which is heated by waste flue gases as well as by supplementary, automatically controlled gas burners. It is thereby preheated, out of contact with air, nearly but not quite to the pasty stage. In order to economise heat as far as possible the flue gases after leaving the preheater pass through flues in the hopper itself before being discharged to air. The coal is transferred directly from the preheater into vertical retorts, which are already heated to the desired temperature (750°), and is therein carbonised. The retorts are of metal, circular in cross-section and slightly tapered. They are heated principally by radiation from the walls of the heating chamber in which are flues provided with gas burners. After circulating in the flues the hot gases pass up around the retorts and thence to the preheater or to a recuperator for heating the air supplied for combustion. When the coal is completely coked it is discharged into a storage chamber in which it is cooled; air passages surround this chamber so that the sensible heat of the coke may be utilised in preheating the air required for combustion.

A. B. MANNING.

Treatment of carbonaceous material. R. V. FARNHAM (B.P. 283,717, 18.12.26).—An apparatus for drying or carbonising fuels or other materials consists of a number of elongated rectangular sections bolted together one above the other. A "retarder," consisting of a tray of channel or H-section which can be rotated about a horizontal axis, is arranged in each section in such a manner that by its rotation the material is delivered from that section to the one below, and by suitably adjusting the periods of rotation the material can be subjected to the heating medium for any desired period. The material is fed into the top section by means of arms rotating in a cylindrical hopper, and may be withdrawn similarly at the bottom. Means are provided for passing heated gases through the apparatus.

A. B. MANNING.

Apparatus for the manufacture of briquettes. S. R. ILLINGWORTH, and ILLINGWORTH CARBONIZATION Co., LTD. (B.P. 284,007, 19.10.26. Addn. to B.P. 258,327; B., 1926, 1003).—The main patent is modified in that the moulds on the outside of the rotating drum are detachable, being secured in position by endless links engaging the walls of the moulds. The moulds may consist of trays divided into a number of compartments, each of which forms a mould, the links then engaging the outside walls of the end compartments of each tray. The links have teeth on their outer surfaces, which mesh with teeth on the rollers supporting the drum, and by which the latter is driven. The coal, after being fed into the moulds, may be compressed by a ram working in a cylinder carried in the casing wall.

A. B. MANNING.

Formation of reconstructed carbonaceous fuel. W. B. RULON (U.S.P. 1,656,364, 17.1.28. Appl., 28.7.25).—Finely-divided solid carbonaceous material is agitated with a liquid towards which it is inert, and a volatile liquid which has an affinity for the solid is then added. The plastic "amalgam" produced in the form of small pieces is compressed into relatively large lumps, which are heated to render them solid and to reduce the percentage of volatile matter therein.

A. B. MANNING.

Utilising powdered fuel. Utilising pulverulent or powdered carbonaceous material. J. J. C. BRAND and B. LAING (B.P. 281,098 and 281,588, [A, B] 26.5.26).—(A) Powdered fuel of size such that 85% is below 200-mesh is fed by worm conveyor into a vessel and is kept in agitation by the admission of air or inert gas at the bottom, so that the normal inter-air space is increased by 30–60%. Air at 5–100 lb./in.² is used to force the powdered fuel to the burners, where the supply is controlled by an inverted needle valve. Baffles impart a whirling motion to the fuel-air mixture, and gauze diaphragms are used to avoid flash-backs from the burner. A portable form of apparatus is described. (B) For the burning of powdered fuel in existing marine etc. boiler furnaces a preliminary combustion chamber lined with firebrick is used. The chamber has an inclined front wall with suitable burners pointing vertically downwards. The fuel and primary air are met at the front wall by the secondary air, which diverts the burning fuel to the rear wall of the chamber over the fire-bridge into the furnace. The temperature is maintained below that of slag formation by cooling externally with air or water.

A. C. MONKHOUSE.

Producing or reactivating active carbon. VER. F. CHEM. IND. A.-G., Assees. of E. KÜCHLER (B.P. 260,567 and Addn. B.P. 265,916, 11.10.26. Ger., [A] 27.10.25, [E] 15.2.26).—(A) Carbon is heated in a container with porous or permeable walls, outside which is circulated a gas containing oxygen, the material being activated by the action of the gas which diffuses through the walls. At the same time a current of the gas is passed directly into the container, in quantity, however, insufficient in itself for activation. (B) In a modification of the above process the container is made of narrow dimensions or is provided with internal channels for the passage of the gases in order to accelerate the activation. The container may be heated by furnace gases, and, if desired, the activating gases may be preheated.

A. B. MANNING.

Manufacture of producer gas. W. M. CROSS (U.S.P. 1,657,371, 24.1.28. Appl., 28.4.27).—Producer gas is obtained by passing a mixture of air and steam (automatically maintained at a temperature above the predetermined temperature of reaction) along with pulverised carbonaceous material to a reaction zone.

C. O. HARVEY.

Roasting of used or waste gas-purifying substances. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 271,854, 12.5.27. Ger., 25.5.26).—Waste gas-purifying masses containing sulphur are roasted in a furnace through which they pass in the same direction as the roasting gases. The oxidation of the volatile organic compounds and ammonia is thereby practically

completed, and the resulting gases can be used directly in the lead chamber process without unduly increasing the consumption of nitric acid. A further improvement is effected by preheating the air used for roasting, *e.g.*, by passage over the hot roasted mass. A. B. MANNING.

Catalytic process for desulphurising hot gases. H. FLEISSNER (Austr. P. 106,443, 18.6.25).—A mixture of the hot gas with air is passed over a catalyst obtained by roasting spathic iron ore or other minerals containing ferrous carbonate. L. A. COLES.

Hot desulphurisation of gases derived from the distillation or pyrogeneration of solid, liquid, or other combustibles. SOC. INTERNAT. DES PROC. PRUD-HOMME HOUDRY (B.P. 267,138, 4.3.27. Fr., 4.3.26).—The gases are passed at 250° through a purifier containing a metal or metallic oxide, *e.g.*, nickel oxide, to remove hydrogen sulphide, a purifier containing oxide of iron or copper to remove organic sulphur, and, if the gases contain water vapour, a third purifier similar to the first to remove hydrogen sulphide evolved from the second purifier. Regeneration is effected by air or by water-gas mixed with steam and heated at about 350°.

A. C. MONKHOUSE.

Low-temperature distillation of coal. INTERNAT. COMBUSTION ENGINEERING CORP., Assees. of W. RUNGE (B.P. 267,095, 2.2.27. U.S., 4.3.26; cf. B.P. 242,623; B., 1927, 162).—Finely-powdered coal passes down a vertical retort counter-current to an ascending stream of hot gases, and is thereby carbonised to give a coke consisting substantially of hollow particles filled with combustible gas. The coke is discharged into a conduit through which it is conveyed by a current of cool combustible gas, the latter thereby taking up part of the heat of the coke. After separation of the coke, the gas is passed to a specially designed preheater in which part of it is burnt and mixed with the remainder, and with other gases if required, in such proportion as to bring their temperature to that necessary for the distillation process. The mixed gases thence pass to the retort.

A. B. MANNING.

Continuous distillation of tree branches, vines, etc. E. A. BARBET and R. MALEAX (F.P. 621,350, 6.1.26).—The wood, cut up into small pieces, is carbonised in a continuous vertical retort, and the gases formed are separated into two portions, one containing the water evolved during the first stage of the heating, and the other suitable for use, *e.g.*, for driving agricultural machinery. L. A. COLES.

Distillation of pyroligneous acid. SOC. DES PROD. CHIM. DE CLAMECY (B.P. 269,927, 22.4.27. Fr., 22.4.26).—Crude pyroligneous acid is distilled and the vapours evolved during the first stage of the distillation are led into a column in which they come into contact with benzene or a similar liquid with which methyl alcohol forms a binary mixture of minimum b.p. The vapours from this column are condensed, the methyl alcohol, together with some acetone and methyl acetate, extracted with water, and the benzene is returned to treat a further quantity of the pyroligneous acid vapours. The vapours evolved during the next stage of the distillation are led to a second column in which, by suitable adjustment of the reflux, the bulk of the acetic acid is condensed in a

concentrated form, water, allyl alcohol, and oily impurities passing to the receiver. These oily impurities are further treated with water to recover allyl alcohol.

A. B. MANNING.

Resolution of aqueous emulsions of tar. B. JOHNSON (B.P. 284,401, 27.10.26).—Tar emulsions are resolved by the addition of a small quantity of a carboxylic acid, *e.g.*, resin acid, oleic acid, stearic acid, etc., which is soluble in the tar, but insoluble in the water, the mixture being then maintained at 60–70° until separation is complete. When treating tars which contain no ammonia it is convenient to add a small quantity of an alkali so that the separated water has an alkaline reaction.

A. B. MANNING.

Recovery of sulphuric acid from the acid tar of benzol purification. C. STILL (B.P. 277,619 and Addn. B.P. 281,547, [A] 16.5.27, [B] 31.5.27. Ger., [A] 20.9.26, [B] 22.12.26).—(A) The acid tar is mixed with concentrated ammonium sulphate liquor from the saturator, and is maintained at 55° by means of a steam coil. The acid resinous constituents rise to the surface on being kept, and after separation and removal of the ammonium sulphate liquor are heated to remove benzol hydrocarbons. Tar or tar oils are added to the product, and the mixture is washed with water before discharge. (B) The operations take place at moderate temperature without the application of external heat.

A. C. MONKHOUSE.

Manufacture of water-soluble humic acid derivatives. O. L. BEER (G.P. 443,339, 29.3.23).—Humic acid or one of its alkali salts is treated with sulphur dioxide or sulphites at ordinary or increased pressure in presence or absence of aldehydes, and, if desired, is treated with chlorine. *E.g.*, an alkaline extract of brown coal is boiled with 38% sodium hydrogen sulphite solution; the resulting brown solution has tanning properties, gives a soluble residue on evaporation, and forms insoluble heavy-metal salts. Tanning materials are also obtained by heating alkali humate with sodium sulphite and formaldehyde solutions, or by the action of chlorine on alkali humate solution. Raw humus (peat etc.) may be warmed with sodium carbonate solution, sulphite added, and heating continued under pressure; sulphur dioxide then precipitates a sulphited humic acid soluble in water.

C. HOLLINS.

Manufacture of a bituminous composition. W. P. BENTLEY (U.S.P. 1,659,554, 14.2.28. Appl., 18.4.24).—A suitably graded non-bituminous mineral is mixed with a crushed bitumen-containing rock, and there is added to the mixture first a volatile liquid capable of liquefying the bitumen in the rock, and then such a quantity of a bituminous flux in liquid condition that the total quantity of bitumen is just sufficient to coat the mineral and fill the voids in the mixture.

A. B. MANNING.

Ammonia-saturator apparatus. J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,654,159, 27.12.27. Appl., 11.2.21).—An apparatus for use in the application of the process described in U.S.P. 1,375,483 (B., 1921, 426 A) comprises a tank supplied with sulphuric acid and gas containing ammonia, and fitted with a rotor which promotes intimate mixture and eliminates back-

pressure. The precipitated salt falls to the bottom of the tank, from which it is removed by a compressed air ejector.

T. S. WHEELER.

Cracking of petroleum oil. R. T. POLLOCK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,658,415, 7.2.28. Appl., 20.12.20).—The oil is cracked under pressure by passage through a heating coil and then enters a vapour chamber whence the vapours pass to a series of dephlegmating columns, the condensates from which are passed, under applied pressure, to independent heating zones, maintained at temperatures above that of the original heating coil.

C. O. HARVEY.

Cracking of [hydrocarbon] oils. J. C. CLANCY (U.S.P. 1,658,116, 7.2.28. Appl., 2.9.22).—Gasoline-like products are produced from heavier hydrocarbon oils by passing them (in the presence of hydrogen) in admixture with molten material containing an alkali cyanide through a retort packed with a metallic catalyst (nickel) maintained at a cracking temperature. The molten material is supplied to, and the mixture of this material with unvaporised oil is withdrawn from, the retort through liquid-sealed passages. The evolved vapours are collected.

C. O. HARVEY.

Treatment of natural-gas, casing-head, and natural gasolines etc. H. B. BERNARD, Assr. to SINCLAIR OIL & GAS Co. (U.S.P. 1,658,322, 7.2.28. Appl., 22.7.24).—The crude gasoline product flows in a helical path down a tower in counter-current to vaporised constituents (which are flowing upwardly), no mixing of vapours and liquid being attempted (*i.e.*, by spraying or bubbling). The vapours and gases are withdrawn at the top and the treated gasoline product at the base of the tower.

C. O. HARVEY.

Manufacture of valuable liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of mineral coal and other solid carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 256,964, 10.8.26. Ger., 14.8.25).—In the catalytic hydrogenation of coal and other carbonaceous materials (other than lignite or peat) at elevated temperatures and under pressures of at least 50 atm., an increased yield of liquid products is obtained and choking of the apparatus by coke-like products is avoided by mixing the initial material with lignite or peat or both, whereby a material is obtained which, under high pressures, may be caused to flow like a viscous liquid.

C. O. HARVEY.

Manufacture of valuable liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 257,256, 10.8.26. Ger., 20.8.25).—Motor fuel of a non-detonating character, lubricating oils, etc. are produced from carbonaceous materials (coal, wood, bone tar, mineral oil residues, bitumen, resins, etc.) by interaction at high temperatures and pressures with water vapour or with gases containing hydrogen and oxides of carbon, in the presence of catalysts capable of forming methyl alcohol by the catalytic hydrogenation of oxides of carbon (vanadic acid, manganese, uranium, zinc, and titanite oxides, etc.). The initial material may be submitted to a preliminary hydrogenation, and it is preferable to allow only gases

or finely-divided material to come into contact with the catalyst (in a separate chamber).

C. O. HARVEY.

Distillation of oils from oil shale or coal or similar material. R. H. CROZIER (B.P. 283,639, 13.10.26).—The distillation of oil from shales and coals of an intumescent character is facilitated and the yield is improved by carrying out the operation in a continuous vertical retort heated by gas burners, the upper portion of the retort, into which the raw material is introduced, being the hottest portion (about 400°). By this arrangement the pitch-like constituents are rapidly vaporised and a semi-coke is produced which readily flows downwards into the cooler zones of the retort, where it is further distilled.

C. O. HARVEY.

Refining of oil. E. C. R. MARKS. From SIMPLEX REFINING Co. (B.P. 283,383, 20.4.27).—In the treatment of oils under reduced pressure, loss of vacuum due to leakage through mechanical pumps is avoided by causing the oil to circulate automatically without mechanical aid. *E.g.*, in the distillation of lubricating oil, the vapours pass upwards through a dephlegmating column down which the condensate is caused to flow. The condensate collects in a chamber at the base of the column which is connected with a U-shaped pipe which passes downwards for some distance and then upwardly, finally discharging into the top of the dephlegmating column. The condensate collects in the U-tube and a hydrostatic head is established. By introducing a vapour (*e.g.*, steam) into the longer arm of the tube, a lighter mixture is produced, and the oil is thereby caused to rise and to discharge itself into the dephlegmator.

C. O. HARVEY.

Refining of oil. J. C. BLACK (U.S.P. 1,656,997, 24.1.28. Appl., 5.4.20).—The oil is treated at a reactive temperature with acid-treated clay.

C. O. HARVEY.

Refining of [hydrocarbon] oils. A. SCHWARZ, Assr. to PETROLEUM SAND PRODUCTS CORP. (U.S.P. 1,656,990, 24.1.28. Appl., 14.6.24. Renewed 3.12.27).—Hydrocarbon oils containing desirable and undesirable unsaturated hydrocarbons are freed from the latter by treatment with a suitable mixture of sulphuric acid and saturated hydrocarbons.

C. O. HARVEY.

Refining of petroleum oils. P. McMICHAEL, Assr. to HYDROCARBON REFINING PROCESS Co. INC. (U.S.P. 1,658,171, 7.2.28. Appl., 17.11.24).—Cracked petroleum oils are treated in a series of washing chambers successively with a solution of caustic soda (with or without the addition of sodium sulphide), an alkaline sodium chromate solution, water, a 70–83% solution of sulphuric acid, water, and finally with a solution of caustic soda.

C. O. HARVEY.

Removal of sulphur and sulphur compounds from hydrocarbon oil. G. L. WENDT, Assr. to STANDARD OIL Co. (U.S.P. 1,658,505, 7.2.28. Appl., 16.7.24).—The oil is treated with an alcoholic solution of sodium plumbite.

C. O. HARVEY.

Purification of crude benzol. I. G. FARBENIND. A.-G. (B.P. 258,576, 9.9.26. Ger., 15.9.25).—Crude benzol is substantially freed from sulphur compounds and converted into a mixture suitable for distillation for the production of pure benzol and other low-boiling hydrocarbons by treatment with hydrogen (or gases

containing it) under pressure (150—200 atm.) at temperatures of about 450° and in the presence of suitable catalysts (mixtures of molybdic acid with magnesium or zinc oxide etc.). Under these conditions the benzol itself is practically unaffected, and the emulsion losses inherent in the acid-refining process are avoided.

C. O. HARVEY.

Mineral oil compositions for lubricating, insulating, and other purposes. R. T. VANDERBILT CO. INC., Assees. of A. A. SOMERVILLE (B.P. 269,840, 9.10.26. U.S., 20.4.26).—Lubricating oils (particularly those for use at high temperatures or in air compressors) and insulating oils are rendered more stable and less liable to undergo atmospheric oxidation by the addition of small amounts (0.5 to a few per cent.) of condensation products of aldehydes with nitrogenous bases (e.g., the condensation product obtained by the interaction of acetaldehyde and aniline).

C. O. HARVEY.

Production of clear [lubricating] oils soluble in water. A. HORN (B.P. 281,476, 19.7.27).—A vegetable oil, such as castor oil, is heated to about 300°, allowed to cool, and then heated to 100—110° with hydrochloric acid vapour with the addition of a contact substance, e.g., calcined aluminium chloride. After cooling, oxygen, air, or ozone is blown through the oil at 150—200° in an open vessel to remove the acid. A contact substance is added such as hydroxides, carbonates, percarbonates, or perborates of the alkalis, stannous chloride, or bases such as resorcinol, pyridine, quinone, quinol, quinoline, or hydroxylamine and trimethylamine in hydrochloric acid or alone. Treatment is continued at 200° and lasts 4—5 hrs.

A. C. MONKHOUSE.

Manufacture of motor fuels. I. G. FARBENIND. A.-G. (B.P. 259,944, 2.10.26. Ger., 19.10.25).—Fuels for use in internal-combustion engines are improved in regard to absence of detonation, more suitable boiling range, ease of starting the engine, etc. by the addition of suitable proportions of products obtained by the destructive hydrogenation of coal, tars, oils, or other carbonaceous materials.

C. O. HARVEY.

Fuel for internal-combustion engines. S. ISERMAN, W. VERNET, and E. Q. MOSES, Assrs. to BOYCE & VEEDER Co., INC. (U.S.P. 1,654,259, 27.12.27. Appl., 22.7.24).—*o*-Nitrotoluene (0.1%) is added to petrol as a decarbonising agent. The use of 1:4-ditoluidino-anthraquinone (0.001%) to give a distinctive greenish-blue colour to petrol so treated is also claimed.

T. S. WHEELER.

Reactivation of adsorptive material. W. S. BAYLIS, Assr. to FILTROL Co. (U.S.P. 1,654,629, 3.1.28. Appl., 5.4.26).—Adsorptive material used to treat oil is removed in a filter-press in which the filter-medium is formed of a metallic fabric, and is then treated *in situ* with a mixture of superheated steam and air at 400°.

T. S. WHEELER.

Manufacture of gasoline. O. BEHMER, Assr. to TEXAS Co. (Re-issue 16,877, 14.2.28, of U.S.P. 1,585,496, 18.5.26).—See B., 1926, 623.

Recovery of acid and resinous matter from acid sludges formed in the refining of mineral oils.

W. DEMANN (B.P. 258,241, 25.8.26. Ger., 9.9.25).—See G.P. 436,242; B., 1927, 436.

Sulphonic acids from tar oils (G.P. 445,645).
Cresols etc. from creosote liquors (G.P. 444,971).—See III. **Absolute alcohol** (B.P. 283,701).—See XVIII.

III.—ORGANIC INTERMEDIATES.

Development of synthetic phenol from benzene halides. W. J. HALE and E. C. BRITTON (Ind. Eng. Chem., 1928, 20, 114—124).—The hydrolysis of halogenobenzenes with caustic soda solution at high pressures and temperatures up to 370° was studied by means of a rotating bomb of heavy piping. The optimum concentration of sodium hydroxide was concluded to be 8%. Higher strengths give an undue formation of tar, whilst lower strengths increase the production of diphenyl ether in place of phenol. The reaction is found to be unimolecular, and the concentration of alkali has no effect on the velocity of hydrolysis. The effect of catalysts is not great except at lower temperatures; metallic copper and cuprous oxide were the most efficient. Normally 6—10% of the product was diphenyl ether. This is an equilibrium value, and if such a proportion be added to the reaction mixture previously the whole of the phenyl halide may be recovered as phenol. At 370° diphenyl ether is rapidly hydrolysed by sodium phenoxide solution or other alkali. The hydrolysis of chlorobenzene is effected just as well with sodium carbonate as with sodium hydroxide. In this case there is no need for subsequent acidification. Under reaction conditions either iron, copper, or nickel is appreciably acted on by alkali with generation of hydrogen, and this hydrogen with phenol yields cyclohexanol, whence arises loss of product as tar. An iron bomb becomes protected by a coating of magnetic oxide, but any change in heat transmission causes this to flake. Benzenesulphonic acid and sodium hydroxide solution undergo a strictly analogous reaction under similar conditions, and the introduction of diphenyl ether has a similar effect upon the yield. The difficulties which have up to the present prevented the operation of a continuous process on these lines are discussed.

