

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 29 and SEPT. 5, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Economics of high-pressure steam. G. A. ORROK (Engineering, 1930, 130, 25—26).—Two sets of curves are plotted correlating: (1) maximum steam temperatures and B.Th.U. per kw.-hr., for the Carnot cycle, and for regenerative-reheat cycles at five selected pressures and both 100% and 80% of the theoretical efficiency, the 80% being considered to be sufficient allowance for all losses from the fuel to the switchboard; (2) steam pressures and savings over a standard of 200 lb./in.² for two selected temperatures (750° and 1000° F.). Three tables are also given showing operating data of stations using steam at 250—1450 lb./in.² It is concluded that raising the pressure increases the economy very largely at the lower ranges, the effect being much reduced at 800 and almost negligible at 1600 lb./in.²; raising the temperature has a much smaller effect. The installation costs of high-pressure stations are at present higher than for low-pressure, but the difference is likely to disappear; there is already no difference in maintenance costs. B. M. VENABLES.

Evaporation tests with models of Ruths' steam accumulator. K. NESSELMANN and F. DARDIN (Wiss. Veröff. Siemens-Konz., 1930, 9, [1], 369—389).—It is shown that with suitable precautions surface charges of more than 400 m.³/m.²/hr. may be obtained without overflowing; e.g., by fixing a cone just below the water surface and extending it into a cylinder reaching almost to the bottom of the accumulator it is possible to work with surface charges of 1650 m.³/m.²/hr. without causing foaming and the formation of large steam bubbles which break explosively from the surface of the water. This arrangement produces good circulation of the water, avoids the tendency to the formation of numerous layers of varying temperature, and reduces the capacity losses.

A. R. POWELL.

Chemistry of disintegration. A. CHWALA (Kolloid-chem. Beih., 1930, 31, 222—290).—Emphasis is laid on the importance of producing a satisfactory state of subdivision of various industrial products, such as paints, dyes, disinfectants, foodstuffs, etc., and the means of effecting the subdivision are considered. These are treated under three headings: (1) purely mechanical disintegration, (2) mechanical disintegration aided by the presence of almost chemically inert substances, (3) colloid-chemical processes aided by mechanical action. Types of colloid mills are described and the results obtained and their applications are outlined. The mechanism of peptisation is discussed.

E. S. HEDGES.

Adhesives and adhesion. Mechanical properties of materials and glued metal joints. W. B. LEE

(Ind. Eng. Chem., 1930, 22, 778—780).—In continuance of the work of McBain and co-workers (cf. B., 1928, 695), the tensile strengths of celluloid, cellophane, and fishing gut were measured, and the technique of the preparation of thin test-films and slabs is described. The strengths of the materials were higher as the rate of loading increased (cf. "lithographic gelatin"), and the strength of cellophane and the gut were decreased by increased relative humidity. A shellac-cresote cement and Bakelite "C" resin ($E = 34,600$ kg./cm.²) were studied in compression and bending. Of several common adhesives examined, shellac compositions gave the strongest joints between smooth nickel surfaces, but the adhesion between metal surfaces (in contrast to wooden ones) was less the rougher were the surfaces. The strengths of the joints were lower if the load was applied very slowly. The adhesion between optically polished quartz surfaces "wrung" with some highly purified ethylalkylcarbinols was measured (2.8—4.9 kg./cm.²).

E. LEWKOWITSCH.

Revivification of foam solutions for fire protection with carbon dioxide. C. B. WHITE (Chem. and Met. Eng., 1930, 37, 379—380).—By a few simple additions to the fire-protection pump the solution of sodium bicarbonate is circulated from the bottom of the storage tank, through a small tower, and back to the upper part of the tank; it is recarbonated in the tower by gas from a cylinder. The method is preferable to adding additional bicarbonate because if that is done the aluminium sulphate or other acid solution which must also be added has to be increased *pro rata* and eventually both will have to be scrapped. B. M. VENABLES.

Measurement of the heat expansion of solid bodies with the aid of photographic and mechanical recording devices. W. M. COHN (Chem. Fabr., 1930, 257—259).—The literature of the subject is briefly reviewed and a photographic recorder, devised by the author for measuring the thermal expansion of solids, using fused silica or Marquardt refractory as the standard rod, is described and illustrated. A. R. POWELL.

See also A., July, 867, **Catalytic reactions at high pressures** (MORGAN). 883, **Thermoregulators** (BEATTIE and JACOBUS; HIBBEN; WESSON; EGERTON). **Protection of ventilating fans** (DREIFUSS and STAALÉ). 884, **High-vacuum distillation with the aid of liquid air and active charcoal** (LEWIN). 885, **Rapid determination of density of small amounts of solids** (CALEY).

Tower-absorption coefficients. SIMMONS and LONG. —See II. **Purifying gases.** HEALY. —See XI. **Measurement of colour-tone.** VAN WÜLLEN-SCHOLTEN. —See XIII.

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

PATENTS.

Muffle furnaces. GIBBONS BROS., LTD., and T. E. BRIDGFORD (B.P. 329,787, 19.4.29).—The roof and sides of the muffle are built of hollow, triangular shaped bricks so arranged as to form a number of transverse, parallel flues, each of which communicates with a passage extending across the furnace below the floor of the muffle and all of which are spaced away from the roof and outer structure of the furnace. The hot gases are admitted to the flues at one side of the roof of the muffle and pass across the roof, down the opposite side, under the muffle, and up the other side to the main flues.
A. R. POWELL.

Muffle furnaces etc. E. A. HAILWOOD (B.P. 330,512, 4.3.29).—The walls of the muffle proper are constructed of metal frames with panels of thin sheet metal, such as rustless iron or steel, preferably corrugated.
B. M. VENABLES.

Heat-exchange device. G. KRÄNZLEIN, and SAMESREUTHER & Co., G.M.B.H. (B.P. 309,445, 8.4.29. Ger., 10.4.28).—A number of parallel tubes are connected to transverse tubes at the ends and the whole is sandwiched between two plates which may be either flat or corrugated and are provided with apertures for the outer fluid or for the leads of electric heaters.
B. M. VENABLES.

Steam generators, accumulators, etc. RUTHS-ACCUMULATOR AKTIEBOLAGET (B.P. 306,135, 7.2.29. Ger., 17.2.28).—In a steam accumulator priming may be caused by steam evolved from irregularities in the lower part passing through superheated water in the upper part and causing violent ebullition of the latter. In this invention evolution of steam is promoted originally in the upper part by means of injected gas bubbles or by causing the water to circulate through steel brushes or other spiky device. [Stat. ref.]
B. M. VENABLES.

Apparatus for carrying out endothermic catalytic gas reactions. I. G. FARBEININD. A.-G. (B.P. 330,872, 9.12.29. Ger., 15.12.28).—The catalysing units comprise return-flow, twin-concentric tubes; the closure of the return end of the outer tube is detachable and protrudes outside the heating chamber or furnace, and the other end only is secured to the furnace wall.
B. M. VENABLES.

Centrifugal apparatus for effecting physical or chemical changes [e.g., evaporation of liquids]. J. E. NYROP (B.P. 330,805, 17.7.29).—A liquid is spread centrifugally in a thin layer on the interior of a rotating cylinder, and gases or vapours are caused to pass in intimate contact with it by means of an interior device which is either stationary or rotated in the opposite direction, or is shaped so that the gases acquire an eddy-motion.
B. M. VENABLES.

Tube mills. MASCHINENBAU-ANSTALT HUMBOLDT (B.P. 310,342, 23.4.29. Ger., 23.4.28).—In a tube mill with internal sifting means from which the ground material is removed by air, the stream of air is divided into two currents, one of which expands into the grinding space and picks up the material; the other passes straight through the mill in an axial pipe and does not entrain any dust until it mixes with the other stream in the

outlet pipe and produces a strong current for delivery. A damper is provided in the axial pipe to regulate the second stream.
B. M. VENABLES.

Pulverising machines. L. MELLERSH-JACKSON. FROM JEFFREY MANUF. CO. (B.P. 330,802, 12.7.29).—A disintegrator is provided with an inlet and a pocket for uncrushable material at the upper periphery and an abutment between the two. Within the pocket are shelves with upturned edges or like devices to retain the uncrushable material flung off the abutment.
B. M. VENABLES.

Drying of [expressing liquid from] pulp and the like. BLAIR CAMPBELL & MCLEAN, LTD., and S. HUTCHIN (B.P. 330,663, 20.3.29).—The apparatus comprises a vessel with a rising strainer bottom and with a lid that can be swung aside also formed as a strainer. After filling, the bottom is pressed upwards by a hydraulic ram, and, after pressing, the lid is swung aside and the upward motion continued to discharge the cake. The apparatus is suitable for use as a filtering medium in the brewing industry.
B. M. VENABLES.

Automatically regulating the concentration of materials [e.g., paper pulp] suspended in liquids. T. KÄLE (B.P. 307,300, 4.3.29. Swed., 3.3.28).—The control device is situated in the outlet pipe of a pump and the additional water or other diluent is added to the inlet pipe so that the pump forms an effective mixer. A portion of the mixed liquid is drawn through a by-pass to the main pipe by means of a propeller that produces a forward and rotating movement; the greater the concentration of the fluid, the less is the rotation. After passing through an orifice situated within the pipe, the fluid impinges on a flexibly supported target; the outer end of the support operates electrical contacts the current through which controls the supply of dilution water.
B. M. VENABLES.

Screening devices for liquids. H. J. VAN DER BIJL (B.P. 330,576, 11.3.29).—A screen (usually drum-shaped) for circulating water or other liquids is formed with apertures of which the diameter is not greater than their length in the direction of flow and is less than the diameter of the tubes of the condenser or other device to be protected. The apertures may be formed by drilling holes in a thick plate or by fastening short pieces of tube in a thin plate; the device is intended to catch long splinters, leaves, etc. that might pass through ordinary mesh-work.
B. M. VENABLES.

Emulsifiers, mixers, etc. H. A. HARRISON (B.P. 330,314, 12.3. and 4.6.29).—The material is forced by a plunger through a cylinder head or plug having a number of concentric grooves connected by small-bore passages which are preferably not quite radial, so as to produce a swirling action.
B. M. VENABLES.

Treatment of liquids with gases. KUPFERHÜTTE ERTEL, BIEBER & Co. (B.P. 308,254, 4.3.29. Ger., 20.3.28).—For aerating a liquid contained in a tank (or treating it with a gas which forms the atmosphere at the surface of the liquid), the liquid is circulated from the bottom of the tank, and then through a pump which forces it through a jet downwardly directed upon the surface of the liquid.
B. M. VENABLES.

Effecting intimate contact between gases and liquids. J. R. ANGER (B.P. 330,770, 14.6.29).—The gas is blown upon the surface of a foam-producing liquid below an inclined baffle in the containing chamber which forms, above it, a quickly expanding space for the outgoing gases and is provided with gutters which lead muddy condensed foam to a sump. B. M. VENABLES.

Continuous gas dryer. (SIR) G. C. MARKS. From H. A. BRASSERT & Co. (B.P. 330,284, 5.3.29).—A pair of moisture eliminators are connected to the mains by water-seals which are emptied to bring an eliminator into use, and filled when it is desired to cut one out for cleaning. B. M. VENABLES.

Devices for distributing air or other gases. Apparatus for separating solid substances from gases. ANEMOSTAT, LTD., and L. M. HIRCHBERG (B.P. 330,270 and 330,713, [A] 4.3.29, [B] 4.5.29).—(A) A device comprising a number of conical sheet guides, of common axis but differing angle, is rendered self-cleaning from adherent dust by dividing the cones into sectors each of which is pivoted near its narrow end and has the centre of gravity (when clean) brought to the axis of the pivot by addition of a counter-weight at the point. Any dirt accumulating will cause the sectors to hang vertically when the air current is off, but they will swing up towards their normal angle when the air current restarts, the resultant shaking dislodging the dust. (B) The gases are admitted downwardly at one end and exhausted upwardly at the other end of a tank which has a baffle depending from the top and a layer of fluid on the bottom. Distributing devices as described in (A) are used at both entrance and exit. B. M. VENABLES.

Means for extracting dust, grit, and like solid matter from gases. O. STOTT, and MATTHEWS & YATES, LTD. (B.P. 330,405, 11.6.29).—The gases are passed in a curved path between a number of curved plates and are moistened by sprays at the entrance and, if desired, intermediately. The cleaner may surround or be adjacent to a suction fan, the outlet of the former acting as the inlet of the latter.

B. M. VENABLES.

Methods and apparatus for gas analysis. I. FAGELSTON (B.P. 330,799, 12.7.29).—A sensitive form of viscosity-effusion bridge and accessories are described for comparing a standard gas with a sample. The regulating and indicating manometers are across the ends of the capillaries; beyond the latter, but before the gases unite, are orifices, preferably of rather smaller diameter than that of the capillaries, causing a decrease of pressure of at least 0.5 and preferably 0.6 of the pressure at entry to the orifices. The apparatus is suitable for all pressures and will indicate 2–3 mm. of water for every 1% change of oxygen in an oxygen-air mixture. B. M. VENABLES.

Substantially suppressing phosgene-formation when extinguishing fires with carbon tetrachloride. I. G. FARBERIND. A.-G. (B.P. 319,320 and Addn. B.P. 331,147, [A] 19.8.29, [B] 30.9.29. Ger., [A] 20.9.28, [B] 11.10.28).—(A) Alkyl or aryl phosphates (e.g., 1–3% of tritolyl phosphate), 0.1–3% of ammonia,

and, if desired, small quantities of hydrocarbons, or (B) animal or vegetable oils or fats (e.g., 2–4% of coconut oil or beef fat), alkyl or aryl phosphates, and ammonia, are added to the carbon tetrachloride (cf. B.P. 317,843; B., 1930, 169). L. A. COLES.

Heat-exchange apparatus. C. W. STANCLIFFE (U.S.P. 1,769,808, 1.7.30. Appl., 2.11.27. U.K., 12.11.26).—See B.P. 286,757; B., 1928, 352.

Cooling towers. J. M. SEYMOUR (B.P. 331,427, 28.8.29).—See U.S.P. 1,739,867; B., 1930, 398.

Grinding and mixing apparatus. R. O. CHILD, ASST. to D. ANDERSON & SONS, LTD. (U.S.P. 1,770,459, 15.7.30. Appl., 1.7.29. U.K., 20.7.28).—See B.P. 302,561; B., 1929, 267.

[Rotary drum] machine for treating granular material. H. BEHRENS, ASST. to MASCHINEN- U. WERKZEUGFABR. KABEL VOGEL & SCHEMMANN A.-G. (U.S.P. 1,769,670, 1.7.30. Appl., 27.3.29. Ger., 11.10.27).—See B.P. 322,405; B., 1930, 125.

Mixing and emulsifying apparatus. A. W. EMPSON (U.S.P. 1,770,302, 8.7.30. Appl., 25.6.27. U.K., 26.6.26).—See B.P. 282,466; B., 1928, 175.

Treatment of pulp. W. C. WEBER, ASST. to DORR Co. (U.S.P. 1,770,353, 8.7.30. Appl., 5.12.25).—See B.P. 262,479; B., 1928, 40.

Rotary spraying separator. F. H. LUENSE, ASST. to ROTOSPRAY MANUF. Co. (U.S.P. 1,769,588, 1.7.30. Appl., 4.5.27).—See B.P. 289,847; B., 1929, 268.

Liquid mixture of low f.p. G. REINHART, JUN., ASST. to GES. F. KÄLTECHEMIE GES. (U.S.P. 1,768,632, 1.7.30. Appl., 3.10.28. Ger., 17.11.27).—See B.P. 322,859; B., 1930, 127.

Air-heating installation for [boiler] furnaces. J. HOWDEN & Co., LTD., and J. H. HUME (B.P. 331,583, 6.4.29 and 3.2.30).

Heat-exchanging means particularly applicable to refrigerators. GEN. ELECTRIC Co., LTD., and F. H. BRITTAIN (B.P. 331,565, 4.3.29).

Absorption refrigerating machine with neutral circulating gases. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 304,122, 29.12.28. Ger., 14.1.28).

Refrigerating methods and apparatus. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 308,692, 26.3.29. Ger., 26.3.28).

Absorption refrigerating machines applicable also for heating purposes. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 309,443, 21.3.29. Ger., 10.4.28).

Condensers for refrigerating systems. BRIT. THOMSON-HOUSTON Co., LTD., ASSEES. of C. STEENSTRUP (B.P. 331,449, 3.10.29. U.S., 4.10.28).

Containers for storing and transporting liquefied gases and applicable for cooling air or other gases. S. BRAUNSTEIN (B.P. 308,621, 23.3.29. Ger., 24.3.28).

Frothing agents (B.P. 329,622).—See III. Heating of gases (B.P. 307,027).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coal-washing investigations—methods and tests.

H. F. YANCEY and T. FRASER (U.S. Bur. Mines Bull. 300, 1929, 259 pp.).—The results of an investigation of the washing characteristics of typical bituminous coals from the eastern, central, and western coalfields of the United States are described. The advantages of clean coal over raw coal and the effect of impurities in coal on its utilisation are discussed. The relation of the coal-bed structure, the physical and chemical impurities in the coal, and its washability are described. Systematic methods for testing the washability of a coal and the factors to be considered in the construction of a satisfactory cleaning plant are given. The sp. gr. of U.S. coals and the physical forms of their impurities vary greatly, consequently they vary in washing characteristics and in the method of treatment required. The sp. gr. of a coal is the chief factor on which ease or difficulty of washing depends. The two most important impurities in coal, viz., ash and sulphur, are not always associated in equal degree in the same sp.-gr. fraction of the raw coal, hence reduction of ash and reduction of sulphur are often separate and distinct problems. Methods for the treatment of fines and the results obtained are reviewed and the following subjects are briefly discussed: minimum sizes cleaned in jig and table operation, re-washing of fines, re-treatment of table middlings by hindered-settling operations, advantages of pretreatment, size classification, rheolaveur, Trent and froth-flotation processes, methods of drying washed coals, and dewatering tests of slurry with vacuum filters. C. B. MARSON.

Sp. gr. and proximate composition of some

Indian vitrains. L. L. FERMOR (Fuel, 1930, 9, 246—265; cf. B., 1929, 155).—Indian vitrains from the following stratigraphical horizons have been analysed: Barakar, Raniganj, Eocene, Miocene, and Siwalik. Taking all the data used, the limits of moisture are 0.63% and 16.61%, and the limits of sp. gr. are 1.214 (moisture 2.25%) and 1.414 (moisture 15.15%), calculated on the ash-free basis; the ash contents varied from 0.32% to 6.22%. Most of the vitrains with less than 9–10% of moisture are caking, whilst those of higher moisture content have no caking power; there is no correlation between caking properties and either volatile matter or fixed carbon content. The sp. gr. increases with the moisture content, irrespective of the age of the coal or of variations of moisture content on keeping the specimen. The relation between sp. gr. and moisture content is discussed in detail from the viewpoint of the colloidal character of vitrain. Within each coalfield the analyses show an orderly progression of properties in stratigraphical sequence; no such orderly progression is observed, however, when the whole of the data are so arranged. It is concluded that the variation in character is due to changes in the conditions of original deposition rather than to subsequent metamorphism. A. B. MANNING.

Chemical examination of lignite. W. FUCHS (Brennstoff-Chem., 1930, 11, 205—208).—A number of samples of lignite from the surface workings of a brown-coal mine at Tünnich have been analysed. When the methoxyl, lignin, cellulose, and humic acid contents

(on the ash- and bitumen-free basis) are plotted the results show clearly the general direction of the processes occurring in the gradual transformation of wood through lignite to brown coal, e.g., the progressive diminution in the percentage of cellulose, the accompanying increase in the percentage of lignin and methoxyl, and the transformation of lignin into humic acids (cf. B., 1930, 540). Only one sample deviated markedly from the general scheme; this was characterised by a high content of bitumen (36.4%), which had apparently retarded the decomposition of the cellulose and to some extent the transformation of the lignin.

A. B. MANNING.

Coal-dust explosibility factors indicated by experimental mine investigations, 1911—1929. G. S. RICE and H. P. GREENWALD (U.S. Bur. Mines Tech. Paper 464, 1929, 45 pp.).—The knowledge gained from experiments on the explosibility of coal dust under various conditions and from studies, following explosion disasters, is classified and summarised and methods for minimising or preventing mine disasters are described (cf. B., 1925, 433; 1926, 178). C. B. MARSON.

Production of fuels from brown coals and waste bituminous coals in the Lurgi plant. O. HUBMANN (Brennstoff-Chem., 1930, 11, 219—222).—The coal is carbonised in a vertical shaft wherein it is brought into direct contact with the heating gases. Units of this type have now been constructed capable of carbonising 200 tons per day. The semi-coke is briquetted, using as binding agent the pitch produced in the process supplemented, as is generally necessary, with other coal-tar pitch, or asphalt. It is preferable to separate the finer material from the raw coal before carbonisation and use this directly for steam-raising and power production; the throughput of the retort is thereby increased, whilst the ash content of the coke and the free carbon in the tar are lowered. The semi-coke should be allowed to come into contact with air before it is briquetted in order that its tendency to spontaneous ignition should be diminished by a limited oxidation. The semi-coke has a marked wearing action on the transporting and briquetting apparatus; the conveyors are preferably made of rubber, and the briquetting machine of the type forming rectangular briquettes. A considerable economy in pitch is achieved by mixing the coal with the hot atomised pitch in a suitable apparatus, the necessary proportion of pitch being about 7.5%. If desired, the larger-sized coke may be separated and marketed without briquetting. The briquettes behave well in the fire and are ideal for the open grate. Although high in sulphur (2%), 70% of this is non-volatile and is retained in the ash. A. B. MANNING.

Dehydration of manufactured gas. F. W. SPERR (Fuel, 1930, 9, 266—281).—The various methods available for drying gas, e.g., by compression, by refrigeration, by treatment with hygroscopic substances, or by treatment with adsorbents, are described and compared (cf. B., 1927, 243). Several methods are available, the cost of which should not exceed 1 cent per 1000 cub. ft. The importance of oil-fog lubrication as a protection against corrosion in the distribution of either moist or dry gas is emphasised. A. B. MANNING.

Removal of sulphur dioxide from flue gases.

A. THAU (Brennstoff-Chem., 1930, 11, 222—226).—By scrubbing the flue gases from a plant burning a North-umberland or Scottish coal, of sulphur content 3.9% and 1.23% respectively, with cold water, complete removal of the sulphur dioxide was effected when the time of contact between the water and the gases was 12 sec., corresponding with a water consumption of 45 tons per ton of coal fired. The sulphur content of the flue gases, expressed as H_2SO_4 , varied from 1—5 mg./m.³. To avoid too great a cooling of the flue gases it was found preferable to heat the water used for scrubbing; this had the further advantage of greatly accelerating the oxidation of the sulphur dioxide to trioxide, so that the water remained almost odourless. A design for a flue gas washing plant suitable for the proposed power station at Battersea is described.

A. B. MANNING.

"Flüssiggas." C. GERDES (Chem.-Ztg., 1930, 54, 509—510).—"Flüssiggas" is the fraction of oil-gas which may be made available for transport by compression into steel cylinders at ordinary temperatures, the resulting liquid contents of the cylinder reverting to gas on releasing the pressure. The properties and composition of the gas are discussed. The preparation of the crude gas by cracking oil in retorts or oil-gas generators (for large quantities) is described, and the subsequent elimination of difficultly condensable gases (chiefly methane, with some hydrogen and traces of carbon monoxide) and undesirable, easily liquefied hydrocarbons, is outlined. Apparatus of the Linde type may be used for fractionating the crude gas. The application of "Flüssiggas" for the lighting of buoys, railway carriages, etc. is discussed, and its use for aircraft propulsion suggested. The heating and storage properties of the gas are compared with those of coal gas, oil gas, and acetylene.

H. E. BLAYDEN.

Analysis of complex gaseous mixtures. W. L. WOOD (Fuel, 1930, 9, 288—291).—A preliminary analysis is carried out in the Bone and Wheeler apparatus. Attention is directed to the following possible causes of error: (a) the appreciable solubility of the higher hydrocarbons in the dilute acid used for washing out the absorption vessel, (b) the absorption of olefines by ammoniacal silver chloride when this is used to determine acetylene, and (c) the solubility of the paraffin hydrocarbons in the ammoniacal cuprous chloride used to absorb the carbon monoxide, all traces of which must be removed to avoid subsequent poisoning of the colloidal palladium used for the absorption of the hydrogen (cf. Beet, B., 1928, 113). Acetylene is removed by a 20% solution of mercuric cyanide in 2*N*-potassium hydroxide. The higher olefines are removed separately by using sulphuric acid of d 1.80, and introducing a suitable correction for the absorption of ethylene. The first stage of the more complete analysis consists of the removal of hydrocarbons higher in the series than butane and butylene, by cooling the gas in an ice and salt freezing mixture, and subsequently allowing the condensed liquid to warm up to 10° to eliminate dissolved gases. A known volume (5—6 litres) of the gas is then passed through alkaline mercuric cyanide to remove acetylene, through bromine to remove olefines, and

through a tube cooled to -76° (solid carbon dioxide and ether), wherein the propane and butane are condensed. The residual gas, and the mixture of propane and butane obtained by volatilising the condensed gas, are analysed by explosion with air or oxygen. The olefines are recovered from their bromides and determined by a slight modification of the method described by Manning, King, and Sinnatt (B., 1928, 217).

A. B. MANNING.

Position of the activated carbon process for the recovery of benzol from coke-oven gas and coal gas. E. REISEMANN (Brennstoff-Chem., 1930, 11, 226—229).—Some account is given of the Bayer process, and of the experiments carried out by the Gas Light and Coke Co. (cf. B., 1930, 42; Hollings and others, *ibid.*, 43).

A. B. MANNING.

Tower-absorption coefficients. III. Absorption of benzene by mineral oil. C. W. SIMMONS and J. D. LONG (Ind. Eng. Chem., 1930, 22, 718—721; cf. Cantelo and co-workers, B., 1927, 799).—The absorption by petroleum wash-oil of benzene from air as the inert carrier has been investigated. The rate of absorption varies linearly with the rate of flow of the absorbent; it decreases rapidly with an increase in flow ratio until a critical flow ratio is reached, after which it remains constant.

L. S. THEOBALD.

Artificial ageing of mineral oils. F. EVERS and R. SCHMIDT (Brennstoff-Chem., 1930, 11, 214—219; cf. B., 1928, 841).—The method of testing insulating and turbine oils previously described has been further developed. The oil (15.5 g.) is uniformly distributed over the catalyst (60 g. of silica impregnated with 1.59 g. of ferric oxide) contained in a glass tube, which is immersed in a water-bath at 100°; the tube is connected to an apparatus supplying electrolytic oxygen, and the oxygen absorbed by the oil is determined at intervals over a period of about 100 min. The current passing through the electrolytic cell is used to control and to measure the quantity of oxygen supplied to the reaction tube. The graph obtained by plotting the oxygen absorbed against time is only slightly curved, and may be replaced by a straight line without appreciable error; the slope of this line is taken as a measure of the rate of ageing ("ageing constant") of the oil. With time expressed in min. and oxygen absorbed as amp.-min., the values for the ageing constant of good insulating oils averages 0.13; it should not exceed 0.21. The ratio of ferric oxide to silica used in the catalyst is that giving approximately the maximum ageing constant. The same constant is obtained if the ferric oxide is replaced by the equivalent amount of copper oxide; lead oxide gives lower figures. The tar value (V_t) and the saponification value (V_s) of an oil are related to its "age" (A), i.e., its oxygen absorption as expressed above, by the equations $V_t = 1.0 \times 10^{-3} AM$, and $V_s = 0.56A$, where M is the mean mol. wt. of the oil.

A. B. MANNING.

Artificial ageing of mineral oils. III. F. EVERS and R. SCHMIDT (Wiss. Veröff. Siemens-Konz., 1930, 9, [1], 357—368; cf. B., 1928, 841).—Using the catalyst and procedure previously recommended for ageing tests on mineral oils, it is shown that the ageing curve

may be expressed by the equation $y = 2.3 \log x \cdot C + K$, where $C = 10.1$ and $K = -11.3$. From the ageing constant of a mineral oil the saponification value, acid value, and tendency to polymerisation during various stages in ageing can be calculated, and a close approximation made of the average life of the oil under electrical conditions.

A. R. POWELL.

Decolorisation of oils with mixed adsorbents.

J. K. CHOWDHURY and S. C. DAS (J. Indian Chem. Soc., 1930, 7, 379—400; cf. B., 1928, 324).—The mixed adsorbents studied—bauxite-silica, alumina-silica, and carbon-alumina—were prepared in each case by precipitating one component (silica or alumina) on the surface of the second, suspended in the liquid, followed by activation by roasting. The maximum decolorising power (for kerosene or ground-nut oil) with bauxite-silica mixtures corresponded with addition of 21.88% of silica, and with alumina-silica mixtures, 83.3% of silica. With activated carbon-alumina mixtures only a slight increase in efficiency was observed, but 30% of alumina may be added without loss of efficiency. The mixtures are readily regenerated by roasting, with unimpaired efficiency. The temperature of roasting (for regeneration) and the water content are interconnected, completely dehydrated mixtures having the lowest efficiency. The power of decolorisation attains a maximum with 6.33% of water for bauxite, 5.95% for the optimum bauxite-silica mixture, and 4.27% for the optimum alumina-silica mixture. The action of the mixed adsorbent is explained by assuming that the pores of one adsorbent are filled up with the second deposited on it, thus forming a large number of narrow capillaries, the two walls of which are oppositely charged. The water present may prevent the molecules of the adsorbed substance from coming into close contact with those of the adsorbent, whilst complete absence of water will affect the electrical properties and thus impair the adsorption capacity. J. R. I. HEPBURN.

Chemical examination of gasolines and light mineral oils. D. A. HOWES (J. Inst. Petroleum Tech., 1930, 16, 54—88).—The most important analytical methods that have from time to time been proposed are collected together, and these are now briefly described, examined, and criticised. The paper is divided into three parts, dealing with the determination of unsaturated, aromatic, and naphthene and paraffin hydrocarbons. A full bibliography is appended.

Motor-fuel volatility. II. Starting volatility.

G. G. BROWN, C. L. NICKOLS, and P. BIGBY (Ind. Eng. Chem., 1930, 22, 649—652; cf. B., 1930, 544).

III. Effective volatility under driving conditions.

J. E. MILLER and G. G. BROWN (*Ibid.*, 653—662).

IV. Relation between atmospheric temperature, fuel volatility, and engine performance. C. L. NICKOLS and G. G. BROWN (*Ibid.*, 662—671). **V. Vapour pressure and vapour lock.** E. A. CLARKE, H. B. COATS, and G. G. BROWN (*Ibid.*, 672—680).—II. Equilibrium volatility, as previously described (*loc. cit.*), is related to ease of starting as determined by actual engine tests.

III. Effective volatility under driving conditions is defined, and methods for its determination from the

equilibrium volatility or A.S.T.M. distillation data are described.

IV. Effective volatility is related to engine performance, and the A.S.T.M. distillation characteristics required for satisfactory performance under different conditions of atmospheric temperature are described.

V. The relation of vapour pressure to vapour lock is discussed, and fuel characteristics necessary to eliminate this trouble are suggested. L. S. THEOBALD.

Anti-knock fuels. L. EDELEANU and W. GROTE (Brennstoff-Chem., 1930, 11, 212—214).—The anti-knock value of a cracked spirit is greatly increased if the fraction boiling above 175° is extracted with liquid sulphur dioxide, and the extract, consisting principally of aromatic and unsaturated hydrocarbons, is recovered and added to the fraction boiling up to 175° (cf. U.S.P. 1,661,566; B., 1928, 326). Engine tests on such treated spirits show them to compare favourably in anti-knock value with straight-run spirits to which lead tetraethyl has been added. A. B. MANNING.

Detection and determination of carbon disulphide in fuels.

F. SPAUSTA (Petroleum, 1930, 26; Motoren-betrieb, 3, No. 7, 7—10).—The presence of as little as 0.05% of carbon disulphide in such fuels as benzene, benzol, etc. may be detected by means of alcoholic caustic potash or soda, the xanthate formed being determined by means of an aqueous ferric chloride solution acidified with sulphuric acid. Even 1 mg. of iron xanthate dissolved in the fuel imparts a red colour. 5 C.c. of the fuel and 3 c.c. of *N*-caustic potash solution (in 96% ethyl alcohol) are shaken vigorously for 2 min., then diluted to dissolve the xanthate, and ferric chloride solution acidified with sulphuric acid is added. In the presence of carbon disulphide a brownish-black precipitate is formed, becoming red on shaking with the fuel. A colorimetric method of determination is given. Homologues of ethyl alcohol give the same results. In place of the ferric chloride, salts of nickel or cobalt may be used, the xanthates of which are yellow; nickel salts are soluble only when benzol, alcohol, or acetone is present, and cobalt salts require the addition of ether. The "doctor test" is unsuitable for the detection of small amounts up to 0.2% of carbon disulphide, owing to the slow rate of precipitation of the lead sulphide.

W. S. E. CLARKE.

Properties of tar-petroleum asphalt mixtures.

P. SCHLÄPFER (Petroleum, 1929, 25, 1457—1464, 1585—1590).—Nine tars of varying characteristics have been blended in different proportions (10, 20, and 30%), with each of two petroleum asphalts. The physical properties of these tars and asphalts are tabulated and the more important characteristics of the mixtures, *e.g.*, viscosity and dropping point, are shown graphically in relation to varying temperatures and percentage of asphalt. Up to a 30% asphalt content the viscosity of the mixture is fundamentally a property of the original tar. In a second series of tests mixtures of asphalts and tars were made of 20, 40, 60, and 80% asphalt content. Irregularities in the curves for the 40% mixture suggest that disturbances occur. This is confirmed microscopically. Generally, tar-asphalt mixtures of less than

30% asphalt content were homogeneous under the microscope, with the carbon uniformly distributed, and did not change appreciably even after 1 year, but in individual cases poor ductility was shown, the carbon was flocculent, and emulsions were visible under the microscope. Mixtures containing 40% of asphalt showed, with few exceptions, a deposit of flocculent carbon and unevenly distributed emulsions. W. S. E. CLARKE.

Purification of normal paraffin hydrocarbons by chlorosulphonic acid treatment. A. F. SHEPARD and A. L. HENNE (Ind. Eng. Chem., 1930, 22, 356—357).—Aschan's method (B., 1898, 798) for the purification of normal paraffins has been applied. The decane fraction obtained from gasoline by fractionation was treated with chlorosulphonic acid, the mixture being stirred during the day and the used acid replaced by fresh every 3 or 4 days. After treatment for 4 weeks the material, on being fractionated, gave a fraction having d_{4}^{20} 0.7264, which compares well with values of 0.72686 and 0.7266 obtained by Simon and Krafft, respectively, on synthetic samples of decane. The authors state that this method of purification is superior to any other they have tried, and can be used to remove hydrocarbons with side chains from the normal paraffins. A. SHORE.

Direct oxidation of hydrocarbons by air. P. MONDAIN-MONVAL and B. QUANQUIN (Bull. Soc. Ind. Mulhouse, 1930, 96, 265—283; cf. B., 1928, 917).—Observations have been made of the changes of pressure and temperature which occurred when a mixture of a hydrocarbon and air was heated in a steel bomb. The bomb was of 700 c.c. capacity, and the initial pressure was 5.5 kg./cm.² Using petrol, no spontaneous ignition took place with less than 0.35 c.c. With mixtures containing between 0.35 and 0.8 c.c. a violent explosion accompanied by a marked rise in temperature occurred when the temperature reached 220—225°. With from 0.8 to 3.6 c.c. of petrol the sudden increase in pressure occurred at gradually falling temperatures, while the magnitude of the effect at first diminished, passed through a minimum, and thereafter again increased rapidly; also over this range carbon was deposited in the bomb. With 3.7 c.c. a remarkable discontinuity occurred in the phenomena observed; the pressure and temperature increase fell to a lower value, which was approximately constant for higher concentrations of the hydrocarbon, and the deposition of carbon no longer took place. This discontinuity corresponded also with a discontinuity in the composition of the combustion gases, and in the appearance of the flame in the bomb. In a second series of experiments the hydrocarbon-air mixtures were passed through a heated tube, at the ordinary pressure, and the oxidation products were examined. Interaction began at 160° and proceeded rapidly at 300°, with the production of carbon dioxide, alcohols, aldehydes (formaldehyde, acetaldehyde, butaldehyde, etc.), and acids. No true ignition of the mixture occurred even at 360°. It is concluded that the initial reaction in the bomb is of a similar nature, consisting of an exothermic reaction unaccompanied by flame; the temperature and pressure of the mixture then rise, and, if the concentration of the

hydrocarbon is within the requisite limits, ignition follows. A. B. MANNING.

Formation of methane from carbon monoxide and hydrogen by bacteria. R. LIESKE and E. HOFMANN (Brennstoff-Chem., 1930, 11, 208—212).—Bacteria have been found in the mud of ditches, in sewage sludge, etc. which bring about the reaction $\text{CO} + 3\text{H}_2 = \text{CH}_4 + \text{H}_2\text{O}$. They are Gram-positive, non-spore-forming bacilli, without cilia, identical with or closely related to the carbon dioxide-reducing bacteria described by Söhngen (B., 1910, 980). Cultures of these bacteria convert the carbon dioxide and carbon monoxide of water-gas or coal gas into methane, the yield of which amounts to over 90% of the theoretical. These results have been obtained from experiments carried out in closed vessels; it has not yet been possible to bring about a similar conversion of carbon monoxide into methane when a continuous current of the gas is passed over the culture. A. B. MANNING.

See also A., July, 851, **Activity of charcoal** (ALEXEEVSKI and MUSAKIN). 868, **Catalytic reactions at high pressures** (EGERTON). 869, **Reduction of carbon dioxide** (BAHR). 875, **Graphite and graphitic acid** (THIELE; HOFMANN and FRENZEL).

Determination of hydrogen in gases. AMBLER.—See VII.

PATENTS.

Treatment of coal [especially anthracite]. DELAWARE, LACKAWANNA, & WESTERN COAL CO., Assees. of (A) E. FARLEY and N. R. LANDON, (B) N. R. LANDON (U.S.P. 1,748,335 and 1,748,384, 25.2.30. Appl., [A, B] 25.2.29).—To restore the lustre and improve the appearance of anthracite or other coal which has become dull on storage, the material is treated with solutions of (A) oxalic or other acid, or (B) glycerin or dextrin, at the ordinary or at higher temperatures. A. B. MANNING.

Method of identifying fuel. H. S. MORK, Assr. to DELAWARE, LACKAWANNA, & WESTERN COAL CO. (U.S.P. 1,748,787, 25.2.30. Appl., 17.1.28).—Coal is coloured for purposes of identification by dissolving a basic dye in water, adding a clay which has the property of adsorbing the dye from solution, and spraying the coal with the suspension of coloured clay. A. B. MANNING.

Fuel briquettes. J. S. FORD and F. MURRAY (B.P. 329,437, 18.3.29. N.Z., 17.1.29).—Coal dust, or other carbonaceous material, is mixed with 5% of its weight of molasses, the mixture is formed into briquettes without using appreciable pressure, and the briquettes are heated at 500—700° for 10—20 min. in order to draw a portion of the binding material to the surface of the briquettes where it is carbonised to form a hard compact crust. A. B. MANNING.

Recovery of waste heat in the drying of brown coal and the like. TELEX APPARATEBAU-GES.M.B.H. (G.P. 462,779, 3.2.24).—A condensing system is provided wherein the heat in the steam evolved during drying is transferred to air, and the warm air is then used in the preliminary drying of the raw brown coal supplied to the plant. A. B. MANNING.

Heating coal and like furnaces. W. SCHAEFER and W. POHL, Assrs. to KERAMISCHE IND.-BEDARFS A.-G.

(U.S.P. 1,747,846, 18.2.30. Appl., 26.10.26. Ger., 13.2.25).—Separate currents of cold and hot air are introduced into the combustion gases supplied for heating the furnace at a point prior to their introduction into the furnace. The amount and temperature of each of the air currents are regulated in such a way that any desired length of flame and a uniform temperature distribution are attained within the heating zone. A. B. MANNING.

Apparatus for mixing air and pulverised coal applicable to other similar operations. G. H. ROBINSON (B.P. 329,483, 27.4.29).—The apparatus comprises a Venturi tube, through which the air is made to pass, and a device for spraying the pulverised fuel into this tube through an aperture therein at the point of minimum cross-section. The spraying device consists of a wire-brush rotating within a cylinder into which the powdered coal is fed from the storage bin.

A. B. MANNING.

Energy production and distribution in the working up of brown coal. P. SCHMIDT (G.P. 462,388, 14.11.25).—The electrical energy produced by the utilisation of the steam from a brown coal drying plant is used to produce hydrogen by electrolysis under pressure, and the hydrogen is then transported to the points of consumption by high-pressure distance transmission.

A. B. MANNING.

Coal-distillation and coke-production plant. BABCOCK & WILCOX, LTD., E. G. WEEKS, and W. A. RILEY (B.P. 329,441, 23.3.29).—The coal is delivered from a bunker to a preheater and thence to a retort provided with means whereby the rate of extraction is controlled by the movement of a float on the surface of the fuel bed (cf. B.P. 324,853; B., 1930, 357). The coke is discharged into a quenching device in the form of a closed container (cf. B.P. 324,852; B., 1930, 357), wherein the cooling of the coke is completed by the admission of a controlled quantity of air. The process proceeds automatically from the delivery of the coal to the bunker to the discharge of the quenched coke out of contact with air other than that intentionally supplied.

A. B. MANNING.

Carbonisation of bituminous materials. DEMAG A.-G. (G.P. 462,876, 3.9.24).—Bituminous or brown coal etc. is first converted into semi-coke by low-temperature carbonisation in known manner, and is then subjected to further carbonisation at above 600° in a special chamber. The heat required for the second stage of the process is provided by the combustion of the necessary quantity of the still hot semi-coke produced in the first stage.

A. B. MANNING.

Low-temperature distillation of bituminous coal and lignite. COMP. GÉN. DE DISTILLATION ET COKÉFACTION À BASSE TEMPERATURE ET MINIERE (INTERTRUST) SOC. ANON., ASSEES. OF INTERNAT. HOLDING DE DISTILLATION ET COKÉFACTION À BASSE TEMPERATURE ET MINIERE (HOLCOBAMI) SOC. ANON. (B.P. 329,542, 17.6.29. Belg., 15.3.29).—The material, in layers not greater than 8–10 cm. thick, is heated gradually until the outside temperature reaches 450°, and this temperature is maintained until that of the coolest part of the material reaches 350°. The temperature is then quickly raised to 500–550°. The process, which is most conveniently

carried out in a retort of the type described in B.P. 229,880 (B., 1925, 345) or B.P. 308,760 (B., 1929, 841), yields a hard, compact, and easily ignitable semi-coke, a low-temperature tar which has undergone no cracking, and a gas of high calorific value.

A. B. MANNING.

Retorts for the low-temperature distillation of carbonaceous material. CARLTON MAIN COLLIERY CO., LTD., R. ADDY, and S. GILL (B.P. 329,464, 11.4.29).—Each retort consists of two vertical, slightly coned, concentric, metal chambers, the coal being charged into the narrow annular space so formed. The inner chamber is movable vertically and is provided at its lower end with a projection which serves to close the lower end of the annular space during carbonisation. When carbonisation is complete the inner chamber is lowered to permit discharge of the coke. The outer surface of the inner chamber may be provided with vertical ribs which divide up the annular space into vertical compartments for receiving the coal. The wall of the inner chamber is perforated by upwardly directed slots for the passage of a portion of the distillates into the interior of the chamber and thence to the offtake. The retorts are mounted in a brickwork setting and adapted to be heated externally, or both externally and internally, by heating gases.

A. B. MANNING.

Gas producer. J. U. M. VOITEUR (B.P. 329,573, 30.7.29. Fr., 29.4.29).—A gas producer for use with moist fuels, particularly wood, operates with reversed draft, the air entering through apertures in the wall of the shaft at a suitable height above the grate, and the combustible gas being withdrawn through a conduit opening into the ash pit. The upper part of the fuel shaft is connected with a condenser by means of an elbow-coupling hinged so that it may be moved aside laterally to permit charging.

A. B. MANNING.

Production of gas of high calorific value, using bituminous coal in a gas producer. R. NÜBLING and R. MEZGER (G.P. 462,309, 6.5.25).—In a plant in which the waste heat is used for preheating the circulating gases and for superheating the steam in known manner, the "blow" period is replaced by a "half-water-gas" period by adding steam to the air. The heat produced by the combustion of this gas and used for preheating purposes is thereby increased; the ratio of carbonisation gas to water-gas, and therefore the calorific value of the resultant gas, are correspondingly increased.

A. B. MANNING.

Gas producer for wet brown coal. A. KADEN (G.P. 462,607, 5.4.25).—The producer is provided with a ring-shaped, internally and externally heated, carbonising and drying chamber, and with separate off-takes for the carbonisation and producer gases. Below the grate is a rotatable ash-tray which forms also a water seal. A vertical grate surrounds the gasification zone and connects the inner part of the shaft with the ring chamber. The producer gas off-take is formed by a central, telescopically adjustable tube.

A. B. MANNING.

Production of enriched water-gas. A. SCHWARZ, ASSR. to COAL & OIL PRODUCTS CORP. (U.S.P. 1,745,454, 4.2.30. Appl., 18.11.25).—The apparatus comprises two generators, an oil still and circulating pump, a

steam generator, and preheating devices for the oil and steam. The heat of the "blow" gases is used to distil the oil, the heavy unvaporised portion of which is introduced into the generator during the "run" in order to enrich the water-gas produced. The residual heat in the blow gases serves to generate the steam which is superheated by being passed through tubes in the furnace of the oil still. A. B. MANNING.

Purification of coal-distillation gas. GEWERKSCHAFT M. STINNES (B.P. 304,145, 14.1.29. Ger., 14.1.28).—Coal gas which is intended for long-distance transmission is compressed, charged with a naphthalene solvent, and then cooled to such a temperature that the solvent separates out again together with the naphthalene. The solvent may also extract traces of benzene from the gas, and the cooling may be carried to such a point that the gas is also dried. A. B. MANNING.

Purification of gases [e.g., coal gas] containing hydrogen sulphide. W. D'LENY, J. R. PARK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,381, 15.5.29).—The gases are scrubbed with an alkaline suspension of 0.02 g.-mol. of ferric hydroxide and 0.005 g.-mol. of nickel carbonate per litre of solution containing 0.15 g.-mol. of sodium carbonate. A. R. POWELL.

Removing soot from gases. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. DANTSIZEN (B.P. 306,043, 14.2.29. U.S., 14.2.28).—The soot is electrically precipitated and is conveyed by the circulation of a current of water to a quiescent pool in which the soot particles are separated by flotation and from which they are removed intermittently or continuously. In order to avoid the accumulation of soluble products in the water, a small fraction may be continuously withdrawn and replaced by fresh water. The flotation of the soot may be assisted by introducing oil in atomised form into the smoke to be treated. A. B. MANNING.

Separation of ammonia and benzol hydrocarbons from coal gases containing hydrogen sulphide, carbon dioxide, and oxygen. GES. F. KOHLENTCHNIK M.B.H., Assees. of F. HÄUSSER (G.P. 462,186, 8.7.21).—The corrosive components of the gas, ammonia, hydrogen sulphide, and carbon dioxide, are removed first, in apparatus constructed of chemically resistant material but possessing small mechanical strength. The separation of the benzol is carried out subsequently under pressure in mechanically stronger apparatus constructed of less chemically resistant material. Methods of carrying out the process are described. A. B. MANNING.

Distillation of wash oil. ZECH M. STINNES (G.P. 462,592, 18.3.24).—The wash oil is preheated in an apparatus offering as large a surface as possible to the oil, which is thereby freed from gases and the lower-boiling constituents. It is then further heated and distilled in the usual manner. Heat recovered from the vapours or from the hot oil is utilised in the preheating. The gases and vapours evolved are then fractionally condensed. The oil, freed from gases and low-boiling constituents, or fractions thereof, are washed in vessels serving also as coolers. A. B. MANNING.

Mine-gas detector. E. HECKERT (U.S.P. 1,746,425, 11.2.30. Appl., 16.10.24. Ger., 25.10.23).—The apparatus comprises a number of diffusion cells, each consisting of a pair of cell chambers separated by a porous division wall, a pressure gauge, and means for connecting one chamber of each pair to the pressure gauge while the gas to be tested is simultaneously admitted to the other chamber. The difference in pressure gives a measure of the firedamp in the mine air being tested. A. B. MANNING.

Apparatus for production of carbon black. J. MACHTOLF, Assr. to C. HOSTMANN-STEINBERG'SCHE FARBENFABR. G.M.B.H. (U.S.P. 1,746,003, 4.2.30. Appl., 9.12.26. Ger., 11.12.25).—An apparatus for splitting acetylene to produce carbon black consists of a number of pipes capable of withstanding high pressures, arranged horizontally one beside the other, and connected at their ends to form one continuous pipe. The acetylene is introduced into the apparatus under pressure, and is decomposed by means of an electric spark. The apparatus is preferably immersed in a cooling bath. A. B. MANNING.

Manufacture of products from ethylene and/or its homologues. H. D. ELKINGTON. From N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 331,186, 18.3.29).—The ethylene etc. is subjected to polymerisation treatment under pressure and at elevated temperatures in the absence of catalysts but in the presence of an inert liquid medium such as mineral or vegetable oils or products obtained therefrom which remain substantially unchanged under the conditions of the reaction. H. S. GARLICK.