C. IRWIN.

Phosgene. M. and L. JACQUÉ (Chim. et Ind., 1928, 19, 24—38).—Phosgene is universally manufactured to-day by passing a mixture of chlorine with a slight excess of carbon monoxide over activated carbon. External cooling is necessary; the optimum temperature is 125°, and 150° must not be exceeded. Atmospheric oxygen if present results in a loss of yield. Carbon monoxide is prepared by the action of carbon dioxide and oxygen alternately on good quality anthracite or by passing carbon dioxide over heated wood charcoal. It must be freed from dust, carbon dioxide, and moisture. The gases are mixed in lead cylinders fitted with diaphragms and pass to the catalyst chambers, which are also of lead. In the usual process, in which a 37% excess of carbon monoxide suffices, 90% of the phosgene is condensed direct and the remainder absorbed in tetrachloroethane. If oxygen is dispensed with in the manufacture of carbon monoxide a 30% excess of the latter is necessary, and a large quantity of tetra-

chloroethane must be maintained for phosgene absorption with increase both in working losses and in the size of the apparatus. The design of a plant to produce 50 tons of phosgene per week, working with oxygen, is discussed in detail. Small gas generators fed with coke and fired by a current of oxygen with or without admixture of carbon dioxide are used. The gas exit pipe is water-cooled. The apparatus also includes coke scrubber, centrifugal dust separator, and alkaline and acid washers. The activated carbon used in the catalysers gradually deteriorates, but each kg. of carbon should yield at least 2 tons of phosgene. The catalyser proposed consists of horizontal lead cylinders in which the carbon rests on a grid. The exit gases after a preliminary cooling are reduced to -20° by passing through a coil cooled by calcium chloride solution. The unliquified portion passes to a tower in which it is washed with tetrachloroethane. The suitability of a sample of carbon for the process may be judged by determining its power of adsorbing the gases or phosgene. Phosgene is conveniently determined by passing the gas through a saturated aqueous solution of aniline, when crystals of diphenylurea which are almost insoluble in cold water are produced. A concentration of 45 mg./m.³ of phosgene in the atmosphere is dangerous. It can be removed by a spray of caustic alkali or of hexamethylenetetramine. The latter is also suitable for use in gas-masks.

C. IRWIN.

Guaiacolsulphonic acid. TURSKI and others. **Determination of aldehydes.** RADCLIFFE and SWANN.—See XX.

PATENTS.

Manufacture of formaldehyde. FABR. DE PROD. CHIM. "KALA" SOC. ANON., and N. KHARTSCHEV (F.P. 620,754, 7.8.26).—Methyl alcohol vapour and air are led over red-hot carbon. The catalyst may be externally heated or may be mixed with sufficient active carbon to heat it to redness on admission of the reaction mixture. Methyl alcohol which contains acetone or pyridine bases may be employed.

C. HOLLINS.

Manufacture of oxygen-containing carbon compounds. BRIT. CELANESE, LTD., H. DREYFUS, and W. VADER (B.P. 283,989, 20.7.26).—Methyl alcohol or a substance capable of generating it (such as dimethyl ether or methyl formate) is treated with carbon monoxide with or without hydrogen at 300–400° preferably under pressure and in presence of water vapour in contact with an inorganic acid or acid salt, especially phosphoric acid or aluminium phosphate ($Al_2O_3 \cdot 12H_3PO_4$). Acetic acid or methyl acetate is obtained, and may respectively be converted into acetone or hydrolysed to acetic acid. The methyl alcohol may be prepared catalytically from carbon monoxide and hydrogen, the crude gaseous product being applied. The process may be extended to the manufacture of aliphatic acids (or esters) generally from the next lower alcohols.

C. HOLLINS.

Manufacture of lead alkyls. M. A. YOUTZ, Assr. to GEN. MOTORS CORP. (U.S.P. 1,658,544, 7.2.28. Appl., 7.9.23).—Lead tetra-alkyls are prepared by reducing with nascent hydrogen a mixture of lead and an alkyl chloride.

B. FULLMAN.

Manufacture of aliphatic secondary amino-

alcohols. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of H. SCHOTTE (G.P. 442,413, 25.3.25).—Amino-alcohols, $R \cdot NH \cdot CH_2 \cdot CH_2 \cdot OH$, are obtained by the action of alkali on β -chlorourethanes, $R \cdot NH \cdot CO_2 \cdot CH_2 \cdot CH_2Cl$, an oxazolidone being probably formed intermediately. β -Chloroethyl methylcarbamate, b.p. 110–112°/15 mm., obtained from β -chloroethyl chloroformate and methylamine, gives β -methylaminoethyl alcohol, b.p. 159°/750 mm. (*picrate*, m.p. 148–150°). β -Chloroethyl ethylcarbamate, b.p. 110–111°/12 mm., β -chloroethyl isoamylcarbamate, b.p. 106°/1.5 mm., β -chloroethyl benzylcarbamate, b.p. 105°/0.8 mm., or 218–220°/15 mm., β -ethylaminoethyl alcohol, b.p. 169–170° (*picrate*, m.p. 125–126°), β -isoamylaminoethyl alcohol, b.p. 105–106°/15 mm. (*picrate*, m.p. 94–95°), and β -benzylaminoethyl alcohol, b.p. 148–149°/13 mm. or 105°/1 mm. [*picrate*, m.p. 104–106° (+ H₂O) or 134–135° (anhyd.)], are also described.

C. HOLLINS.

Treatment [concentration] of [dilute] lower aliphatic acids. BRIT. CELANESE, LTD., H. DREYFUS, and C. I. HANEY (B.P. 283,702, 30.11.26).—Dilute acetic acid (etc.) is extracted with a mixture of a hydrocarbon (*e.g.*, light petroleum, b.p. 40–70°, petrol, b.p. 70–90°, or benzene) and a solvent for the acid (*e.g.*, ether, chloroform, acetone oils), both of lower b.p. than the acid. The extracting mixture may be used in vapour form, and the acid is recovered by distilling off the solvent.

C. HOLLINS.

Preparation of pure lactic acid. C. H. BOEHRINGER SOHN (G.P. 444,956, 27.3.21).—Crude calcium lactate is treated with kieserite (in place of magnesium sulphate crystals, to avoid dilution), and the resulting magnesium lactate is converted directly into lactic acid or, better, since magnesium sulphate is rather soluble in concentrated lactic acid, into calcium lactate by stirring with milk of lime. In the latter case the precipitated magnesium hydroxide carries down coloured and odorous impurities.

C. HOLLINS.

Saponification of esters of organic acids. BRIT. CELANESE, LTD., and W. BADER (B.P. 284,582, 20.7.26).—Organic acids are obtained in concentrated form by hydrolysis of esters with the theoretical amount of water in presence of mineral acid, particularly sulphuric or phosphoric acid, at 150–300°. Ortho- or pyro-phosphoric acid contains sufficient water for hydrolysis (with conversion into metaphosphoric acid), and the process may be made continuous, ester and water in theoretical proportions being added to the metaphosphoric acid residue. *E.g.*, methyl acetate vapour and steam are introduced near the bottom of a cylindrical copper vessel, fitted with stirrer and filled with phosphoric acid at 180–220°; glacial acetic acid distils from the vessel (with a little unchanged ester) and the escaping gas is pure dimethyl ether according to the equation: $2Me \cdot CO_2Me + H_2O = 2Me \cdot CO_2H + Me_2O$. Methyl acetate vapour may also be passed up a tower with copper plates against a counter-current of phosphoric acid which is circulated and hydrated by a steam jet.

C. HOLLINS.

Production of sulphonic acids [from neutral low-temperature tar oils]. GELSENKIRCHENER BERGWERKS-A.-G., and F. SCHÜTZ (G.P. 445,645, 20.9.22).—

Products suitable for use, *e.g.*, as disinfectants, as frothing agents, and for impregnating wood, are obtained by sulphonating neutral low-temperature tar oils. Examples are given of the production of sulphonic acids from oils of b.p. 111° and b.p. 80°. L. A. COLES.

Preparation of cresols and other hydroxylated benzene compounds from creosote liquors. BRAUN-KOHLER-PROD. A.-G. (G.P. 444,971, 26.3.24).—Creosotes are distilled over magnesium or aluminium or their alloys (particularly magnalium or electron metal) at 500–600° to give cresols and xylenols. *E.g.*, a crude lignite tar, with 30% of benzine asphalt, distilled with aluminium turnings at 560–580° gave fractions (a) b.p. below 210°, (b) b.p. 210–280°. Alkali extracted 10% of (a), consisting of *o*-, *m*-, and *p*-cresols, and 15–20% of (b), consisting mainly of xylenols. C. HOLLINS.

Manufacture of highly chlorinated paraffins. A. SCHAARSCHMIDT (G.P. 442,829, 27.3.20).—Molten paraffin is chlorinated above 150° until the chlorine content exceeds 61.4%. At 150–160° a yellowish transparent mass is obtained. If this is heated at 180–200° hydrogen chloride is evolved, and further treatment with chlorine at 220° gives a brown, hard mass of high m.p. Free chlorine and hydrogen chloride are removed by bubbling air through the mass. The products serve for manufacture of lacquers, non-inflammable films, and plastic masses. C. HOLLINS.

Chlorination of saturated hydrocarbons. E. KRAUSE and K. ROKA, ASSRS. to HOLZVERKOHLUNGS-IND. A.-G. (U.S.P. 1,654,821, 3.1.28. Appl., 4.6.24. Ger., 14.11.23. Cf. U.S.P. 1,591,984; B., 1926, 898).—The process of the patent cited is modified in that hydrogen chloride is used in place of chlorine. T. S. WHEELER.

Manufacture of hydrogenated compounds. J. D. RIEDEL. A.-G. (G.P. 444,665, 19.1.19. Addn. to G.P. 369,374; B., 1923, 543 A).—The catalysts of the prior patent, with the exception of mixed elementary metals, are used for reduction of phenols, aldehydes, and ketones. *E.g.*, mixed carbonates of nickel, cobalt, and copper in atomic proportion, 60:30:10, at 130–170°, catalyse the conversion of benzaldehyde into benzyl alcohol by hydrogen at 25 atm.; at 210–220° cyclohexylmethylcarbinol is obtained in good yield without formation of benzhydrol. Acetone with hydrogen at 15 atm. gives at 100° in presence of the same catalyst isopropyl alcohol; β -naphthol is similarly converted by hydrogen at 190–200° and 10–30 atm. in 3 hrs. into tetrahydro- and in 5 hrs. into decahydro- β -naphthol. cycloHexanol is obtained from phenol and hydrogen at 190–215° and 25 atm. in presence of mixed oxides of the same metals. Cinnamaldehyde with hydrogen under pressure at 140° yields heptyl alcohol, b.p. 174–178°, and some diheptyl ether, b.p. 145°/9 mm. C. HOLLINS.

Catalytic decomposition of cyclic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 283,600, 12.7.26).—Simple benzene hydrocarbons are obtained by passing more complex compounds with hydrogen at 300–550° over mixed dehydrogenating catalysts, especially those containing iron, cobalt, nickel, molybdenum, vanadium, tungsten, manganese, chromium, or their compounds, activated by admixture

with difficultly reducible oxides of metals. Carbazole, passed with hydrogen at 350° over alumina and iron oxide, gives benzene and ammonia, with some aniline; cresol gives benzene and toluene; naphthalene gives benzene. Similarly, mixed benzene hydrocarbons are obtained from low-temperature tar of b.p. 180–250°.

C. HOLLINS.

3'-Nitro-4'-hydroxy-*o*-benzoylbenzoic acid.
3'-Amino-4'-hydroxy-*o*-benzoylbenzoic acid.
4'-Hydroxy-*o*-benzoylbenzoic acid. **4'-Amino-*o*-benzoylbenzoic acid.** [A, B, C] I. GUBELMANN, H. J. WEILAND, and O. STALLMANN, and [D] I. GUBELMANN, ASSRS. to NEWPORT CO. (U.S.P. 1,654,287–1,654,290, 27.12.27. Appl., [A, B] 28.6.26, [C] 29.11.26, [D] 8.12.26).—(A) 3'-Nitro-4'-hydroxy-*o*-benzoylbenzoic acid, from the corresponding chloro-compound by treatment with boiling aqueous sodium hydroxide solution, has m.p. 175°. (B) 3'-Amino-4'-hydroxy-*o*-benzoylbenzoic acid is formed from the nitro-compound (A) by reduction. (C) 4'-Hydroxy-*o*-benzoylbenzoic acid, from the corresponding chloro-compound by treatment with sodium hydroxide solution under pressure at 180° for 12 hrs., has m.p. 212–213°. (D) 4'-Amino-*o*-benzoylbenzoic acid, from the corresponding chloro-compound by treatment with ammonia solution in presence of copper or of a copper salt under pressure at 180° for 18 hrs., has m.p. 200° [decomp.] (*acetyl* derivative, m.p. 277°).

T. S. WHEELER.

Manufacture of condensation products of arylamines. P. HALER and H. KAPPELER (B.P. 266,358, 17.2.27. Switz., 17.2.26).—An arylamine (aniline, toluidine) is condensed in an acid medium with formaldehyde below 100°, and the orange-red product is washed and treated with alkali (solution of sodium hydroxide, ammonia, lime, sodium carbonate, or sulphide) to give a bone- or horn-substitute. Alternatively, the base and formaldehyde are condensed first in alkaline medium and the product is condensed with more formaldehyde in acid medium, followed by washing and treatment with alkali. Modified products are obtained by adding to the reaction mixture a diluent (water, glycerol, acetone, etc.), a filler (carbamide, tannin, cellulose, leather powder, asbestos, calcium phosphate, barium sulphate, emery, etc.), or a pigment (indigo, nigrosine, chrome yellow, etc.). The final product may further be heated above 100° with or without linseed oil etc. C. HOLLINS.

Manufacture of isatins and *N*-arylsulphonyl derivatives thereof. I. G. FARBENIND. A.-G. (B.P. 265,224, 28.1.27. Ger., 28.1.26).—Arylsulphonyl amides in the form of alkali salts are condensed with oxalyl chloride in a solvent (carbon disulphide) and the products are cyclised by means of aluminium chloride etc. to give *N*-arylsulphonylisatins, from which isatins are obtained by hydrolysis with alkali or cold concentrated sulphuric acid. *N-p-Toluenesulphonylisatins* are thus prepared from the *p*-toluenesulphonyl derivatives of *p*-toluidine (m.p. 202–205°), *m*-toluidine (mixture, m.p. 161–163°), and 3-chloro-*p*-toluidine (m.p. 180–190°); α -naphthylamine gives 1:8-naphthisatin, m.p. above 300°, instead of the expected 1:2-compound. *Sodium p-toluenesulphon-3-chloro-p-toluidide*, C₇H₇·SO₃·NNa·C₇H₆Cl, has m.p. 141–142°. C. HOLLINS.

Manufacture of 4-nitrobenzthiazoles. I. G. FARBENIND. A.-G., Assees. of K. BRAND (G.P. 442,773, 8.7.24).—Acylated 2-chloro-5-nitroanilines are treated with sodium sulphide (or with sodium disulphide, followed by reduction of the disulphide to a thiophenol) and then acidified to give 4-nitrobenzthiazoles. Examples are: 4-nitrobenzthiazole from 2-chloro-5-nitroformanilide, and its 1-methyl (m.p. 136—137°) and 1-phenyl (m.p. 192°) derivatives from the corresponding acetanilide and benzanilide, respectively. C. HOLLINS.

Manufacture of indophenols. I. G. FARBENIND. A.-G., Assees. of F. BALLAUF (G.P. 443,685, 3.11.25).—Indophenols are obtained from dihydroindoles by the usual general methods, and may, if desired, be reduced to leuco-indophenols. The products are intermediates for sulphide and vat dyes. 2-Methyldihydroindole is oxidised with chromic acid in presence of *p*-aminophenol, or is condensed with quinonechloroimide in concentrated sulphuric acid at 10°, and the product, after reduction to the leuco-indophenol, is made acid to Congo paper and salted out. Dihydroindole, 2-phenyldihydroindole, and 1:2-dimethyldihydroindole are similarly oxidised with *p*-aminophenol, and 2-methyldihydroindole with 2:6-dichloro-4-aminophenol. C. HOLLINS.

Manufacture of chlorine-substitution products of 1-amino-2:4-dimethylbenzene [chlorinated *m*-4-xylidines]. I. G. FARBENIND. A.-G. (B.P. 278,761, 11.10.27. Ger., 11.10.26).—4-Nitro-*m*-xylene is chlorinated in carbon tetrachloride solution in presence of ferric chloride, giving at 0° crystals of 2-chloro- and an oily 6-chloro-derivative, which are reduced to 2-chloro-*m*-4-xylidine, m.p. 47—48°, and 6-chloro-*m*-4-xylidine, m.p. 96—97°, respectively. Further chlorination, or chlorination without cooling in ice, gives 2:6-dichloro-4-nitro-*m*-xylene, reducible to 2:6-dichloro-*m*-4-xylidine, m.p. 56—57°. The bases are volatile in steam. C. HOLLINS.

Oxidation of arylalkyldithiocarbamic acids to the corresponding disulphides. SILESIA VER. CHEM. FABR. (B.P. 279,790, 11.7.27. Ger., 30.10.26).—The dithiocarbamates obtained by interaction of carbon disulphide and alkylarylamines are oxidised in alkaline solution to the thiuram disulphides by a current of nitric oxide mixed with air. C. HOLLINS.

Manufacture of pure urea. L. BUB, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,659,190, 14.2.28. Appl., 9.11.25. Ger., 15.11.24).—See B.P. 249,041; B., 1926, 420.

Manufacture of sulphuric acid esters of aromatic hydroxyalkyl ethers. W. HENTRICH and M. HARDTMANN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,644,524, 4.10.27. Appl., 21.6.26. Ger., 25.6.23).—See B.P. 266,940; B., 1927, 324.

Catalysts (B.P. 281,218).—See X. **Detergents** (B.P. 284,367).—See XII. **Mercaptobenzthiazoles** (B.P. 283,661 and 283,679).—See XIV.

IV.—DYESTUFFS.

Oxidation of organic dyestuffs and of cellulose on exposure to light. V. V. SCHARVIN and A. B. PAK-

SCHVER (J. Russ. Phys. Chem. Soc., 1927, 59, 459—463).—See B., 1927, 837.

Arsenic in coated papers etc. STERN.—See V.

PATENTS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 265,232 and 265,964, [A] 29.1.27, [B] 8.2.27. Ger., [A] 29.1.26, [B] 9.2.26. Addn. [A, B] to B.P. 237,294; B., 1925, 840).—The dyes of the prior patent are obtained by condensing 1:4:5:8-naphthalene-tetracarboxylic acid or its anhydride with (A) an *o*-nitroarylamine, and reducing the di-*o*-nitroarylimide so formed, whereby 3-nitro-*p*-phenetidine gives a brown vat dye, *o*-nitroaniline a red; or with (B) an *o*-diamine in alcohol or other low-boiling solvent, and heating the resulting product in acetic acid or other high-boiling solvent. C. HOLLINS.

Production of benzantrones and their derivatives. BRIT. ALIZARINE CO., LTD., W. H. DAWSON, C. W. SOUTAR, and J. ANDERSON (B.P. 284,035, 3.11.26).—Glycerol is treated with thionyl chloride, sulphuryl chloride, or sulphur mono- or di-chloride, preferably in equimolecular quantities, and the reaction mixture is then condensed in the usual way with anthranol and sulphuric acid to give a benzantrone. Alkaline fusion of the product obtained by using thionyl chloride gives a bright blue vat dye. When sulphur dichloride is used a benzantrone containing sulphur results. By using sulphur monochloride, sulphur is introduced in the Bz-ring and a dibenzanthronyl is obtained by alkaline fusion of the product. C. HOLLINS.

Manufacture of vat dyes of the dibenzanthrone series. I. G. FARBENIND. A.-G. (G.P. 443,610, 24.3.23).—The hydroxylated dibenzanthrones of G.P. 414,203 and 414,924 (B., 1925, 799; 1926, 234) are alkylated or arylated. By the action of methyl *p*-toluenesulphonate on tetrahydroxydibenzanthrone a violet vat dye is obtained, on dihydroxydibenzanthrone a grey-blue vat dye. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 260,588, 25.10.26. Ger., 29.10.25).—Anthraquinone-acridones (B.P. 894 of 1911) are obtained in a single operation by heating 1-chloroanthraquinone-2-carboxylic acid with an arylamine (β -naphthylamine, *p*-chloroaniline), preferably in presence of a mild condensing agent (borax, potassium hydrogen sulphate, boric acid, oxalic acid, arsenious oxide, sodium acetate with boric acid, etc.), in a solvent (nitrobenzene, trichlorobenzene). C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 263,494, 21.12.26. Ger., 22.12.25).—The alkylation products of pyrazolanthrones give by alkaline fusion red vat dyes. C. HOLLINS.