Conversion of hydrocarbons into lighter hydrocarbons. A. LOSEY, P. GOTTLIEB, and L. L. HAUPT (B.P. 330,570, 11.3.29. U.S., 7.1.29).—A hydrocarbon distillate and a suitable conversion catalyst, e.g., a volatile metallic chloride, is held under superatmospheric pressure while heated by indirect contact with a flow of vaporised "bottoms" under a lower pressure, being fractionally condensed thereby. The resulting vapours are subjected to thermodynamic expansion, the uncondensed vapours are drawn off, and the condensate is returned for further conversion. The unvaporised portion of the original charge is recycled in heat-exchange relation with the separate flow of hydrocarbon vapours which are fractionally condensed thereby, the fractions being collected and withdrawn, the vapours drawn off and fractionally condensed, and the condensate recycled. The heaviest fraction formed by the loss of latent heat from the heating vapour is collected, withdrawn, and reheated, and the vapours are returned in fractionating relation with the hottest portion of the separate flow of hydrocarbon condensate, thereby forming further vapour consisting of a lighter series of hydrocarbons evolved from the high-temperature heating vapours. The "bottoms" formed by reheating the heaviest fraction is separately reheated to a higher temperature than that of the catalyst and the latent heat is employed to effect recuperation of the impaired catalyst and conversion of the original mixture. H. S. GARLICK.

Recovery of refined products from carbonaceous materials such as coal, tars, mineral oils, etc.

J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 329,331, 12.11.28. Cf. B.P. 328,997; B., 1930, 752).—The reaction products from the destructive hydrogenation of carbonaceous materials, or from the extraction under pressure of bituminous materials, are fractionated by passing them directly from the reaction chamber into several vertical columns wherein they are cooled by means of gases or liquids flowing countercurrent thereto. Thus the waste gases from the process may be used as the cooling agent by circulating them through tubes arranged within the fractionating columns. The fractionation may be effected without releasing the pressure on the reaction products. The heavier condensates may be returned to the reaction chamber for re-treatment. A. B. MANNING.

Catalytic hydrogenation of heavy hydrocarbons, tars, crude petroleum, etc. SOC. INTERNAT. DES PROC. PRUDHOMME-HOUDRY (F.P. 637,022, 26.10.26).—In order to prevent poisoning of the catalyst, and to remove any material deposited thereon, light hydrocarbon vapours, especially those of the lighter fractions of the hydrogenation product, are passed, periodically or continuously, through the hydrogenation chamber.

A. B. MANNING.

Production of hydrocarbons of low b.p. from coal, oil, etc. H. S. WAITE (B.P. 330,934, 20.2.29).—The volatile products from the distillation of coal are passed direct from the retorts into a chamber where they come in contact with absorbent material, such as coke, maintained at 400–550°. The coke is supported on travelling screens and is fed into and removed from the chamber at a regular rate. The yield of condensible hydrocarbons in the gases is increased. T. A. SMITH.

Transformation of petroleum hydrocarbons into gasoline. JENKINS PETROLEUM PROCESS Co. (B.P. 330,539, 8.3.29. U.S., 16.11.28).—A mixture of the hydrocarbons with an absorptive binder of ash-forming content is subjected to incipient cracking conditions and then transferred to a non-extraneously heated zone of reduced pressure where separation occurs into a readily convertible distillate vapour and a residuary compound in liquid form. This residuum is reheated under such conditions of temperature and pressure that when transferred to a further non-extraneously heated zone of suitably reduced pressure it separates into additional light vapours and leaves a liquid residue that on cooling solidifies to a dry, coal-like structure.

H. S. GARLICK.

Apparatus for cracking oils. W. S. HADAWAY, JUN., ASSR. to TEXAS Co. (U.S.P. 1,751,148, 18.3.30. Appl., 28.8.25).—The presence of free carbon in the oil in cracking stills is determined by the fall in electrical resistance of the oil. Electrodes are placed in a conduit through which the oil is being passed and the power absorbed is measured on a wattmeter. Increase in the power absorbed shows that it is necessary to remove the oil from the still if the deposition of carbon is to be prevented. Several such indicators placed in different parts of the plant enable the cracking operation to be carefully controlled. T. A. SMITH.

Simultaneously obtaining refined mineral oils and pure bitumen from crude mineral oils. N.V.

MIJNBOW-EN CULTUURMAATS. "BOETON" (B.P. 330,565, 9.3.29. Holl., 26.10.28).—Crude oil is treated with a concentrated solution of iron or zinc chloride (800 pts. of the salt to 200 pts. of water). The oil is separated and the unsaturated substances which remain in the salt solution are coagulated by agitation. The product, steamed or washed with boiling water, yields a "carbene"-free bitumen, 18% of the oil being so recovered. T. A. SMITH.

Manufacture of petroleum products of the type known as "white oils." STANDARD OIL DEVELOPMENT Co., Assees. of F. A. HOWARD (B.P. 308,297, 9.2.29. U.S., 21.3.28).—"Petrolatum liquidum" is prepared from hydrogenated crude oil. The oil is hydrogenated catalytically, if desired, at 360–455° and under 50–200 atm. A fraction of suitable viscosity (50–600 sec. Saybolt at 100° F.) is then treated with fuming sulphuric acid (10–50 vol.-%), washed, dried, and filtered through fuller's earth. T. A. SMITH.

Apparatus for refining used lubricating oil. W. D. HARRIS and R. V. AYCOCK (B.P. 331,290, 10.4.29).—Oil from a still is discharged below a perforated contact plate, supporting a layer of fuller's earth and a neutralising agent, and passes upwardly through the clarifying material. Reaction with the fuller's earth causes coagulation of a sludge which sinks through openings in the contact plate and rests on and is supported by a screen. The oil is filtered by opening a valve and allowing the oil to percolate through the bed of coagulated sludge. H. S. GARLICK.

Manufacture of motor fuels. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 330,593, 9.1.29).—Approx. 1–2 pts. of liquid hydrocarbons, b.p. up to 105°, recovered from natural or cracking gases is blended with 3–4 pts. of the heavy benzine, b.p. 100–180°, obtained by the destructive hydrogenation of carbonaceous materials. [Stat. ref.] H. S. GARLICK.

Manufacture of fuel mixtures. P. E. and F. A. WEBB (U.S.P. 1,746,172, 4.2.30. Appl., 7.5.28).—The fuel consists of a mixture of acetylene and pentane produced by passing the former through the liquid pentane and subsequently adjusting the mixture ratio by adding a further quantity of acetylene. Other volatile liquid paraffin hydrocarbons may be used in place of pentane.

A. B. MANNING.

Refrigeration of gases for recovery of gasoline. F. E. HOSMER (B.P. 309,121, 14.1.29. U.S., 5.4.28).—In systems in which gasoline is recovered from natural gas by refrigeration, the formation of ice from the moisture contained in the gas is prevented by bringing the gas in direct contact with cold brine, which removes the moisture and cools the gas. The gas is then further compressed and cooled to remove the gasoline. After removal of the gasoline the residual gas is passed through the weakened brine solution to remove water and reconcentrate the brine for further use. The traces of water which remain in the gas and are precipitated with the gasoline are prevented from freezing by the addition of a little brine. T. A. SMITH.

Drying or carbonising machine [for fuel agglomerates]. F. M. CROSSMAN (U.S.P. 1,769,491, 1.7.30.

Appl., 18.5.29. U.K., 24.5.28).—See B.P. 318,506; B., 1929, 968.

Manufacture of oil gas. K. N. WANNEBO (U.S.P. 1,770,563, 15.7.30. Appl., 26.3.26. Ger., 11.9.23).—See B.P. 269,711; B., 1927, 468.

Conversion of heavy mineral oils into lower-boiling products. W. G. LEAMON (U.S.P. 1,769,789, 1.7.30. Appl., 26.6.25).—See B.P. 317,868; B., 1929, 882.

Liquid fuel. A. LAURENT (U.S.P. 1,770,315, 8.7.30. Appl., 6.7.26. Belg., 21.11.25).—See F.P. 615,749; B., 1927, 835.

Clouderising coal dust for power purposes. C. H. VERITY (B.P. 331,221, 12.3.29).

Apparatus for making illuminating and heating gas [from volatile liquid fuels]. J. WHITEHART (U.S.P. 1,747,094, 11.2.30. Appl., 1.7.27).

Gas-analysis apparatus (B.P. 330,799).—See I. **Acetic acid from pyroligneous acid** (B.P. 330,026).—See III. **Treatment of non-fibrous materials** (B.P. 330,649).—See V. **Ammonium sulphate** (B.P. 330,945 and 330,947).—See VII. **Castor oil soluble in mineral oil** (B.P. 317,391).—See XII. **Stoving lacquers** (B.P. 329,954).—See XIII.

III.—ORGANIC INTERMEDIATES.

See A., July, 867—8, **Catalytic reactions at high pressures** (MORGAN; BONE; GREEN). 903, **Detection of some primary arylamines** (RUZICKA). 908, **Reaction of α -naphthol** (CARLETTI). 919, **Preparation of tri- and tetra-chloro-*p*-benzoquinones and *s*-trichloroaniline** (ERDELYI). **Synthesis of 1-methylantraquinones** (FAIRBOURNE and FOSTER). **Nitration of 2-hydroxy-3-methylantraquinone** (MITTER and PAL).

Direct oxidation of hydrocarbons by air. MONDAIN-MONVAL and QUANQUIN.—See II. **Insecticidal pyridine and pyrrolidine derivatives.** RICHARDSON and SHEPARD.—See XVI.

PATENTS.

Catalytic oxidation of organic compounds. SELDEN Co., Assees. of A. O. JAEGER (B.P. 315,854, 29.5.29. U.S., 21.7.28).—The "base-exchange" catalyst preparations of earlier patents are effective after lixiviation with acid, which removes part or all the basic constituents, but leaves the porous physical structure.

C. HOLLINS.

Manufacture of butadiene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,396, 22.2.29).— γ -Butylene glycol vapour is passed with steam over a hydrating catalyst, composed of an oxide or borate (or a compound furnishing the oxide), with or without activators such as copper sulphate or red phosphorus, at 300°. Alumina, *e.g.*, gives 65% conversion when 30 pts. of the glycol vapour are mixed with 70 pts. of steam.

C. HOLLINS.

Production of 1:3- $[\alpha\gamma]$ -butadiene. C. J. LEYES (B.P. 329,748, 20.3.29).—Vinyl bromide vapour is

passed through a bath of molten tin, stannous bromide and $\alpha\gamma$ -butadiene being withdrawn as formed.

C. HOLLINS.

Production of acetylene dichloride of low b.p. from acetylene and chlorine. I. G. FARBENIND. A.-G. (B.P. 310,964, 29.4.29. Austr., 5.5.28).—*trans*-s-Dichloroethylene, b.p. 48–51°, is the main product when acetylene and chlorine are led over active carbon below 100° (optimum 40°). A high velocity is preferred, and the excess of acetylene is adjusted so that the unused chlorine does not exceed 5% in the exit gases after removal of the products. The exit gas is re-circulated with fresh acetylene and chlorine, which are conveniently added as equimolecular mixture.

C. HOLLINS.

Extraction of acetic acid from pyroligneous acid. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (B.P. 330,026, 16.3.29. Belg., 25.1.29).—A single solvent is employed (*a*) to wash out tars, (*b*) to extract the acetic acid, the solvent being chosen so that the amount used in the first operation is only a small fraction of that used in the second; the amount of solvent to be recovered from the tar washing is thus minimised. Suitable solvents are amyl acetate, mesityl oxide, dipropyl ketone, cyclohexanones, or mixtures of these.

C. HOLLINS.

Manufacture of soluble wood ethers. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,011, 5.3.29).—A wood ether, obtained, *e.g.*, by heating with ethyl chloride under pressure a mixture of pine sawdust and 3 pts. of 50% soda lye, is made completely soluble in alcohol-benzene (1:1–9) by treatment with chlorine dioxide in aqueous or acetic acid solution. If water is used chalk may be added. The chlorine dioxide may be produced *in situ* from chloric acid by reduction with vanadous sulphate or electrolytically.

C. HOLLINS.

Manufacture of acetaldehyde [from acetylene]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,867, 12.6.29).—Acetylene and steam are passed at 300–400° over a tungsten catalyst, *e.g.*, silicotungstic acid on clay or ammonium tungstate ignited on clay or zinc oxide.

C. HOLLINS.

Manufacture of crotonaldehyde and its homologues. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (F.P. 637,517, 12.7.27. Belg., 14.7.26).—Acetaldehyde (*etc.*) is aldolised with alkali at 5°, and the product is neutralised with phosphoric acid and distilled; aqueous crotonaldehyde passes over at 84–85° in 92% yield on unrecovered acetaldehyde.

C. HOLLINS.

Manufacture of acetone from acetylene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,350, 13.4.29).—In the catalytic production of acetone from acetylene the gases emerging from the catalyst chamber (which may contain, *e.g.*, zinc oxide at 400°) are passed without cooling over a similar catalyst to convert acetic acid, acetaldehyde, and unconverted acetylene into acetone.

C. HOLLINS.

Manufacture of organic acyl halides. G. B. ELLIS. From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 329,721, 1.3.29).— α -Halogeno- and $\alpha\beta$ -dihalogeno-ethyl

esters, obtained, *e.g.*, by addition of hydrogen halide and halogen, respectively, to vinyl esters, are distilled slowly with an acid catalyst (0.01–0.02% of zinc chloride, sulphuric acid, phosphoric acid). From α -chloroethyl acetate there are obtained acetyl chloride and acetaldehyde; from α -chloroethyl chloroacetate, chloroacetyl chloride and acetaldehyde; from α -chloroethyl butyrate, butyryl chloride and acetaldehyde; from $\alpha\beta$ -dichloroethyl acetate, acetyl chloride and chloroacetaldehyde; from $\alpha\beta$ -dibromoethyl acetate, acetyl chloride and bromoacetaldehyde, etc.

C. HOLLINS.

Manufacture of pure benzoic acid. I. G. FARBENIND. A.-G. (B.P. [A] 329,375 and [B] 329,389, 18.2.29. [A] Addn. to B.P. 307,343; B., 1930, 603).—(A) Crude benzoic acid is reduced with nascent hydrogen (*e.g.*, with magnesium–aluminium and acetic acid), or is dissolved in benzene, xylene, etc. and shaken with aqueous bisulphite, or is melted under aqueous bisulphite in a stirring autoclave (1–2 atm.). Phthalic acid may be removed by dissolution in the calculated quantity of alkali. (B) The reduction may be omitted if the crude acid is free from naphthaquinone, in which case it is melted under water containing the requisite amount of sodium hydroxide or lime.

C. HOLLINS.

Manufacture of benzoylhydroperoxides [perbenzoic acid] etc. P. A. A. VAN DER BEEK and W. P. JORISSEN (Dutch P. 17,393, 17.2.26).—Benzaldehyde (etc.) in solution in acetone, benzene, chloroform, carbon tetrachloride, etc. is treated with oxygen or air in sunlight or other source of active rays.

C. HOLLINS.

Manufacture of carboxylic acid halides. I. G. FARBENIND. A.-G. (B.P. 308,666, 25.3.29. Ger., 23.3.28).—Methyl chloride is led with 8 vols. of carbon monoxide over a phosphoric or boric acid catalyst (sodium metaphosphate on pumice) at 700–800°; acetyl chloride is obtained in 10–12% yield. Other halogenated hydrocarbons behave similarly.

C. HOLLINS.

Manufacture of alkylene-substituted phenols. SCHERING-KAHLBAUM A.-G. (B.P. 304,727, 22.1.29. Ger., 25.1.28).—A dihydroxydiphenylmethane of the type $(\text{OH} \cdot \text{C}_6\text{H}_4)_2 \cdot \text{CR} \cdot \text{CHR}'$ loses a mol. of phenol when heated at 130–230° with a porous catalyst (tonsil, frankonite), yielding substituted hydroxystyrenes, $\text{OH} \cdot \text{C}_6\text{H}_4 \cdot \text{CR} \cdot \text{CR}'$. Thus the dihydroxydiaryl-methanes from acetone and phenol and *m*-cresol give, respectively, 4-hydroxyisopropenylbenzene and 3-hydroxy-4-isopropenyltoluene; the product from methyl ethyl ketone and phenol gives 4-hydroxyisobutenylbenzene. From such products perfumes, pharmaceuticals, and disinfectants may be derived.

C. HOLLINS.

Manufacture of hydrogenated amines. I. G. FARBENIND. A.-G. (B.P. 306,414, 18.2.29. Ger., 18.2.28).—A phenol or a hydrogenated phenol is mixed with ammonia or an amine and either heated at 180–190° with reduced nickel in an autoclave with hydrogen under pressure or passed in vapour form with hydrogen over nickel at 180–190°. The hydrogen is omitted when cyclohexanols are employed. The preparation of mono- and di-cyclohexylamine mixture is described.

C. HOLLINS.

Manufacture of amino-alkyl compounds. I. G. FARBENIND. A.-G. (B.P. 307,305, 4.3.29. Ger., 2.3.28).—Aromatic or heterocyclic magnesyl compounds are treated with amino- or alkylated amino-alkyl halides. β -Diethylaminoethylbenzene, b.p. 100°/10 mm., is obtained from magnesium phenyl bromide. The preparation of δ -diethylaminobutylbenzene, b.p. 132°/10 mm., 4- β -diethylaminoethylanisole, b.p. 140°/10 mm., 4-bromo-1- β -diethylaminobutylbenzene, b.p. 140°/10 mm., and 2-methyl-3- β -diethylaminoethylindole, b.p. 171°/2 mm., is described.

C. HOLLINS.

Manufacture of amino-substituted tertiary alcohols. I. G. FARBENIND. A.-G. (B.P. 307,307, 4.3.29. Ger., 2.3.28).—Esters of aromatic carboxylic acids, carrying as nuclear substituent an alkylated amino-alkylamino- or aminoalkoxyl group, are treated with a Grignard reagent (2 mols.) to give basic tertiary alcohols which form salts soluble in water to neutral solutions. Ethyl *N*- β -diethylaminoethyl-*p*-aminobenzoate, b.p. 171°/4 mm., reacts with magnesium phenyl bromide (2 mols.) to give 4- β -diethylaminoethylaminotriphenylcarbinol, b.p. 215°/2 mm. The preparation of 2- β -diethylaminoethoxyphenyldiethylcarbinol, b.p. 160–164°/3 mm., and 4 : 4'-diethoxy-4''- β -diethylaminoethoxytriphenylcarbinol, b.p. 245–249°/4 mm., from ethyl *o*- β -diethylaminoethoxybenzoate, b.p. 180–183°/10 mm., is also described.

C. HOLLINS.

Manufacture of amino-alcohols. I. G. FARBENIND. A.-G. (B.P. 307,304, 4.3.29. Ger., 2.3.28).—Secondary and tertiary alcohols containing as substituent an aromatic residue carrying an aminoalkyl or alkylated aminoalkyl group are obtained from corresponding ketones by the Grignard reaction. *N*-Methyl-*N*- β -diethylaminoethyl-*p*-aminobenzaldehyde reacts with magnesium *p*-anisyl bromide to give 4-methyl- β -diethylaminoethylamino-4'-methoxybenzhydrol, b.p. 240°/2 mm. Similar compounds are obtained from *N*- β -diethylaminoethyl-*p*-aminoacetophenone, b.p. 171°/2 mm., and magnesium *p*-phenetyl bromide (product, m.p. 101°, b.p. about 170°/2 mm.); from *o*- β -diethylaminoethoxybenzaldehyde, b.p. 152–155°/3 mm., and magnesium ethyl bromide (product, b.p. 142–145°/3 mm.); and from 2- β -diethylaminoethoxy-3-allylbenzaldehyde and magnesium ethyl bromide (product, b.p. 184–187°/5 mm.). The salts of the new bases give neutral solutions in water and have therapeutic properties.

C. HOLLINS.

Manufacture of naphthylaminocarboxylic acid derivatives [naphthylaminosalicylic acids]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 329,741, 13.3.29).—The Kolbe reaction is applied to 4- β -naphthylaminophenols. The preparation of 5- β -naphthylaminosalicylic acid, m.p. 175°, 6- β -naphthylamino-*m*-cresotic acid, 213–214°, and 5- β -naphthylamino-*o*-cresotic acid, m.p. 214–215°, is described.

C. HOLLINS.

Manufacture of *o*-hydroxycarboxylic acids of fluorene. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,305, 6.3.29).—2-Hydroxyfluorene is readily converted by the Kolbe reaction into 2-hydroxyfluorene-3-carboxylic acid, m.p. 256–260° (decomp.), and a smaller amount of the 1-carboxylic acid, m.p. 236–240°. These are separated by crystallisation from

alcohol. The 3-carboxylic acid gives an *o*-aniside, m.p. 157°. C. HOLLINS.

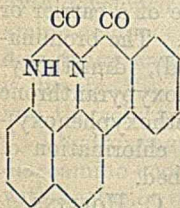
Manufacture of *o*-aminocarboxylic esters of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 314,028, 20.6.29. Ger., 21.6.28. Addn. to B.P. 267,164; B., 1928, 361).—Anthraquinone-1:2-*isooxazole* is heated with an alcohol or phenol in presence of an alkaline agent (potassium cyanide, sodium alkoxides). The *isooxazole* ring is opened and an ester of 1-aminoanthraquinone-2-carboxylic acid is produced. The methyl (m.p. 228°), ethyl (m.p. 198°), and phenyl (m.p. 198°) esters are described. C. HOLLINS.

Manufacture of β -anthraquinonecarboxylic acids and esters thereof. I. G. FARBENIND. A.-G. (B.P. 314,020, 13.6.29. Ger., 21.6.28).—2-Aldehydoanthraquinones react in presence of alkali cyanide with water to give leucoanthraquinone-2-carboxylic acids, or with alcohols or phenols to give the corresponding esters; air oxidation of the resulting vats yields anthraquinone-2-carboxylic acids or esters. The preparation of anthraquinone-2-carboxylic acid, m.p. 286–287°; its methyl and ethyl esters, m.p. 170° and 147°, respectively; methyl 1-iodoanthraquinone-2-carboxylate, m.p. 172–175°; methyl, ethyl, and phenyl 1-aminoanthraquinone-2-carboxylates, m.p. 228°, 198°, and 198°, respectively, is described. C. HOLLINS.

Dye intermediates [alkyl ethers of leuco-acylaminoanthraquinones]. J. THOMAS, D. A. W. FAIRWEATHER, and SCOTTISH DYES, LTD. (B.P. 330,215, 27.11.28).—An acylaminoanthraquinone is reduced with zinc and acetic acid and the leuco-compound is alkylated without isolation. The dimethyl ethers of leuco-derivatives of 1- and 2-acetamido- and 2-benzamidoanthraquinones, and of 1- and 3-chloro-2-acetamidoanthraquinones, are described. C. HOLLINS.

Condensation product of the perimidone series.

W. MIEG and R. HEIDENREICH, ASSS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,749,955, 11.3.30. Appl., 18.6.28. Ger., 16.12.25).—*N*-Benzoylperimidone is heated at 150–170° with sodium aluminium chloride or is boiled in carbon disulphide with aluminium chloride, to give a compound (annexed formula) or an isomeride. Similar products are obtained from the *m*-nitrobenzoyl and α -naphthoyl derivatives. C. HOLLINS.



Manufacture of indoles. IMPERIAL CHEM. INDUSTRIES, LTD., H. A. PIGGOTT, and E. H. RODD (B.P. 330,332, 26.3.29).—Acylated arylamines containing an *o*-methyl or -methylene group are cyclised to 2-substituted indoles by boiling with an alkali metal in an inert solvent (*e.g.*, diethylaniline or tetrahydronaphthalene); a copper catalyst (copper bronze) may be added. The preparation of 2-methylindole from acet-*o*-toluidide; 2-ethylindole, m.p. 35°, b.p. 160–170°/25 mm., from propion-*o*-toluidide; 2:5-dimethylindole, m.p. 114–115°, b.p. 188°/40 mm., from acet-*m*-4-xylylide; 2:7-dimethylindole, m.p. 35–37°, b.p. 146–148°/10 mm., from acet-*m*-5-xylylide; 2-phenylindole, m.p. 185°, from benz-*o*-toluidide; 2:2'-di-indolylmethane, b.p. 152–

153°/15 mm., from malondi-*o*-toluidide; and 2:3-dimethylindole, m.p. 104–105°, b.p. 188–196°/20 mm. (picrate, m.p. 153°), from *o*-ethylacetanilide, is described. C. HOLLINS.

Iodination of *o*-oxyquinoline-*ana*-[8-hydroxyquinoline-5-]sulphonic acid. UNION CHIM. BELGE Soc. ANON. (B.P. 329,888, 5.7.29. Belg., 7.6.29).—A mixture of aqueous hydrogen peroxide and sulphuric acid is added to a boiling aqueous solution of 8-hydroxyquinoline-5-sulphonic acid, potassium carbonate, and potassium iodide. The iodo-derivative crystallises out on cooling. C. HOLLINS.