Manufacture of dyes of the anthanthrone series. L. CASSELLA & Co., G.M.B.H. (B.P. 260,998 and Addn. B.P. 280,217, [A] 3.11.26, [B] 2.11.27. Ger., [A] 7.11.25, Switz., [B] 2.11.26).—(A) Anthanthrone (G.P. 280,787) is halogenated under milder conditions than those described in G.P. 287,250 (B., 1916, 354). *E.g.*, chlorine in trichlorobenzene at 150°, or in sulphuryl chloride at 60° in presence of iodine, gives a yellowish-orange

vat dye; bromine in nitrobenzene at 160°, or in concentrated sulphuric acid at 50–100° in presence of iodine, gives a reddish-orange vat dye. (B) The dyes of the prior patent are obtained by cyclisation of 1:1'-dinaphthyl-8:8'-dicarboxylic acid in sulphuric acid followed by halogenation in the same medium without isolation. Economy of bromine may be effected by adding sufficient sulphur trioxide (as high-strength oleum) to oxidise the hydrogen bromide, before bromination.

C. HOLLINS.

Preparation of condensation products [vat dyes] of the anthraquinone series. I. G. FARBENIND. A.-G., Assees. of H. TESCHE and P. TUST (G.P. 443,584, 14.2.25).—*o*-Diaminoanthraquinones are heated with metal salts or oxides, preferably in a solvent. 1:2:4-Triaminoanthraquinone heated with ferric chloride in nitrobenzene with addition of sodium acetate yields a brown to brown-black vat dye, also obtained by using copper chloride or by using red lead in acetic acid, or by the action of hydrochloric acid or sodium hydroxide on the intermediate product prepared by action of copper chloride and sodium acetate on the triaminoanthraquinone in acetic acid. 1:2-Diamino-4-benzamidoanthraquinone is similarly converted into a red-brown vat dye, 1:2-diaminoanthraquinone a brown, and 2:3-diaminoanthraquinone a brown-olive.

C. HOLLINS.

Purification of vat dyes. SOC. CHEM. IND. IN BASLE [GES. F. CHEM. IND. IN BASEL] (B.P. 278,728, 6.10.27. Switz., 6.10.26).—The vat dyes made from aminoanthraquinones and cyanuric chloride etc. (B.P. 205,525 [see U.S.P. 1,437,783], 231,688, 234,086, 237,872; B., 1923, 490 A; 1925, 438, 624, 839) are improved in purity and fastness by treatment with aqueous hypochlorite solutions.

C. HOLLINS.

Vat dyes of the isatin-oxthyionaphthen group. C. KRAUHS, Assr. to GRASSELLI DYE STUFF CORP. (U.S.P. 1,650,139, 22.11.27. Appl., 5.1.26. Ger., 8.1.25).—7-Methylisatin or a chloro-derivative is condensed with an oxthyionaphthen to give dyes yielding in the vat yellow to orange to brown shades. T. S. WHEELER.

Manufacture of indigoid vat dyes. KALLE & Co. A.-G., Assees. of M. P. SCHMIDT and O. HERRMANN (G.P. 441,439, 27.9.24).—An isatin α -anil or α -chloride is condensed with a tetrahydronaphthacarbazole, with subsequent halogenation if desired. *E.g.*, 4-hydroxy-7:8:9:10-tetrahydro- α -naphthacarbazole (as annexed formula), prepared by alkaline fusion of the product from cyclohexanone and α -naphthylhydrazine-5-sulphonic acid, gives with 5-chloroisatin α -anil a violet vat dye; 3-hydroxy-7:8:9:10-tetrahydro- β -naphthacarbazole with isatin α -anil a blue; 3-hydroxy-8(?)-methyl-7:8:9:10-tetrahydro- β -naphthacarbazole with isatin α -anil a blue-violet.

C. HOLLINS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G., Assees. of FARBENFABR. VORM. F. BAYER & Co. (B.P. 259,970, 13.10.26. Ger., 14.10.25).—Direct developing

green dyes of the type *p*-diamine \rightarrow middle component \rightarrow an aminonaphtholsulphonic acid carrying an external amino-group \rightarrow 2-methylindole or 1-phenyl-3-methylpyrazolone, are described. The *p*-diamine may be *p*-nitroaniline-*o*-sulphonic acid (reduced after final coupling), or *p*-aminoacetanilide-3-sulphonic acid or other acylated *p*-diamines of the benzene or naphthalene series (hydrolysed after final coupling), or derivatives of these. As middle components are used cresidine, α -naphthylamine, α -naphthylamine-6(or 7)-sulphonic acid or its 2-ethoxy-derivative, 1-amino-5-naphthol-7-sulphonic acid, 1-amino-2-naphthol ethers, etc., a middle component of the benzene series or of the naphthalene series being employed with a first component of the same series. Suitable third components with an external amino-group are the *p*-aminobenzoyl derivatives of 2:5:7-, 2:8:6-, or 1:8:4-aminonaphtholsulphonic acids, or the naphthimazoles containing an aminophenyl substituent in the hetero-ring, derived from 1:2:5:7-, 1:2:8:6-, and 1:2:8:4-diaminonaphtholsulphonic acids, and the corresponding naphthathiazoles. The dyes may be diazotised and developed on the fibre with β -naphthol or 1-phenyl-3-methylpyrazolone.

C. HOLLINS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G. (B.P. 263,164, 15.12.26. Ger., 15.12.25).—Diazo compounds are coupled with arylamides of sulphonated 2:3-hydroxynaphthoic acid (B.P. 183,428; B., 1922, 853 A) in substance or on the fibre, the process of B.P. 260,339 (B., 1927, 9) being excluded. The azo dyes so formed dye animal or vegetable fibres or may be used for lake-formation. Examples are: sulphonated 2:3-hydroxynaphthoic anilide with diazotised aniline (orange), *m*-aminobenzanilide (yellowish-red), *m*-aminobenzaldehyde (yellowish-red), or aniline-*o*-sulphonic acid (red); *o*-toluidide or 4-chloro-*o*-anisidide with diazotised *o*-phenetidine or *m*-4-xylydine (bluish-red); *o*-toluidide with tetrazotised dianisidine (bluish-violet); 4-chloro-*o*-toluidide with diazotised 4-nitro-*o*-toluidide (ruby-red); β -naphthylamide with *o*-phenetoleazo- α -naphthylamine (black); and sulphonated bis-2:3-hydroxynaphtholylbenzidine with diazotised *p*-nitroaniline (brownish-red ice colour on cotton).

C. HOLLINS.

Manufacture of [azo] dyes containing chromium. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 283,979, 17.7.26).—Azo dyes, made by coupling a diazotised *o*-aminophenol with a naphthol or an arylaminonaphtholsulphonic acid, are heated with a chromium solution under pressure, with or without subsequent treatment with alkali. The chromium compounds of (a) 1:2:4-aminonaphtholsulphonic acid \rightarrow β -naphthol and (b) *p*-nitro-*o*-aminophenol \rightarrow 2-phenylamino-8-naphthol-6-sulphonic acid, dye wool in pure blue and dark brown shades, respectively.

C. HOLLINS.

Manufacture of black copying colours. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 283,777, 24.5.27).—Black copying colours are made by coupling a diazotised dialkylated phenosafranine with a cresol, particularly diethylphenosafranine with *p*-cresol (cf. B.P. 14,687 of 1895).

C. HOLLINS.

Chromiable brown disazo [mordant] dyes. W. NEELMEIER, T. NOCKEN, and W. REBNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,643,222, 20.9.27. Appl., 22.4.26. Ger., 28.4.25).—See B.P. 251,637; B., 1927, 771.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemistry of *Phormium tenax*. P. W. AITKEN (New Zealand J. Sci. Tech., 1927, 9, 226—228).—The present state of knowledge of the constituents of *Phormium tenax* (New Zealand flax) is summarised.

J. R. NICHOLLS.

Manufacture of ground-wood pulp. H. NAKAMURA and Y. SASAOKA (Sexagint [Osaka celebration], Kyoto, 1927, 181—184).—Analyses of ground-wood pulp coming directly from the stone and of that collected on the centrifugal pulp screens show the latter to contain more cellulose and less pentosan than the former.

H. F. GILLBE.

Arsenic in coated papers and boards. H. J. STERN (Analyst, 1928, 53, 83—86).—A paper or board used for wrapping in its uncoated state is not likely to contain any dangerous amount of arsenic, nor are the adhesives employed for coating sources of danger. Of the synthetic pigments used, antimony oxide may be employed as part of the base for colour lakes, and the so-called arsenical greens may contain over 30% As_2O_3 . There appears to be no danger from the lakes of natural dyewoods, and, except for dyestuffs (such as Magenta and Methyl Violet) precipitated by arsenious oxide in alkali solution, or those produced by means of tartar emetic, synthetic dyestuffs and pigments are generally free from arsenic, although contamination during manufacturing processes is possible. Pigment Scarlet 3B and Orange II quite commonly contain 50—100 pts. of arsenic per million, and Pigment Fast Red PRL, Fast Orange RL, and colours of the type of Patent Blue A (Disulphine Blue A) are also liable to contain arsenic. A limit for arsenic in coated boards and papers in force with certain users is 10 pts. per million.

D. G. HEWER.

PATENTS.

Preparation of fibrous vegetable materials for textile and other purposes. VICKERS, LTD., and O. D. LUCAS (B.P. 283,285, 8.10.26, 4.12.26, and 2.4.27).—The material, after scutching, is boiled first with caustic soda solution of less than 2% strength and then, with or without washing, with a similar solution of alkali containing 0.1% of an oil such as linseed oil, suitable times of treatment for retted flax being 1 hr. and 0.25 hr. respectively. After washing with cold water the fibres are treated with weak acid, e.g., 0.2—0.4% hydrochloric acid, optionally containing a small proportion of sodium hypochlorite, and, after further washing, are dried. The operations are preferably carried out in open vats with the fibres loosely arranged in reticulated trays, to which latter a vertical reciprocating movement is given to ensure efficient circulation of the treating liquor.

D. J. NORMAN.

Improvement of half-wool fibrous material which cannot be felted. A. S. KESSLER (G.P. 446,267, 7.4.26).—Low-grade material containing an excess of

cotton is treated cold with strong alkali solutions containing strengthening and moistening agents, e.g., glycerin, alcohol, sulphite-cellulose waste liquor, etc., as well as material for protecting the wool.

L. A. COLES.

Improvement of cloth by the galvanic deposition of metals upon it. W. ENZ (Austr. P. 106,449, 11.1.26. Ger., 15.1.25).—The cloth during the electrolytic treatment is tightly packed on an electrode constructed of plastic, conducting or partially conducting material surrounding a solid core; e.g., a mixture of freshly-precipitated copper sulphide and oleic acid packed around a sheet of copper is used for coppering the cloth.

L. A. COLES.

Impregnation of fibres and fibrous materials. P. HEERMANN (G.P. 445,771, 18.5.26).—Heterocyclic bases, such as pyridine and its derivatives, are used as solvents in impregnating fibrous materials with oleates, stearates, resinates, etc. in water.

L. A. COLES.

Removal of fat from raw sheep's wool. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 282,164, 17.9.26).—The organic esters of carbonic acid, particularly the methyl and ethyl esters, are used, either alone or in conjunction with the more usual reagents, for extracting fat from raw wool. These esters differ from other solvents in that they do not cause twisting and tangling of the wool nor do they remove dirt. The solvent adhering to the wool may be washed out with water and recovered by extracting the aqueous liquor with a suitable solvent, e.g., dichlorobenzene.

D. J. NORMAN.

Treatment of fibrous material to render it proof against moth. L. E. JACKSON and H. E. WASSLE (B.P. 263,092, 29.9.26. U.S., 17.12.25; cf. B., 1927, 870).—The material is treated with a cinchona alkaloid or a derivative thereof. Quinidine salts are particularly suitable, and may be applied in solution in water (as hydrochloride), alcohol (as sulphate), or petroleum naphtha or other organic solvent (as oleate). The strength of the solution should be at least 1% calculated as quinidine. [Stat. ref. to B.P. 146,225, 104,684, and 8858 of 1895.]

D. J. NORMAN.

Moth-proofing compounds. I. G. FARBENIND. A.-G. (G.P. 442,901, 17.7.25).—Phthalic acid and its derivatives, especially neutral or acid phthalic esters, phthalimide, halogenophthalic esters or acids, hydroxy, phthalic acids, methylamine and pyridine phthalates are claimed as moth-proofing agents. They may be used as solids, or in solution in volatile solvents. The butyl and amyl esters are specially mentioned.

C. HOLLINS.

Production of figures on vegetable textile material by mercerisation. E. GUMDER (B.P. 267,566, 14.3.27. Ger., 13.3.26).—The fabric is impregnated with a mercerising solution, stretched, and printed while under tension with a suitable neutralising medium, e.g., dilute hydrochloric acid in the case of alkaline mercerising agents. The printed fabric is then allowed to shrink.

D. J. NORMAN.

Treatment [weighting] of yarns, fabrics, films, etc. [containing cellulose esters or ethers]. H. DREYFUS (B.P. 281,084, 26.5.26).—Increased quantities

of loading agents can be introduced into cellulose esters or ethers if the material is treated with aqueous solutions of swelling agents, particularly organic swelling agents, *e.g.*, glycollic acid, lactic acid, etc., before or during each stage of the loading operation or repetition thereof. Suitable swelling solutions are aqueous solutions containing 7–10% of acetone, 50% of ethyl alcohol, 15–20% of diacetone alcohol, or 10–12% of ammonium thiocyanate. D. J. NORMAN.

Manufacture of artificial [silk] threads. L. LILIENFELD (B.P. 281,352, 30.6.26).—The extensibility of artificial threads produced by the process described in B.P. 274,521, 274,690 (B., 1927, 745), and 264,161 can be increased to at least 8% without deleteriously affecting the strength or lustre by treating the threads with shrinking agents under conditions which permit complete or partial shrinkage to occur. Suitable shrinking agents include mercerising solutions, *e.g.*, caustic soda solutions (18–25%) at 15–18° with or without the addition of neutral or alkaline salts or mono- or poly-hydric alcohols, sodium sulphide solution (50–58%), and mixtures of caustic soda and sodium sulphide. If desired the threads may be pretreated with protective agents such as starch, gelatin, etc. The mercerised threads are centrifuged, transferred (with or without washing) to a neutralising bath, washed, bleached, and finally dried without tension. The process is also applicable to artificial silk in fabric form, and to mixtures of artificial silk with vegetable or animal fibres. D. J. NORMAN.

Manufacture of threads, filaments, etc. from viscose. COURTAULDS, LTD., H. J. HEGAN, and E. HAZELEY (B.P. 282,973, 20.12.26).—Filaments which show a subdued lustre and a certain roughness of surface resulting in improved felting properties are made by spinning a viscose solution containing a small proportion of added soluble carbonate (2–4%) and sufficient caustic soda, *e.g.*, over 7%, to give a ratio NaOH:CO₂ of, *e.g.*, 4:1 or more into a sulphuric acid bath containing a high proportion, *e.g.*, 25% or more, of sodium sulphate. The filaments show little inflation as evidenced by their density (1.45–1.53). D. J. NORMAN.

Manufacture of artificial materials from viscose. L. LILIENFELD (B.P. 281,351, 29.6.26. Addn. to B.P. 274,521; B., 1927, 745).—Artificial threads having a strength of more than 2 g. per denier in the dry state are made by spinning a viscose solution containing not more than 5% of caustic alkali calculated as NaOH into a bath containing 45–55% of sulphuric acid, as H₂SO₄. D. J. NORMAN.

Production of alkali-cellulose from sheets of cellulose impregnated with alkali solution. I. G. FARBENIND. A.-G. (G.P. 445,728, 27.5.24).—The vats in which the sheets of cellulose have been steeped are drained and tilted until the sheets, which during the steeping hang vertically, lie in a horizontal or nearly horizontal position. Gentle pressure is applied to the sheets by means of partition walls which slide in grooves in the sides of the vat, thus expressing the greater part of the excess liquor, the remainder being subsequently removed in presses. L. A. COLES.

Treatment of natural and artificial cellulose fibres with alkali. CHEM. FABR. VORM. SANDOZ (B.P.

279,784, 10.6.27. Ger., 29.10.26).—Raw and sized cotton fabrics may be satisfactorily mercerised without a preliminary bowking if a small quantity (1–2%) of a suitable mixture of phenols (particularly the cresols and higher homologues) and hydrogenated aromatic compounds, *e.g.*, tetrahydronaphthalene, cyclohexanol, decahydro- β -naphthol, is added to the mercerising liquor. Mixtures containing 88–98% of phenols and 12–2% of a hydroaromatic compound give particularly good results. D. J. NORMAN.

Treatment of precipitated acetylcellulose. VER. F. CHEM. IND. A.-G. (B.P. 269,543, 12.4.27. Ger., 15.4.26).—The precipitated cellulose acetate is formed into a continuous coherent web, and, after squeezing between heavy rollers to remove as much as possible of the adherent acid liquor, is washed with water in a series of vats working on the counter-current principle. The web is finally dried on drying cylinders. The process is continuous and facilitates the recovery of acetic acid. D. J. NORMAN.

Spinning of artificial threads according to the stretch-spinning process. W. SCHULZ (B.P. 261,365, 4.11.26. Ger., 11.11.25).—During the stretch-spinning of, *e.g.*, cuprammonium hydroxide solutions of cellulose into a bath at about 40°, there is a tendency for the gases initially dissolved in the coagulant to form bubbles which intermingle with the filaments. To avoid this, sufficient pressure is applied to the precipitating liquor to prevent the liberation of gases therefrom at the operating temperature. Thus the liquor may be supplied to the spinning funnel under any desired head of pressure through a pipe, the diameter of which is large compared with that of the outlet of the funnel. D. J. NORMAN.

Treatment of artificial silk yarns after spinning. F. J. GAHLERT (B.P. 283,752, 10.2.27).—Artificial silk yarn, closely resembling natural silk in appearance and capable of giving a solid washable fabric showing no irregularities after dyeing and finishing, is obtained by passing ordinary twisted artificial silk yarn under tension through a bath of, *e.g.*, water, and then imparting to it a further mechanical twist. D. J. NORMAN.

Treatment of rayon [artificial silk]. E. K. GLADDING and T. E. SHARPE, Assfs. to DU PONT RAYON Co., INC. (U.S.P. 1,655,097, 3.1.28. Appl., 27.3.26).—Artificial silk is freed from copper by treatment with dilute (0.2%) sodium cyanide solution containing 0.1% of ammonia. T. S. WHEELER.

[Weighting of] cellulose [acetate] fibres, fabrics, and articles. BRIT. CELANESE, LTD. (B.P. 258,874, 21.9.26. U.S., 22.9.25).—Fabrics composed of or containing cellulose esters or ethers are impregnated at, *e.g.*, 45–65° with an aqueous solution of a metal salt, which is a swelling agent for the ester or ether, and are then treated with a reagent to precipitate the metal as an insoluble derivative. *E.g.*, 1 pt. of cellulose acetate silk is soaked in 30 pts. of stannic chloride solution (*d* 1.2) for 0.5 hr. at 40–50°, and, after rinsing, is transferred to a solution of disodium hydrogen phosphate (*d* 1.035). After a further rinse the fabric is treated with sodium silicate solution (*d* 1.035), and is finally washed with or without soap solution. Metal salts which are not swelling agents for cellulose acetate may be used in

conjunction with such swelling agents as dilute acetic acid. The treated silk shows not only increased weight and volume, but also an increased resistance to heat.

D. J. NORMAN.

Compositions containing cellulose esters and ethers etc. H. J. HANDS, and SPICERS, LTD. (B.P. 279,139, 21.4.26).—Chloropropanes having more than three atoms of chlorine per mol., *e.g.*, penta-, hexa-, or hepta-chloropropane (including all isomerides) or mixtures of these, are used as plasticising agents for cellulose esters or ethers. When used in small quantities, *e.g.*, 2–3% on the weight of ester, in association with other known plasticising agents, they are effective in reducing inflammability.

D. J. NORMAN.

Treatment of bagasse fibres preparatory to pulp board making. H. T. PRICE (U.S.P. 1,656,829, 17.1.28. Austral., 16.7.26).—The material is suspended in water and heated at 100°. Caustic soda is added and the mixture boiled for about 1 hr. with stirring. The bagasse is then passed between rollers and is again boiled for 1 hr.

D. J. NORMAN.

Drying of waste sulphite liquors and other viscid liquids to obtain dry granular solids. H. G. C. FAIRWEATHER. From INDUSTRIAL WASTE PRODUCTS CORP. (B.P. 282,480, 6.9.26).—The heated or superheated liquor is sprayed into the hottest part of a current of heated gas and is carried along therewith through a drying chamber until desiccation is complete. The temperature of the drying gas may be up to 538° at its hottest part, and should at all parts be above the b.p. of the liquor. The separated dry particles have a glazed surface and consequently show a reduced tendency to coalesce. [Stat. ref. to B.P. 215,315, 209,148, 190,099, 151,965, 145,079, and 19,350 of 1913.]

D. J. NORMAN.

Manufacture of transparent papers, particularly those used for the packing of edible articles. O. KLOTZ (B.P. 283,751, 7.2.27).—Pergamyn, parchment, tissue, or other suitable paper is coated on one or both sides with a solution of gelatin.

D. J. NORMAN.

Testing of pulp suspensions. M. O. SCHUR, Assr. to BROWN Co. (U.S.P. 1,653,125, 20.12.27. Appl., 20.6.24).—The suspension is poured on to a foraminous screen and the thickness of the resulting layer of pulp is noted.

D. J. NORMAN.

Continuous manufacture of cellulose acetate. SOC. CHIM. DES USINES DU RHÔNE (B.P. 270,656, 24.3.27. Fr., 10.5.26).—See F.P. 615,879; B., 1927, 811.

Manufacture of [multiple-ply] textile fabrics. E. WEINHEIM (B.P. 255,476, 16.7.26. Ger., 17.7.25).

Production of combed materials, rovings, fine yarns, etc. from artificial fibres. C. NIETHAMMER (B.P. 275,540, 15.10.26. Ger., 9.8.26).

[Apparatus for] impregnating and rendering rigid pieces of paper or fabric of any form. MANUF. DE MACHINES AUXILIAIRES POUR L'ÉLECTRICITÉ ET L'IND. (B.P. 280,386, 10.12.26. Adn. to B.P. 215,726).

Keratin threads (G.P. 445,503).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Development of vat dyes on the fibre by means of nitrites. I. G. FARBENIND. A.-G., Asses. of K. JELLINEK (G.P. 441,984, 20.6.25).—Leuco-vat dyes are oxidised with nitrite in a bath containing formic acid and an electrolyte. This makes possible the use of vat colours with ice-colours for printing. Bright, fast prints are obtained.

C. HOLLINS.

Dyeing of animal fibres, in particular silk, by means of ice-colours. J. W. LEITCH & Co., LTD., A. E. EVEREST, and J. A. WALLWORK (B.P. 283,347, 8.7.26).—Silk or wool, or a mixture of these or a mixture with cotton or viscose, in loose fibre, yarn, or piece goods, is padded with a dilute neutral or alkaline solution of α - or β -naphthol containing soap, Turkey-red oil, etc. and, after squeezing or hydro-extraction, is treated with a diazo solution at 0°, or is printed with a diazo paste. Thus a 2-litre padding bath may contain 1 g. of β -naphthol, 5 g. of olein or stearin soap, and 10 g. of sodium carbonate. The naphthol may with less advantage be applied in dilute aqueous alcohol, acetone, or pyridine solution.

C. HOLLINS.

Dyeing of wool or silk by means of ice-colours. J. W. LEITCH & Co., LTD., A. E. EVEREST, and J. A. WALLWORK (B.P. 283,838—9, 8.7.26).—(A) Wool, alone or in mixtures (except with silk), (B) silk, alone or in mixtures, is dyed by the process described in B.P. 283,347 (preceding), using in place of a naphthol a 2 : 3-hydroxy-naphthoic arylamide.

C. HOLLINS.

Dyeing of cellulose acetate and materials made therefrom. I. G. FARBENIND. A.-G., Asses. of H. KESSELER and E. DÖRING (G.P. 444,961, 22.2.24).—Acetate silk is dyed with basic dyes with addition of not more than 5 g. of a soluble thiocyanate per litre of dye bath, together with the requisite water-soluble colloid. Dyeings fast to rubbing are obtained.

C. HOLLINS.

Dyeing, printing, or stencilling of materials composed of or containing cellulose esters. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 284,376, 21.10.26).—The processes for the dyeing etc. of cellulose acetate materials with dispersions of insoluble coloured organic compounds (B.P. 219,349, 224,925, 242,393, 242,711, 269,960, 273,819, 273,820; B., 1924, 906; 1925, 39; 1926, 87, 50; 1927, 475, 650) are extended to the dyeing etc. of materials made from other cellulose esters, except cellulose formate obtained by formylation with strong formic acid and a catalyst below 5° (cf. B.P. 260,650, B., 1927, 103). Examples are: cellulose formate silk dyed with 4-chloro-2-nitro-4'-ethoxydiphenylamine or 5 : 7-dibromo-3-indole-2'-thionaphthene-indigo in presence of ammonium sulphoricinoleate; cellulose propionate silk dyed with 5 : 8-dimethyldiaminoalizarin in presence of sulphonated naphthalene-ricinoleic acid condensation product, or with 4-chloro-2-nitro-4'-methoxydiphenylamine in presence of xylene and Turkey-red oil.

C. HOLLINS.

Printing with vat dyes. I. G. FARBENIND A.-G., Asses. of H. GOSSLER (G.P. 444,962, 26.2.25).—Vat dyes are printed with alkalis and excess of zinc dust (or other metal which develops hydrogen in presence of alkali),

and steamed. The prints before steaming are quite stable, and the shade obtained is fuller than by the usual methods.

C. HOLLINS.

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

Manufacture of hydrated baryta from the carbonate. P. BAUD (Compt. rend., 1928, 186, 438—441).—A mixture of barium carbonate (100 pts.) and ferric oxide (12—15 pts.) heated in a tube-furnace at 1150—1180° for 1—2 hrs. yielded a black product containing 56—58% BaO (water-soluble). The addition of calcium carbonate rendered the mass porous as a result of the liberation of carbon dioxide, whilst the use of a mineral containing 76% Fe₂O₃ yielded a clinker containing 132% BaO.9H₂O. High yields were obtained on the commercial scale, and the process might be used for the preparation of alkali and barium sulphate: $\text{Ba(OH)}_2 + \text{Na}_2\text{SO}_4 = \text{BaSO}_4 + 2\text{NaOH}$.

J. GRANT.

Phosgene. JACQUÉ.—See III. **Germicidal efficiency of sodium hydroxide.** LEVINE and others. **o-Tolidine test for chlorine.** McCrUMB.—See XXIII.

PATENTS.

Catalytic production of hydrocyanic acid from formamide. I. G. FARBENIND. A.-G. (B.P. 269,166, 1.4.27. Ger., 10.4.26).—Vapours of formamide or formamide containing ammonium formate are highly diluted with ammonia and/or inert gases, and are passed at high velocity at reduced pressure, and above 300°, over solid metals the catalytic action of which is not impaired by the temperature and reaction materials, or are passed through tubes made of, or lined with, such metals. Examples of such catalysts are iron, aluminium, nickel, or V2A steel tubes, alone or filled with various metallic turnings.

W. G. CAREY.

Manufacture of active silica. I. G. FARBENIND. A.-G. (B.P. 255,864, 19.7.26. Ger., 23.7.25).—Silica is precipitated from suitable silicon compounds without any intermediate formation of a sol or gel in an acid, neutral, or only slightly alkaline solution, the precipitate is freed from mother-liquor and subjected to a pressure of 100 atm. or more without heating.

W. G. CAREY.

Effecting [purifying hydrogen by] catalytic reactions for the production of ammonia. LAZOTE, INC., Assees. of R. WILLIAMS (B.P. 258,887, 23.9.26. U.S., 24.9.25).—Hydrogen containing 2—10% CO is passed under a pressure of 900 atm. at 400° over a catalyst made by heating zinc carbonate or a mixture of zinc and chromic hydroxides at 400°. In this way the carbon monoxide is converted into methyl alcohol, which is removed by cooling the gases, still under pressure, and at the same time acts as a solvent for traces of other impurities in the gases. After passing through the cooler, the last traces of water and methyl alcohol are removed by means of active charcoal, and the hydrogen-nitrogen mixture is then passed directly over the ferric oxide catalyst to obtain ammonia.

A. R. POWELL.

Production of salts from brines and solutions. O. V. MARTIN, Assr. to MARTIN-COLVIN Co. (U.S.P. 1,657,633, 31.1.28. Appl., 19.5.26).—The solution is heated and a portion is converted into a mist over the

rest of the solution, thereby warming the air with which the spray comes into intimate contact and keeping it above its dew point. Rapid evaporation of the solution occurs in the zone of mist.

W. G. CAREY.

Manufacture of alkali nitrates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 283,771—2, [A] 25.4.27, [B] 29.4.27).—(A) In manufacturing alkali nitrates by the action of oxides of nitrogen on the corresponding chlorides, the vapours evolved contain about 1% of the oxides of nitrogen and 3% of hydrogen chloride; by treatment with sulphuric acid under pressure the nitrogen oxides are absorbed with the formation of nitrosyl-sulphuric acid, from which they may be recovered by known methods. The acid will absorb up to 5% of its weight of nitrogen as oxide without separation of crystals, and only 0.025% of hydrogen chloride is simultaneously absorbed. (B) An acid solution of alkali chloride saturated at 15° and containing some nitrate is treated under pressure (e.g., 6 atm.) at 35° with a mixture of nitric oxide, oxygen, and nitrogen (e.g., in the volume ratio of 9 : 15 : 76). When absorption of the nitric oxide is complete the pressure is released and solid alkali chloride equivalent to the nitric acid formed is added; on cooling to 15° the corresponding nitrate crystallises out.

A. R. POWELL.

Production of nitrate of lime. APPAREILS ET ÉVAPORATEURS KESTNER (B.P. 279,037, 26.8.27. Fr., 16.10.26).—Coarsely crushed limestone is treated with nitric acid in a tower provided for forced circulation of the carbon dioxide in the direction of the general movement of the materials, thus avoiding the opposition of the gas to the flow of nitric acid and nitrate, a partial vacuum being produced at the outlet of the tower or an increased pressure at its inlet. A heater causes complete disengagement of the carbon dioxide from the newly-prepared calcium nitrate liquor.

Production of zinc salts [sulphate]. METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 278,747, 7.10.27. Ger., 7.10.26).—Liquors obtained by leaching with dilute sulphuric acid the product resulting from the chloridising roasting of zinciferous pyrites are digested with cement copper to remove dissolved copper as cuprous chloride. The solution is used for leaching a further quantity of roasted material, again denuded of copper, cooled to 5° to allow Glauber's salt to crystallise out, and put through the whole process again to obtain a solution containing 400 g./litre of zinc sulphate. This liquor is freed from iron by treatment with lime and from copper and cobalt by boiling with zinc dust; on cooling to 10° crystals of zinc sulphate free from sodium sulphate are obtained. Alternatively, the purified liquor may be concentrated until it contains 620 g./litre of zinc sulphate and cooled to 60°, whereby sodium sulphate in excess of 70 g./litre crystallises out as the double salt with zinc sulphate. Further cooling to 10° yields pure zinc sulphate. A third method of treating the solution comprises adding sufficient sulphuric acid to provide an excess of 20% over that required to form sodium hydrogen sulphate and evaporating the solution to obtain zinc sulphate monohydrate.

A. R. POWELL.

Manufacture of alumina [from bauxite, clays, etc.]. J. C. SEAILLES (B.P. 277,697, 19.9.27. Fr.,

18.9.26).—To render the alumina content more readily soluble, clays are calcined at 650–750° and bauxite at 500–600°. In either case the calcined product is heated with a solution or suspension in water of an alkaline-earth hydroxide either with or without pressure. With barium hydroxide a solution of barium aluminate is obtained which is separated from the insoluble portion and treated with sodium carbonate or sulphate to obtain a solution of sodium aluminate, from which alumina may be precipitated and sodium carbonate regenerated by known methods; the barium sulphate or carbonate precipitate is converted into hydroxide for further use in the process. An alternative procedure consists in treating the barium aluminate solution with lime to precipitate calcium aluminate (which is converted into the sodium salt as described above), and to regenerate directly barium hydroxide. In case calcium hydroxide is used to decompose the ore the whole of the alumina remains insoluble as calcium aluminate, which is converted into the sodium salt by digestion with sodium carbonate under pressure. The aluminium hydroxide obtained by the above processes is claimed to be practically free from silica and a high recovery is effected.

A. R. POWELL.

Purification of alumina. ALUMINUM CO. OF AMERICA, Asses. of B. T. HORSFIELD (B.P. 262,405, 3.11.26. U.S., 4.12.25. Addn. to B.P. 248,360; B., 1927, 682).—In the electrothermic treatment of clays and bauxites to obtain a ferrosilicon-titanium alloy and a fluid alumina slag, the charge is so adjusted that the latter contains at least 95% Al_2O_3 . During pouring it is blown with an oxidising blast of air and/or steam and the hollow globules so obtained are treated with an acid solution which will remove all the lime and most of the ferric oxide, titania, and silica. As leach liquor, sulphuric, hydrochloric, or hydrofluoric acid may be used, or mixtures which generate these acids, e.g., sodium chloride and sulphur dioxide in the presence of air. The purified alumina so obtained contains 99.5–99.8% Al_2O_3 .

A. R. POWELL.

Disintegration of liquid alumina. A. L. MOND. From METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 284,131, 7.6.27).—Fused alumina is transformed into small, easily separable crystals suitable for fusion electrolysis by disintegration of the molten mass with rapidly rotating discs or paddles, or by blowing with air or other gases, followed by rapid cooling by water as rain or spray, or by projecting the disintegrated material into a chamber irrigated with water, the particles being collected in a container through which water flows. Means are provided for preventing both premature solidification of the molten alumina and the steam evolved from coming into contact with the hot or non-disintegrated material.

W. G. CAREY.

Production of chromyl chloride. PERMUTT A.-G. (B.P. 270,711, 3.5.27. Ger., 5.5.26).—An acid chloride, e.g., chlorosulphonic acid, reacts with chromic acid or a chromate in the presence of concentrated sulphuric acid or other diluting agent which does not decompose chromyl chloride; the acid anhydride formed, e.g., sulphur trioxide when chlorosulphonic acid is used, is converted into an acid chloride by the intro-

duction of hydrochloric acid, and then acts on fresh quantities of chromic acid.

W. G. CAREY.

Manufacture of titanium compounds. TITAN CO. A./S. (B.P. 271,085; 11.5.27. Norw., 12.5.26).—The hot solution of titanium and ferrous sulphates obtained by treating ilmenite with sulphuric acid is added slowly with continuous stirring to a quantity of hot mother-liquor from which most of the titanium has been removed by hydrolysis under such conditions that the composition of the solution remains constant while the titanium salt is gradually hydrolysed. As hydrolysis proceeds the liquor containing the suspended precipitate is withdrawn from the precipitation vessel, the precipitate is removed, and the hot mother-liquor returned to the precipitation vessel, or treated separately at increased temperature and pressure in order to precipitate its remaining titanium content.

A. R. POWELL.

Manufacture of a radium preparation. O. HAHN, Assr. to U.S. RADIUM CORP. (U.S.P. 1,655,184, 3.1.28. Appl., 29.7.25. Ger., 15.8.24).—A solution of a lanthanum salt containing a small quantity of a radium salt is treated with hydrofluoric acid to precipitate lanthanum fluoride having radium fluoride dispersed through it. Alternatively, a solution containing radium bromide and thorium nitrate is evaporated to dryness and the residue heated at 500° to give a mixture of radium bromide and thorium oxide. The dispersed radium in such mixtures liberates radon freely.

T. S. WHEELER.

Manufacture of ammonium phosphate. R. GRIESSBACH and K. RÖHRE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,659,198, 14.2.28. Appl., 17.3.26. Ger., 23.3.25).—See B. P. 255,766; B., 1926, 821.

Roasting of gas-purifying substances (B.P. 271,854). **Sulphuric acid from acid tar** (B.P. 277,619 and 281,547). **Ammonia saturator** (U.S.P. 1,654,159).—See II. Alkali arsenates (U.S.P. 1,654,527–8).—See X.

VIII.—GLASS; CERAMICS.

Effectiveness of eye protection glasses and its characterisation. L. BLOCH (Gas- u. Wasserfach, 1927, 70, 1229–1232).—The penetrability of eye-protection glasses is most easily determined for the visible rays, by measuring with a photometer the intensity of a source of light before and after interposing the given glass. The value usually recorded is the "dimming index," i.e., the negative logarithm of the penetrability. Instead of taking the visible spectrum as a whole, separate observations may be made with red, green, and blue rays by placing a red, green, or blue glass in front of the eyepiece of the photometer. The dimming indices for the three colours afford a means of characterising the colour of the given glass. Similar measurements for infra-red rays are easily made by determining the diminution caused by interposing the given glass between an electrically-heated glowing wire and a thermopile. For measurements with ultra-violet rays the light from a quartz lamp is passed through a screen (e.g., "black glass" of the Sendlinger Optischer Glaswerke) which allows only the ultra-violet rays to pass. When these rays fall on to white drawing paper the latter shines out

brightly, and if a test glass is interposed its dimming effect may be measured. The dimming indices of different coloured glasses are less widely different for infra-red rays than for visible rays, whilst the indices for ultra-violet rays bear no close relation to those for visible and infra-red rays.

W. T. K. BRAUNHOLTZ.

PATENTS.

Manufacture of acid metal phosphate [glass].

H. BLUMENBERG, JUN., Assr. to STOCKHOLDERS' SYND. (U.S.P. 1,654,404, 27.12.27. Appl., 23.12.24).—A metallic compound, *e.g.*, lead oxide or potassium carbonate, is heated at 400–700° with phosphoric acid (4–14 mols.) to yield a glass of value in the manufacture of enamels with low m.p.

T. S. WHEELER.

Manufacture of ceramic material. G. KNUDSEN (B.P. 260,298, 23.10.26. Norw., 24.10.25).—Shaped ceramic or refractory products are made from a mixture of talc and raw or burnt magnesite mixed in such proportions as to obtain magnesium orthosilicate after firing. The materials are finely ground, mixed with a binding agent such as tar or molasses to obtain a suitable degree of plasticity and with a filler such as olivine or artificial magnesium orthosilicate from a previous charge, formed into shape, and fired at 1000–1400° for 48 hrs. or until the greater part of the charge is converted into Mg_2SiO_4 . A. R. POWELL.

Manufacture of heat-insulating material. W. W. ODELL (U.S.P. 1,657,582, 31.1.28. Appl., 23.4.25).—Plastic clay and prepared alkaline peat containing only sufficient water to give plasticity for working and moulding are intimately mixed and macerated, the mixture then being moulded, dried, and burnt.

W. G. CAREY.

Refractory material. V. M. GOLDSCHMIDT and R. KNUDSEN (B.P. 283,791, 22.6.27).—A furnace or other apparatus which is exposed to high temperatures and chemical action is made from natural olivine rock containing less than 10% FeO, and/or from blocks made of olivine fragments rammed and heated with or without a binding agent, *e.g.*, colloidal magnesium silicate, magnesium oxide, tar, pitch, etc.

W. G. CAREY.

Production of vitreous silica. BRIT. THOMSON-HOUSTON Co., LTD., Asses. of L. B. MILLER (B.P. 282,733, 5.12.27. U.S., 29.12.26).—See U.S.P. 1,628,468; B., 1927, 523.

Production of highly refractory magnesite masses. F. BAUMHAUER, Assr. to DYNAMIDON-WERK ENGELHORN & Co., G.M.B.H. (U.S.P. 1,659,476, 14.2.28. Appl., 16.2.24. Ger., 20.2.23).—See B.P. 211,873; B., 1925, 243.

IX.—BUILDING MATERIALS.

Flow of heat through limestone and lime. R. T. HASLAM and V. C. SMITH (Ind. Eng. Chem., 1928, 20, 170–174).—The mathematical treatment of the rate of heat flow through solids is developed for the case of limestone (in which dissociation occurs), but only on the assumption of the limestone being a "semi-infinite block." The rate of heat transference

through slabs of moderate size was determined experimentally by means of embedded thermocouples, the sides of the slabs being insulated so that heat transference was in one direction only. The black-body coefficients were determined as about 0.5 using temperatures below that of the dissociation of limestone. With a furnace temperature of 1060° the ratio between the time required for the dissociation temperature to penetrate a given distance and the calculated time (ignoring the thermal requirements of dissociation) was found to be 0.425. This experimental figure was then used for the calculation of the time required for the complete dissociation of spheres of limestone of given diameter at given temperatures. The (linear) relationship between the radius of such a sphere and the square root of the time required is depicted in a series of curves. C. IRWIN.

Laboratory tests on physical properties of water-bearing materials. N. D. STEARNS (U.S. Geol. Survey, Water-Supply Paper 596F, 1927, 121–176).—Methods of sampling water-bearing strata and of determining their apparent sp. gr., mechanical composition, porosity, moisture equivalent, and permeability to water are described in detail with reference to certain American water-bearing gravels, tables showing the physical properties of which are included.

A. R. POWELL.

Principles of kiln-seasoning of timber. I. Types of commercial kilns in use. S. T. C. STILLWELL (Dept. Sci. Ind. Res., Forest Prods. Res., Spec. Rept. No. 2, 1928, 11 pp.).

PATENTS.

Impregnation of wood with two or more liquids.

A. DESSEMOND (B.P. 283,703, 1.12.26).—The wood is subjected to the action of a vacuum after each impregnation and the liquid extracted is measured so that the degree and duration of the vacuum are controlled, and the quantity of impregnating liquid in the wood is proportioned. Suitable apparatus is described.

W. G. CAREY.

Composition for impregnation of wood. B.

WURZSCHMITT, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,654,229, 27.12.27. Appl., 17.6.26. Holl., 15.6.25).—The process of U.S.P. 1,500,066 (B., 1924, 749) is modified in that formaldehyde is added to the solutions therein described to inhibit their corrosive action on iron.

T. S. WHEELER.

Varnishing of wood. J. PAISSEAU (Addn. No. 31,459, 3.8.25, to F.P. 613,502).—Nitrocellulose lacquers adhere better to wood which has previously been treated with nitric or mixed acid, washed, and dried.