Manufacture of mercaptobenzthiazoles. I. G. FARBENIND. A.-G. (B.P. 306,492, 21.2.29. Ger., 21.2.28).—An *o*-chloronitrobenzene is treated with hot aqueous or alcoholic sodium hydrogen sulphide; the mixture is then cooled, carbon disulphide is added, and reaction completed at 80–100°. The preparation of 2-thiolbenzthiazole and its 5-methyl (m.p. 170–172°), 5-nitro (m.p. 223°), and 5-chloro-derivatives, and of 2:5-dithiolbenzthiazole (from 1-chloro-2:4-dinitrobenzene), is described. C. HOLLINS.

Gas-generating chemical [lachrymators etc.]. B. C. GOSS, ASSR. to LAKE ERIE CHEM. CO. (U.S.P. 1,750,101, 11.3.30. Appl., 19.1.27).—Chloroacetophenone or other solid irritant is dissolved in methyl or ethyl bromoacetate or other liquid irritant, and the solution is used in shells, bombs, sprays, etc. C. HOLLINS.

Production of substances which may be used in the textile, leather, and allied industries as wetting, cleaning, foaming, and dispersion agents. H. T. BÖHME A.-G. (B.P. 308,824, 28.3.29. Ger., 30.3.28).—Alcohols corresponding with the higher fatty acids, *e.g.*, oleyl alcohol, $C_8H_{15}\cdot CH:CH\cdot[CH_2]_7\cdot CH_2\cdot OH$, are treated with concentrated or fuming sulphuric acid at 0°, whereby the alcohol group is sulphated and $H\cdot SO_4H$ is added at the double linking. C. HOLLINS.

Production of water-soluble substances having capillary - active properties [solvents, wetting, emulsifying, and frothing agents]. R. SAJITZ and E. THIEL (CHEM. FABR. POTT & CO.) (B.P. 329,622, 15.1.29. Ger., 28.11.28).—A secondary or tertiary alcohol (C_3 and higher, especially *tert.*-butyl alcohol) is converted by acid agents (concentrated sulphuric acid at 100°) into polymerised hydrocarbons, which are then vigorously sulphonated, *e.g.*, with chlorosulphonic acid in carbon tetrachloride, or with concentrated sulphuric acid in presence of acetic anhydride. C. HOLLINS.

Manufacture of wetting, cleansing, emulsifying, and dispersing agents and preparations containing the same. I. G. FARBENIND. A.-G. (B.P. 306,116, 29.11.28. Ger., 17.2.28).—Amides or esters (or ester amides) which are useful wetting agents etc. are made by heating together organic carboxylic or sulphonic acids having at least 8 carbon atoms with amines containing at least two hydroxylated radicals. Stearic acid, *e.g.*, is heated with "triethanolamine" at 160–180°, or oleic acid with the product from ethylenediamine and ethylene oxide. The wetting agents may be mixed with soaps, glues, gums, sulphite-cellulose waste liquor, organic solvents, etc. [Stat. ref.] C. HOLLINS.

Production of organic compounds containing oxygen [higher alcohols]. G. PATART, Assr. to DU PONT AMMONIA CORP. (U.S.P. 1,770,165, 8.7.30. Appl., 29.1.26. Fr., 7.2.25).—See F.P. 593,649; B., 1927, 347.

Production of esters. G. F. HORSLEY, Assr. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,770,779, 15.7.30. Appl., 9.8.28. U.K., 3.8.27).—See B.P. 301,523; B., 1929, 122.

Manufacture of [poly]sulphides of aromatic [carboxylic acid] compounds. R. EDER, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,769,423, 1.7.30. Appl., 17.5.28. Switz., 27.5.27).—See B.P. 291,100; B., 1929, 889.

Manufacture of [6-]chlorothymol. F. RASCHIG (U.S.P. 1,769,648, 1.7.30. Appl., 2.4.27. Ger., 29.4.26).—See B.P. 270,283; B., 1928, 36.

Products from ethylene and/or its homologues (B.P. 331,186).—See II. **Halogenated indanthrones (B.P. 330,217—8).** **Benzanthrone dyes (B.P. 311,661).** **Yellow azo-dye intermediate (B.P. 313,927).**—See IV. **Phosphoric acid esters (B.P. 330,228).**—See VII.

IV.—DYESTUFFS.

See A., July, 904, **Effect of substituents on shade of derivatives of stilbenesulphonic acids (WAHL and JONICA).** 906, **Azo dyes of the fluorene series (COURTOT).** **Substantive dyes derived from 2:7-diaminofluorene (NOVELLI).** **Colour and constitution (HODGSON).** 919, **Azo dyes from 2:6-diaceto-resorcinol (ALGAR and BOYLAN).** 924, **Condensations of ethyl carbamate, phenyl carbamate, and diphenyl carbamate with resorcinol (SEN and MUKHERJI).** **Methylisopropylthioindigoid dyes from *p*-cymene (HIXSON and CAUWENBERG).** 930, **Cyanine dyes from quaternary salts (HAMER).**

Disintegration. CHWALA.—See I.

PATENTS.

Manufacture of anthraquinone derivatives [wool dyes]. I. G. FARBENIND. A.-G. (B.P. 306,963, 27.2.29. Ger., 29.2.28).— α -Arylamino- β -cyanoanthraquinones are prepared by introduction of either group by suitable reactions, and are then sulphonated to give wool dyes. 3-Bromo-1-*p*-toluidinoanthraquinone is heated with cuprous cyanide in quinoline and the product is sulphonated with weak oleum for a wool red (bluer than the sulphonated starting material). 2-Bromo-1-amino-4-*p*-toluidinoanthraquinone similarly gives a greenish-blue wool dye. 4-Bromo-1-methylamino-2-cyanoanthraquinone is condensed with *p*-toluidine in presence of sodium acetate; sulphonation of the product yields a green wool dye of good shade in artificial light.

C. HOLLINS.

Manufacture of intermediate products of the benzanthrone series and dyes therefrom. Soc. CHEM. IND. IN BASLE (B.P. 311,661, 13.5.29. Switz., 12.5.28).—Benzanthrone is oxidised with manganic sulphate in 100% sulphuric acid at 60–65° to a dihydroxy-3:3'-dibenzanthronyl. This is converted by

alkaline fusion into dihydroxydibenzanthrone, which by alkylation yields grey-blue vat dyes.

C. HOLLINS.

Production of anthraquinone derivatives [halogenated indanthrones]. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 330,217, 27.11.28).—3:3'-Dichloro- and dibromo-indanthrones are halogenated; the new halogens enter α -positions and are reactive. 3:3'-Dichloroindanthrone is treated with chlorine in anhydrous nitrobenzene at 15° to give the 3:3':4:4'-tetrachloro- or the 3:3':4-trichloro-compound; 3:3'-dibromoindanthrone gives similar products. The hydroazine may first be converted into the azine and then halogenated; or the azine may be heated with concentrated hydrochloric acid at 160–165°, whereby the hydrogen chloride additive product is converted into 3:3':4-trichloroindanthrone. Chlorination of 3:3':6(7):6'(7')-tetrachloroindanthrone gives a green vat dye.

C. HOLLINS.

Production of anthraquinone derivatives [from halogenated indanthrones]. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 330,218, 28.11.28).— α -Halogenated indanthrones are made to react with phenols or with ammonia or primary or secondary amines, particularly with 1-aminoanthraquinone, methylamine, or aniline, with or without diluents, acid-absorbing agents, and/or copper or copper compounds. 3:3':4:4'-Tetrachloroindanthrone (cf. preceding abstract) is condensed with aniline for a blue-green vat dye, with 1-aminoanthraquinone (blue-green), and in the azine form with ammonia (blue-green). 4-Chloro- or 4-bromo-indanthrone with phenol gives a greenish-blue vat dye.

C. HOLLINS.

Manufacture of derivatives of pyranthrone. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,357, 17.12.28).—The ethers of hydroxypyranthrone (B.P. 325,250; B., 1930, 453) are halogenated, preferably in chlorosulphonic acid in presence of a carrier or with sulphuryl chloride in nitrobenzene. The bromination of ethoxypyranthrone (bluish-red), dimethoxypyranthrone (orange), β -chloroethoxypyranthrone (bluish-red), diphenoxy- and di-(*o*-methoxyphenoxy)-pyranthrone (reddish-brown), and the chlorination of methoxypyranthrone (brown) are described.

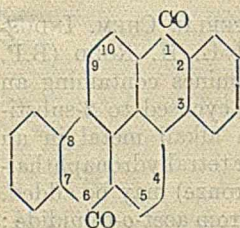
C. HOLLINS.

Manufacture of nitrogen-containing derivatives of the benzanthrone series. I. G. FARBENIND. A.-G.

(B.P. 307,926, 13.3.29. Ger., 16.3.28).—Dibenzpyrenequinones are nitrated to give products reducible to amino-compounds which are vat dyes. 2:3:7:8-Dibenzpyrene-1:6-quinone (annexed formula) or its dibromo-derivative gives a violet vat dye, 1:2:7:8-dibenzpyrene-3:6-quinone a greyish-blue (dinitro)-becoming deep brown with hypochlorite, and the methyl derivative (from 4-*p*-toluylbenzanthrone) an olive-green.

C. HOLLINS.

Production of dye derivatives [sulphuric leuco-esters] of the flavanthrone series and their use



R. S. BARNES, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 329,356, 11.12.28).—Red leuco-esters are obtained when flavanthrone or 3:3'-dichloroflavanthrone is treated with pyridine-sulphuric anhydride and copper in presence of an alkaline-reacting salt (sodium acetate).

C. HOLLINS.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. I. G. FARBENIND. A.-G., E. HOFFA, and E. THOMA (B.P. 329,960, 25.2.29. Addn. to B.P. 286,274; B., 1929, 675).—The process of the prior patent is extended to *p*-aminodiaryl amines in general. Examples are: *p*-aminophenyl- β -naphthylamine \rightarrow 2:3-hydroxynaphthoic anilide (navy-blue); methyl 3-amino-6- β -naphthylaminobenzoate \rightarrow *p*-chloroanilide (reddish-dark blue); 3-chloro- or 3:5-dichloro-4-aminophenyl- β -naphthylamine \rightarrow *p*-chloroanilide (dark blue); 4-amino-4'-phenoxydiphenylamine \rightarrow *o*-toluidide (indigo-blue), etc. 4-Aminodiphenylamines containing a cyano-, nitro-, or acylamino-group in 4'-position, and 2:3-hydroxynaphthoic anilide, *o*-toluidide, *o*- and *p*-anisidides, *p*-chloroanilide, and 4-chloro-*o*-toluidide are specially claimed as components.

C. HOLLINS.

Manufacture of azo dyes [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 306,415, 18.2.29. Ger., 18.2.28).—Diazo compounds of the benzene series containing at least one negative group are coupled in substance or on the fibre with a 2:3-hydroxynaphthoic 4-alkoxy- α -naphthylamide. The 4-methoxy- α -naphthylamide is coupled, *e.g.*, with diazotised 5-nitro-*o*-anisidine (garnet) or 4-chloro-*o*-toluidine (claret).

C. HOLLINS.

Manufacture of azo dyes [ice colours etc.]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 329,353, 16.11.28).—Diazoamino-compounds obtainable by the process of B.P. 324,041 (B., 1930, 364) are coupled in substance or on the fibre with suitable coupling components, the diazoamino-compound being split or the dyes developed by means of acid reagents. The diazoamino-compound derived from 4-chloro-*o*-toluidine (by diazotising and coupling with 4-sulphoanthranilic acid) is dissolved in water, acetic acid and alum are added, and the solution is used for coupling on the fibre with 2:3-hydroxynaphthoic β -naphthylamide for red. The diazoamino-compound from 4-chloro-*o*-anisidine is mixed with a solution of the *o*-anisidine in the calculated amount of alkali, and the dry mixture after evaporation is made into a printing paste; the printed colour is steamed and developed with dilute formic or acetic acid. The diazoamino-compound may be pasted with hot water containing thiodiglycol ($\beta\beta$ -dihydroxydiethyl sulphide) and alkali and thickening agent added to form the printing paste; the print is steamed or hung, and developed in dilute acetic, formic, or oxalic acid. Amongst the coupling components mentioned are: 2:3-hydroxynaphthoic α - and β -naphthylamides, *o*-anisidide, *o*-toluidide, *o*-phenetide, 2:5-dimethoxyanilide; 2-hydroxycarbazole-*o*-carboxylic *o*-anisidide, *o*-toluidide, acetoacetic toluidide; phenyl 4-hydroxy- α -naphthyl ketone; 2-hydroxy- β -naphthacarbazole; acylamino-naphthols; etc.

C. HOLLINS.

Manufacture of azo dyes and their application to the dyeing of regenerated cellulose materials.

IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 329,961, 25.2.29).—Level shades on viscose are obtained with disazo dyes of the type: non-phenolic *m*-nitro- or *m*-acylamino-arylamine \rightarrow B, reduced or hydrolysed, \rightarrow C, in which B is a phenol or naphthol, with or without carboxyl or sulphonic substituents, or an *N*-arylamino-naphtholsulphonic acid, whilst C is a *m*-phenylenediamine or an *N*-aryl- or *N*-aroyl-2:5- or -2:8-aminonaphtholsulphonic acid, or a 1:8- or 1:5:7-aminonaphtholsulphonic acid; the *N*-substituents must be free from sulphonic groups. Examples are: *m*-nitroaniline \rightarrow salicylic acid, reduced \rightarrow phenyl- γ -acid (brown); 4-nitro-*o*-anisidine \rightarrow phenyl- γ -acid, reduced \rightarrow H-acid (blue); *m*-aminoacetanilide \rightarrow salicylic acid, hydrolysed, \rightarrow benzoyl- γ -acid (orange); *m*-nitroaniline \rightarrow phenyl- γ -acid, reduced, \rightarrow *m*-phenylenediamine (yellowish-brown).

C. HOLLINS.

Manufacture of yellow monoazo dyes [for chrome leather], material dyed therewith, and an intermediate product therefor. I. G. FARBENIND. A.-G. (B.P. 313,927, 19.6.29. Ger., 19.6.28).—2-Chloro-*p*-toluidine-6-sulphonic acid, obtained by sulphonation and reduction of *o*-chloro-*p*-nitrotoluene, is diazotised and coupled with resorcinol (1 mol.) to give an intense orange-yellow dye for chrome leather, equal to Phosphine in purity of shade. Substituted resorcinols may be used.

C. HOLLINS.

Manufacture of azo dyes containing chromium. I. G. FARBENIND. A.-G. (B.P. 306,843, 25.2.29. Ger., 25.2.28).—The azo dyes of B.P. 245,765 (B., 1926, 734), containing as coupling components pyrazolones derived from aminoarylsulphonylsalicylic acids, are boiled with chromium formate (etc.). Examples are *o*-chloro-*p*-toluidine or metanilic acid \rightarrow phenylmethylpyrazolone from 2-amino-4'-hydroxy-4-sulphodiphenylsulphone-3'-carboxylic acid (greenish-yellow).

C. HOLLINS.

Manufacture of sulphide dyes. IMPERIAL CHEM. INDUSTRIES, LTD., and M. WYLER (B.P. 329,716, 26.2.29).—Aceto-*p*-xylylide is nitrated with mixed acid, and the nitro-compound, or the monoacetyldiamino-*p*-xylene, m.p. 141–142°, obtained on reduction, is heated with benzidine and sulphur to give an orange sulphide dye.

C. HOLLINS.

Manufacture of vat dyes [from naphthalene-1:4:5:8-tetracarboxylic dianhydride and *o*-diamines]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,364, 19.1.29).—The red vat dye from naphthalene-1:4:5:8-tetracarboxylic dianhydride and *o*-phenylenediamine is converted by acid oxidants (chromic acid; permanganate and sulphuric acid) into bright orange vat dyes.

C. HOLLINS.

Manufacture of vat dyes of the violanthrone series. H. SIEBENBÜRGER, ASSI. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,770,815, 15.7.30. Appl., 21.7.25. Switz., 9.8.24).—See B.P. 238,225; B., 1925, 876.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of small quantities of copper in materials [fabrics]. P. KLUCKOW and SIEBNER (Kautschuk, 1930, 6, 161–162).—The area of 10–20 g. of the fabric is measured in sq. cm. The material is then ashed, treated with 10 c.c. of nitric acid (*d* 1.4),

and evaporated with 5 c.c. of sulphuric acid until fumes of the latter are evolved. The treatment with nitric and sulphuric acids is repeated once or twice, and the solution containing 3–5 c.c. of concentrated sulphuric acid per 100 c.c. is precipitated with hydrogen sulphide. The precipitated copper sulphide is filtered off, care being taken to exclude air as far as possible. The precipitate is washed with as little water as possible into a colorimetric tube, and after warming with a few drops of nitric acid and addition of excess of ammonia, and standardisation of the volume, the copper is determined colorimetrically by comparison with a solution of known copper content. The suggested permissible maximum for fabrics intended to be proofed with rubber is 0.002 g. Cu/m.² D. F. TWISS.

Mechanism of the plasticising of natural cellulose fibres. K. HESS (Z. angew. Chem., 1930, 43, 471–481).—The behaviour of natural cellulose fibres from various sources when subjected to the action of cuprammine solutions has been followed röntgenographically. The results indicate that, besides diffusion phenomena, two compounds are formed between the cellulose and the copper ammine which subsequently undergo hydration to an extent which depends on the alkali concentration. During the latter process the fibres assume a spiral structure, the micelles lose their regular orientation, and the fibres swell by osmosis and become plastic and elastic. Similar tests made on alkali-cellulose in carbon disulphide appear to indicate that a compound is formed, but the reaction is much more complex than in the case of cuprammine solutions. Cellulose acetate and nitrate in various organic solvents, e.g., chloroform-methyl alcohol, cyclohexanone, camphor, give röntgenograms which show lines apparently due to the formation of complexes, the presence of which causes mechanical changes to take place in the micelles of the fibre structure; liquid is drawn into the fibre and serves as a lubricant allowing the micelle series to slip over one another, thus producing the characteristic plastic properties.

A. R. POWELL.

Bamboo. I. Fine structure of the bamboo fibre. K. SISIDO (J. Cellulose Inst., Tokyo, 1930, 6, 148–150).—A theoretical discussion of the results of previous workers.

Composition of *Cassia siamea*, L. Y. SHINODA (J. Cellulose Inst., Tokyo, 1930, 6, 155–156).—The following percentage composition has been found. Moisture content 11.2, alcohol-benzene extract 18.2, lignin (König) 37.3 (on the extracted, dry product), ash content 0.3, impure cellulose content 40.3, hydrolysis value (Ost) 92.8%, and pentosan content (Tollens-Krüger) 15.6. Before chlorination the wood meal was steeped in 10% sodium hydroxide solution for 48 hrs. and its cellulose content redetermined, when the value 33.8%, of purity 100%, was found. Xylose and mannose were found in the hydrolysis liquor, but no galactose was present.

B. P. RIDGE.

Factors during spinning which influence the physical properties of rayon. P. C. SCHERER, JUN., and R. E. HUSSEY (Ind. Eng. Chem., 1930, 22, 594–596).—Complete regeneration has been attained on the spool by the adhering liquor at a relatively

low temperature. The effects of time of contact in the acid bath and of the ripeness of viscose on the properties of rayon are recorded. Complete regeneration by control of the time of contact with the bath is desirable in existing processes. L. S. THEOBALD.

Removal of hemicelluloses from wood by sodium hydroxide. O. HORN (Cellulosechem., 1930, 11, 151–152).—Beech-wood meal, previously extracted with 1:1 benzene-alcohol, is treated 4 times for 36–48 hrs. with 5% sodium hydroxide solution. Determinations of the lignin and methoxyl content show that a portion of the lignin together with the hemicellulose have been removed during treatment with the soda. (Cf. Friedrich and Diwald, B., 1925, 151.)

T. H. MORTON.

Determination of the acetate content of cellulose acetate. D. KRUEGER (Farben-Ztg., 1930, 35, 2032–2033).—The methods which have been proposed for this determination can be divided into two classes involving acid and alkaline hydrolysis. Accurate determinations can be achieved only by choice of a method whereby adsorption of the hydrolysing agent and decomposition of the cellulose are kept at a minimum. The methods advocated involve the careful standardisation of the conditions of hydrolysis.

J. O. CUTTER.

Nitrates of cellulose benzoate. M. SENDO and J. KONDO (J. Cellulose Inst., Tokyo, 1930, 6, 150–155).—Cellulose monobenzoate was treated at 18–20° with mixed acids of the following percentage composition in, respectively, sulphuric, nitric, nitrous acid, and water: (1) 71.14, 20.81, 0.13, 7.92; (2) 65.6, 18.1, 0.15, 16.15; and (3) 59.58, 21.39, 0.23, 18.8, when cellulose dinitrate mononitrobenzoate [$C_6H_7O_2(NO_3)_2 \cdot CO_2 \cdot C_6H_4 \cdot NO_2$] was obtained by means of acids (1) and (2) and the mononitrate [$C_6H_8O_3(NO_3) \cdot CO_2 \cdot C_6H_4 \cdot NO_2$] by acid (3). The explosive properties of these nitrates are similar to those of the normal cellulose nitrates, but, compared with equally nitrated normal cellulose, these mixed esters are more stable, less hygroscopic, less soluble in ether-alcohol, and burn at a slower rate.

B. P. RIDGE.

Cellulose oxalates. G. VON FRANK and W. CARO (Ber., 1930, 63, 1532–1543).—Alkyl cellulose oxalates are readily prepared by the action of alkoxalyl chlorides on cellulose (paper, ramie fibre, or Girard's hydrocellulose) in presence of pyridine and nitrobenzene. Provided that the degree of esterification is uniform, increase in the weight of the alkyl group is accompanied by diminution in density, increase in solubility in non-polar solvents, and decrease in polar media, diminution in softening temperature, increase in plasticity of films, and decrease in their tenacity. The products obtained are mixtures of the di- and tri-esters. When hydrolysed they yield hydrocellulose. The following substances are prepared by the action of oxalyl chloride, purified from phosphorus trichloride by distillation, on the requisite alcohol; in the case of solid alcohols, a solution in chloroform is used: allyloxalyl chloride, b.p. 50–51°/12 mm.; cyclohexoxalyl chloride, b.p. 120–122°/34 mm.; octoxalyl chloride, b.p. 104–106°/9 mm.; cetoxalyl chloride, b.p. 155–160°/0.01 mm.; nitrobenzoyloxalyl chloride, b.p. 183°/11–12 mm.

H. WREN.

Beating of [paper] pulp. XI. A characteristic of vegetable fibres as papermaking materials. M. NAKANO (J. Cellulose Inst., Tokyo, 1930, 6, 144—147; cf. B., 1930, 608).—Wool, silk, asbestos, and vegetable fibres were beaten, and their paper-forming properties compared. Wool cannot be split longitudinally, but is reduced to short lengths showing no fibrillation, from which it is impossible to form a sheet. Silk and asbestos split up into fibrils, but the sheets formed from the products are weak owing to the slippage of the fibres. It follows, therefore, that fibrillation is not necessarily accompanied by the development of the adhesive properties which are essential for the production of satisfactory sheets. Only vegetable fibres have these characteristic properties, which are due to the colloidal state of the surface of the fibrils, and their felting capacity is of secondary importance.

B. P. RIDGE.

Distribution of loading in paper. H. SCHILDE (Papier-Fabr., 1930, 28, 409—415, 423—427, 439—444).—The factors controlling the concentration of loading material in a sheet of paper, particularly the change in concentration from the "top-side" to the "under-side," have been investigated. It is found that the particles of loading do not fall to the under-side according to the usual laws, but are affected by the state of the fibres, the substance of the sheet, the speed of the paper-machine, and the mode of removal of water by mechanical means from the wire-part. The amount of loading lost through the wire almost invariably exceeds that retained in the sheet, and the loss is increased at high machine speeds and by the use of vacuum couch rolls. No rules are formulated, but the effects of the various factors are illustrated by a series of solid diagrams.

T. T. POTTS.

Adhesives and adhesion. LEE.—See I. **Conversion of cellulose into sugar.** LÜERS.—See XVII.

See also A., July, 1940, **Silk fibroin** (GOLDSCHMIDT and STRAUSS). 968, **X-Ray analysis of cotton fibres** (CLARK and others). **Destruction of coniferous wood by the larva of the common beetle** (FALCK).

PATENTS.

Treatment of coconut fibre. E. ELÖD (B.P. 308,720, 15.3.29. Ger., 27.3.28).—The fibres are softened without loss of strength and rendered suitable for spinning if after removal of the embedding material they are digested for about 40 min. at 120—150° with dilute solutions of alkalis. After washing and drying, the fibres may be treated with hygroscopic agents, oils, etc.