C. HOLLINS.

Protection of wood. P. BARTSCH (Re-issue 16,880, 14.2.28, of U.S.P. 1,374,806, 12.4.21).—See B., 1921, 434 A.

Bituminous composition (U.S.P. 1,659,554).—See II.

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

Heat of the A2 and A3 transformations in carbon steels. S. UMINO (Sci. Rep. Tôhoku Imp. Univ., 1927, 16, 1009–1030).—From determinations of the heat

content and true sp. heat at high temperatures of steels containing 0.04—0.077% C the heat of transformation of iron at the A2 point is deduced as 3.63 g.-cal./g. and at the A3 point, 5.35 g.-cal./g. The A2 transformation in pure iron takes place over a range of 130—140°, and the A3 transformation requires an appreciable time to become complete at 930°. Both heats of transformation are decreased by the presence of carbon, being 3.23 and 3.50 g.-cal./g. respectively for the 0.35% C steel, and 3.18 and 0.8 g.-cal./g. for the 0.77% C steel. The heat of the A1 transformation increases rapidly with the carbon content from 0.67 g.-cal./g. with 0.04% C to 13.6 g.-cal./g. with 0.77% C. A. R. POWELL.

Tempering changes in carbon steels. R. HAY and R. HIGGINS (J. Roy. Tech. Coll., Glasgow, 1927, [4], 62—76).—The tempering changes which take place in a 0.26% carbon steel have been investigated by determining the Brinell hardness, specific volume, yield stress, maximum stress, percentage elongation, and Charpy impact value of specimens which had been water-quenched from 1000° and then tempered from laboratory temperatures to 650°. Pronounced maxima or minima occur in the property-temperature curve at about 100°, 250°, and 350°. Brinell hardness and specific volume determinations carried out on steel quenched at temperatures from 400° to 1150° show that an appreciable amount of iron carbide goes into solution below 650°. When such steels are kept for two months, those specimens quenched below 650° undergo a considerable increase in hardness and specific volume, probably owing to deposition of finely dispersed carbide through the α -iron. Tempering at 50° brings about a fall in hardness, which is ascribed to coagulation of this carbide. The change at 100° is thought to be due to decomposition of iron carbide in α -iron, whereas the change at 250° is attributed to the decomposition of residual austenite in the quenched specimens. The change at 350° is accounted for by the assumption that all the remaining austenite is not transformed immediately to martensite and then to troostite, but that the change is periodic and takes a certain time to go to completion. L. M. CLARK.

Action of water, air, oxygen, and carbon dioxide on the corrosion of iron. K. INAMURA (Sci. Rep. Tôhoku Imp. Univ., 1927, 16, 979—986).—Pure water free from dissolved gases has only a very slight rusting action on iron which is barely influenced by the addition of pure sodium chloride. Passage of pure oxygen through either the water or the salt solution causes a rapid increase in the rate of corrosion. Carbon dioxide alone increases the rate of corrosion, but not nearly to the same extent as oxygen alone; it, however, accelerates the corrosion due to oxygen, but apparently to a smaller degree than is generally accepted. A. R. POWELL.

Investigation of the corrosion of metals [iron] with a thermobalance. K. INAMURA (Sci. Rep. Tôhoku Imp. Univ., 1927, 16, 987—997).—The rate of corrosion of a sample of commercial iron in various salt and acid solutions has been followed by means of the Honda thermobalance (*ibid.*, 1915, 4, 97). In solutions of sodium carbonate and sodium hydrogen carbonate, the specimen gained in weight linearly with the time of immersion owing to the slow formation of

ferrous hydroxide over the surface; in dilute acid solutions the decrease in weight was also a linear function of the time, but, owing to adhesion of hydrogen bubbles, observation in very dilute acids (0.0025*N*) was uncertain. In all the solutions tested, corrosion started at several spots and slowly spread from these over the surface of the metal. A. R. POWELL.

Manganese in steel and pig iron. Volumetric determination by the vanadate method. L. E. STOUT and G. C. WHITAKER (Ind. Eng. Chem., 1928, 20, 210—212).—The method proposed is the inverse of the determination of vanadium in steel by oxidation with permanganate. Standard vanadyl sulphate solution is prepared by dissolving ammonium metavanadate in dilute sulphuric acid and reducing with sulphurous acid in excess. It is standardised on steel of known manganese content and is very stable. A 0.2 g. sample of steel is dissolved in 20 c.c. of 1:3 sulphuric acid, and 5 c.c. of 10% ammonium persulphate solution are added. After cooling, 10 c.c. of silver sulphate solution are added, the mixture is shaken, and 10 c.c. of ammonium persulphate are added. After 30 min., 10 c.c. of sodium chloride solution are added. The two solutions are equivalent so that the silver ion is quantitatively removed. The solution is then titrated with vanadyl sulphate solution. The first oxidation converts iron into ferric sulphate. The second oxidation in presence of silver sulphate converts manganese into permanganic acid. If pig iron is to be tested, the only variation is in removing undissolved graphite before adding silver sulphate. A large number of results of good accuracy are tabulated, and details as to the time required for complete oxidation with varying quantities of silver sulphate are given. C. IRWIN.

Determination of minute amounts of cobalt in steel. W. J. AGNEW (Analyst, 1928, 53, 31—32).—1 g. of steel is dissolved in aqua regia, the solution evaporated to dryness, the residue ignited, dissolved in hydrochloric acid, the solution evaporated to a small bulk, slightly diluted, iron (or chromium) separated by an emulsion of zinc oxide, the volume made up to 200 c.c., and 100 c.c. (0.5 g. of steel) are filtered off. To the filtrate 5 c.c. of concentrated hydrochloric acid are added, hydrogen sulphide is passed through to remove copper, and, after filtration, the filtrate is boiled and 0.2 g. of α -nitroso- β -naphthol dissolved in 10 c.c. of strong acetic acid is added. After boiling and allowing to settle for $\frac{1}{2}$ hr. the precipitate is collected on a filter, washed with 5% hydrochloric acid and hot water, ignited at about 800°, the residue dissolved in about 10 drops of hot concentrated hydrochloric acid, and the diluted solution placed in a Nessler tube. The colour produced by adding 10 c.c. of 1:1 ammonia solution and 5 c.c. of 0.1% potassium ferricyanide solution is matched by adding a cobalt solution (containing 0.001 g. Co per c.c.) to 10 c.c. of ammonia, 10 drops of hydrochloric acid, and 5 c.c. of the ferricyanide solution. If more than 2 mg. or 0.4% Co is present, a gravimetric method is more suitable. The sensitiveness of the test is 1 part in 5×10^5 . D. G. HEWER.

Corrosion of copper and brass. K. INAMURA (Sci. Rep. Tôhoku Imp. Univ., 1927, 16, 999—1008).—

During a period of five weeks the rate of corrosion of copper is a linear function of the time of immersion in *N*-ammonium chloride, 0.01*N*-hydrochloric acid, 0.01*N*-nitric acid, 0.01*N*-sulphuric acid, and distilled water, the rate decreasing in the above order. In 0.01*N*-solutions of ammonia, sodium chloride, ammonium chloride, sodium carbonate, and potassium hydroxide the daily loss in weight decreases slowly at first, then more and more rapidly until finally corrosion ceases. The behaviour of 70 : 30 brass to the first of the above series of reagents is similar to that of copper, but in the case of the second series the loss in weight-time curves are straight lines, corrosion proceeding at a steady rate over the whole period.

A. R. POWELL.

Colorimetric determination of small amounts of iron in zinc. W. J. ANGEW (Analyst, 1928, 53, 30).—Zinc (2 g.) is dissolved in 20 c.c. of hydrochloric acid, a few c.c. of nitric acid are added to oxidise any iron, the solution is treated with 0.01 g. of uranium (as uranyl nitrate solution) to agglomerate the iron, ammonia solution is added in slight excess, the liquid boiled, and, after 20 min., the precipitated ferric and uranium hydroxides are collected on a filter and dissolved in 10 c.c. of 1 : 1 hydrochloric acid. The solution is then placed in a Nessler tube, made almost alkaline with ammonia solution, and 1 c.c. of a 10% aqueous solution of sodium salicylate added, followed by ammonia in slight excess, as seen by the yellow colour due to uranium. Finally, it is acidified with acetic acid until the purple colour, due to iron, is developed, and the uranium yellow is destroyed. After addition of 10 c.c. of 1 : 1 acetic acid the colour is compared with that of a solution made by adding a standard iron solution (made from ferric sulphate and containing 0.0001 g. of iron per c.c.) to a Nessler glass containing the same quantity of uranium solution of the same acidity. The sensitiveness of the test is 1 pt. in 2.5×10^6 , and the purple colour is stable for 48 hrs. in the presence of acetic acid, but is destroyed by sulphuric, hydrochloric, and nitric acids.

D. G. HEWER.

PATENTS.

Production of soft workable grey iron. L. MELLERSH-JACKSON. From MASCHINENFABRIK ESSLINGEN (B.P. 260,619, 29.10.26).—The smelted iron is superheated at 1400° or more, the superheating temperature increasing with the content of carbon and silicon. The product is afterwards cast at the normal casting temperature. The wall-thickness of the castings and the composition of the iron with regard to its carbon and silicon content must be taken into account when selecting the superheating temperature.

M. E. NOTTAGE.

Heat-treatment of metals [hardening of steel]. C. L. IPSEN, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,658,682, 7.2.28. Appl., 10.7.25).—An empty furnace is heated to a definite quenching temperature and a charge temperature indicator is set to a definite reference mark at this temperature. The charge is introduced, heated, and quenched when the indicator reaches approximately the reference mark.

J. S. G. THOMAS.

Preventing the dissolution of iron and steel in sulphuric acid. NEWPORT Co. (B.P. 259,200, 20.9.26.

U.S., 30.9.25).—The dissolution of iron and steel in sulphuric acid of strength less than 90% may be prevented or retarded by the addition of 1% or less of thiourea or its substitution products.

M. E. NOTTAGE.

Alloy steel. W. R. SHIMER and R. H. CHRIST, Assrs. to BETHLEHEM STEEL Co. (U.S.P. 1,659,055, 14.2.28. Appl., 5.5.25).—An alloy containing up to 2% Cr, up to 2% W, and not more than 0.3% C is claimed.

F. G. CROSSE.

Nickel-cobalt-iron alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 282,901, 1.10.26).—An alloy, having a high magnetic permeability in a minute field of force and also a high magnetic induction in a moderately strong field, is composed of 55–65% Ni, 20–30% Co, and 15–25% Fe, with or without 1–5% of a fourth element, *e.g.*, chromium, which increases the electrical resistance; a small quantity of a deoxidiser such as magnesium may also be added.

M. E. NOTTAGE.

Hydrometallurgical process. G. H. BUCHANAN, G. B. WINNER, and E. L. TUCKER, Assrs. to AMER. CYANAMID Co. (U.S.P. 1,654,918, 3.1.28. Appl., 16.11.23).—Ore containing copper, silver, and gold is cyanided, and the solution is treated with zinc to precipitate silver and gold. The residual solution is acidified to separate copper cyanide, which is treated with sodium chloride, sulphuric acid, and iron to form copper and hydrogen cyanide.

T. S. WHEELER.

Copper-extraction process. W. E. GREENAWALT (U.S.P. 1,654,930, 3.1.28. Appl., 13.8.26).—The process of U.S.P. 1,483,056 (B., 1924, 340) is modified in that the solution to be electrolysed is treated with hydrogen sulphide to decompose any sulphur dioxide present.

T. S. WHEELER.

Aluminium alloys. T. GOLDSCHMIDT A.-G. (B.P. 260,275, 20.10.26. Ger., 21.10.25).—The mechanical and physical properties of aluminium alloys are improved by heating the alloy to redness and quenching, followed by a sequence of ageing treatments at atmospheric and higher temperatures. Alloys containing 88–98% Al may possess tensile strengths of 36–55 kg./mm.² and elongations of 18–26%.

C. A. KING.

Aluminium alloys. J. STONE & Co., LTD., and H. J. MAYBREY (B.P. 283,760, 26.2.27).—The structure of aluminium alloys, particularly those containing silicon, is improved by treating the alloy with boron exothermically. A suitable reaction mixture contains a compound of boron, powdered aluminium, and oxygen carrier, *e.g.*, potassium chlorate, barium peroxide, or manganese dioxide, if the final presence of manganese is desired.

C. A. KING.

Manufacture of aluminium-nickel-silver alloys. R. K. HEZLET and R. GENDERS (B.P. 283,994, 16.10.26).—In a "nickel-silver" alloy the combined content of nickel and aluminium is more than 15%, up to 2.5% Al being present. A particular composition consists of 20% Ni, 50% Cu, 0.2–0.3% Al, and the remainder zinc. When casting the alloy the cross-sectional dimensions of the molten stream should be approximately the same as that of the mould.

C. A. KING.

Metal compositions [aluminium alloys]. K. BERNHOEFT, Assr. to GEN. ELECTRIC Co. (U.S.P.

1,658,702 and 1,658,757, 7.2.28. Appl., [A] 19.8.26, [B] 7.1.27. Ger., [A] 17.12.25, [B] 19.3.26.—(A) An alloy of 98.4% Al and 1.6% Cd. (B) Aluminium is alloyed with 0.1–1% Cu and 0.5–3% Cd. F. G. CROSSE.

Alteration of the colour of the magnesium flame. BOEHM-WERKE A.-G. (G.P. 445,181, 1.10.25).—Magnesium foil or ribbon is coated with salts which impart the desired colour to the flame when it is burnt.

L. A. COLES.

Production of metallic antimony, alloys of antimony, and alkali arsenates. H. M. BURKEY, Assr. to AMER. METAL CO., LTD. (U.S.P. 1,654,527—8, 3.1.28. Appl., [A] 11.1.26, [B] 27.10.26).—(A) A mixture of antimony trioxide, arsenic trioxide, and sodium carbonate is fused to yield antimony, sodium arsenate, and carbon dioxide. By treating the fused mixture with air a relatively small quantity of antimony trioxide can be used. (B) The process is performed in presence of molten lead to alloy the antimony formed.

T. S. WHEELER.

Removing, purifying, and recovering oil from tin plate. Separating oil from aqueous emulsions. G. B. ELLIS. From AMER. SHEET & TIN PLATE CO. (B.P. 283,614 and 283,830, 13.9.26).—(A) In an apparatus for cleansing tinned plates and recovering palm oil therefrom, the plates, after passing through the tinning and oiling baths, are passed through a dilute, hot, alkaline, aqueous solution prior to the final cleansing and polishing with bran. By this treatment the oil is also freed from flux (zinc chloride) and thus rendered fit for further use. (B) The emulsion is destroyed by acidification or mechanical treatment (centrifuging), and the oil is separated and recovered in a special apparatus described.

C. O. HARVEY.

Treatment of manganese ores. S. G. S. DICKER. From J. C. WIARDA & Co. (B.P. 284,098, 1.3.27).—Manganese ore (carbonate or dioxide) is ground to 200-mesh and heated at 600° for several hours in a reducing atmosphere or with a reducing agent, whereby the manganese content of the ore is converted into manganous oxide. The hot material is discharged directly into a hot 15% solution of ammonium sulphate or chloride, which dissolves the manganous oxide with evolution of ammonia. The mixture is digested at 80° with agitation until all the ammonium salt is decomposed and the liberated ammonia expelled from the solution. Manganous hydroxide is precipitated from the filtered solution by treatment with an aqueous solution of the ammonia expelled in the first stage, thereby regenerating ammonium sulphate for a subsequent leaching operation. The precipitate is collected, washed, dried, and dehydrated to manganous oxide by heating at 825°; air is excluded throughout the treatment.

A. R. POWELL.

Roasting of vanadiferous ores and compounds. H. T. KOENIG (U.S.P. 1,654,820, 3.1.28. Appl., 13.10.25).—The ore is heated with sodium chloride and sulphuric acid at 600–900° to liberate free vanadic acid.

T. S. WHEELER.

Desulphidising and desulphurising of sulphur minerals. M. F. COOLBAUGH and J. B. READ, Assrs. to COMPLEX ORES RECOVERIES Co. (U.S.P. 1,657,711, 31.1.28. Appl., 4.8.22).—In a multi-stage roasting

operation sulphide minerals are heated in an atmosphere containing an excess of sulphur trioxide to form sulphates, which are then rapidly dissociated by withdrawing the partial pressure of sulphur trioxide. C. A. KING.

Production of rock wool. E. R. POWELL (U.S.P. 1,656,828, 17.1.28. Appl., 5.1.27).—Suitable raw material is melted in the presence of hot gases from an oxidising flame before being run off and converted into the form of wool. M. E. NOTTAGE.

Deflocculation of colloids. W. O. BORCHERT, Assr. to NEW JERSEY ZINC Co. (U.S.P. 1,655,045, 3.1.28. Appl., 1.4.25).—Runaway extract, a waste product of tanneries, is used to deflocculate the colloidal matter present in mineral pulps prior to froth flotation.

T. S. WHEELER.

Manufacture of [metallic] catalysts. I. G. FARBENIND. A.-G. (B.P. 281,218, 5.5.27. Ger., 27.11.26).—Catalysts consisting of metals such as nickel, cobalt, or copper, or mixtures of these, may be made from aqueous solutions of their salts by means of a more electro-positive metal such as iron, zinc, or aluminium in the presence or absence of a non-metallic carrier such as kieselguhr, silica gel, or carbon. The metal may be separated in a neutral, acid, or alkaline solution; in an alkaline solution the oxide of the electro-positive metal is simultaneously produced and itself activates the catalyst. M. E. NOTTAGE.

Rust-proofing process. W. H. ALLEN (U.S.P. 1,654,716, 3.1.28. Appl., 7.5.26).—The process of B.P. 8667 of 1906 (B., 1907, 207) is modified in that the surface of the article under treatment is rendered smooth by rubbing it with cotton fabric during immersion in the rust-proofing solution. T. S. WHEELER.

Disintegration of metals. E. J. HALL, Assr. to METALS DISINTEGRATING Co., INC. (U.S.P. 1,659,291, 14.2.28. Appl., 20.12.17. Renewed 30.10.22).—See B.P. 121,600; B., 1919, 826.

Zirconium alloy. G. R. FONDA, Assr. to GENERAL ELECTRIC Co. (U.S.P. 1,658,712, 7.2.28. Appl., 30.8.24).—See B.P. 239,235; B., 1925, 908.

Washing of ores (B.P. 266,723).—See II. **Cleaning of metal surfaces (U.S.P. 1,658,222).**—See XI.

XI.—ELECTROTECHNICS.

Electrolytic precipitation of latices. SCHOLZ.—See XIV.

PATENTS.

Electric furnace. C. A. CADWELL, Assr. to ELECTRIC RAILWAY IMPROVEMENT Co. (U.S.P. 1,657,785, 31.1.28. Appl., 29.4.20).—Electrodes extend into a chamber, and a resistor providing paths of varying resistance for passage of current from one electrode to the other is arranged in the chamber between the electrodes.

J. S. G. THOMAS.

Electric furnace. J. L. BERNARD (U.S.P. 1,658,071—2, 7.2.28. Appl., [A, B] 15.12.26).—(A) A wheel-supported furnace body runs on tracks on a tilting base from which a frame carrying a vertically adjustable roof extends upwards. Means are provided for adjusting the roof and for locking the furnace body in the frame. (B) In a furnace constructed in accordance with (A) jacks are

attached to the tilting base for raising the furnace body into contact with the roof, and means are provided on the frame for guiding the furnace body.

J. S. G. THOMAS.

Electro-cleaning [of metal surfaces]. R. M. BURNS and C. W. WARNER, Assrs. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,658,222, 7.2.28. Appl., 10.2.25).—Electric current is passed between suitable cathodes and metallic surfaces to be cleaned, which are immersed in a solution of phosphoric acid maintained at 50° and at 70% concentration.

J. S. G. THOMAS.

Construction of vacuum tubes. H. E. METCALF, Assr. to MAGNAVOX Co. (U.S.P. 1,657,636, 31.1.28. Appl., 23.7.24).—One of the electrodes of a vacuum tube is of German silver, and is heated to throw off the copper and zinc and deposit them on the walls of the tube, the nickel of the alloy being left in place to form an electrode.

W. G. CAREY.

XII.—FATS; OILS; WAXES.

Examination of residues from the refining of oils and fats. J. DE TOURNADRE (Chim. et Ind., 1928, 19, 39—40).—The process of oil refining includes the removal of free fatty acids, the bleaching of the neutral oil, and the removal by steam distillation of substances giving an offensive odour or taste. The chief by-product is obtained in the first stage, and is either a free fatty acid (from decomposition of a lime soap) or a soda soap containing a large quantity of neutral oils. These substances are sold to the soapmaker on the basis of their fatty acid content. The free acid is determined by difference from a determination of moisture and of residue insoluble in carbon disulphide. In the case of the soda soap a given quantity is decomposed with strong hydrochloric acid. If a clear separation is obtained wax or stearic acid is added if necessary to bring the acid to the solid state, when it is separated. If separation is not good the whole is dissolved in hot water, cooled, and the fatty acid extracted with carbon disulphide.

C. IRWIN.

Halphen reaction for cottonseed oil as a general reaction for oils of the families *Malvaceæ*, *Tiliaceæ*, and *Bombacaceæ*. S. IVANOV (Ber. Deut. bot. Ges., 1927, 45, 588—591).—The substance causing the Halphen test colour in cottonseed oil is located in the seed, and is extracted with the oil. It is not present in the petals or colouring matter. The reaction is specific for the order *Columnifera*, including *Malvaceæ*, *Tiliaceæ*, and *Bombacaceæ*, but not *Sterculiaceæ*.

A. G. POLLARD.

Constitution of elæostearic acid. F. FRITZ (Farben-Ztg., 1928, 33, 1224—1225).—A brief account of the work of various investigators on the constitution of elæostearic acid as revealed by its oxidation products. The validity of Böeseken and Ravenswaay's formula for the acid (cf. B., 1925, 813) is considered to be upheld.

S. S. WOOLF.

Determination of oleic and linoleic acids in an oil; determination of the bromine index. Y. VOLMAR and B. SAMPAHL (J. Pharm. Chim., 1928, [viii], 7, 106—109).—For the determination of oleic and linoleic acids in oils containing no other unsaturated

acids, calculation of the percentage amounts from the iodine value of the mixture gives very discordant results compared with those given by the gravimetric tetrabromide method of Eibner and Muggenthaler for the determination of linoleic acid. The following volumetric method gives results comparable with those of the above gravimetric method. The unsaturated acids (0.5 g.) in 15 c.c. of dry ether are cooled to -10° , and bromine is added drop by drop until the brown colour persists. After keeping at -10° for 2 hrs. the excess of bromine is removed with 5% aqueous sodium thiosulphate solution, the ether evaporated, and the residue dissolved in 100 c.c. of 95% alcohol, 20 c.c. of the solution are boiled for 5 hrs. with 5 c.c. of 60% aqueous potassium hydroxide solution, and, after cooling, the bromine is determined by Volhard's method. E. H. SHARPLES.

Oil bromide films and their use in determining the halogen absorption of oils. H. TOMS (Analyst, 1928, 53, 69—77).—The "insoluble bromide" obtained from most drying oils has been debrominated with difficulty and the presence of hexabromostearic acid confirmed, whilst the presence of tetrabromostearic acid has been rendered more probable by the isolation of a thick oil, apparently a liquid form of this acid. A satisfactory micro-method for determining the unsaturation value of non-volatile oils consists in spreading a thin film of the oil in a single drop, about 0.2 mm. thick, on a weighed microscope slide, and placing the slide in a wide tube closed at each end with a waxed cork and containing a boat with a few drops of bromine. After 20—30 min. the slide is withdrawn, excess of bromine removed, and the slide again weighed; the bromine value is calculated from the increase in weight. This value multiplied by the at. wt. of iodine and divided by the at. wt. of bromine gives the ordinary iodine value, and agrees well with theory except in the peculiar case of tung oil, the appearance of the brominated films of which is characteristic. A method is given for calculating the percentage of α -elæostearic triglyceride in tung oil assuming the presence in the oil of two types of substances, viz., (a) those containing non-conjugated systems of double linkings and giving the same iodine value regardless of the method used, and (b) the glyceride of α -elæostearic acid which gives only two thirds its theoretical value with Wijs' solution in 2 hrs., but its full value by the bromide vapour method.

D. G. HEWER.

Drying of oils. EIBNER.—See XIII. **Determination of mustard oil.** SACCARDI and PRERI.—See XIX.

PATENTS.

Detergent, cleansing, and polishing compositions. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, and E. CHAPMAN (B.P. 284,367, 21.9.26).—A cleansing composition for tiles, walls, etc. comprises a sulphonated (or sulphonated and alkylated) high-boiling mineral oil fraction in powder or paste form or in solution together with abrasive material (whiting, pumice), with or without addition of thickening and tinting agents, an organic solvent, soap, or a sulphonated oil. The first-mentioned ingredient increases the cleansing efficiency by ensuring rapid and complete contact with the surface to be cleansed.

C. HOLLINS.

Manufacture of cleansing and emulsifying agents.

I. G. FARBEININD. A.-G. (B.P. 260,243 and Addn. B.P. 283,786; 13.10.26. Ger., [B] 21.10.25).—(A) The oily products practically insoluble in water and consisting chiefly of higher aliphatic alcohols, obtained by catalytic hydrogenation of oxides of carbon or by cracking operations employing sulphuric acid, are mixed with a soap and an aliphatic alcohol of low b.p., and, if desired, an aliphatic, aromatic, or hydroaromatic hydrocarbon. (B) Soft soap, grain soap, or other soap-like substance is used as the "soap" constituent of the mixture.

S. S. WOOLF.

Digesters for use in the whale oil industry.

T. ARENTZ (B.P. 271,111, 14.5.27. Norw., 15.5.26).

Waterproofing composition (U.S.P. 1,658,540).

See II. Recovery of oil from tin plate (B.P. 283,614 and 283,830).—See X. Resins from cashew nut-shell oil (B.P. 283,803).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Negative catalysis as a means of improving the drying of oils. A. EIBNER (Farben.-Ztg., 1928, 33, 1222—1224).—A general discussion of the functions of negative catalysts, with special reference to the effects of undercoats on coats subsequently applied and the neutralisation of such effects by "flattening" the undercoats.

S. S. WOOLF.

Protective paint from rubber. H. GRAY (Ind. Eng. Chem., 1928, 20, 156—158; cf. Fisher, B., 1928, 61).—Films of thermoprene on metal exhibit good adhesion and low permeability and exert a marked protective action against corrosion; they are tough and free from brittleness at 0°.

D. F. TWISS.

Arsenic in coated papers etc. STERN.—See V. **Pigments and rubber.** WINKELMANN and CROAKMAN.—See XIV.

PATENTS.

Protective compositions particularly for anti-corrosive purposes. A. A. VAN DER MEULEN (B.P. 283,664, 28.10.26).—A mixture of 1—2 pts. of phenol and 40 pts. of linseed oil is claimed.

S. S. WOOLF.

Fluorescent paint. L. J. BUTTOLPH, Assr. to COOPER HEWITT ELECTRIC Co. (U.S.P. 1,658,476, 7.2.28. Appl., 17.12.24).—A mixture of benzene, anthracene, rubber, and a vulcaniser is used.

F. G. CROSSE.

Production of [pigment] mixtures containing metal powder. M. RAGG and F. RAHTJEN (B.P. 284,172, 1.10.27).—Mixtures containing metal powder are prepared by atomising the molten metal or alloy and projecting it against the remaining ingredients of the mixture, e.g., pigments, fillers, in suitable apparatus.

S. S. WOOLF.

Plastic and lacquer compositions of nitrocellulose. CANADIAN ELECTRO PRODUCTS Co., LTD., Assees. of H. W. MATHESON (B.P. 270,650, 9.3.27. U.S., 4.5.26).—Esters of mandelic acid are used as plasticisers for plastic and lacquer compositions of nitrocellulose.

S. S. WOOLF.

Compositions of matter containing esters or ethers of carbohydrates. J. SCHINDELMEISER (B.P. 283,619, 6.10.26).—The use of esters of borneol, iso-

borneol, and terpineol in paint or varnish compositions containing esters or ethers of cellulose or other similar carbohydrates is claimed.

S. S. WOOLF.

Manufacture of solutions of organic compounds.

I. G. FARBEININD. A.-G. (B.P. 256,229, 26.7.26. Ger., 1.8.25).—Dialkyl ethers of ethylene, propylene, or butylene glycol are used as solvents for natural or artificial resins, e.g., in cellulose lacquers.

S. S. WOOLF.

Reaction products of cashew nut-shell oil.

M. T. HARVEY (B.P. 283,803, 11.10.26).—Cashew nut-shell oil is condensed with aldehydes, in the presence or absence of catalysts, to produce synthetic resins that may subsequently be hardened.

S. S. WOOLF.

Manufacture of condensation products of carbamide and formaldehyde. I. G. FARBEININD. A.-G. (B.P. 260,253, 16.10.26. Ger., 21.10.25).—Lacquers are prepared directly by mixing solutions of carbamide and paraformaldehyde in an organic solvent, water being excluded as far as possible, and incorporating natural or artificial resins, cellulose esters, and colouring matters before or during the carbamide-aldehyde condensation.

S. S. WOOLF.

Manufacture of a condensation product.

P. W. GRIFFITH, Assr. to AMER. CYANAMID Co. (U.S.P. 1,658,597, 7.2.28. Appl., 16.8.24).—A product is formed by the interaction of guanidine, carbamide, and formaldehyde.

R. FULLMAN.

Varnishing of wood (Addn. F.P. 31,459).—See IX.**XIV.—INDIA-RUBBER; GUTTA-PERCHA.**

Absorption of hydrogen under high pressure by rubber, and behaviour of rubber after release of pressure. G. TAMMANN and K. BOCHOW (Z. anorg. Chem., 1928, 168, 322—324).—If rubber and zinc are placed in 2*N*-sulphuric acid, and the whole is subjected to a pressure of 550 or 1150 kg./cm.², the rubber takes up a considerable amount of hydrogen. If now the pressure is released, the rubber swells considerably, numerous small bubbles appearing in it, but after 15—20 min. about 0.8% of the hydrogen has escaped. After this point, the amount of hydrogen evolved is proportional to the pressure at which the rubber was saturated with the gas, so that the absorption coefficient must be practically independent of the pressure, but transparent rubber and black unvulcanised rubber give up their hydrogen more rapidly than do the red and grey varieties.

R. CUTHILL.

Effect of certain metallic salts on the ageing of a [rubber] "tread compound." B. S. TAYLOR and W. N. JONES (Ind. Eng. Chem., 1928, 20, 132—133).—Ageing experiments with an otherwise typical black "tread compound" by the Geer oven and Bierer-Davis bomb methods show that the presence of as little as 0.1% of copper oleate or manganese oleate is distinctly detrimental. Ferric stearate was less deleterious, but 0.5% had a distinct adverse effect. Mercuric stearate between 0.1 and 1% was relatively innocuous. The bomb test at 70% was much more severe than the oven test at the same temperature. Application of cupric chloride to the surface of the test strips has a similar effect to the incorporation of cupric stearate.

D. F. TWISS.

Effect of the proportion of sulphur in vulcanising [rubber] reclaim. R. E. CARLIDGE and H. L. SNYDER (Ind. Eng. Chem., 1928, 20, 137—140).—Comparison is made of the tensile characteristics and of the proportion of additional combined sulphur in vulcanised products obtained from an alkali "tyre reclaim," when vulcanised with various percentages of sulphur (from 1½ to 6%) or for different periods. The best physical properties with the simple mixture of reclaim and sulphur are obtained with 3% S; in a mixture of equal proportions of the reclaim and new rubber together with carbon black and other typical ingredients, and allowing 4% of sulphur for the vulcanisation of the new rubber, the best proportion for sulphur to reclaim is 2—2½%.

D. F. TWISS.

Hardness tester for rubber. E. C. ZIMMERMAN and R. W. BROWN (Ind. Eng. Chem., 1928, 20, 216—218).—A "penetrometer" is described in which the alteration in stress between minimum and maximum penetration is made negligibly small by the use of a long spring. In order to render the penetrometer applicable over a wide range of hardness, three springs corresponding with different loads can be used.

D. F. TWISS.

Pigment reinforcement of reclaimed rubber. H. A. WINKELMANN and E. G. CROAKMAN (Ind. Eng. Chem., 1928, 20, 134—137).—"Whole tyre" reclaim (containing 57% of rubber) was mixed with 5% of sulphur, and with various compounding ingredients in proportions between 2½ and 20 vols. relative to the reclaim (100 vols.). Comparison was made of the influence of "mineral rubber," whiting, barytes, *blanc fixe*, clay (including catalpo), zinc oxides (including kadox), thermatomic carbon, and carbon black on the stress-strain curve and the resistance to tearing and abrasion. Only carbon black showed any marked reinforcing action, the effect increasing with the proportion added, the resistance to tear and to abrasion being also much higher than that for any other ingredient. Measurement was also made of the plasticity of the various mixtures and of the influence of organic accelerators. The effect of an accelerator is dependent to some extent on the nature of the filler present; generally, however, with a mild ultra-accelerator improved physical characteristics are obtained in the vulcanised product.

D. F. TWISS.

Value of the rubber hydrocarbon in reclaimed rubber. W. W. VOGT (Ind. Eng. Chem., 1928, 20, 140—143).—Mixtures suitable for tyre treads, containing reclaimed rubber and of the same ultimate composition except for the proportion of new to old caoutchouc in a constant inclusive total of rubber hydrocarbon, were vulcanised to their respective technical optima in equal times. Tensile and abrasion tests indicated that the value of the caoutchouc contributed by the reclaimed material ranges from nil for small proportions up to 50% of that of new rubber for larger proportions. For the substitution of new rubber by reclaimed rubber without loss in quality it may therefore be necessary to make other adjustments in the composition of the mixture.

D. F. TWISS.

Rate of vulcanisation of reclaimed rubber. N. A. SHEPARD, H. F. PALMER, and G. W. MILLER (Ind. Eng. Chem., 1928, 20, 143—152).—When mixed with a

standard proportion of 8.3% of sulphur on the actual rubber content reclaimed rubber produced from motor covers, solid tyres, or motor tubes vulcanises much more rapidly (as judged by physical tests) than smoked sheet rubber; this characteristic causes mixtures containing new rubber together with such reclaimed rubber to vulcanise remarkably rapidly. Although residual alkali from the reclaiming process may contribute to this feature, it is not the prime cause, the identity of which is, however, still uncertain. The proportion of sulphur already in combination with the rubber hydrocarbon in the reclaimed material appears to have little influence on the rate of subsequent vulcanisation, but has a marked effect on the ageing qualities of the vulcanised product. When using rubber mixtures containing reclaimed rubber, a low concentration of sulphur and a minimum time or temperature of vulcanisation are desirable.

D. F. TWISS.

Factors in processing reclaimed rubber. P. S. SHOAFF (Ind. Eng. Chem., 1928, 20, 152—155).—For the production of satisfactory reclaimed rubber, it is desirable to ensure uniform grinding of the scrap rubber, devulcanisation at a moderate temperature, efficient removal of any residual alkali, drying at as low a temperature as possible, and a minimum of milling and refining. Types of equipment are discussed and possible developments indicated.

D. F. TWISS.

Paint from rubber. GRAY.—See XIII.

PATENTS.

Vulcanisation of rubber. W. A. GIBBONS, Assr. to NAUGATUCK CHEMICAL CO. (U.S.P. 1,654,167, 27.12.27. Appl., 17.12.23).—Latex mixed with sulphur, zinc oxide, and an organic accelerator active at low temperatures is evaporated to dryness and heated at 100° for 40 min.

T. S. WHEELER.

Manufacture of an accelerator for the vulcanisation of rubber. W. J. KELLY (B.P. 283,679, 9.11.26).—Mercaptobenzthiazole may be obtained in a yield 95—97% of the theoretical by heating a mixture of aniline, sulphur, and carbon disulphide in an autoclave at 280—285° so that the pressure increases to between 600 and 700 lb./in.²

D. F. TWISS.

Production of an accelerator for the vulcanisation of rubber. L. B. SEBRELL (B.P. 283,661, 26.10.26).—Mercaptobenzthiazole, without undesirable by-products, is produced by heating a mixture of ammonia (gaseous or aqueous), aniline, carbon disulphide, and sulphur at a temperature between 100° and 250° and a pressure between 100 lb. and 1000 lb./in.²

D. F. TWISS.

Manufacture of rubber articles. DUNLOP RUBBER CO., and D. F. TWISS (B.P. 283,984, 17.9.26).—Aqueous dispersions of rubber, e.g., latex, with or without additional substances such as vulcanising agents, pigments, etc. are sprayed in a wet unheated condition on to an unheated former or rubber article, the latter of which may be a damp, unvulcanised deposit produced from latex. The articles so obtained are then dried and may be vulcanised.

D. F. TWISS.

Regeneration of rubber contained in the beads of used tyres, etc. SYND. FRANCO-NEERLANDAIS (B.P. 276,626, 16.6.27. Fr., 25.8.26. Cf. B.P. 269,127; B., 1927, 635).—The beads of discarded tyres are cut

into pieces and soaked in the oily distillate obtained by the dry distillation of waste rubber, *e.g.*, old rubber tubing. The treated beads are then placed in an acid, such as hydrochloric acid, which causes polymerisation of the absorbed oil and also renders the canvas wrapping easily removable from the hard core. The rubber present in the canvas undergoes regeneration by this treatment; the cores can be distilled for the treatment of further beads.

D. F. TWISS.

Vulcanisation of rubber. GOODYEAR TIRE & RUBBER Co., Assec. of J. TEPPEMA (B.P. 265,919, 16.11.26. U.S., 12.2.26).—See U.S.P. 1,637,790—1; B., 1927, 789.

Manufacture of material for use in repairing punctures in the air-tubes of pneumatic tyres and other pneumatically inflatable articles. R. SURRIDGE (B.P. 284,095, 17.2.27).

XV.—LEATHER; GLUE.

PATENTS.

Cleaning of leather goods and furs. ACHILLE SERRE, LTD., and J. B. ARGENT (B.P. 283,709, 11.12.26).—Articles of leather or fur are placed in a drum with a suitable quantity of a dry, powdered, abrasive medium, *e.g.*, hardwood dust, with or without fine sand. A small proportion of a non-inflammable solvent may be added. Part of the periphery of the drum is perforated and provided with a movable cover, or covers, by means of which the perforations are closed for cleaning the goods and opened to allow the sawdust and sand to be separated from the articles.

D. WOODROFFE.

Dissolution and reprecipitation of keratins. M. BERGMANN, Assec. of H. KOESTER (G.P. 445,503, 1.2.25).—Keratin (horns, hoofs, feathers, hair, or wool), with or without a previous treatment with alkali, is stirred with an ammoniacal solution of copper or nickel oxide until dissolved. It may then be precipitated by acids or acid salts in a form suitable for production of threads or artificial masses, formaldehyde or tanning materials being added if desired. The solution may be mixed with a cuprammonium cellulose solution and the mixture spun into threads which differ considerably from other artificial silks in mechanical and dyeing properties.

C. HOLLINS.

Humic acid derivatives (G.P. 443,339).—See II. **Condensation products of arylamines** (B.P. 266,358).—See III. **Deflocculation of colloids** (U.S.P. 1,655,045).—See X.

XVI.—AGRICULTURE.

Microbiological analysis of soils. W. BENECKE and H. SÖDING (Z. Pflanz. Dung., 1927, 10A, 129—159).—The methods of Mitscherlich and of Christensen for determining the nutrient content of soils are extended. The method is based on the increased growth of algae and fungi on series of artificial media to which known amounts of soil are added. The nutrient values of various soils as indicated by this method vary somewhat with the organism used and with the duration of the experiment. Results differ from those obtained in the Mitscherlich process for soils of high organic content.

In mineral soils the two methods agree in differentiating lacking nutrients in soils, but do not indicate the same quantitative fertiliser requirement. Differences in this respect are attributed in part to results of sterilisation during the experiment and the somewhat abrupt changes in p_H value occurring during the vegetation period in some instances. In general, the relative nitrogen and phosphate contents of soils as indicated by the growth of *Aspergillus niger* under the conditions described, agree more closely with the results of chemical analyses than with values obtained by the Mitscherlich method.

A. G. POLLARD.

Soil microbiology. III. Fixative power of soil. S. WINOGRADSKY and J. ZIEMIECKA (Ann. Inst. Pasteur, 1928, 42, 36—62).—A method is described for determining the nitrogen-fixing power of organisms present in the soil which consists in determining (1) the growth obtained from 1 g. of the soil on a plate of a selective medium of silica gel and (2) the total growth obtained spontaneously in a sample of the soil.

W. O. KERMAK.

Optimum soil reaction of the sugar beet. O. ARRHENIUS (Ind. Eng. Chem., 1928, 20, 219).—The relative yield of sugar beet, as determined by pot experiments and also by field investigations over a wide area, reaches a maximum for soils which are neutral or slightly alkaline (p_H 7.0—7.5).

F. R. ENNOS.

Effect of fertiliser constituents on the eye-spot disease of sugar-cane. H. A. LEE and J. P. MARTIN (Ind. Eng. Chem., 1928, 20, 220—224).—During the winter months when conditions are favourable for infection of the sugar cane with eye-spot disease, applications of nitrogenous fertilisers tend to increase considerably the degree of infection, and should be avoided at this time in fields subject to the disease. The use of phosphoric acid or of materials containing potash does not result in increased eye-spot, and in the case of the latter appears to diminish it slightly.

F. R. ENNOS.

Relation between water and potash in plant production. F. W. MORSE (J. Agric. Res., 1927, 35, 939—946).—Pot experiments with millet and soya bean were made using soils starved of potash. Water supply was maintained at three rates, low, medium, and high. An additional series of pots received potassium sulphate. In this second series millet was nearly indifferent to the varying water supply, whilst in the first series water was a limiting factor. Soya beans were about equally affected by both potash and water.

H. J. C. HINES.

Growth of *Bacillus radicum* on artificial media containing various plant extracts. F. E. ALLISON (J. Agric. Res., 1927, 35, 915—923).—The addition of water extracts of various plants caused marked increase in the growth of a red clover strain of the nodule organism on a sugar-salt medium. Both leguminous and non-leguminous plant extracts were used with success, wide variation occurring between different parts of the same plant. Extracts of legume roots were the most stimulating.

H. J. G. HINES.

PATENTS.