D. J. NORMAN.

Treatment of threads for receiving sound records. W. F. CROLL (B.P. 330,679, 3.4.29).—Suitable thread, e.g., artificial horse hair, cotton, silk, or soft copper wire coated with cellulose, is impregnated with a compound containing water, glycerin, soap, and wax and is then passed through dies of successively varying diameter.

D. J. NORMAN.

Preparation of artificial silk. COMP. DE PROD. CHIM. ET ELECTROMÉTALL. ALAIS, FROGES, ET CAMARGUE (B.P. 305,468, 29.1.29. Fr., 4.2.28).—Artificial filaments are made from products obtained by condensing

urea or its derivatives and/or cyanamide or its derivatives with formaldehyde, the condensation being conducted in an aqueous medium until the product, when poured into water, neither dissolves nor flocculates, but collects into a homogeneous viscous mass. The solution is simultaneously or subsequently concentrated until it has a viscosity suitable for spinning. The condensation reaction is better controlled if carried out in neutral solution; when condensation has reached the desired stage the solution should be stabilised before concentration by adding an alkali or alkaline-reacting salt such as an acetate or phosphate. A suitable coagulating bath comprises an acidified saturated solution of, e.g., magnesium, sodium, or aluminium sulphate.

D. J. NORMAN.

Producing [from animal proteins] solutions adapted to be spun. HEBERLEIN & Co. A.-G. (B.P. 306,464, 20.2.29. Ger., 20.2.28).—Animal proteins of sparing solubility and high mol. wt., e.g., horn, skin, flesh, waste wool, or silk, are made to swell by treatment with hot water, if necessary under pressure, or with an acid solution which does not cause degradation of the protein, and are simultaneously or subsequently dissolved in liquid phenols or their homologues or derivatives.

F. R. ENNOS.

Preventing the evolution of disagreeable smelling gases in the viscose artificial silk manufacture.

BRIT. ENKA ARTIFICIAL SILK Co., LTD. (B.P. 316,971, 20.7.29. Holl., 7.8.28).—A small quantity of alkali nitrite is added to the viscose or to the lye used for dissolving the cellulose xanthate, and the nitrous vapours formed therefrom during spinning are mixed in the air-discharge pipes of the spinning room with oxides of nitrogen from another source, so that the noxious gases are oxidised.

F. R. ENNOS.

Manufacture of threads or filaments of cellulose derivatives. BRIT. CELANESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 331,229, 20.12.28).—The extruded filaments of organic derivatives of cellulose while

traversing a countercurrent of evaporative medium in the spinning cell are subjected to a controlled and uniform amount of stretch by passing around two or more rollers, the first receiving the filaments while they are still plastic and contain 20—40% of residual solvent, and the subsequent rollers drawing the filaments at a greater speed than the first.

F. R. ENNOS.

Production of artificial materials from organic esters of cellulose. BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. SOWTER (B.P. 330,950, 18.1. and 12.3.29).—Threads of the cellulose derivative, while still

in a swollen condition, are partially hydrolysed by passing through an aqueous or alcoholic solution of caustic alkali or ammonia; the desired swollen condition is produced in the case of the wet-spinning process by incorporation of a high-boiling solvent with the spinning solution, the coagulating bath, or a subsequent treatment bath, and in the case of the dry-spinning process by use of a high-boiling solvent in the spinning solution.

F. R. ENNOS.

Manufacture of artificial silk from viscose. W. W. TRIGGS. FROM SPINNSTOFFFABR. ZEHLFENDORF

GES.M.B.H. (B.P. 331,097, 28.6.29).—The thread is drawn from the spinning nozzle by means of a roller and is thence wound on to the bobbin, the circumferential velocity of the roller being greater than that of the bobbin, so that uniform shrinking and structure of the thread is ensured. F. R. ENNOS.

Manufacture of opaque films. NON-INFLAMMABLE FILM Co., LTD., and D. J. P. PHILLIPS (B.P. 331,214, 25.3.29).—Two films of regenerated cellulose or cellulose esters or ethers are superimposed, the one containing finely-divided metallic powder, *e.g.*, gold or aluminium, and the other ordinary fillers with or without pigments, *e.g.*, zinc oxide. F. R. ENNOS.

Manufacture of viscose products. I. G. FARBENIND. A.-G. (B.P. 307,848, 14.3.29. Ger., 14.3.28).—In order to remove heavy-metal (copper or iron) compounds, viscose products, after coagulation and washing, are treated with oxalic, lactic, tartaric, or citric acids or their salts. F. R. ENNOS.

Manufacture of soluble cellulose esters and ethers. I. G. FARBENIND. A.-G. (B.P. 305,947, 11.2.29. Ger., 11.2.28. Addn. to B.P. 283,181 and 297,766; B., 1928, 637; 1929, 51).—Cellulose is heated, above or below 100° and in the presence of a base such as pyridine, with a number of higher fatty acid halides, either in admixture or successively, which may also be replaced wholly or in part by a halide of a cyclic-substituted fatty acid (phenylacetic acid) or of an alicyclic carboxylic acid, *e.g.*, cyclohexanecarboxylic acid; where esterification takes place below 100°, the reaction mixture is afterwards heated above 100° until the desired solubility of the cellulose ester is attained. F. R. ENNOS.

Manufacture of readily soluble acylcelluloses yielding clear solutions. I. G. FARBENIND. A.-G. (B.P. 305,674, 8.2.29. Ger., 9.2.28).—Cellulose is acetylated below 50° in the presence of a relatively small amount of catalyst (sulphuric acid, less than 2% by wt. of the cellulose), part of the fatty acid normally present being replaced by monochloroacetic acid as a diluent. F. R. ENNOS.

Manufacture of finely-comminuted masses from cellulose derivatives. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,957, 16.3.29).—Cellulose derivatives are subjected to mechanical tearing by friction by repeated passage through a friction roller, in the presence of a resin and/or a non-solvent moistening agent (water, alcohol, hydrocarbons) which may contain small amounts of gelatinising agents insufficient to dissolve the cellulose derivatives, and the mass so obtained is afterwards ground in a ball- or hopper-mill. Addition may also be made during either process of dispersing agents, protective colloids, or emulsifying agents, together with dyes and fillers which have no chemical action on the cellulose derivatives. F. R. ENNOS.

Manufacture of artificial sponges [from viscose]. I. G. FARBENIND. A.-G., Assees of R. SKUTEZKY (B.P. 308,838, 28.3.29. Austr., 30.3.28).—Viscose, to which about 10% of its weight of fibrous material (cotton, hemp, flax, etc.) may be added if desired, is mixed with pore-forming fillers in the form of neutral salts

with or without water of crystallisation, *e.g.*, sodium sulphate, and salts reacting with alkali (acid or ammonium salts), moulded to the required shape, heated for $\frac{1}{2}$ —3 hrs. at 180° under pressure in a moist atmosphere, and washed with hot water. F. R. ENNOS.

Controlling the moisture content of paper. F. E. P. KLAGES (B.P. 330,596, 8.3.29).—Air is continuously withdrawn from the surface of the partly dried paper, preferably at the beginning of the last nest of dryers, and is passed over a wet-bulb thermostat which controls the steam supply to the drying cylinders. D. J. NORMAN.

Manufacture of pulp [boards]. S. D. WELLS, ASSR. to PAPER MILL LABORATORIES, INC. (U.S.P. 1,746,451, 11.2.30. Appl., 7.7.26).—Compact and waterproof products are obtained from cereal straws or other fibrous vegetable material by digesting the raw material with, *e.g.*, lime and then treating the unwashed or partly-washed pulp in a beating engine with acids or acid salts to neutralise the cooking agent, at the same time converting it, if desired, into a filler, *e.g.*, calcium sulphate, and to coagulate colloidal organic matter. When a waterproof product is required aluminium sulphate should be used to complete the acidification of the mass. Further quantities of colloidal matter may be coagulated if, after the liquor has been acidified, it is again rendered slightly alkaline. D. J. NORMAN.

[Rubber]-coated paper. G. J. ESSELEN, JUN., and R. P. ROSE, ASSRS. to GEN. RUBBER Co. (U.S.P. 1,746,888, 11.2.30. Appl., 8.4.25).—A coating mixture comprises rubber latex, a filler (clay), and a resinous adhesive (shellac), which has been dispersed in an aqueous solution of, *e.g.*, borax. Preferably the amount of rubber solids, together with that of any other binders present, should exceed 12% of the weight of the filler. Vulcanising agents may be added, and vulcanisation effected at below 93°. D. J. NORMAN.

Treatment of artificial silk. B. BORZYKOWSKI (U.S.P. 1,770,729, 15.7.30. Appl., 29.10.28. Ger., 12.3.27).—See B.P. 287,073; B., 1929, 677.

[Sliver]-drying apparatus. W. PRINCE-SMITH and D. WATERHOUSE (B.P. 331,403, 15.7.29).

Production of pulp [from wood or other fibrous material]. (SIR) G. C. MARKS. From BAUER BROS. Co. (B.P. 331,567, 5.3.29).

Concentration of paper pulp (B.P. 307,300).—See I. Soluble wood ethers (B.P. 330,011).—See III. Laminated glass (B.P. 306,891, 324,898, 325,048, and 331,030). Safety glass (B.P. 315,667).—See VIII. Celluloid from scrap films (B.P. 331,006).—See XXI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Plant for bleaching. R. WEISS (Sealed Notes Nos. 1723 and 1730, 23.3. and 2.4.07. Bull. Soc. Ind. Mulhouse, 1930, 96, 284—286). Report by J. LÉONHART (*Ibid.*, 286—287).—The apparatus comprises two boilers, a circulating pump, a tubular heater, and a compensating vessel. The bleaching liquid is pumped through the heater to the bottom of one of the boilers, and up

through the textiles therein; it passes then through the compensating vessel, wherein it is freed from air, to the top of the second boiler, and is sprayed on to the textiles therein; from the bottom of the second boiler it returns to the pump. The direction of circulation is periodically reversed so that the materials in both boilers receive identical treatment. Uniformity of operation, avoidance of the formation of oxycellulose, and an economy of steam and chemicals is claimed. LÉONHART reports favourably on the plant.

A. B. MANNING.

Dyeing of artificial silks. W. WELTZIEN (Kolloid-Z., 1930, 52, 110—116).—A review embracing recent work on the dyeing of cellulose and acetate silks.

E. S. HEDGES.

PATENTS.

Cleansing composition [for fabrics]. A. R. JENNINGS (B.P. 306,119, 22.12.28. U.S., 17.2.28).—A mixture of a powdered mineral absorbent, such as talc, with eucalyptus oil.

A. J. HALL.

Production of coloured reserve effects on textile materials. I. G. FARBENIND. A.-G. (B.P. 311,740, 15.5.29. Ger., 15.5.28).—Coloured reserves on aniline-black are obtained by using a vat-dye paste thickened with cellulose esters or ethers (or other thickening agents precipitated by alkali), to which a reserving agent (sodium acetate, sodium hydrogen sulphite, zinc oxide) is added.

C. HOLLINS.

Production of coloured discharge effects on dyed goods. DURAND & HUGUENIN A.-G. (B.P. 310,893, 2.5.29. Ger., 2.5.28).—The galloxyanines obtainable according to B.P. 301,329 (B., 1930, 502) from *m*-substituted alkylanilines (ethyl-*m*-toluidine, *m*-chlorodimethylaniline, dimethyl-*m*-toluidine) give vivid coloured discharges by the usual hyposulphite reduction method.

C. HOLLINS.

Production of effects on vegetable yarns and fabrics. HEBERLEIN & Co. A.-G. (B.P. 313,616, 14.6.29. Ger., 15.6.28).—Vegetable fibrous materials are esterified superficially so that they retain their strength and gain a woolly handle and resistance to wetting and direct dyes. The material is treated with alkali and esterified by means of an acyl halide in an indifferent solvent in the proportion of 0.5, 2.0, or 3.0 mols. of the halide per mol. of $C_6H_{10}O_5$ (cf. Karrer and Zega, B., 1923, 1125 A).

A. J. HALL.

Manufacture of coloured fabrics. J. MORTON, J. E. G. HARRIS, and MORTON SUNDOWN FABRICS, LTD. (B.P. 329,334, 13.11. and 28.12.28).—Yarn is treated with oxidants (potassium chlorate, ammonium thiocyanate) suitable for developing leuco-vat-dye esters of the Soledon or Indigosol type, and is then woven with untreated yarn. The fabric is printed with a leuco-ester printing paste preferably containing a catalyst (ammonium vanadate). It is advantageous to pass the printed steamed fabric through a dilute alkaline bath to prevent development of the leuco-ester on untreated portions during the rinsing operations.

C. HOLLINS.

Printing with aniline-black etc. I. G. FARBENIND. A.-G. (B.P. 311,779, 16.5.29. Ger., 16.5.28).—A vat dye

can be printed simultaneously with aniline-black if a cellulose ester or ether or other thickening agent precipitated by alkali is used. The aniline-black is developed, and the material is then padded in an alkaline bath containing hyposulphite and a wetting agent, the vat dye being then fixed by steaming without intermediate drying.

C. HOLLINS.

Printing with vat dyes. I. G. FARBENIND. A.-G. (B.P. 314,904, 4.7.29. Ger., 4.7.28. Addn. to B.P. 279,864; B., 1929, 15).—Cotton is printed with a paste containing vat dye, a stable reducing agent (glucose), and a cellulose ether or ester as thickener (cellulose methyl ether), then padded in a caustic alkali bath, and without drying steamed, washed, oxidised, and soaped.

C. HOLLINS.

Fixation of insoluble metal compounds on textile materials [weighting or mordanting of acetate silk etc.]. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 329,659, 16.11.28).—Acetate silk or other textile is impregnated, *e.g.*, in a padding mangle, with a soluble metal salt (stannic chloride, ferric chloride, chromium chloride, etc.), and, after drying, a precipitant (particularly trisodium phosphate) is similarly applied.

C. HOLLINS.

Ornamentation of textile fabrics etc. I. G. FARBENIND. A.-G. (B.P. 311,741, 15.5.29. Ger., 15.5.28).—A flexible support such as paper is treated with a removable double layer of pliable materials pigmented as desired, *e.g.*, cellulose derivatives or resins, the layer in contact with the support being of higher m.p. than the other, which latter softens and becomes sticky at raised temperatures. The prepared sheet is laid on the fabric, and ironed hot, when the layer of lower m.p. is absorbed by the fabric, after which the paper support is removed to expose the layer of higher m.p.

F. R. ENNOS.

Dyeing of cellulose acetate threads. O. SPENGLER and W. MÜLLER, Assis. to GEN. ANILINE WORKS, INC. (U.S.P. 1,770,714, 15.7.30. Appl., 14.8.26. Ger., 15.8.25).—See F.P. 619,329; B., 1928, 86.

Delustring of artificial silk fibres or rayon. NYANZA COLOR & CHEM. CO., INC. (B.P. 315,432, 13.7.29. U.S., 14.7.28).—See U.S.P. 1,705,490; B., 1929, 640.

Apparatus for bleaching and dyeing etc. TISSAGE DEWITTE-LIETAER (B.P. 319,672, 25.9.29. Belg., 25.9.28).

Machine for dyeing, bleaching, and the like of loose yarns or bobbins. F. G. SCHUMACHER and J. J. MÜLLER (B.P. 331,706, 20.7.29).

Dyeing machines. C. F. KRANEY, and C. HEINZE MASCHINENFABR. A.-G. (B.P. 331,520, 28.12.28).

Dyeing of fibre laps. C. OBERMAIER (OBERMAIER & Co.) (B.P. 316,182, 16.7.29. Ger., 24.7.28).

Methods and apparatus for coating fabrics [with rubber etc.]. NAUGATUCK CHEM. CO., Asses. of J. MCGAVACK (B.P. 319,726, 5.7.29. U.S., 27.9.28).

Wetting etc. agents (B.P. 306,116, 308,824, and 329,622).—See III. **Sulphuric leuco-esters** (B.P. 329,356). **Dyeing of regenerated cellulose** (B.P. 329,961).—See IV.

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

Volumetric determination of phosphoric acid.

W. H. ROSS (J. Assoc. Off. Agric. Chem., 1930, 13, 203—207).—The accuracy of the official methods for the volumetric determination of phosphoric acid in the presence of sulphates and in materials high in organic matter has been studied collaboratively, using pure monopotassium phosphate, Tennessee phosphate rock, and a commercial grade of cottonseed meal. The results showed that, in the presence of sulphates, precipitation at 45–50° leads to high values, but precipitation at 25–30° with continuous stirring gives values in good agreement with those obtained in the absence of sulphates. Dissolution of the sample in dilute sulphuric acid gave good results for the material high in organic matter, provided precipitation was performed at 25–30° with continuous stirring.

H. J. DOWDEN.

Causticising. W. E. PIPER (Chem. Met. Eng., 1930, 37, 362—365).—The evolution of the Solvay process into a modern continuous system (based on identical reactions), utilising agitators, thickeners, and vacuum filters, is indicated and typical flow sheets of continuous and batch systems are given. The method of slaking the lime is vital as determining the size of the plant because of the effect it has on the settling rate of the carbonate residue. When milk of lime was prepared by slaking with all the water needed to make the soda solution it yielded a carbonate precipitate that settled at an initial rate of about 0.2 ft./hr.; if slaked dry with water and then creamed with the remainder of the solution the rate was 1.5; and when slaked dry with dilute caustic solution and then creamed the rate was 8 ft./hr. The use of a raised temperature increases both the settling rate and the percentage conversion (causticity), but there is no advantage in exceeding 85°; the conversion will then be about 95% when making a 10–15% caustic soda solution. The heating steam should be in a coil and the vessels well lagged to preserve the heat and prevent convection currents. It is advisable to avoid either too rapid or too prolonged agitation; 90 min. at twice the normal speed halved the rate of settling compared with 30 min. at normal speed. Excess of lime does not seem to have much effect on the percentage conversion of the soda (which depends on the concentration of solution made), but it increases the speed of reaction. It is recommended, in the continuous process, to have an additional causticising agitator between the first and second thickeners, to which a portion of the more concentrated sodium carbonate solution is added, the primary agitators giving a rapid and high conversion of the soda with excess of lime and the secondary an incomplete conversion of soda, but complete conversion of lime. Of the dilute caustic carbonate solution from the second thickener part is used to make milk of lime and the remainder to dissolve soda ash; the fractions unite in a drag classifier which is used to remove grit etc.

B. M. VENABLES.

Aluminium sulphate. G. KULLERUD and A. D. HÖRLÜCK (Dansk Tidsskr. Farm., 1930, 4, 157—171).—Commercial aluminium sulphate frequently contains basic salt, and it is difficult to prepare a pure sulphate

from such material by repeated recrystallisation unless sufficient sulphuric acid be previously added to transform the basic salt present into the normal sulphate. A number of p_H determinations have been carried out by various methods on aluminium sulphate solutions, and the results are tabulated; when using the quinhydrone electrode, several hours are necessary before a constant value is attained. It is recommended that aluminium sulphate should be employed, for pharmaceutical purposes, in the form of a solution, which must always be tested to see that it contains alumina and sulphuric acid in equivalent amounts; this may be conveniently ascertained by making a colorimetric p_H determination, using *p*-sulphobenzeneazobenzylaniline. The above method will detect the presence of 0.15% of free acid in the salt, and the presence of basic salt is also indicated, which is not the case when the usual thio-sulphate method is employed.

H. F. HARWOOD.

Sources of error in the determination of hydrogen in gases.

H. R. AMBLER (Analyst, 1930, 55, 436—443).—In determining hydrogen by the explosion method, error introduced by the oxidation of nitrogen is negligible for mixtures containing less than 20% of hydrogen, but above this the amount of nitrogen peroxide increases rapidly with increase of hydrogen content. With the slow-combustion method the oxidation is negligible. Completeness of combustion in the explosion method varies; in mixtures containing 10–20% of hydrogen at least 99% of the hydrogen is oxidised, but for poorer mixtures combustion may be incomplete even when there is a definitely visible explosion, the error even extending to 40% on the total hydrogen when less than 10% of hydrogen is present. The degree of excess of oxygen makes no apparent difference to the completeness of combustion. Even when 6.5% of methane is present the amount of unburnt hydrogen is not increased.

D. G. HEWER.

Decolorisation of oils. CHOWDHURY and DAS.—See II. Purifying gases. HEALY.—See XI. Coloured-light recipes. VANINO.—See XXII.

See also A., July, 861, System K_2CO_3 - $Ca(OH)_2$ - KOH - $CaCO_3$ (USANOVITSCH and BOROVIK). 867, Catalytic reactions at high pressures (MORGAN; APPLEBEY). 868, Electrochemical production of ammonium persulphate (IZGARISCHEV and GRUZDEVA). 872, Production of potassium phosphates (ASKENASY and NESSLER). 875, Metal carbonyls (HIEBER and others).

PATENTS.

Production of phosphoric acid and hydrogen. BAYERISCHE STICKSTOFF-WERKE A.-G. (B.P. 308,599, 19.3.29. Ger., 24.3.28. Addn. to B.P. 308,598; B., 1930, 660).—In the process described in the prior patent a certain amount of phosphorous acid is produced; this is converted into phosphoric acid and hydrogen by heating under about 600 atm. at below 600°.

A. R. POWELL.

Heating of gases [for ammonia synthesis]. GAS-VERARBEITUNGSGES. M.B.H. (B.P. 307,027, 28.2.29. Ger., 1.3.28).—The gas is first preheated to 400° in a heat exchanger by the hot gases from the ammonia catalysis

chambers, and then heated to the desired temperature by passing it countercurrent to a stream of nitrogen heated above 500° by passing it through a metal coil immersed in molten lead. Alternatively, or in addition, the gas mixture may be preheated during the purification process by adding small quantities of oxygen or of carbon monoxide and passing the mixture over heated catalysts comprising metals of group VIII.

A. R. POWELL.

Preheating of ammonia gas in the execution of reactions therewith at elevated temperatures.

I. G. FARBEIND. A.-G. (B.P. 307,843, 13.3.29. Ger., 14.3.28).—The preheater consists of coils of copper, silver, aluminium, or magnesium, or alloys of these metals with one another or with silicon, *e.g.*, 10% silicon-copper. Mixtures of ammonia and carbon monoxide may be preheated at 590–620° without appreciable dissociation of the ammonia taking place.

A. R. POWELL.

Production of ammonium sulphate. W. G. ADAM, D. G. MURDOCH, and GAS LIGHT & COKE Co. (B.P. 330,945 and 330,947, 20.3.29).—A solution of ammonium sulphate produced (A) by the interaction of ammonia, carbon dioxide, and calcium sulphate, or (B) by saturation of sulphuric acid with ammonia, is evaporated until it contains 45% of ammonium sulphate; ferric sulphate equivalent to 0.003% Fe on the amount of ammonium sulphate present and sulphuric acid equal to 0.2% of the solution are added, and the solution is boiled at constant temperature to give a controlled rate of crystallisation. In this way a loose crystalline mass is obtained which does not cake.

A. R. POWELL.

Manufacture of carbamates and conversion products thereof. J. Y. JOHNSON. From I. G. FARBEIND. A.-G. (B.P. 329,737, 11.3.29).—Ammonia and carbon dioxide are made to react in an inert liquid in presence of metal salts, or of alkaline-earth oxides or hydroxides, soluble to some extent in the liquid, the latter being one in which the metal carbamate is substantially insoluble. *E.g.*, carbon dioxide and ammonia are passed at ordinary temperature and pressure into methyl-alcoholic calcium chloride solution, or carbon dioxide into a suspension of calcium hydroxide in alcohol saturated with ammonia. Calcium, lead, or sodium nitrate in methyl alcohol, lead acetate in ethyl alcohol, or calcium nitrate and chloride in formamide may also be used. The metal carbamate gives the cyanamide when heated in ammonia gas.

C. HOLLINS.

Electrolysis of fused halogen salts. J. Y. JOHNSON. From I. G. FARBEIND. A.-G. (B.P. 330,791, 1.7.29).—Electrolysis is effected at a temperature below red heat, using a tungsten anode or a metal or graphite anode coated with tungsten. Pure beryllium may thus be obtained from a bath containing 73% BeCl_2 and 27% NaCl at 420–450°, and pure aluminium from a bath containing 82% AlCl_3 and 18% NaCl at 200°.

A. R. POWELL.

Production of potassium nitrate with simultaneous obtention of dimagnesium phosphate. CHEMIEVERFAHREN-GES.M.B.H. (B.P. 329,939, 11.10.29. Ger., 28.11.28).—Crude phosphate is decomposed with nitric acid and potassium sulphate, according to the

known process. A solution containing potassium nitrate and phosphoric acid results; the bulk of the phosphoric acid is separated out from this as dimagnesium phosphate by neutralisation with oxide, hydroxide, or carbonate of magnesium (or a mixture or compound thereof). The phosphoric acid remaining in the solution (preferably to the extent of about 1%) is then removed by neutralisation, preferably by being precipitated as magnesium orthophosphate, and the potassium nitrate is separated out by crystallisation or evaporation. The magnesium orthophosphate obtained is used to assist in the precipitation of magnesium diposphate from fresh initial materials.

S. K. TWEEDY.

Manufacture of crystalline phosphates. VICTOR CHEM. WORKS, ASSEES. OF N. C. LINDBERG and G. A. McDONALD (B.P. 316,136, 9.7.29. U.S., 23.7.28).—The aluminium and iron phosphates present in phosphate crystals obtained by neutralising concentrated phosphoric acid are removed by washing or flotation. Any liquid which does not impair the crystals may be used, preferably the filtered mother-liquor or a saturated solution of the desired phosphate. The wash-liquor may be passed through oxidising tanks in order to precipitate any ferrous phosphate as ferric phosphate; it may then be added to the phosphoric acid before neutralisation.

S. K. TWEEDY.

Production of diammonium phosphate. FEDERAL PHOSPHORUS Co., ASSEES. OF B. G. KLUGH and W. R. SEYFRIED (B.P. 315,700, 20.6.29. U.S., 16.7.28).—A mixture of 5–6 pts. of phosphoric acid (as a 70% solution) and 1 pt. of diammonium phosphate (as a cold saturated solution) is treated in a heat-insulated vessel with ammonia gas until the composition of the salts in solution corresponds with a 5:1 mixture of mono- and diammonium phosphates. The hot, highly concentrated solution is run into a closed vessel containing about half its volume of a cold saturated solution of the diammonium salt, whereby the temperature of the mixture falls to 80°. An atmosphere of ammonia is maintained above the solution, which is agitated until all the phosphate is converted into the di-salt. The ammonia is then replaced by air and the mixture cooled with agitation at the rate of 10°/hr. The crystals of di-salt are separated and about one third of the mother-liquor is returned to the first saturator and the remainder to the second saturator.

A. R. POWELL.

Manufacture of triple superphosphates. F. C. PALAZZO (B.P. 310,479, 26.4.29. It., 26.4.28).—Substantially pure precipitated dicalcium phosphate of high phosphoric anhydride content (*e.g.*, 38%) is mixed with phosphoric acid, preferably concentrated, in quantity not exceeding that necessary for the formation of monocalcium phosphate. When the reaction mixture corresponds to that represented by the equation: $\text{H}_3\text{PO}_4 + \text{CaHPO}_4 \cdot 2\text{H}_2\text{O} = \text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O} + \text{H}_2\text{O}$, the products contain at least 50% of water-soluble P_2O_5 . Preferably, the reaction components are mixed, then dried after a few hours at about 50°, and crushed.

S. K. TWEEDY.

Manufacture of phosphoric acid esters. J. Y. JOHNSON. From I. G. FARBEIND. A.-G. (B.P. 330,228,

31.1.29).—Phosphorus oxychloride is allowed to react with an aliphatic or aromatic alcohol or mixtures thereof at 0–5°, the mixture is then heated at about 40°, the hydrochloric acid formed removed by washing with water and then alkali, and the ester purified by distillation under reduced pressure. The preparation of the following esters of phosphoric acid is described: tributyl, *n*-butyl diisocamyl (b.p. 145–146°/4.5 mm.), diisocamyl cyclohexyl (b.p. 142°/0.5 mm.), and phenyl dibutyl (b.p. 183–185°/15 mm.).
A. R. POWELL.

Production of calcined borax. AMER. POTASH & CHEM. CORP. (B.P. 330,146, 14.6.29. U.S., 11.3.29).—Borax is calcined while retaining its shape and size by subjecting it in stages to a stream of air, or other dry gas, having a temperature at each stage such that the borax cannot melt or dissolve in its water of crystallisation and having a humidity such that water of crystallisation is picked up at each stage. Five stages may be employed, the temperature and humidity (in lb. of water vapour per lb. of air) for each being: (1) 50–100°, 0.08; (2) 65–150°, 0.06–0.08; (3) 125–200°, 0.04–0.06; (4) 175–260°, 0.02–0.04; (5) 225–325°, 0.0–0.02. Two mols. of water are lost in each stage except the last, where only one is lost, the final product being the monohydrate having an apparent sp. gr. of 0.5–0.9. Borax containing 8–12% H₂O, or having 1–3 mols. of water, is most advantageously produced by the process.
S. K. TWEEDY.

Borax product and its production. AMER. POTASH & CHEM. CORP. (B.P. 330,453, 2.8.29. U.S., 9.4.29. Addn. to B.P. 330,146; preceding).—Borax is heated at 50–100° in a current of warm air containing less than 10% of moisture until 20% of the combined water is removed; the temperature of the air is raised to 65–150° and its humidity decreased below 8% to remove a further 20% of water, then to 125–200° with less than 6% humidity, and finally to 175–260° with less than 4% of moisture, whereby a cellular, dense product, Na₂B₄O₇·3H₂O, is obtained which is readily soluble in water.
A. R. POWELL.

Production of [crystalline] aluminium oxide. VEREIN. ALUMINIUM-WERKE, A.-G. (B.P. 313,409, 10.6.29. Ger., 9.6.28).—A mixture of bauxite and sulphur-containing materials (pyrites) is subjected to a reducing smelting treatment, and the molten product, consisting to a large extent of aluminium sulphide, is chilled, e.g., by pouring on to a cooled surface, or by pouring into water or into molten aluminium.
S. K. TWEEDY.

Simultaneous preparation of alumina and pure carbon dioxide. C. D'ASSEEV (B.P. 318,976, 13.9.29. Belg., 13.9.28).—A solution of crude aluminium sulphate containing iron and obtained by treating aluminium ores with sulphuric acid is treated cold with magnesium hydrogen carbonate, whereby basic iron and aluminium carbonates are precipitated, carbon dioxide is evolved, and a solution of magnesium sulphate is obtained. The precipitate is separated from the mother-liquor and heated at 90–100° to obtain pure carbon dioxide; the residual hydroxides are digested with 5% sodium hydroxide solution to obtain ferric hydroxide for use in gas purification and a solution of sodium aluminate from

which the alumina is reprecipitated in a pure form by treatment with the carbon dioxide evolved in the first stage of the process or by agitation with alumina.

A. R. POWELL.

Preparation of alumina. W. J. MÜLLER and H. HILLER (B.P. 330,661, 19.3.29).—Bauxite is heated under pressure at above 190° with a solution of sodium hydroxide of *d* 1.16–1.18. After removal of the insoluble red slime the aluminate solution so obtained may be directly inoculated with aluminium hydroxide as in the Bayer process and, after separation of the alumina, the alkaline liquor may be returned to the autoclaves without being concentrated further.
A. R. POWELL.

Removal of one or more metals of the iron group from solutions containing salts of one or more of these metals [e.g., iron from aluminium salts]. K. W. PALMAER (B.P. 318,149, 12.8.29. Appl., 28.8.28).—Iron is removed from solutions of aluminium sulphate obtained by the acid decomposition of aluminium ores by reducing any ferric salt to the ferrous state, neutralising the solution, and electrolysing it between a lead anode and a mercury cathode which is kept continuously in movement. The iron is thus deposited as an amalgam and a pure solution of aluminium sulphate is obtained.
A. R. POWELL.

Production of magnesium oxide from kieserite and other salts or salt mixtures containing magnesium. KALI-FORSCHUNGS-ANSTALT G.M.B.H. (B.P. 330,715, 7.5.29. Ger., 12.1.29).—Kieserite, schönite, or other natural or artificial sulphates containing magnesium, potassium, and sulphate are made into a slurry with water and the latter is saturated with ammonia, whereby ammonium sulphate is formed in solution and an insoluble residue of crystalline anhydrite and/or potassium sulphate and magnesium hydroxide is obtained. By continuous decantation of the mother-liquor the magnesium hydroxide may be separated from the other insoluble constituents. After filtering off the magnesia the mother-liquor is returned to the saturator for use again.
A. R. POWELL.

Separating magnesium chloride from its aqueous solutions and obtaining anhydrous magnesium chloride. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,650, 15.2.29. Addn. to B.P. 329,032; B., 1930, 644).—A 30% solution of magnesium chloride is treated with 64% of its weight of ammonium chloride, saturated with ammonia, and cooled to –30°. The crystals of MgCl₂·6NH₃ which separate are collected, washed with liquid ammonia, and heated at 500° to remove the ammonia for use again; the residue consists of anhydrous magnesium chloride.
A. R. POWELL.

Obtaining hydrogen peroxide by distillation. DEUTS. GOLD-UND SILBER-SCHNEIDANSTALT VORM. ROESSLER, G. BAUM, and OESTERR. CHEM. WERKE GES.M.B.H. (B.P. 330,255, 20.2.29).—About 200–250 litres/hr. of dilute sulphuric acid containing 25% of persulphuric acid are drawn by suction under 650–700 mm. of vacuum through a lead pipe, 60 m. long and 75 mm. internal diam., wound into 15–25 coils and immersed in a steam jacket. The hydrogen peroxide evolved is recovered by fractional condensation of the vapours evolved.
A. R. POWELL.

Manufacture of manganese dioxide. Y. KATO and K. YAMAMOTO (B.P. 330,257, 26.2.29).—Lower oxides of manganese or materials containing them are heated with 3–5% of an alkali hydroxide or carbonate in the presence of air or oxygen at 500°. The product is cooled, ground, and leached, first with water and then with dilute acid. [Stat. ref.] A. R. POWELL.

Production of zirconium oxide [from zirconium mineral]. A. KARL (B.P. 314,526, 18.6.29. Belg., 30.6.28).—The mineral is fused with sodium carbonate. The product (sodium zirconate), after removing the sodium silicate by washing, is dissolved in excess sulphuric acid and the iron and titanium sulphates contained in the resulting solution are reduced (*e.g.*, by means of nascent hydrogen, sulphur dioxide, or sodium hyposulphite). Pure zirconia is then precipitated out as the result of hydrolysis by neutralising the solution, *e.g.*, with sodium carbonate. The initial fusion is conveniently effected in a furnace lined with zircon, the melt being allowed to run straight into water. The precipitated sodium zirconate is preferably dissolved at 100–400° in sulphuric acid having a concentration between that of fuming acid and acid of *d* 1.71.

S. K. TWEEDY.

Production of hydrogen peroxide. L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 309,602, 11.4.29. Fr., 14.4.28).—Electrolytic hydrogen containing 4% O₂ is passed over a platinum catalyst at 500–600° under 400 atm. and thence through a condenser maintained at 0°. The condensate is a 2.9% solution of hydrogen peroxide. Combustion of oxygen under high pressure in an atmosphere of hydrogen under pressure also affords a dilute solution of hydrogen peroxide.

A. R. POWELL.

Removal of free chlorine and bromine from fluid mixtures. J. B. PAYMAN and H. A. PIGGOTT, ASSRS. TO BRIT. DYE-STUFFS CORP., LTD. (U.S.P. 1,770,010, 8.7.30. Appl., 23.4.28. U.K., 27.4.27).—See B.P. 292,307; B., 1928, 604.

Processes and apparatus for liquefying solid carbon dioxide. MASCHINENFABR. ESSLINGEN, and J. STOFFELS (B.P. 331,750, 3.9.29).

Gas-analysis apparatus (B.P. 330,799).—See I. [Hydrogen from] brown coal (G.P. 462,388). Separation of ammonia from coal gas (G.P. 462,186).—See II. Activation of chemical reactions (B.P. 330,226). Treatment of mica (B.P. 329,878).—See XI. Lakes or pigments (B.P. 307,436).—See XIII. Compressed iodine (B.P. 331,118).—See XX.

VIII.—GLASS; CERAMICS.

Effects of additions of zinc oxide and cadmium oxide on simple glasses. C. W. PARMELEE and R. G. EHMAN (J. Amer. Ceram. Soc., 1930, 13, 475–488).—The small batches of glass were melted and fired in a vacuum electric furnace, which is fully described. A simple Na₂O–PbO–SiO₂ glass was used, the Na₂O content varying from 5% to 20% in steps of 5%. This constituent content was replaced by additions of zinc and cadmium oxides, and the effect of such variations on thermal expansion, refractive index, acid-resistance,

and solubility was determined. The difference in expansion indicated occurs only in the higher temperature ranges. Glasses containing above 10% ZnO (or CdO) showed a greatly increased refractoriness and viscosity. These oxides are stated to impart desirable properties to enamels, and additions of up to 10% act as fluxes, decrease the viscosity, increase the acid-resistance, and improve the appearance.

R. J. CARTLIDGE.

Effect of additions of calcium and magnesium oxides on enamel glasses. C. W. PARMELEE and D. T. H. SHAW (J. Amer. Ceram. Soc., 1930, 13, 498–508).—A simple glass containing SiO₂ 58%, PbO 22%, and Na₂O 20% was used, and various amounts of calcium and magnesium oxides were added. The tests consisted of measurements of the thermal expansion, refractoriness, compressive strength, modulus of elasticity, solubility, and index of refraction of the enamels produced. A glass consisting of SiO₂ 58%, PbO 22%, CaO (or MgO) 5–10%, Na₂O remainder gave the best enamel.

R. J. CARTLIDGE.

Systematic method for the investigation of sheet-iron enamels. A. I. ANDREWS (J. Amer. Ceram. Soc., 1930, 13, 489–497).—Selected compositions of 24 sheet-iron ground coats and 24 cover enamels are considered. Tables and triaxial diagrams are given to simplify the study of relative amounts of felspar, borax, and flint. A description of the utility of the three most common triaxial diagrams is appended.

R. J. CARTLIDGE.

Reactivity test for determining the value of barium carbonate as a scum preventive [on glazed ware]. A. L. BENNETT and H. R. GOODRICH (J. Amer. Ceram. Soc., 1930, 13, 461–469).—A discussion of the causes of scumming and glaze peeling is followed by a description of the Howat and Williams reactivity test for barium carbonate (Bull. Amer. Ceram. Soc., 1923, 2, 161) and a method for determining soluble sulphates in clays. Tables given indicate the reactivities of various barium carbonates, and comparative effects of barium carbonate and barium chloride in a terra-cotta body. A reactivity value of 22–23% is stated to be desirable.

R. J. CARTLIDGE.

Rate of vitrification of porcelain. C. W. PARMELEE and A. E. BADGER (Ind. Eng. Chem., 1930, 22, 781–785).—The rate of vitrification of a typical porcelain has been followed by determining the changes in volume and in porosity brought about by different conditions of heating in an electric furnace. At 890° the initial increase in volume is maintained at a constant value, whilst at 995° it is followed by a gradual decrease in volume as the time of heating is increased. At higher temperatures, 1090° and 1200°, this decrease is greater and sets in more rapidly. The porosity varies in a similar manner, the decrease being the greater the higher is the temperature (up to 1200°) at which the sample is heated. These changes are in accord with the transformations which are known to take place when a porcelain body is fired.

L. S. THEOBALD.

Thermal expansion of silica brick and mortars. S. S. COLE (J. Amer. Ceram. Soc., 1930, 13, 437–446).—A more complete study of the thermal expansion of

American and European silica brick and mortars is presented. Data are given in the form of curves showing values for the expansion of silica brick varying between 1.15% and 1.30% at 950° and of mortars between 1.30% to 1.52%, depending on the content of clay, quartzite, and bats. Apparent sp. gr. and porosity values are also given; porosity appears to have little effect on expansion. R. J. CARTLIDGE.

Special refractories for electric furnace linings.

(a) **Silica, corundum, and carborundum bricks.** O. KUKLA (Stahl u. Eisen, 1930, 50, 800—803).

(b) **Sillimanite bricks.** F. SOMMER (*Ibid.*, 804).—

(a) Experiments with a 7-ton Héroult-Lindenburg electric steel furnace show that carborundum bricks behave well as a refractory in the roof of the furnace in a reducing atmosphere, but are not as good as silica bricks for the body of the furnace in contact with the molten charge. In this position the carborundum is rapidly burned at the surface of the bricks, thus causing rapid wear. Corundum bricks are unsatisfactory owing to their great tendency to spalling.

(b) Sillimanite bricks behave no better than good silica bricks in electric steel furnaces, and are four times as expensive. A. R. POWELL.

Clays. BERRIDGE.—See IX.

See also A., July, 847, **Fusion diagrams of highly refractive oxides** (VON WARTENBERG and WERTH). 857, **Production of kaolin and kaolinite** (ENK). 874, **Silvering of glass** (VON WARTENBERG). 885, **Determination of coefficients of expansion of glasses and ceramic materials** (THILENIUS and HOLZMANN). 968, **Testing of ampoule glass for alkalinity** (STICH).

PATENTS.

Stratified bodies such as strengthened glass.

C. R. CROSS, C. J. G. FOX, L. G. S. HEBBS, and TRIPLEX SAFETY GLASS CO., LTD. (B.P. 324,898, 3.11.28).—Sheets of cellulose acetate may serve to unite one or more sheets of glass if diacetone alcohol is used as the medium, the coating being applied to the nitrocellulose enamel with which the gelatin-coated glass surface is normally covered. In place of diacetone alcohol, "acetone oil," the monomethyl ether of ethylene glycol, or the monoethyl ether of diethylene glycol may be used. M. PARKIN.

Manufacture of safety glass. I. G. FARBERIND. A.-G. (B.P. 315,667, 15.7.29. Ger., 14.7.28).—The middle layer in safety glass is made of the cellulose ester of a fatty acid containing more than 3 carbon atoms, *e.g.*, laurate, acetate-butyrate, butyrate-laurate, these esters being fast to light and water. Adhesion may be increased by addition of resin, such as polymerised vinyl acetate, or other softener, *e.g.*, resorcinol monoacetate; or a sticky layer, such as cellulose naphthenate with vinyl acetate resin and resorcinol monoacetate, may be spread on the two glass plates, which are then applied to a film of the cellulose ester. C. HOLLINS.

Making compound transparent sheets [laminated glass]. L. BARTELSTONE (B.P. 331,030, 27.4.29. U.S., 13.3.29).—A sheet of celluloid, conditioned by baking for 5 days in a current of warm air at about 37°, is cleaned while "earthed" to prevent electrification,

resurfaced by spraying with a free-flowing celluloid solution, and the residual solvent from this removed by baking for 30 min. at 65—71°. Two plates of cleaned glass are then given a thick coating of castor oil on their upper faces, the celluloid sheet is laid on one, and the other quickly turned over on to it, the process being completed by subjecting to suitable temperature and pressure. M. PARKIN.

Manufacture of [laminated] safety glass. DU PONT VISCOLOID CO. (B.P. 306,891, 14.1.29. U.S., 27.2.28).—Laminated glass possessing better adherence and not so liable to discoloration is produced by using a cellulose ester plastic from the sheeting machine without polishing, coating it with a cement (*e.g.*, gelatin) to give a film not greater than 0.0005 in., placing it between the polished glass sheets and subjecting the sandwich to hot water (110—115°) under pressure (150—500 lb./in.²). M. PARKIN.

Luminous enamels and their application. A. ANDRÉ (B.P. 325,072, 10.1.29).—An aluminous flux (2 pts.), for which three preferred compositions are given, is finely powdered after melting at 900—1100°, and mixed with 1 pt. of phosphorescent material, such as zinc sulphide, together with 6—7% of white clay (*e.g.*, kaolin), the mixture being made into a paste with water. The clay should be pretreated with potassium permanganate solution to destroy organic matter. Surfaces of metal, porcelain, or ceramic bodies may be decorated by applying this paste and, when dry, firing as usual. Oxidisable metal parts should be covered with a protective coating, preferably having a cobalt base. M. PARKIN.

Production of hollow bodies from silica and other refractory materials. W. C. HERAEUS GES.M.B.H., and A. KRÖNER (B.P. 330,943, 20.3.29).—Fused silica tubes etc. are produced by feeding pulverulent quartz into a furnace rotating about a horizontal axis, which carries a mould either of water-cooled metal or of refractory material readily detachable from the product afterwards, or the lining may consist of granules of the material itself. A carbon arc is struck to play along the mould, and the fused quartz particles are spread by centrifugal force (100 r.p.m. for a tube 100 mm. in diam.) against the sides of the mould. M. PARKIN.

Manufacture of objects [glass, enamel] from non-conducting materials. A. UHLMANN, ASSR. to A. T. OTTO & SONS (U.S.P. 1,768,869, 1.7.30. Appl., 14.7.26. Ger., 18.7.25).—See B.P. 255,497; B., 1928, 264.

Forming ceramic articles and the like. T. S. CURTIS, ASSR. to VITREFRAX CORP. (U.S.P. 1,768,546, 1.7.30. Appl., 16.11.26).—See B.P. 291,523; B., 1928, 605.

Weatherproof laminated glass. L. BARTELSTONE (B.P. 325,048, 13.12.28).

IX.—BUILDING MATERIALS.

Colloidal nature and water content of clays. H. BERRIDGE (Engineering, 1930, 130, 5—7, 61—63).—Whilst most investigations of clay have been carried out from the ceramic point of view, a study of clay has now been made so far as it affects the foundations of engineering and architectural structures. Data

obtained by the author and by other workers for the water content, volume weight, tensile strength, and compressive strength of various clays in different states are listed. The volume weight, or density of the total system, affords for engineering purposes a valuable means of classification which has so far not been adopted. When dried, clay shrinks until its water content has fallen to about 12.5%; its volume then ceases to diminish, although evaporation of water proceeds until the porous mass contains about 5% of water, which can be driven off at 110°. If the temperature is kept below 415° the resulting "argil" re-absorbs moisture, but not if heated above that temperature. The effect of absorption of water on the mechanical properties and volume changes of clay is discussed in relation to the digging of trenches for foundations and the construction of clay embankments, and the most efficient and economical courses are indicated. Dried clay expands when wetted, even when loaded to above 5 tons per sq. ft. If left exposed to the atmosphere, the clay absorbs about 6% of water without volume change. E. S. HEDGES.

Corrosion of structural steel and steel reinforcing rods encased in concrete. E. A. CROSS (Engineering, 1930, 130, 30).—An engine-room floor composed of continuous concrete 6 in. thick supported on 18-in. steel joists (covered with concrete) was originally provided with reinforcement in the lower part of the slabs only, *i.e.*, no steel was provided to take tensional stresses in the upper half of the floor, where it passed over the joists. The floor carried a brine tank, which for some years had been leaking; there were external signs of severe corrosion, especially along the joists where tension cracks were noticed. A large area of floor was removed, and the joists and rods were found to have corroded to vanishing point in places. A minor contributory cause was considered to be condensation around ventilating holes, which had been left in the floor, for the room below was at a much higher temperature. A method of repair, which provides a dense and impervious concrete with adequate reinforcement, is described. B. M. VENABLES.

Corrosion of steel by breeze and clinker concretes. F. L. BRADY (Dept. Sci. Ind. Res., Building Res., Rep. No. 15, 1930, 15 pp.).—Breeze and clinker aggregates promote the rusting of steel even under dry indoor conditions, the rate of corrosion of steel in contact with these aggregates being roughly proportional to their sulphur content. The deleterious effect of breeze and clinker in reinforced concrete is due to their high porosity, which permits the ready diffusion of oxygen and moisture into the mass with consequent rapid oxidation of the sulphur compounds present. The substitution of sand for the finer portion of coal residue aggregates reduces rusting owing to the diminution in the amount of accessible sulphur compounds, and not to the reduced porosity. When clinker low in sulphur and free from fines is used together with sand in concrete aggregates, only very slow rusting of embedded steel reinforcement occurs; if, however, the fines are not removed, fairly rapid corrosion occurs. These results indicate that coal residues are unsuitable for the

preparation of concrete which is to come into contact with steel.

A. R. POWELL.

Action of salts, acids, and organic substances on cement and concrete. R. GRÜN (Z. angew. Chem., 1930, 43, 496–500).—Addition of calcium, barium, aluminium, ferric, or hydrogen chloride to the water used in making cement or concrete mixtures increases appreciably the rate of setting and the tensile and compressive strengths, probably owing to the formation of calcium chloride which acts as an accelerator. Formaldehyde, sugars, and similar organic compounds, even in small quantities, retard or almost completely prevent the hardening of cement. Oxalic acid tends to stabilise cement mixtures by combining with the free lime, and has no action whatever on hardened cements. Phosphoric acid, on the other hand, slowly disintegrates cement structures by forming soluble acid phosphates, but its action in low concentrations is exceedingly slow.

A. R. POWELL.

See also A., July, 872, **Hydrated calcium aluminates** (TRAVERS and SEENOUTKA). 967, **Drying of timber** (CAMPBELL and BOOTH). 968, **Destruction of coniferous wood by beetle larva** (FALCK).

Tar-asphalt mixtures. SCHLÄPFER.—See II. **Thermal expansion of mortars.** COLE.—See VIII.

PATENTS.

Piston pump for raising viscous mixtures such as concrete, mortar, etc. M. GIESE and F. HELL (B.P. 319,347, 30.5.29. Ger., 21.9.28).

Pulp-board (U.S.P. 1,746,451).—See V.