Manufacture of a dry and non-hygroscopic fertiliser from the vinasses of distilleries and sugar factories.—SELBI (Soc. d'Exploit. de Licences de

BREVETS IND.) (B.P. 279,022, 22.4.27. Fr., 16.10.26).—The preheated vinasses mixed with a distributing carrier, *e.g.*, sawdust, is stirred up with an equal weight of preheated superphosphate in a mixer at 60–70° until homogeneous. The product is then dried at 125–130°, crushed, and screened. F. R. ENNOS.

Bactericides and fungicides for treatment of seeds. CHEM. FABR. L. MEYER (G.P. 443,507, 13.3.23).—“Chloromethyl alcohol” or “fluoromethyl alcohol” the mixture of products obtained by interaction of 4 pts. of 40% aqueous formaldehyde and 6 pts. of 40% hydrochloric or hydrofluoric acid, is used for treating seeds. These are pickled for 15 min. in a 0.25–0.33% solution of the halogen compound alone or mixed with 5% of mercuric chloride, and are then covered for 8 hrs. C. HOLLINS.

Manufacture of agents for protection of plants and destruction of parasites. CHEM. FABR. DR. HEPPES & Co., G.M.B.H., Assees. of J. B. CARPZOW (G.P. 442,738, 14.9.24).—Salt- or fresh-water slimes, consisting of colloidal silicic acid and vegetable cells of microorganisms, are dried at low temperature, mixed with insecticides etc., dried, and powdered. The colloid adsorbs copper salts, arsenic compounds, formaldehyde generators (*e.g.*, hexamethylenetetramine), chlorine generators (*e.g.*, bleaching powder, trichloroethylene), or oxygen generators (*e.g.*, perborates), and in the last three cases liberates formaldehyde, chlorine, and oxygen, respectively, by catalytic action. Unlike humus extracts, the slime material does not clog the leaf-pores. C. HOLLINS.

Parasiticide and disinfectant. E. MERCK (Swiss P. 118,144, 3.6.25. Ger., 21.7.24. Addn. to Swiss P. 108,018. Cf. G.P.401,413; B., 1925, 187).—Copper salts of cyclic organic compounds, *e.g.*, copper naphthenate, salicylate, or resinate, are used instead of copper salts of fatty acids. L. A. COLES.

Means for combating animal pests. I. G. FARBENIND. A.-G. (G.P. 443,391, 10.1.22).—The mixtures of pyridine bases obtained from acetaldehyde and ammonia or amines are efficient substitutes for nicotine. The mixture consisting essentially of 2-methyl-5-ethylpyridine is very effective against worms, caterpillars, etc. The pyridine base or its tannate or resinate is dissolved or suspended in water, to which magnesia, alumina, calcium carbonate, copper hydroxide, and soaps, resin salts, ligninsulphonic acid, etc., may be added. The *N*-methyl and *N*-ethyl derivatives are for some purposes still more effective. C. HOLLINS.

Means for destroying animal pests. K. RÜLKE (G.P. 443,612, 25.6.22).—Introduction of an amino- or substituted amino-group into the pyridine or quinoline molecule increases the insecticidal powers of these bases, 2-Amino- or 2-ethylamino-pyridine and 2-aminoquinoline are recommended against plant-lice. Amino-derivatives of heterocyclic bases generally are claimed, alone or with soaps, saponin, alcohols, ketones, etc. and in solution. C. HOLLINS.

Products containing arsenic for combating plant pests. I. G. FARBENIND. A.-G. (Swiss P. 120,424–6, 8.4.25. Ger., 10.4.24. Addn. to Swiss P. 116,298).—The product contains aluminium compounds in place of

the iron compounds in the product described previously, and is prepared, *e.g.*, by stirring arsenic trioxide, aluminium sulphate, and calcium carbonate in the presence of a little water, and drying the product. L. A. COLES.

Insecticides. I. G. FARBENIND. A.-G., Assees. of K. BRODERSEN and W. EXT (G.P. 442,432, 11.9.23).—Additive compounds of phenols with quinones or amines have strong insecticidal action. *E.g.*, the compound from *p*-chlorophenol and benzoquinone at 10% dilution kills in 15 min. even the resistant beet-leaf bug, *Zosmenus capitatus*, Wolff, and small ants, etc. More certain, but less rapid, are the compounds from quinol and *p*-toluidine, 3:5-dinitro-*o*-cresol and pyridine, chlorodinitrophenol and pyridine, or trichlorophenol and aniline. C. HOLLINS.

XVII.—SUGARS; STARCHES; GUMS.

PATENTS.

Halogen-calcium-starch preparations. HENKEL & CIE., G.M.B.H. (B.P. 276,340, 16.8.27. Ger., 17.8.26).—The halogen-calcium-starch preparations described in B.P. 244,708 (B., 1926, 561) are treated with 50–80% alcohol to remove the excess of calcium halide. After removal of the solvent by filtration the product is dried at 45°, in which form it is capable of yielding a size with cold water which retains its covering and spreading power after being left unused for several days. F. R. ENNOS.

Fertiliser from vinasses (B.P. 279,022).—See XVI.

XVIII.—FERMENTATION INDUSTRIES.

Abnormal wines. FONZES-DIACON (Ann. Falsif., 1928, 21, 17–19).—The low temperatures and excessive rainfall in parts of France during 1927 retarded the maturing of the grapes and resulted in wines of lower alcoholic strength than normal. With the usual criteria some of these wines might be considered to be watered, but the “tartaric acid index” (B., 1928, 31) shows them to be merely abnormal. J. R. NICHOLLS.

Wines from grapes attacked by *Eudemis*. HUGUES (Ann. Falsif., 1928, 21, 19–20).—The conclusions drawn by Fabre and Bremond on this subject (B., 1928, 104) are criticised. J. R. NICHOLLS.

Methyl alcohol in spirits and tinctures. R. VIVARO (Ann. Falsif., 1928, 21, 22–24).—The methyl alcohol is oxidised to formaldehyde, which is condensed with hydroxylamine. The formaldoxime is decomposed and the resulting hydrocyanic acid is detected. The largest and most constant proportion of formaldehyde is obtained by boiling with potassium dichromate and sulphuric acid, when under the conditions specified, 10–15% of the theoretical quantity is obtained. 50 c.c. of the alcoholic solution is distilled, 20 c.c. of distillate being collected and mixed with 20 g. of powdered potassium dichromate and 200 c.c. of 10% sulphuric acid. When the dichromate has dissolved the mixture is distilled, the first 40 c.c. of distillate (containing only acetaldehyde) being discarded. The next 120 c.c. are mixed with 1 g. of hydroxylamine hydrochloride, neutralised to phenolphthalein, and 5 g. of sodium carbonate are added. After boiling for $\frac{1}{2}$ hr. with a reflux condenser the

solution is acidified with sulphuric acid and distilled into sodium hydroxide solution. The hydrocyanic acid is characterised by the ferrocyanide reaction. Furfuraldehyde, if present, must be removed by means of *m*-phenylenediamine hydrochloride or aniline phosphate.

J. R. NICHOLLS.

Relation of temperature to rate and type of fermentation, and to quality of commercial sauerkraut. H. B. PARMELE, E. B. FRED, W. H. PETERSON, J. E. MCCONKIE, and W. E. VAUGHN (*J. Agric. Res.*, 1927, **35**, 1021—1038).—Definite heat production during the fermentation of sauerkraut is indicated. The temperature of the fermenting mass governs the rate of acid production. When the temperature of the shredded cabbage is 65—75° acid production is rapid in the early stages, and becomes slow later. Below 65° the fermentation and acid production have a more uniform rate. Best-quality sauerkraut is produced under the latter conditions. The degree of fermentation is most suitably measured by means of titratable acid determinations. Measurements of p_H are less satisfactory for the purpose, being largely influenced in the initial stages of fermentation by the carbon dioxide produced. The process of fermentation is accomplished by a sequence of micro-organisms, including at least two distinct types of lactic acid-producing bacteria. Bacterial numbers may be roughly determined during the fermentation by observations of the rate of reduction of methylene-blue.

A. G. POLLARD.

PATENTS.

Production of absolute alcohol. E. C. R. MARKS. From U.S. INDUSTRIAL ALCOHOL Co. (B.P. 283,701, 29.11.26).—A continuous process for obtaining absolute alcohol from dilute alcohol (beer) is described. The beer is first distilled to give 95—96% alcohol vapours in the upper part of the rectifying unit, fusel oil being run off into a separate rectifying column from which water and fusel oil are separately drawn off, whilst alcohol vapours pass on to a dehydrating column. Here the 95—96% alcohol from the beer still is dehydrated by means of benzene (etc.), the benzene layer of the condensate being returned to the dehydrating column; the aqueous layer is diluted with water to recover more benzene and the wash waters (25% alcohol) are returned if desired to the beer still. C. HOLLINS.

Ointments (G.P. 443,756).—See XX.

XIX.—FOODS.

Different chemical constituents of the crust and crumb of Roman wheat bread. L. SETTIMI (*Annali Chim. Appl.*, 1928, **18**, 19—31).—Analyses show that the carbohydrates and nitrogenous substances in the crust and crumb of wheaten bread exhibit markedly different molecular constitutions. The crust contains greater proportions of degraded starch compounds and degraded protein derivatives than the crumb, and hence should have the greater nutritive value.

T. H. POPE.

Cheshire butters and cheeses of low Reichert-Meissl value. H. LOWE (*Analyst*, 1928, **53**, 89—90).—Samples of the butter and cheese were examined during November, and found to have low Reichert-Meissl

values, viz., 22.9 and 23.4 for two cheeses, and 22.6, 23.9, and 23.5 for three butters. A gallon sample of milk was churned in the laboratory, and the resulting butter fat had a Reichert-Meissl value of 22.1. Similar results had been obtained in previous years for November and December samples.

D. G. HEWER.

Accurate determination of the protein content of butter. B. J. HOLWERDA (*Chem. Weekblad*, 1928, **25**, 102—103).—A micro-method and apparatus are described, in which 1 g. of butter is treated with 1 c.c. of 50 vol. % sulphuric acid, the mixture kept for at least 30 min. at 40°, and then extracted twice with light petroleum to remove the fat. The residue is then treated in a micro-Kjeldahl apparatus and the nitrogen content determined in the usual way. The results are more consistent than those obtained by the macro-method, and experience shows that butter is usually sufficiently homogeneous for a 1 g. sample to be representative.

S. I. LEVY.

Determination of salt in butter and margarine. G. VAN B. GILMOUR (*Analyst*, 1928, **53**, 34).—A mixture of 10 g. of the melted butter with 20 c.c. of industrial methylated spirits is heated to the b.p., 40 c.c. of water are added, and the whole is mixed without reheating, filtered, and 30 c.c. of the filtrate are titrated with 0.1*N*-silver nitrate solution; the number of c.c. multiplied by 0.119 (assuming a water content of 15%) gives the percentage of salt. The method is satisfactory even in the presence of emulsifying agents.

D. G. HEWER.

Arsenic in New Zealand-grown apples. R. L. ANDREW (*New Zealand J. Sci. Tech.*, 1927, **9**, 206—209).—Apples sprayed with lead arsenate according to the usual New Zealand practice were picked at varying intervals from the last spraying and examined for arsenic. The peeled fruit (including core and seeds) was treated separately and the quantity of arsenic did not exceed 1/700 grain per lb. of whole fruit, and in most cases was negligible. The peel (including stem and calyx) contained more, but with an interval of 12—15 days between the last spraying and picking the total arsenic in the apple did not exceed 1/100 grain per lb. Ordinary wiping with a dry cloth reduced the arsenic but little, except where patches of dry spray were visible. The arsenic appeared evenly distributed over the whole skin, there being no accumulation at the stem or calyx.

J. R. NICHOLLS.

Relation of atmospheric humidity to deterioration of evaporated apples in storage. C. W. CULPEPPER and J. S. CALDWELL (*J. Agric. Res.*, 1927, **35**, 889—906).—Dried samples of five varieties of apples were stored at 20—30° under conditions such that atmospheric humidity ranged from 0 to 100%. At relative humidities above 80.5%, fungal growths appeared rapidly; between 47% and 80.5% there was no fungal growth, but changes occurred which made the fruit unmarketable before the end of the storage period. At 18.5% R.H. or less, evaporated apples maintained their original qualities indefinitely. Pretreatment of the samples with sulphur dioxide or sodium chloride, or by heating in steam at 80°, delayed deterioration at the higher

humidities, sulphur dioxide being the most effective reagent.

H. J. G. HINES.

Occurrence of acetaldehyde in Bartlett pears and its relation to pear scald and breakdown. C. P. HARLEY and D. F. FISHER (*J. Agric. Res.*, 1927, **35**, 983—993).—A definite relationship exists between the amount of acetaldehyde in pear tissue and the severity of attacks of both scald and core breakdown, all pears having more than 14 mg. of acetaldehyde per 100 g. of fresh tissue being thus affected. Highest concentrations of acetaldehyde were found in the tissues within the breakdown area, and also in the flesh immediately beneath the scald. Acetaldehyde was demonstrated to be a normal metabolic product of healthy pears, and it is suggested that when local accumulations exceed limiting values, browning of the tissues occurs. The toxicity of acetaldehyde to pear tissue was shown artificially.

A. G. POLLARD.

Vitamins in canned foods. VI. Strawberries. E. F. KOHMAN, W. H. EDDY, and N. HALLIDAY (*Ind. Eng. Chem.*, 1928, **20**, 202—204; cf. B., 1926, 213).—By means of feeding experiments on animals it is shown that strawberries are a rich source of vitamin-C, similar in this respect to tomatoes. The canning of strawberries results in no apparent loss of vitamin-C, probably on account of the effective removal of oxygen by the exhaust. The content of strawberries in vitamin-A is only one fortieth and in vitamin-B one fourth of that of tomatoes.

F. R. ENNOS.

Composition of fruit. L. H. LAMPITT and E. B. HUGHES (*Analyst*, 1928, **53**, 32—34).—A summary of analyses of various fresh fruits for the years 1925—1927.

D. G. HEWER.

Determination of mustard oil. P. SACCARDI and M. PIERI (*Arch. Farm. sperim. Sci. aff.*, 1927, **44**, 1—10).—The Kuntze-Dieterich method of determining mustard oil gives results varying with the time during which the precipitated silver sulphide is dried. Experiment shows that the sulphide may undergo partial conversion into oxide or into sulphate, which may then lose sulphur dioxide and oxygen to form the oxide. Either of the two following methods gives satisfactory results: (1) 5 g. of the pounded mustard are macerated with 100 c.c. of water at the ordinary temperature for 30 min., three fourths of the liquid being then distilled off and made up to 100 c.c. Of this solution, 50 c.c. are mixed with 20 c.c. of 0.5N-alcoholic potassium hydroxide solution and heated in a reflux apparatus on a steam-bath for 30 min. The excess of alkali is determined by titration. For ordinary commercial mustard containing 0.71—0.87% of oil, the extract from 5 g. of the mustard requires 0.0224—0.027 g. of potassium hydroxide, or 0.78—0.96 c.c. of the 0.5N-solution for saponification. (2) 10 g. of the mustard flour are macerated for 12 hrs. with water, the mass being separated on a dry filter and washed into a 100 c.c. flask; the liquid is defecated with neutral lead acetate, de-leaded with ammonium carbonate, made up to volume, and filtered. The dextrose in 10 c.c. of the filtrate is determined by means of Fehling's solution, the percentage of dextrose, calculated on the mustard, having the mean value

0.95; for the non-defecated extract 0.94% was obtained.

T. H. POPE.

Biological values of certain types of sea food. II. Vitamins in oysters (*Ostrea virginica*). D. B. JONES, J. C. MURPHY, and E. M. NELSON (*Ind. Eng. Chem.*, 1928, **20**, 205—210).—To convert oysters into a homogeneous, palatable form which would not deteriorate during the course of the experiments, they were drained, frozen, and the frozen product finely ground. Dehydration at 40° under reduced pressure could not be employed as it resulted in the loss of nearly all the vitamin-B properties and a considerable proportion of those of vitamin-A. The content of the frozen oysters in vitamins-A, -B, and -D, as determined by feeding tests on albino rats, compared favourably on the dry basis with that of other vitamin-containing foods. Oysters are deficient in vitamin-E. Some evidence was obtained suggesting that there may be a seasonal variation in the vitamin content of oysters.

F. R. ENNOS.

Fermentation of sauerkraut. PARMELE and others.—See XVIII.

PATENTS.

Manufacture of bread. H. A. KOHMAN (U.S.P. 1,655,707, 10.1.28. Appl., 12.12.24).—The flour, yeast, and other ingredients of the batch, including an oxyhalogen acid compound and a persulphate, e.g., 0.0015% of potassium iodate and 0.075% of potassium persulphate based on the weight of flour used, are vigorously mixed by mechanical means. Without setting the dough aside for any fermentation period, it is at once subdivided, proofed, and baked in the usual way.

F. R. ENNOS.

Manufacture of leavened bread. A. K. EPSTEIN (U.S.P. 1,657,379, 24.1.28. Appl., 14.12.25).—The dough batch is mixed with sufficient yeast-assimilable carbohydrate to enable the yeast to carry on fermentation, with a water-soluble, yeast-assimilable substance containing organic nitrogen, e.g., carbamide, and with potassium chlorate and magnesium sulphate. The amount of yeast used may be either the same as that normally required, in which case the fermentation will be more readily completed, or less than that needed to ferment the dough completely in the ordinary mixture, the fermentation in this case being allowed to continue for the usual period.

F. R. ENNOS.

Bread improver. A. H. FISKE, Assr. to RUMFORD CHEMICAL WORKS (U.S.P. 1,657,116, 24.1.28. Appl., 7.3.27).—A mixture of powdered gum tragacanth and iodic acid, with or without flour, is added to the dough batch.

F. R. ENNOS.

Removal of the cerealin from grain. S. STEINMETZ (B.P. 259,989, 15.10.26. Ger., 19.10.25).—The grain after soaking is strained from the excess of water and subjected to pressure by beater blades in a non-ventilated drum, whereby heat is produced by friction of the grains, which serves to soften both the cellulose and the cerealin layers. The husks are then removed and part of the cerealin layer is washed off by water under pressure. The grain finally passes to a polishing drum, in which it is again subjected to pressure and heating by friction to free it completely from cerealin and to dry it.

F. R. ENNOS.

Preservation of meat. W. H. HOBBS and A. LANE (B.P. 283,626, 11.10.26).—The meat is placed in a closed chamber maintained at 0–1°, whilst a current of air, cooled to the same temperature and previously sterilised by passage through a solution of thymol in acetic acid, is introduced in the form of jets from a travelling pipe at the bottom of the chamber so that it circulates around and between and impinges against the meat, and is finally withdrawn at the top. The moisture content of the air in the chamber should be 62–66%, and, if necessary, the humidity of the circulating air may be reduced by passing over a suitable hygroscopic material, e.g., calcium chloride, and then through a layer of granular thymol prior to introduction into the meat chamber.

F. R. ENNOS.

Preparation of a soluble gum composition. A. LEO (B.P. 283,657, 18.10.26).—To standard pectin is added a mixture of finely-powdered citric acid and sodium bicarbonate containing an excess of the former. On addition of this composition to the boiling fruit juice to be jellified, the carbon dioxide evolved disintegrates the pectin and causes it to pass into solution.

F. R. ENNOS.

Conservation of animal foodstuffs. W. K. GÜNTHER (B.P. 284,130, 7.6.27).—The foodstuff, e.g., fish, is enclosed in a thin textile web and packed in sphagnum (white moss), which has been partially saturated with water cooled by ice and salt.

F. R. ENNOS.

Wrappers for foodstuffs (B.P. 283,751).—See V.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Casein glycerophosphate. J. J. L. ZWIKKER (Pharm. Weekblad, 1928, 65, 134–136).—Analysis of Sanatogen shows it to consist approximately of 5% of sodium glycerophosphate and 95% of sodium caseinate; the latter is somewhat more alkaline in reaction than the sodium caseinate prepared according to the British Pharmaceutical Codex.

S. I. LEVY.

Preparation of guaiacolsulphonic acid from *p*-dichlorobenzene. J. S. TURSKEI, A. PIOTROVSKI, and S. VINAVER (Przemysl Chem., 1927, 11, 365–370; Chem. Zentr., 1927, ii, 809).—*p*-Dichlorobenzene (1 g.-mol.) is nitrated with a mixture of 90% of nitric and 10% of fuming sulphuric acid at 60° to 1 : 4-dichloro-2-nitrobenzene, m.p. 53°, which with methyl-alcoholic potash at 140° and 7 atm. in 5 hrs. yields *p*-chloro-*o*-nitroanisole, m.p. 97°. This, distilled with superheated steam, is reduced with iron filings and hydrochloric acid to *m*-chloro-*o*-anisidine, m.p. 80°, and the diazo compound decomposed in presence of copper sulphate, yielding chloroguaiacol, b.p. 236–241°, m.p. 37°. This, when heated with sodium sulphite at 175–180° for 15–20 hrs., yields 92 g. of guaiacolsulphonic acid (1-hydroxy-6-methoxybenzene-3-sulphonic acid), obtained by evaporation of the acidified product and extraction with alcohol.

A. A. ELDRIDGE.

Determination of iodine in thyroid gland preparations. E. SCHULEK and A. STASIAK (Pharm. Zentr., 1928, 69, 113–115).—The material is heated with potassium hydroxide in a nickel crucible, the melt dissolved,

the filtered solution oxidised with hypochlorite, and iodate determined by addition of potassium iodide and titration with thiosulphate. Only 0.1 g. of the material is taken, and the heating with potassium hydroxide (2.5 g.) requires only 10 min.