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

Potentiometric determination and separation of chromium, vanadium, and molybdenum, applied to steel analysis. W. TRZEBIATOWSKI (Rocz. Chem., 1930, 10, 411–436).—Steel containing chromium, vanadium, molybdenum, and tungsten is dissolved in concentrated hydrochloric acid to which one third of its volume of water has been added, insoluble tungsten being then dissolved by the addition of nitric acid. Tungstic acid is precipitated by dilution and is removed by filtration. The filtrate is concentrated to a syrup, which is transferred to a crucible containing excess of sodium hydroxide, ignited, fused with excess of sodium peroxide, and extracted with boiling 2*N*-sodium hydroxide containing 2–3 c.c. of hydrogen peroxide. The precipitate of hydroxides is filtered off, and the filtrate is concentrated to about 80 c.c., neutralised with 50% sulphuric acid, and the volume made up to 200 c.c. with 25% sulphuric acid. 50 C.c. of the solution are now titrated with stannous chloride solution, in an inert atmosphere, to the first abrupt fall in potential, corresponding to reduction of sexa- to ter-valent chromium, and of quinque- to quadri-valent vanadium. Ferrous ammonium sulphate and concentrated sulphuric acid to a final concentration of 50% are now added, the system is warmed at 90–100°, and stannous chloride is added to the second potential fall, corresponding to reduction of quadri- to ter-valent vanadium, and of sexa- to quadri-valent molybdenum. The solution is

now diluted to 100 c.c., and to 50 c.c. 20 c.c. of 25% ammonium sulphate and 50 c.c. of 4% potassium bromate solutions are added; the solution is then diluted so that for every 5 c.c. of concentrated sulphuric acid present there are 60 c.c. of solution. The solution is then heated for 10 min. at 60° and for 5 min. at boiling, a stream of carbon dioxide being passed in in order to remove bromine. After cooling to 40–50°, 25 c.c. of concentrated sulphuric acid are added and the solution is titrated at 60° with stannous chloride to the third potential fall, corresponding to reduction of quinque- to quadri-valent vanadium. The percentage chromium, vanadium, and molybdenum contents are then given, respectively, by $346 \cdot 6M(b - 2cM'/M)/mi$, $2040M'c/mi$, and $1920M(a - 2cM'/M)/mi$, where m represents the weight of steel taken, a , b , and c the number of c.c. of stannous chloride solution used for the first, second, and third potential falls, i the number of c.c. taken for titration, and M and M' are the titres of the stannous chloride solution determined alone and under conditions analogous to those used in the experiment. Molybdenum catalyses the reduction of quadri- to ter-valent vanadium; should it be absent from the solution it should, therefore, be added.

R. TRUSZKOWSKI.

Pressure-ageing of duralumin. L. PESSEL (Ind. Eng. Chem., 1930, 22, 776–777).—The application of pressure to duralumin during the ageing period reduces its susceptibility to intergranular corrosion. Tensile strength and hardness are negligibly decreased by this treatment, but elongation is increased. After exposure to corrosion, however, the decrease in tensile strength and in elongation is much smaller in duralumin which has been aged under pressure. L. S. THEOBALD.

Resistance of nickel to corrosion. R. KRULLA (Chem.-Ztg., 1930, 54, 429–431).—Curves showing the rate of dissolution of nickel in various corroding media at room temperature are given and the effect of various alloying elements on the resistance of nickel to corrosion is discussed. Up to 4% Fe decreases greatly the rate of dissolution in nitric acid, carbon increases the resistance to attack by molten alkalis, and chromium and molybdenum improve the resistance towards acetic and hydrochloric acids, respectively. A nickel alloy with 20% Mo loses only a few cg./hr. in hot concentrated hydrochloric acid. In contact with reducing flames or hot gases containing sulphur compounds nickel rapidly becomes brittle due to absorption of carbon or sulphur.

A. R. POWELL.

Composition of deposit forming on zinc immersed in cupric sulphate solution. I. A. GALECKI and J. TOMASZEWSKI (Rocz. Chem., 1930, 10, 437–471).—The coherence of the deposit forming on zinc rods immersed in cupric sulphate solution increases with stirring and with the concentration and acidity of the solutions; at the same time the colour of the deposit becomes lighter. The deposit contains two constituents: (a) a metallic, flexible, red, yellow, or white coating, possessing either a dull or a polished surface; and (b) a dark brown to black powdery or flocculent precipitate. The former consists of metallic copper and zinc in various proportions, probably both as a mixture

and as alloys, whilst the latter contains oxides of the two metals, as well as basic sulphates and the free metals.

R. TRUSZKOWSKI.

Technical preparation of rhenium. W. FEIT (Z. angew. Chem., 1930, 43, 459–462).—An account is given of researches made on the extraction of rhenium from a complex sulphide slime obtained as a by-product in an unstated metallurgical process. The slime had weathered in the air for many months and on extraction with water yielded a brownish-green solution from which nickel and copper sulphates were obtained by evaporation. By the addition of regulated quantities of ammonium sulphate most of the copper, nickel, and zinc were separated as double ammonium sulphates. The almost black mother-liquor had $d > 2$, and with more ammonium sulphate yielded dark violet crystals of the ammonium salt of a hetero-poly-acid containing molybdenum, vanadium, and phosphorus; the pale yellow mother-liquor yielded crystals of potassium perrhenate on addition of potassium chloride. Large quantities of the slime are being worked up as follows: it is heated at 100° with constant rabbling for 1 month, whereby almost complete oxidation is effected; the residue is extracted with water and the solution evaporated in stages to remove zinc, copper, nickel, and ferrous sulphates. When traces of potassium perrhenate begin to separate the solution is diluted with mother-liquor from a previous crop of crystals and treated with a large excess of potassium chloride. The grey, impure perrhenate is collected, washed with cold water, and dissolved in hot dilute sodium hydroxide; on filtering off the precipitated hydroxides and cooling the solution relatively pure potassium perrhenate is obtained and may be further purified from traces of molybdenum and vanadium by repeating the recrystallisation twice. In this way a product with 64.6% Re and less than 0.01% of impurities has been obtained at the rate of 10 kg. per month at a cost of 10 s. per g. A. R. POWELL.

Resistance of metals to the system urea-water-ammonium carbamate. J. G. THOMPSON, H. J. KRASE, and K. G. CLARK (Ind. Eng. Chem., 1930, 22, 735–736; cf. Krase and co-workers, B., 1930, 408).—The corrosion of 36 metals, mainly alloys, by this system at 142° under pressure has been investigated.

L. S. THEOBALD.

See also A., July, 846, **Recrystallisation of lead** (GARRE and MÜLLER). 863, **Electrode potentials of silver in cyanide solutions** (FINK and HOGABOOM). 870, **Electrodeposition of metals from their liquid ammonia solutions** (TAFT and BARHAM). 874, **Silvering of glass** (VON WARTENBERG). 882, **Determination of metals by electrodeposition** (TUTUNDŽIĆ). **Test for tin** (MEISSNER).

Adhesives and adhesion. LEE.—See I. **Sheet-iron enamels**. ANDREWS.—See VIII. **Corrosion of structural steel**. CROSS. **Corrosion of steel by concrete**. BRADY.—See IX.

PATENTS.

Siemens-Martin furnaces. H. WADE. From "TERNI" SOC. PER L'IND. E L'ELETTRICITA (B.P. 329,176, 24.5.29).—The furnace has superimposed air and gas

ducts, the lower wall of the upper or air duct being downwardly inclined towards the gas duct, and the upper and lower walls of the air duct diverging towards the mixing and combustion chambers, so that the angle at which the air and gas streams meet can be varied by altering the supply pressures. A. R. POWELL.

Electrical devices for heating a metal melt within a ladle. F. KRUPP A.-G. (B.P. 312,063, 8.5.29. Ger., 18.5.28).—The device comprises a hollow cylinder of refractory material in which is embedded a water-cooled copper coil through which a high-frequency current is passed. The apparatus is immersed in molten steel in the ladle so as to keep it hot by inductive heating until the slag has separated. A. R. POWELL.

Furnace ladles. F. KRUPP A.-G. (B.P. 312,064, 8.5.29. Ger., 18.5.28).—The ladle comprises a shell built up of annular rings of non-magnetic chromium-nickel-iron alloy insulated from one another and provided with holes to prevent generation of eddy currents. The shell is lined with a refractory material in which is embedded a water-cooled copper coil, by the aid of which the ladle contents may be heated by induction. A. R. POWELL.

Crucible furnaces for melting metals. H. BAILEY (B.P. 329,365, 13.2.29).—A crucible furnace comprises a heating chamber tapering inwards towards the top and provided with fire-bars at the bottom on which the crucibles and fuel are placed. Air under pressure is admitted into the ash-pit below the fire-bars and is there preheated before passing into the fuel chamber; the ashes are sprayed with water to damp them down and to provide steam to aid the combustion of the fuel. A. R. POWELL.

Case-hardening furnace with charging device of worm type. H. LINDHORST (B.P. 330,188, 22.8.29).—The case-hardening chamber consists of a horizontal rotating drum closed at one end with a cap carrying a tube inside which is a worm-charging device, the whole rotating round a pyrometer tube through the longitudinal axis of the worm. At the end of the worm-tube outside the drum is a hopper through which more of the case-hardening mixture is fed into the drum during use, so that the case-hardening gases are generated regularly throughout the operation. A. R. POWELL.

Manufacture of tools of homogeneous alloys of great hardness. G. BREWER. From F. KRUPP A.-G. (B.P. 331,121, 25.7.29).—Carbides, borides, or silicides of tungsten, molybdenum, chromium, uranium, titanium, or vanadium are obtained by melting a finely-powdered intimate mixture of the constituents in an atmosphere of hydrogen and casting in a water-cooled copper mould. The fine-textured alloy is then crushed to a fine powder, which is pressed into rods or shaped bodies, and these are heated at 1750° in hydrogen until completely sintered. A. R. POWELL.

[Nickel] alloys. A.C. SPARK PLUG Co. (B.P. 330,401, 7.6.29. U.S., 1.4.29).—An alloy for use as an electrode in sparking plugs comprises 88–94% Ni, 1.5–2% Mn, 4–10% Cr, 0.4–0.6% Ba. A. R. POWELL.

Cadmium plating. C. H. HUMPHRIES (B.P. 330,289, 5.3.29. U.S., 1.10.28).—A bath for cadmium plating comprises a solution of 1–6 oz. of sulphuric acid, 2–8 oz. of aluminium sulphate crystals, 10–20 oz. of sodium sulphate crystals, 0–8 oz. of ammonium sulphate, 2–4 oz. of cadmium oxide, and 10 g. of glue in 1 gal. of water. A. R. POWELL.

Anti-friction metals and bearings thereof. F. L. BALL, F. MOULTON, and L. W. TOOLS Co., LTD. (B.P. 330,577, 11.3.29).—A mixture of powdered or granulated lead, tin, copper, and antimony together with 3–6% of graphite is compressed into a suitable mould and heated at such a temperature that the metals melt. A. R. POWELL.

Soldering [tungsten, molybdenum, or tantalum]. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 330,403, 10.6.29).—The parts to be joined are sheathed with zirconium and the joint is heated electrically until the zirconium melts. A. R. POWELL.

Blast-furnace tuyères and prevention of burning thereof. E. H. HOLZWORTH (B.P. 331,464, 30.10.29).—See U.S.P. 1,738,901; B., 1930, 244.

Preparation of silicon-steel sheets for electrical purposes. A. F. MURPHY and W. JONES (B.P. 331,511, 2.4.29).—See U.S.P. 1,714,038; B., 1929, 603.

Treatment of nickeliferous material [South African copper-nickel-platinum concentrates]. S. C. SMITH (U.S.P. 1,770,866, 15.7.30. Appl., 18.4.29. U.K., 18.4.28).—See B.P. 311,909; B., 1929, 562.

[Electric] furnace for production of aluminium. E. R. LAUBER (U.S.P. 1,769,298, 1.7.30. Appl., 28.6.26. Ger., 30.7.25).—See B.P. 256,193; B., 1927, 449.

Roasting of [tin] ores and the like. F. L. WILDER, E. MORRIS, E. SCHIFF, and E. S. KING, Assrs. to ELECTRO METALLURGICAL ORE REDUCTION, LTD. (U.S.P. 1,769,405, 1.7.30. Appl., 29.3.28. U.K., 6.4.27).—See B.P. 291,568; B., 1928, 574.

Production of aluminothermic mixture and the like. R. W. HYDE (B.P. 319,775, 19.9.29. U.S., 29.9.28).—See U.S.P. 1,751,550; B., 1930, 719.

[Bell-pump] gas-suction apparatus for mineral furnaces. E. SOMOZA (B.P. 317,727, 17.8.29. Spain, 20.8.28).

Apparatus for heating billets etc. H. A. DREFFEIN (B.P. 302,651, 17.11.28. U.S., 19.12.27).

Furnaces particularly adapted for heat-treatment of railway tyres and similar articles. J. BAKER & Co. (ROTHERHAM), 1920, LTD., and S. E. BAKER (B.P. 330,844, 27.9.29).

Removing iron from aluminium salts (B.P. 318,149).—See VII. Luminous enamels (B.P. 325,072).—See VIII. Electrolytic processes (B.P. 329,308).—See XI. Silver from scrap films (B.P. 331,006).—See XXI.

XI.—ELECTROTECHNICS.

Drying of electrolytic pastes in pocket-lamp dry batteries. H. GRIMM (Z. Elektrochem., 1930, 36, 427–428).—In studying the drying of such pastes an impression of the surface is taken by impressing a filter-paper

coated with carbon black thereon, the surface thus coated being re-impressed on a clean filter-paper. Illustrations of results are given. H. I. DOWNES.

Purifying [pyrites] burner gases with electrostatic precipitators. J. J. HEALY, JUN. (Chem. Met. Eng., 1930, 37, 366—368).—Operating and constructional details are given of an installation of both dry and wet Cottrell cleaners used for purifying the gases (10% SO_2) before they pass through a platinum catalyst. The hot or dry precipitators are provided with nickel-steel wire discharge electrodes and steel-mesh collecting electrodes; they operate at between 440° and 340° and collect about 24 cwt. of dust per day from 28 tons of ore. The cold or wet cleaners have lead-covered copper wire and lead-sheet electrodes; they operate at about 30° and deliver an acid drip (d 1.16) containing 9 g. of As_2O_3 per litre from the first stage and 10 g. of H_2SO_4 and 0.05 g. of As_2O_3 per litre from the third and final stage, the arsenious oxide in the cleaned gas being 0.0013 mg./cub. ft., and after passing through a long flue about half that, an amount which is supposed to be at the danger point, though no ill-effects have been noticed. B. M. VENABLES.

See also A., July, 863, **Electrode potentials of silver in cyanide solutions** (FINK and HOGABOOM). 868, **Electrolytic manufacture of ammonium persulphate** (IZGARISCHEV and GRUZDEVA). 870, **Electrodeposition of metals from their liquid ammonia solutions** (TAFT and BARHAM). 882, **Determination of metals by electrodeposition** (TUTUNDŽIĆ). 883, **Thermoregulators** (BEATTIE and JACOBUS; HIBBEN; WESSON; EGERTON). 919, **Preparation of tri- and tetrachloro-*p*-benzoquinones and *s*-trichloroaniline** (ERDÉLYI).

Heat transfer from tube walls. EAGLE and FERGUSON.—See I. **Ageing of mineral oils.** EVERS and SCHMIDT.—See II. **Refractory furnace linings.** KUKLA; SOMMER.—See VIII. **Steel analysis.** TRZEBIA-TOWSKI.—See X. **Polymerised oils.** HOCK and NOTTE-BOHM.—See XII. **Colour of tanning extracts.** DE LA BRUÈRE.—See XV.

PATENTS.

Electrical induction furnaces. N. R. DAVIS, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 331,324, 8.5.29).—In induction furnaces of the type described in B.P. 323,561 (B., 1930, 334), an earthed, hollow, electrically-conducting screen or grid, cooled by circulating fluid and insulated from the inductor coil, is arranged between this coil and the charge. J. S. G. THOMAS.

Electrolytic processes and baths therefor. M. SCHLÖTTER (B.P. 329,308, 14.2.29).—Metals are electrolytically deposited evenly and a satisfactory current-path dispersion is secured by using a plating bath containing acid sulphonates of the metals, *e.g.*, stannous dihydrogen naphthalenetetrasulphonate, or copper hydrogen benzene-*p*-disulphonate. Gelatin and tannin and copper sulphate are preferably also present for copper-plating. C. HOLINS.

[Electrical] activation of chemical reactions. L. Tocco (B.P. 330,226, 29.1.29. Fr., 7.11.28).—Certain reactions, *e.g.*, the oxidation of sulphur dioxide or

hydrogen sulphide to sulphuric acid, are said to be promoted by the presence of an electric field produced by an A.C. of 25,000—150,000 volts at 25—500 periods.

A. R. POWELL.

Dip for dry-cell cores. BURGESS BATTERY Co., Assees. of J. M. HENDERSON (B.P. 331,467, 31.10.29. U.S., 2.11.28).—Cathodes are dipped in a mixture composed of wood pulp (10—30 pts.) and powdered graphite (100—400 pts.) suspended in water (1000—2500 pts.) containing gelatinised starch (25—200 pts.).

J. S. G. THOMAS.

Manufacture of [porous] electrodes for accumulators. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,080, 14.6.29).—Powdered metal mixed with at least 5% of removable material is sintered, if desired under pressure, and the latter material is removed either partially or wholly. Thus, *e.g.*, a slab containing 70% of nickel powder produced from the carbonyl and 30% of powdered ammonium carbonate is heated for 4 hrs. at 600° , then for 1 hr. at 800° in hydrogen. The resulting plate is impregnated with nickel nitrate.

J. S. G. THOMAS.

[Grids, frames, etc. of] electric accumulators. I. G. FARBENIND. A.-G. (B.P. 311,401, 26.4.29. Ger., 12.5.28).—A hollow body composed of an alloy of lead with an alkali or alkaline-earth metal is completely filled with hard lead worked through an extrusion press. [Stat. ref.] J. S. G. THOMAS.

Treatment of mica, or artificial mica, or substances resembling mica. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,878, 28.6.29).—Single sheets of mica for electrical purposes are covered with mixtures of unhardened resinous condensation products of polyhydric alcohols (*e.g.*, glycols, sorbitol, erythritol), with one or more polybasic organic acids (*e.g.*, phthalic, succinic, or maleic acid) and one or more organic or inorganic vapour-absorbing fillers (such as asbestos, iron oxide, zinc oxide, kieselguhr, tale), after dilution, if desired, with an organic solvent (*e.g.*, acetone). The whole is then hardened by prolonged heating.

S. K. TWEDDY.

Manufacture of thermionic cathodes. SOC. ANON. DES ETABL. IND. DE E. C. GRAMMONT ET DE A. GRAMMONT (B.P. 308,649, 25.3.29. Fr., 24.3.28).—Gold-coated or gold wire, after passing through a bath of mercury, is transferred to an electrolytic bath composed, *e.g.*, of a saturated solution of chloride(s) of alkali or alkaline-earth metal(s), the anode being formed of precious metal(s), *e.g.*, platinum or iridium. The wire, thus coated with amalgam of alkali or alkaline-earth metal, is then heated electrically in hydrogen or other gas until the mercury completely evaporates.

J. S. G. THOMAS.

Protection of [iodised starch] pictures [for wireless-transmission purposes] from the effect of light. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 331,154, 9.10.29).—Pictures are covered with or enveloped in protective material consisting of regenerated cellulose soaked in an aqueous solution of æsculin or of an alkali salt of naphthionic acid.

J. S. G. THOMAS.

Induction furnace. E. F. RUSS (U.S.P. 1,770,662, 15.7.30. Appl., 24.5.28. Ger., 21.8.26).—See B.P. 305,388; B., 1929, 360.

[Mounting of ribbon heating-units in] electric furnaces. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF R. M. CHERRY (B.P. 307,031, 1.3.29. U.S., 1.3.28).

Processes and casting moulds for manufacturing zinc containers for galvanic elements, batteries, etc. A. GASSAN and P. RUCK (B.P. 331,650, 22.5.29).

Production of thin insulating coatings on wires. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 304,192 and Addn. B.P. 306,122, 8.1.29. Ger., [A] 16.1.28, [B] 16.2.28).

[Tubular] electric incandescence lamps. GEN. ELECTRIC CO., LTD., ASSEES. OF PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN (B.P. 331,796, 27.11.29. Ger., 30.11.28).

Testing incandescence lamps etc. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF W. L. KUBACK (B.P. 310,339, 23.4.29. U.S., 23.4.28).

Electric-discharge tubes. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 331,318, 6.5.29).

Electron-discharge device. RAYTHEON, INC. (B.P. 317,398, 6.8.29. U.S., 15.8.28).

Electric-discharge lamps. PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H., ASSEES. OF DEUTS. LEUCHTRÖHREN-GES. M.B.H. (B.P. 317,851, 15.8.29. Ger., 25.8.29).

Cathodes for electron-discharge devices. S. LOEWE (B.P. 306,513, 22.2.29. Ger., 22.2.28).

[Exhausting] thermionic valves. F. A. GOULDING and S. F. GAME (B.P. 331,231, 23.7.29).

[Panel system for] electric heating [of buildings etc.]. F. A. L. BILLEREY (B.P. 331,237, 25.3.29).

X-Ray tubes. INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. OF ALLGEM. ELEKTRICITÄTS GES. (B.P. 315,430, 13.7.29. Ger., 14.7.28).

Oil-immersed X-ray apparatus. VICTOR X-RAY CORP., ASSEES. OF J. B. WANTZ (B.P. 313,568, 13.6.29. U.S., 14.6.28).

Measurement of the rate of flow of fluids by means of electric generators. L. MELLERSH-JACKSON. From SIEMENS & HALSKE A.-G. (B.P. 331,040, 8.5.29).

Removing soot from gases (B.P. 306,043). Energy production from brown coal (G.P. 462,388). Cracking oils (U.S.P. 1,751,148).—See II. Electrolysis of fused halogen salts (B.P. 330,791). Removing iron from aluminium salts (B.P. 318,149).—See VII. Silica bodies (B.P. 330,943).—See VIII. Heating metal melts (B.P. 312,063). Nickel alloys (B.P. 330,401). Cadmium plating (B.P. 330,289). Furnace ladles (B.P. 312,064).—See X. Porous sheets (B.P. 331,197 and 331,227).—See XIV. Treatment of tobacco (B.P. 331,026).—See XX.

XII.—FATS; OILS; WAXES.

Spectrographic analysis of the varying fluorescence of olive oil as observed with ultra-violet light. H. MARCELET and H. DEBONO (Compt. rend., 1930, 190, 1552—1553).—The emission and absorption spectra of various North African olive oils, exhibiting very varying fluorescence, have been examined by the method previously described (cf. Marcelet, B., 1930, 674). The emission spectra show a band at 5700—6900 Å. with sometimes a maximum at 6650—6900 Å.; the absorption spectra bands at 4380—4750 and 5640—6900 Å., with a maximum in some cases towards 6900 Å. Slight differences in the spectra seem to be connected with the varying fluorescence, but are not sufficient to modify sensibly the spectra. C. A. SILBERRAD.

Analysis of Philippine lumbang oil. A. O. CRUZ and A. P. WEST (Philippine J. Sci., 1930, 42, 251—257).—A refined, cold-pressed lumbang oil had: d_4^{20} 0.9170, n_D^{20} 1.4740, saponif. value 191.7, iodine value (Wijs, 6 hrs.) 152.7, unsaponifiable matter 0.34%, oxyacids 0.13%, free fatty acids (as oleic) 0.05%, Wesson colorimeter readings 0.1 red and 1.5 yellow. The composition of the oil is given as: essential oil (cf. Santiago and West, B., 1927, 304) 0.3%; unsaponifiable matter 0.3%; glycerides: of saturated acids 2.1%, of oxidised acids 0.1%, of unsaturated acids [α -]linolenic 7.7%, α -linoleic 14.3%, β -linoleic 4.5%, γ -linoleic 15%, δ -linoleic 27.9% (cf. *loc. cit.*); and of oleic 40.4%.

E. LEWKOWITSCH.

Shea nuts from Nigeria. ANON. (Bull. Imp. Inst., 1930, 28, 123—131).—Botanical specimens from Nigerian shea trees (*Butyrospermum Parkii*); no distinct morphological varieties were observed and 14 samples of nuts from the provinces of Kabba and Sokoto were examined, the proportion of kernel (61.3—84.5%, 57.6—75.5% from the respective districts), the oil content (35.5—44.2%, 33.9—50.2%, on moisture-free kernels), and the unsaponifiable matter in the oil (7.7—11.8%, 5.1—11.0%) being determined. From the results it appears that the percentage of unsaponifiable matter in the oil is the greater, the lower are the percentage of kernels and the yield of oil, although it is unconnected with the average weight of the kernels or the shape or colour of the nuts. It would seem, therefore, that the riper are the nuts, the lower is the amount of unsaponifiable matter in the oil. E. LEWKOWITSCH.