S. I. LEVY.

Analysis and chemistry of neoarsphenamine. A. E. JURIST and W. G. CHRISTIANSEN (J. Amer. Chem. Soc., 1928, 50, 191–196; cf. B., 1926, 27, 767).—Both free and combined formaldehyde-sulphoxylate reduce iodine in neutral solution. Consequently, when neoarsphenamine is oxidised with excess of iodine, the residual iodine reduced with sodium arsenite, the solution acidified, and the sulphate precipitated as barium sulphate, the sulphur thus determined is usually greater than that given by the Elvove method. If x be the free sulphoxylate sulphur, y the combined sulphoxylate sulphur, A the sulphur found by the above arsenite method less free sulphate sulphur, and B the iodine titration in neutral solution less the arseno-group iodine equivalent, x and y can be evaluated from the equations $x + y = A$; and $x/0.8 + y/1.6 = B$. By analysis of mixtures, synthetically prepared, containing sulphonic acids, it is shown that the difference between total sulphur and sulphur oxidisable by iodine in alkaline solution is due to nuclear sulphur introduced in the preparation of the arsphenamine base from which the neoarsphenamine is obtained, and not to sulpharsphenamine. Comparative analyses of a number of neoarsphenamines indicate the probable existence of two types of combination between arsphenamine base and sodium formaldehyde-sulphoxylate, one being of the *N*-methylene type, the other resembling double-salt formation. In addition, there is present in neoarsphenamine sulphur other than sulphoxylate, derived from some subsequent chemical change, possibly oxidation.

F. G. WILLSON.

Determination of arsenic in arsenobenzenes. A. KIRCHER and F. VON RUPPERT (Pharm. Zentr., 1928, 69, 17–18).—The authors have carried out comparative tests with their own method (A., 1924, ii, 59; B., 80), that of Stollé and Fechtig (Z. angew. Chem., 1927, 40, 1154), and that of De Myttenaere (B., 1924, 32), and find that the differences are negligible. The authors' method gave results slightly above those obtained by the other methods.

S. I. LEVY.

Determination of chloral in chloral syrup. M. FRANÇOIS (J. Pharm. Chim., 1928, [viii], 7, 54–57).—Determination of chloral in chloral syrups by the author's method (B., 1918, 166 A) gives satisfactory results. The criticisms of Andron (Bull. Soc. Pharm. Bordeaux, 1926, 64, 199) are shown to be invalid. E. H. SHARPLES.

Determination of citral and similar aldehydes [in essential oils]. L. G. RADCLIFFE and W. J. N. SWANN (Perf. Ess. Oil Rec., 1928, 19, 47–51).—The reaction of thiosemicarbazide with aldehydes and ketones (Neuberg and Neiman, A., 1902, i, 572) has been studied quantitatively, particularly with reference to the determination of citral in lemon oils. The following method gives values for citral slightly higher than those obtained by the hydroxylamine method of Bennett. 2–3 g. of the oil are heated on a water-bath with 0.8 g.

of thiosemicarbazide and 50 c.c. of alcohol until most of the thiosemicarbazide is dissolved. The mixture is evaporated to dryness, boiled with 150 c.c. of carbon disulphide, and filtered. The solid, unchanged thiosemicarbazide is washed, dried, and weighed, and the amount of citral thiosemicarbazone formed can be calculated. By the use of suitable solvents the method is applicable to other aldehydes and ketones, *i.e.*, the solvent must remove all substances except the excess of thiosemicarbazide. The method may also be employed for the detection of traces of impurities in commercial aldehydes and ketones. After separating the excess of thiosemicarbazide the presence of any impurity is indicated by the depression of the m.p. of the thiosemicarbazone remaining after removal of the solvent. In a sample of commercial citral the m.p. of the thiosemicarbazone was depressed 10°, and it is suggested that a correlation between m.p. depression and percentage of impurities might be established. The formation and properties of metallic salts of thiosemicarbazones are discussed.

E. H. SHARPLES.

Methyl alcohol in tinctures. VIVARO. See XVIII.

PATENTS.

Preparation of pyridine derivatives. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 259,960, 11.10.26. Austr., 17.10.25).—Chlorination of β -iodopyridines yields β -pyridyl iodochlorides, $\text{NC}_5\text{H}_4\cdot\text{ICl}_2$, from which by hydrolysis with alkali β -iodopyridines are obtainable. *3-Pyridyl iodochloride*, m.p. 128—130° (decomp.), *2-chloro-5-pyridyl iodochloride*, m.p. 104—106° (decomp.), *2-chloro-5-iodopyridine*, decomp. 200—205°, and *2-chloro-5-iodoxypyridine*, detonating at 210—215°, are described. The iodoxy-compound is prepared by distilling in steam the iodoxy-derivative.

C. HOLLINS.

Production of isonaphthyridine and its derivatives. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 259,973, 13.10.26. Aust., 17.10.25).— β -Aminopyridines are subjected to the Skraup or Doebner reaction. *2-Chloro-5-aminopyridine*, with glycerol, arsenic pentoxide, and sulphuric acid, gives *2-hydroxy-1:5-naphthyridine* [*2-chloroisonaphthyridine*], not melted at 250°; *1:5-[or iso]naphthyridine*, m.p. 72°, b.p. 112/12 mm., is similarly obtained from *3-aminopyridine*. *2-Chloro-3-aminopyridine* condenses with pyruvic acid and benzaldehyde to form *2-chloro-6-phenyl-1:5-naphthyridine-8-carboxylic acid*, decomp. 186°. In each case cyclisation occurs *ortho* to the pyridine nitrogen.

C. HOLLINS.

Preparation of quinoline derivatives. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 282,143, 11.9.26).—4-Aminoquinolines, obtained by replacement of a halogen, alkoxy-, hydrazino-, carbonamido-, or carbazido-group in 4-position by an amino-group, are condensed with aromatic aldehydes; or the introduction of the amino-group may follow the condensation. The products are strong bactericides. The following compounds are described: *4-Chloro-2-styrylquinoline*, m.p. 117°; *4-amino-2-styrylquinoline*, m.p. 173—175°; *4-chloro-6-ethoxy-2-styrylquinoline*, m.p. 120°; and the *4-amino*-compound, m.p. 210° (*glycollate*, m.p. 237°); *4:6-di-*

ethoxy-2-styrylquinoline (from the 4-chloro-6-ethoxy-compound and sodium ethoxide); *4-phenylhydrazino-2-styrylquinoline* (from 4-chloro-2-styrylquinoline and phenylhydrazine); *4-phenylhydrazino-6-ethoxy-2-styrylquinoline*: *4-chloro-2-methyl- β -naphthaquinoline*, m.p. 200°; *4-chloro-2-styryl- β -naphthaquinoline*, m.p. 128°; *4-amino-2-styryl- β -naphthaquinoline*, m.p. 226—227°; *4-amino-6-ethoxyquinaldine*, m.p. 195°; *4-amino-6-ethoxy-2-m-nitrostyrylquinoline*, m-aminostyryl compound, m.p. 182°; *2-styrylquinoline-4-carboxylic acid (methyl ester)*, m.p. 98°; *ethyl ester*, m.p. 77°; *amide*, m.p. 274°; *hydrazide*, m.p. 215°; *azide*; *urethane*, m.p. 202°; *4-ethylamino-6-ethoxy-2-styrylquinoline*, m.p. 178° [*glycollate*, m.p. 214—216° (decomp.)]; *4-diethylamino*-compound, m.p. 103°; *4-diethylamino-6-ethoxyquinaldine*, m.p. 74°.

C. HOLLINS.

Manufacture of 2-phenylquinoline-4-carboxylic acid [atophan]. A. J. STEPHENS. From R. VON WULFING (B.P. 283,822, 23.9.27).—By the use of the compound of isatin with sodium or potassium hydrogen sulphite (sodium compound contains 52.9% of isatin) for condensation with acetophenone a very pure atophan, m.p. 212—213°, is obtained.

C. HOLLINS.

Manufacture of crystallised arsenobenzenes. A. ALBERT (B.P. 259,245, 4.10.26. Ger., 3.10.25).—Pure crystalline arseno-compounds are obtained by reducing with hypophosphorous acid (or salts) an arsenic acid, or arsenoxide, or dihalogenoarsine, containing the grouping, $\text{As}\cdot\text{Ar}\cdot\text{COR}$, where R is hydrogen or alkyl, or the hydrazones, semicarbazones, etc. of these. *3-Hydroxybenzaldehyde-4-arsinic acid*, prepared from *3-nitro-4-aminobenzaldehyde* by Bart's reaction followed by reduction of the nitro-group and diazotisation, is reduced in the form of its *semicarbazone*, or of the *dichloroarsine*, to *2:2'-dihydroxy-4:4'-dialdehydoarsenobenzene*. *3-Hydroxyacetophenone-4-arsinic acid semicarbazone*, or the *dichloroarsine*, or the *arsenoxide*, gives *2:2'-dihydroxy-4:4'-diacetylarsenobenzene*, decomp. above 200°.

C. HOLLINS.

Preparation of organo-mercury compounds. F. HEIN and K. WAGLER (G.P. 444,666, 13.8.24).—Organo-mercury salts are treated with metals (copper, silver, iron, nickel, cobalt, zinc) or their alloys in presence of basic nitrogen compounds (ammonia, amines, pyridine bases and homologues) with or without other solvents. Benzyl mercurichloride in pyridine is treated for 1—2 hrs. with copper turnings or warmed for 1 hr. with copper gauze; the pyridine is distilled off in a vacuum and copper chloride removed by means of aqueous ammonia, leaving mercury dibenzyl, $\text{Hg}(\text{CH}_2\text{Ph})_2$, in 90% yield; mercury is recovered by heating the copper in hydrogen. Mercury diphenyl, m.p. 125°, is obtained by treating phenyl mercurichloride with zinc turnings in alcoholic pyridine, or diethylamine or liquid ammonia. *Mercury 2:2'-dinitrodiphenyl*, m.p. 204.5—205°, and *mercury tetramethyldiaminodiphenyl*, m.p. 167—168°, are similarly prepared. The products find pharmaceutical application.

C. HOLLINS.

Benzylresorcinols. A. R. L. DOHME, Assr. to SHARP & DOHME (U.S.P. 1,658,229, 7.2.28. Appl., 9.10.23).—See B.P. 223,190; B., 1925, 378.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Experiments with hyposulphite and Rongalite. F. BÜRKI (Phot. Korr., 1928, 64, 16—17).—A 0.1% solution of hyposulphite acts as a developer, giving a slight fog. A 1% solution acts more quickly but with pronounced fog, whilst more concentrated solutions give mostly fog. If Rongalite, the addition product of hyposulphite and formaldehyde, is used the solution does not act as a developer, and the latent image, after treatment with this solution, can still be developed with a normal developer. Pre-treatment of the latent image with formaldehyde accelerates the subsequent developing action of hyposulphite or of an ordinary developer. Selenium is soluble in a solution of hyposulphite or of Rongalite, giving a neutral, colourless solution, which can be used for toning papers. W. CLARK.

Constitution of the flavinduline desensitisers. G. KÖGEL (Phot. Korr., 1927, 63, 193—195).—The desensitising group is considered to be the azomethine group $>C:N$, of which there are two in flavinduline. W. CLARK.

Development of Daguerre plates by cathode pulverisation. G. SIMON (Compt. rend., 1928, 186, 139).—Daguerre plates (polished silver lightly treated with iodine) may be developed by exposure for a fraction of a second to the pulverisation obtained from a pure or an amalgamated copper cathode, in place of exposure to mercury vapour. Gold or platinum cathodes gave negative results. J. GRANT.

Hypothesis of the latent image. A. P. H. TRIVELLI (Z. wiss. Phot., 1928, 25, 65—78).—See B., 1928, 107.

Fog correction of photographic blackening. H. A. PRITCHARD (Z. wiss. Phot., 1928, 25, 79—113).—See B., 1927, 957.

PATENTS.

Light-sensitive paper etc. A. H. MEURSING and E. J. GRATAMA, Assees. of T. RODY (B.P. 274,075, 1.7.27. Ger., 7.7.26).—For producing a sensitive velour-like material such as paper, linen, silk, etc. an adhesive substance, insoluble in water, is applied to the base, after which fine vegetable or animal fibre dust is applied. The fibre dust is rendered sensitive before or after application, for example by bathing the paper etc. in a silver nitrate solution followed by a solution containing ammonium bromide and potassium iodide. W. CLARK.

Colour photography. W. T. TARBIN (B.P. 283,765, 17.3.27. Ger., 1.2.27).—In a method of colour photography using a number of superimposed layers each sensitive to a different spectral region, the layer sensitive to the blue is arranged so that in exposure it is the farthest from the source of light. The composite plate may be built up as follows. A glass plate is coated with a red-sensitive stripping layer in contact with which is a non-stripping green-sensitive layer on a thin celluloid support, whilst the blue-sensitive layer is coated on glass, the layer being in contact with the back of the celluloid support of the green-sensitive layer. W. CLARK.

Production of a colour photograph or film. F. E. IVES, Assr. to C. W. FENNINGER (U.S.P. 1,655,182, 3.1.28. Appl., 29.10.20).—The process of U.S.P. 1,278,668 (B., 1918, 784 A) is modified in that a blue-to-green, iron-toned image is produced at the interior surface of the colloid layer, which is then treated with potassium bromide to resensitise the silver present, and the red image is produced at the outer surface. T. S. WHEELER.

Preparation of layers for surface-printing processes. U. OSTWALD (B.P. 262,793, 8.12.26. Ger., 8.12.25).—In the preparation of a layer for surface-printing processes, glue, gelatin, or albumin is mixed with a non-precipitating, water-insoluble, organic colloid which strengthens the particles of the glue etc. and reduces its water-absorbing properties. When dry the layer is treated with an agent which tans it on exposure to light. The glue etc. is dissolved in a suitable organic solvent, preferably acetic acid, whilst the organic colloid used may be a cellulose derivative and/or a resin in a solvent such as acetic acid. Rubber or linoleum or other elastic substance is used as the support and solutions of cellulose derivatives containing plastifiers are used as intermediate layers in cases when the main layer will not adhere firmly to the support. After washing out the solvent, sensitising with dichromate, exposing, and washing out the dichromate, the unexposed parts of the layer are rendered porous by treatment with alkaline reagents such as ammonium carbonate or an alkali carbonate. W. CLARK.

Coloured magnesium flame (G.P. 445,181).—See X.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Manufacture of explosives. F. W. GUTHKE (B.P. 275,228, 25.7.27. Ger., 28.7.26).—Explosives are obtained by nitrating mixtures of solid and liquid polyhydric alcohols. In particular, the explosive may contain a mixture of nitrated sorbitol and nitrated ethylene glycol with or without nitrated glycerol. S. BINNING.

Fuse compositions and fuses for shells. CURTIS'S & HARVEY, LTD., and A. J. GRIMWOOD (B.P. 283,741, 21.1.27).—A powder for use in time rings for shell fuses consists of black powder in which all or part of the charcoal is replaced by cuprene. The powder may be compressed into brass-lined channels in rings made of aluminium or its alloy, to form a shell time-fuse which may be combined with a percussion fuse. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Carbon and nitrogen transformation in fresh sewage solids digestion. H. HEUKELEKIAN and W. RUDOLFS (Ind. Eng. Chem., 1928, 20, 177—179).—The changes in carbon, nitrogen, and volatile matter during the fermentation of fresh sewage solids are indicated graphically, the results obtained with and without the addition of lime to maintain a p_H value of 7.4 being compared. It is shown that the total nitrogen content is unchanged, though a decreasing amount of volatile matter causes a rise in the percentage nitrogen

In the case of limed material there is a reduction of 60% in the volatile matter and of 30% in the carbon content during a period of 8 months. The addition of lime facilitates the decomposition of certain types of organic matter, possibly fats, with consequent increase in the volume of gas produced.

C. JEPSON.

Gases from sewage sludge digestion. W. D. HATFIELD, G. E. SYMONS, and R. R. MILLS (Ind. Eng. Chem., 1928, 20, 174—176).—The sewage from Decatur (Illinois) contains half its volume of waste from a corn products factory, and has a temperature of 20—40°. Under these conditions putrefaction is rapid and the gases evolved contain up to 1% by vol. of hydrogen sulphide. Plant and laboratory experiments have shown that the rate of gas formation reaches a maximum about 30°, and that, at 30—35°, half the volume of gas evolved is obtained from sludge deposited during the previous 24 hrs.

C. JEPSON.

Activated sludges. L. CAVEL (Compt. rend., 1928, 186, 433—436).—Determinations of the carbon and nitrogen contents of activated sludges before and after aeration for five months showed that 4.32% and 67.17% were removed, respectively. Similar determinations using the dried and heated sludge have shown that the reduction of organic matter, under the influence of aeration, refers almost exclusively to the nitrogen removed, and indicates that the sludge is de-activated by an excess of nitrogen, since when this is removed by aeration its properties are restored. Unlike that of bacterial beds, the activity of sludges depends largely on the composition of the sewage treated.

J. GRANT.

Natural and artificial black sand for demanganising water. H. LÜHRIG (Gas- u. Wasserfach, 1927, 70, 1277—1281).—The black sand, containing deposited manganese oxides, found in the alluvial bed of the Oder, occurs only in places to which air has access. When water is passed through a filter bed of this sand, part of the oxygen in the former is removed, not bacteriologically but apparently by certain oxides of manganese which give it up again in the process of demanganising water. This is borne out by the observations that the demanganising efficiency of the sand increases with use, and that demanganisation is incomplete if the concentration of manganese in the water exceeds about 45 mg. of manganous oxide per litre. By supplying additional oxygen, however, *e.g.*, as permanganate, demanganisation is complete even with such concentrations. Other per-salts, also chlorine, have the same revivifying effect on the sand, although chlorine may, in certain concentrations, exert a poisoning action. The chemistry of the demanganising process is not yet clear, but the same results are obtained with both natural and artificially prepared black sands.

W. T. K. BRAUNHOLTZ.

Stable colour standards for the determination of nitrites in water. R. DANET (J. Pharm. Chim., 1928, [viii], 7, 113—114).—For the determination of nitrites in water by the Griess method, Fuchsine S is used as colour standard. In four standard tubes are placed 0.3, 0.6, 0.9, and 1.2 c.c., respectively, of a 0.01% solution of Fuchsine S and the volume in each is made up to 10 c.c. with distilled water saturated with camphor and con-

taining 1% of acetic acid. In a similar tube are placed 10 c.c. of the water and 1 c.c. each of the sulphanilic acid and naphthylamine reagents, and the colour formed after 20—30 min. is compared with those of the standards, which represent 0.5, 1.0, 1.5, and 2 mg. of sodium nitrite per litre, respectively.

E. H. SHARPLES.

Vorce's method for determination of small quantities of phenol in impure water. F. LIEBERT and W. M. DEERNS (Chem. Weekblad, 1928, 25, 103—105; cf. B., 1925, 861).—The four stages of the method, *viz.*, purification and concentration of the water, separation of the phenol, and colorimetric determination, have been examined to estimate the errors which may arise. Since phenol itself is readily oxidised by hydrogen peroxide in bright light, the purification should be carried out in the dark. No losses of phenol occur during concentration under the conditions laid down; the method of separation is also satisfactory. Use of corks or rubber stoppers in the apparatus for the distillation gives rise to errors, the results being too high in consequence of the presence in the phenol solution of reducing substances from the stoppers or corks. The tungsten molybdate reagent employed for the colorimetric determination is not specific for phenol, so that errors may arise from this cause; generally, however, the method is sufficiently accurate for ordinary purposes.

S. I. LEVY.

Elimination of errors in the o-tolidine method [for determination of chlorine in water]. F. R. McCURMB (J. New England Water Works' Assoc., 1927, 41, 386—398).—The colour-producing substance is shown to be a hydrogen-ion indicator giving the desired yellow when the liquid under test has pH 2.0 or less. In order to ensure this condition, the maximum amount of hydrochloric acid which can be used in making up the o-tolidine solution has been determined, and a method of preparation is recommended. A comparator set is described by which it is possible to minimise the interference due to colour, turbidity, and the effects of manganese, iron, and nitrites.

C. JEPSON.

Effect of addition of salts on the germicidal efficiency of sodium hydroxide. M. LEVINE, J. H. TOULOUSE, and J. H. BUCHANAN (Ind. Eng. Chem., 1928, 20, 179—181).—The addition of sodium chloride, sodium carbonate, and trisodium phosphate to sodium hydroxide markedly decreases the time required at 50° and 60° to effect a 99.9% removal of bacteria. Trisodium phosphate is less efficient than the other two salts, the effects of which are approximately the same. Reasons to account for these results are suggested (cf. B., 1928, 70).

C. JEPSON.

Eye protection glasses. BLOCH.—See VIII.

PATENTS.

Cleansing agent for teeth. C. H. BOEHRINGER SOHN, Assees. of H. STROBEL (G.P. 442,857, 15.5.25).—Salts which generate small amounts of acid by hydrolysis, especially organic salts, and aluminium lactate in particular, are used with or without addition of chalk, magnesium carbonate, or other acid-binding agent.

C. HOLLINS.

Disinfectant (Swiss P. 118,144).—See XVI.