Vitamins in [fatty] oils. F. S. GERONA (Ann. Falsif., 1930, 23, 284—290).—The presence of vitamins in oils can be determined by their absorption spectra. Olive oil has a greater vitamin content than cod-liver oil, and contains vitamin-B as well; it loses its activity when heated to 200°. Natural grape-seed oil is also rich in vitamins, but the refined product, owing to heat-treatment, is inactive. B. W. TOWN.

Drying oils. XIII. Changes in linseed oil, lipase, and other constituents of the flax seed as it matures (1929). E. R. THEIS, J. S. LONG, and G. F. BEAL (Ind. Eng. Chem., 1930, 22, 768—771; cf. B., 1930, 109).—Oils from seeds of various degrees of maturity were analysed, the content of the individual

saturated and unsaturated (Kaufmann-Keller thiocyanometric method; cf. B., 1929, 401) acids being determined. The rapid production of oil in the early stages of growth of the seed (some 17 days) is accompanied by a marked decrease in the activity of the enzymes present and by desaturation of the first-formed glycerides. The oils from the 1929 North-west (mature) crop (Minnesota) showed an abnormally low iodine value (170—175) and high oleic acid content (15—20%), probably owing to the unusually hot, dry weather in the latter part of the season.

E. LEWKOWITSCH.

Drying tests on soya-bean oil. E. MARKOWICZ (Farben-Ztg., 1930, 35, 2078—2079).—Experiments on the drying of soya boiled oils made with various driers are described. Resinate driers cause slow drying and tend to produce re-softening of the film; manganese driers are also unsuitable as they yield turbid or deposit-forming oils. By the use of cobalt or lead cobalt linoleates better boiled oils can be produced which dry in 6—8 hrs., forming films which scarcely re-soften and have good adhesion, but abrade more easily than linseed oil films. The drying properties are much improved by bodying the soya-bean oil to a stand oil in the absence of air.

E. LEWKOWITSCH.

Physico-chemical investigations on electrically polymerised oils (Voltol process). (Heat of dissolution of caoutchouc.) L. HOCK and C. L. NOTTEBOHM (Kolloidchem. Beih., 1930, 31, 185—221).—There are analogies between caoutchouc and multimolecular oils and a study of the polymerisation of linseed oil under the influence of the silent electric discharge has been made. The final product of this treatment is a solid gelatinous product which is insoluble in all the usual solvents and can be purified from other oils by washing with light petroleum. This substance contained about 70% C and 11% H, but a mol. wt. determination was not possible; X-ray examination indicated an amorphous structure. The course of the polymerisation was followed by measurements of refractive index, viscosity, iodine value, and mol. wt. in various solvents. The heats of dissolution in benzene and in menthol were also measured.

E. S. HEDGES.

See also A., July, 856, Systems soaps-cresol-water (ANGELESCU and POPESCU). 867—8, Catalytic reactions at high pressures (MORGAN; DONNAN). 891, Linoleic acids and their oxidation by peracids (SMITH). [Oil from] seeds of *Casalpinia Bonducella* (KATTI; KATTI and PUNTAMBEKAR). Crystalline coloured wax (KUHN and others).

Decolorisation of oils. CHOWDHURY and DAS.—See II.

PATENTS.

Isolation of fatty acids obtained by the oxidation of waxes. J. Y. JOHNSON. From I.G. FARBENIND. A.-G. (B.P. 330,921, 14.3.29).—The oxidation products of waxes are treated with hot concentrated acetic acid, and the insoluble unsaponifiable matter which separates on cooling to not below 15° is removed, the fatty acids being recovered by freezing or by distilling the solution.

E. LEWKOWITSCH.

Refining vegetable and animal fats and oils by removing the free fatty acids therefrom by distil-

lation. METALLGES. A.-G. (B.P. 307,750, 7.3.29. Ger., 12.3.28).—The oil is treated with dilute mineral acid alone, before distilling off the high-boiling fatty acids, which are recovered in a high degree of purity.

E. LEWKOWITSCH.

Manufacture of edible fatty products with a butter-like aroma. N.V. NEDERL. N.V. FRANSCH-HOLLANDSCHE OLIEFABR. NOUVEAUX ETABL. CALVÉ-DELFT (B.P. 301,750, 3.12.28. Holl., 3.12.27).—A minute amount (4 pts. per million) of diacetyl or other diketone is incorporated directly in the fatty product during any stage of the manufacture, or indirectly, by promoting the formation of butylene glycol, acetyl-methylcarbinol, etc. during the souring of milk and oxidising these compounds to the corresponding diketones.

E. LEWKOWITSCH.

Manufacture of oils which are soluble in mineral oils from castor oil. I. G. FARBENIND. A.-G. (B.P. 317,391, 14.3.29. Ger., 15.8.28).—Castor oil, or a mixture thereof with mineral oil, is heated at about 250—320° in the presence of an inert catalyst, e.g., oxides of aluminium, tungsten, titanium, etc., or sulphates, phosphates, silicates, etc., which split off water; the volatile products are preferably removed during the reaction, e.g., by a stream of carbon dioxide.

E. LEWKOWITSCH.

Detergent composition of oils. H. MENZ, W. STEFFEN, and E. JAAKS-MÜNCHENBURG (B.P. 331,034, 30.4.29).—The composition consists of a sulphonated vegetable oil (e.g., castor oil) mixed with turpentine oil, a hydrogenated hydrocarbon, and pine-needle oil.

H. ROYAL-DAWSON.

Preparation of fine granules of soap. COLGATE-PALMOLIVE-PEET CO. (B.P. 314,483, 18.2.29. U.S., 28.6.28).—Fine soap particles from a shredder are dried by falling through a counter-current of hot air (cf. B.P. 314,482; B., 1930, 725), deposition on the shredder being prevented by a fan suitably arranged to reverse the air current locally. At the lower end of the drying chamber the air inlet comprises a series of ports which are adapted to impart a centrifugal motion to the entering hot air, and are kept clear by a rotary cleaning device.

E. LEWKOWITSCH.

Converting liquid soap into a rapidly soluble, handy form. A. WELTER (B.P. 331,268, 26.3.29).—The dry powder produced by atomising a liquid soap (containing soda, silicate, etc., as required) is rendered homogeneous in roller mills and shaped either to threads by extruding presses or to soap-wool, flakes, etc. by rollers in the usual way.

E. LEWKOWITSCH.

Detergent alcoholic soap or cream. G. MAZZETTI (B.P. 317,819, 6.4.29. It., 23.8.28).—A mixture of alcohol with motor spirit or benzene, light petroleum, turpentine, etc., is added to a potash-coconut oil soap containing additional glycerin.

E. LEWKOWITSCH.

Recovery of grease or fat [from wool-scouring liquors]. F. E. BAILEY (U.S.P. 1,770,476, 15.7.30. Appl., 22.8.23. Austral., 19.9.22).—See B.P. 204,326; B., 1925, 325.

Manufacture of margarine. A. K. EPSTEIN (B.P. 331,562, 5.2.29).—See U.S.P. 1,707,800; B., 1929, 482.

Water-soluble substances (B.P. 329,622).—See III. Treatment of non-fibrous materials (B.P. 330,649).—See V. Deodorising etc. of butter fat (B.P. 330,662). Production of plastic substances (B.P. 330,887). Salad cream (B.P. 330,708).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Measurement of colour-tone. W. VAN WÜLLEN-SCHOLTEN (Farben-Ztg., 1930, 35, 1824—1825).—In examining the use of the Zeiss step-photometer for colour-tone determinations in pigments and varnishes, dry mixtures of lead-white and lampblack are made up into a series of grey varnishes and the tones obtained are analysed. Addition of 0.1 g. of lampblack to 100 g. of lead-white, *e.g.*, gives a grey containing 70% black tone, 0.7 g. 90%, 2.1 g. 94.5%, and 7.0 g. 97.5%. The standard tones in railway colour charts used for testing colour-blindness are found to have 1—5% colour content (green, blue, etc.), and this varies according to the source of the pigments employed. The use of the Zeiss photometer for the standardisation of pigments, especially those intended for greys, by manufacturers is recommended. C. HOLLINS.

Yellowing of interior gloss paints and enamels. S. WERTHAN, A. C. ELM, and R. H. WIEN (Ind. Eng. Chem., 1930, 22, 772—776).—A preliminary report. Moisture and a temperature above 0° are necessary for the reactions which lead to the formation of yellow compounds, but yellowing is independent of atmosphere since the same degree of discoloration occurred in ozonised air, in hydrogen, and in nitrogen. Red light produces a greater discoloration than green or blue, and infra-red radiations have the greatest effect of all. Purification of the oil does not eliminate yellowing, which is also independent of the free acid content. The most unsaturated oil shows the greatest discoloration. L. S. THEOBALD.

Lac industry in India. (Miss) D. NORRIS (J. Oil Col. Chem. Assoc., 1930, 13, 195—199).

See also A., July, 883, Protection of ventilating fans (DREIFUSS and STAAB). 913, *Cannabis Indica* resin (CAHN). 920, Nitration of abietic acid (GOLD-BLATT).

Disintegration. CHWALA. Adhesives and adhesion. LEE.—See I. Drying tests on soya-bean oil. MARKOWICZ.—See XII.

PATENTS.

Manufacture of oil paint. M. W. SAUNDERS and J. J. HYNES (B.P. 330,584, 6.3.29).—An oil paint containing heavy-metal soaps as driers (*e.g.*, lead linoleate and/or resinates) is heated to about 100° and mixed with a hot, aqueous, saline solution (*e.g.*, 4 oz. of sodium or magnesium chloride per gal.) which will form an emulsifier by double decomposition with the drier. E. LEWKOWITSCH.

Production of dull-decoration paints ready for painting. A. RÖHRMANN (B.P. 330,673, 25.3.29).—A matrix consisting of 75 pts. of a mixture of waxes, 125 pts. of a copal varnish-wood oil solution (41 pts. of copal, 20 pts. of wood oil, 39 pts. of solvent), 300 pts. of a linseed oil-wood oil solution (similar proportions,

linseed oil replacing the copal), 485 pts. of solvent (*e.g.*, turpentine), 10 pts. of driers, and 5 pts. of glycerin is mixed with pigments at about 100°.

E. LEWKOWITSCH.

[Dehydration of] pigments. E. C. DE STUBNER (B.P. 330,917, 13.12.28. Cf. B.P. 277,949; B., 1929, 365).—Wet pigments, other than those which have been precipitated in aqueous media, are washed with a non-aqueous liquid miscible with water until the liquid has completely replaced the water. For example, ethyl alcohol, acetone or pyridine, and alcohol or acetone are used for pigments for the manufacture of lacquers, printers' inks, and cellulose acetate, respectively. L. A. COLES.

Manufacture of [aluminium] lakes or pigments.

I. G. FARBENIND. A.-G. (B.P. 307,436, 7.3.29. Ger., 7.3.28).—Colour lakes are obtained by boiling the absorption complex of a basic or acid dye on aluminium hydroxide, silicate, or phosphate with the reduction products of complex phosphotungstomolybdic acids obtained by the process described in B.P. 292,253 (B., 1928, 603). A. R. POWELL.

Transfer and marking composition therefor.

KAUMAGRAPH CO. (B.P. 331,135, 5.9.29. U.S., 19.4.29).—The composition comprises a mixture (*e.g.*, "Amberol G 8") of a phenol-formaldehyde condensation product with a more fusible resin, together with a fusible wax (*e.g.*, beeswax) and a blown oil (*e.g.*, blown rapeseed oil). L. A. COLES.

Manufacture of pearl essence. J. PAISSEAU (B.P. 308,969, 26.3.29. Ger., 2.4.28).—Natatory bladders are pickled in an acid medium, *e.g.*, water containing 1—5% of tartaric or formic acid, in the presence of sufficient common or other neutral salt to prevent the decomposition of the conjunctive tissues. A detergent material, *e.g.*, saponin, is added, the temperature is raised to 30—60° or above for a suitable period, after which a proteolytic or lipolytic ferment is added at 45° or less. The unattacked pearl essence crystals are removed by settling, centrifuging, etc. S. S. WOOLF.

Production of lac [lacquer] products. J. SCHEIBER (B.P. 316,538, 13.2.29. Ger., 30.7.28).—Ricinoleic acid or elaidoricinoleic acid is distilled in a vacuum until 70—80% has passed over in the form of octadecadienoic acid. The residual viscous liquid or its glyceryl ester is a quick-drying varnish, and may usefully be combined with glyceryl octadecadienoate to give a product similar in properties to boiled wood oil; for this purpose the residue and distillate may be mixed and esterified together with glycerol. The residue may also be incorporated with cellulose esters. C. HOLLINS.

Manufacture of stoving lacquers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,954, 22.11.28).—Residues from the distillation of oxidised paraffin waxes, especially residues from products of B.P. 322,437 (B., 1930, 155) in which hydroxycarboxylic acids are present and acids up to C₈ have been distilled off, are dispersed or dissolved in organic diluents, with or without other suitable film-forming substances, and applied as stoving lacquers. Preferably the residues are first heated alone or with a polymerising agent, such as chlorine or sulphur. C. HOLLINS.

Manufacture of lacquers, filling or priming compositions, and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,910, 14.3.29. Cf. B.P. 322,537; B., 1930, 157).—Coating compositions are claimed comprising nitrocellulose and soluble resinous condensation products of aromatic dibasic acids, *e.g.*, phthalic acid, with polyhydric alcohols containing 4–6 hydroxyl groups, *e.g.*, sorbitol or pentaerythritol, incompletely esterified by organic monobasic acids of natural origin and of non-drying character, *e.g.*, lauric, ricinoleic, or abietic acid, and solvents; softeners, fillers, and colouring materials may be added. S. S. WOOLF.

Manufacture of lacquers, filling or priming compositions, and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,909, 14.3.29. Addn. to B.P. 322,466; B., 1930, 157).—Coating compositions are claimed which comprise nitrocellulose and soluble resinous condensation products of aliphatic dibasic acids, *e.g.*, succinic or diglycolic acid, with polyhydric alcohols, *e.g.*, glycerol or glycols, incompletely esterified by high-molecular, aliphatic, monobasic acids of non-drying character, *e.g.*, lauric, ricinoleic, or abietic acid; and solvents; softeners, fillers, and colouring materials may also be added. S. S. WOOLF.

Preparation of coatings. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,897, 16.3.29).—Water-insoluble cellulose derivatives are finely dispersed, mechanically, in water and mixed with aqueous emulsions of assistants (dissolving, softening, or gelatinising agents, or mixtures of these, in amounts which are insufficient for homogenising the mixture). The assistant emulsions are preferably prepared with the use of protective colloids, *e.g.*, gelatin or methylcellulose, or of wetting or emulsifying agents, *e.g.*, alkyl-naphthalene-sulphonic acid salts, resin soaps, etc. Colouring materials and/or rubber latices may be added. The coating material thus formed may be sprayed on to substrata and subjected, if desired, to after-treatment. S. S. WOOLF.

Organic liquid coating compositions [pyroxylin varnishes]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 330,735, 17.5.29).—The varnishes contain at least one organic film-forming substance, *e.g.*, pyroxylin, resin esters, linseed oil, and a solvent containing a ketone or mixture of ketones containing an aliphatic branched-chain connected with the carbonyl group, such as are obtained by the process described in B.P. 310,438 (B., 1929, 635). *E.g.*, the varnish may consist of 50 pts. of pyroxylin, 10 pts. of butyl phthalate, and 30 pts. of castor oil dissolved in 500 pts. of the ketones having b.p. 120–170°, 300 pts. of toluene, and 100 pts. of light petroleum. A. R. POWELL.

Plastic or coating compositions. SELDEN CO., Assees. of A. O. JAEGER (B.P. 312,636, 6.5.29. U.S., 29.5.28).—Phthalide, hydrogenated phthalides, their alkylated, arylated, or alkylidene derivatives are used as plasticisers for synthetic resins of all types. C. HOLLINS.

Preparation of solutions or plastic masses. BRIT. CELANESE, LTD. (B.P. 303,152, 14.12.28. U.S., 29.12.27).—The solid constituent is treated with the

vapour of a solvent (or solute) before mixing with a liquid solvent (or solute), which is the same as, or easily miscible with, that used in the vapour treatment; the process is particularly applicable to the production of solutions etc. of cellulose derivatives. E. LEWKOWITSCH.

Synthetic resin varnishes and synthetic resins. IMPERIAL CHEM. INDUSTRIES, LTD., H. H. MORGAN, and A. A. DRUMMOND (B.P. 329,335, 13.11.28).—Partly esterified or etherified polyhydric alcohols (glycol, glycerol, or erythritol) are condensed with a polybasic acid (phthalic) anhydride in presence of a non-hydroxylated fatty ester of glycerol (preferably a drying oil) and in presence or absence of a solvent. Phthalic anhydride, *e.g.*, is heated at 220–230° with glycerol monobenzoate and wood oil, with α -chlorohydrin and wood oil, or with glycerol monobenzoate, tung oil, and tetrahydronaphthalene. C. HOLLINS.

Production of shaped articles from urea or its derivatives, solid polymerised aldehydes, and fillers. BAKELITE GES.M.B.H. (B.P. 305,211, 28.1.29. Ger., 3.2.28).—The components are mixed with one another in the absence of water or volatile liquids and without heating, and the mixture is hot-pressed with no intermediate treatment. Natural or artificial resins, aldehyde-binding substances, *e.g.*, *p*-toluenesulphonamide, or water-binding substances, *e.g.*, burnt gypsum, dyes, fire-resistant materials, etc., may be incorporated if desired. S. S. WOOLF.

Moulding mixtures [comprising phenol-formaldehyde condensation products]. H. WADE. From BAKELITE CORP. (B.P. 330,968, 20.3.29).—A phenolic resin is kneaded, *e.g.*, at 100°, with a mixture of a filler and a high-boiling aldehyde (*e.g.*, furfuraldehyde), with the addition, when a non-reactive resin is used, of a methylene compound (*e.g.*, hexamethylenetetramine). L. A. COLES.

Manufacture of (A) shellac-like product and (B) artificial shellac. A. W. BURWELL, Assr. to ALOX CHEM. CORP. (U.S.P. 1,770,875–6, 15.7.30. Appl., [A] 20.12.26, [B] 2.1.28. U.K., [A, B] 6.10.27).—See B.P. 309,384; B., 1929, 651.

Compositions for use in marking lines in roads and tennis courts. W. T. BELL and J. F. BENNETT (B.P. 331,207, 23.3.29).

[Flotation method of] surfacing articles with films comprising pigments, varnishes, glazes, enamels, lacquers, etc. G. M. BROWN and H. T. WYSE (B.P. 331,201, 19.2. and 21.5.29).

Azo pigments (B.P. 306,415 and 329,960).—See IV. **Soluble acylcelluloses** (B.P. 305,674). **Artificial silk** (B.P. 305,468). **Treatment of non-fibrous material** (B.P. 330,649).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Structural changes during the processing of rubber. E. A. GRENQUIST (Ind. Eng. Chem., 1930, 22, 759–765).—Plasticity values of raw rubber based on flow and recovery, *e.g.*, in a Williams plastometer, are probably regulated mainly by the state of aggregation of the rubber and the degree of breakdown and fusion

of the rubber globules. Cold milling causes disaggregation of the hydrocarbon and breakdown and fusion of the globules, the resulting softness being permanent. Heating in air causes partial breakdown and fusion of the globules together with thermal disaggregation and oxidation. At temperatures up to 164° the presence of oxygen appears to be necessary for breakdown and fusion of the globules; heating in steam or carbon dioxide leaves the globular structure almost unaltered, and, as the plastic changes are more or less reversible so long as the globular structure remains, the effect of such heating is only temporary, re-aggregation occurring gradually. In the absence of air, rubber exhibits a maximum degree of disaggregation at any temperature. Ultra-violet light concurrently affects the plastic and also the elastic properties of the rubber, both plastic flow and recovery being increased. There exists a correlation between elasticity and double refraction of rubber, the anisotropy at 300% elongation falling parallel to the recovery values after deformation in the plastometer. No agency in this investigation caused such far-reaching and profound destruction of the elastic properties of rubber as prolonged mastication.

D. F. TWISS.

Vulcanisation of rubber. I. Thermochemistry of vulcanisation of rubber. II. Vulcanisation of rubber with nitro-compounds. III. Kinetics of vulcanisation of rubber with sulphur and selenium. J. T. BLAKE. IV. Theory of vulcanisation of rubber. C. R. BOGGS and J. T. BLAKE (Ind. Eng. Chem., 1930, 22, 737—740, 740—744, 744—747, 748—755).—I. By careful measurement of the heat of combustion of a rubber-sulphur mixture before and after vulcanisation and taking precautions to avoid premature atmospheric oxidation of the vulcanised material, it is found that combination of rubber with sulphur up to about 6% occurs without any appreciable energy change in the system. From this point onwards, however, the combination with sulphur is accompanied by liberation of heat, complete formation of vulcanite being attended by a maximum liberation of 442 g.-cal. per g. of rubber. With the additional presence of diphenylguanidine as vulcanisation accelerator, together with zinc oxide, heat evolution is observable even at approx. 4% of combined sulphur. The formation of soft vulcanised rubber and of vulcanite are distinct processes, only the latter involving thermochemical change. Vulcanisation with *m*-dinitrobenzene or selenium does not involve any heat evolution.

II. Vulcanisation of rubber with di- or tri-nitrobenzene involves a definite chemical change, a change in density occurring during the process and a proportion of the vulcanising agent becoming insoluble in acetone. The reaction is one of the first order, and probably comprises a rapid primary combination of rubber and nitro-compound to form a loose molecular compound which undergoes subsequent slower conversion into the vulcanised product. The equivalent weight of rubber per nitro-group in the final product from either di- or tri-nitrobenzene is approx. 1315.

III. Vulcanisation of rubber by selenium is a reaction of the first order. The maximum coefficient of vulcanisation obtainable is about 5.1%, corresponding

with a sulphur coefficient of 2.04%, and with an at. wt. of selenium per 1560 pts. of rubber. No vulcanite product was obtainable from rubber and selenium even on prolonged heating or with the aid of an organic accelerator. The second stage of the combination between rubber and sulphur, which gives rise to vulcanite, is a reaction of the second order and, in any one molecule, occurs after the reaction yielding soft vulcanised rubber. Vulcanisation accelerators expedite the formation of the soft vulcanised product, but have no appreciable influence on the subsequent reaction yielding vulcanite.

IV. All the phenomena observed in vulcanisation are explained by the theory that vulcanisation is an additive chemical reaction capable of yielding two stable products, viz., soft vulcanised rubber and hard vulcanite. The formation of the two products occurs successively in any one molecule during exhaustive vulcanisation by sulphur. Certain vulcanising agents effect only the first stage of the process, and the influence of accelerators is substantially limited to this stage. There are two types of chemical unsaturation in the rubber molecule; after satisfying the valencies responsible for the production of soft vulcanised rubber, further addition of sulphur at first gives rise to an unstable type of material which may be described as "intermediate vulcanite"; the deterioration of ordinary soft vulcanised rubber with age is due to the presence in it of a proportion of molecules of the "intermediate vulcanite" type. There is a definite relation between the physical properties and the proportion of combined sulphur only when "intermediate vulcanite" is not present extensively. The electrical properties of vulcanised rubber are in accord with a view that in the formation of vulcanite the sulphur adds progressively from one end of the rubber molecule to the other, thus giving rise to dipoles which have a maximum effect when the reaction is about half completed. In the "reclaiming" of rubber, the vulcanised rubber molecule undergoes fission into two portions, viz., an insoluble fraction containing practically all the combined sulphur, and a soluble, sulphur-free fraction. The physical properties of ordinary soft vulcanised rubber are determined by the relative proportions of pure soft rubber and "intermediate vulcanite" present, the best product being obtained when the latter is at a minimum. Vulcanisation accelerators, by affecting mainly the reaction yielding the soft rubber constituent, lead to relative reduction of the proportion of "intermediate vulcanite," and so result in a product of superior physical properties, but with a lower total proportion of combined sulphur. For the production of pure soft rubber, the proportion of combined sulphur necessary would be approx. 0.5%, which corresponds with a composition $(C_5H_8)_{200}S_2$.

D. F. TWISS.

Determination of copper in fabrics. KLICKOW and SIEBNER.—See V. **Heat of dissolution of caoutchouc.** HOCK and NOTTEBOHM.—See XII.

PATENTS.

Manufacture of rubber. SOC. ITAL. PIRELLI, and U. PESTALOZZA (B.P. 330,520, 6.3.29. Addn. to B.P. 284,608; B., 1929, 612).—The addition of an aromatic disubstituted guanidine, e.g., *s*-diphenylguanidine,

renders latex capable of thickening and of coagulation in regions where localised heating, *e.g.*, to 70–95°, is applied. Small quantities of other substances such as ammonium salts, or salts of bi- or ter-valent metals may expedite the thickening and coagulating process. The rubber layer or article produced, even in the presence of sulphur, is unvulcanised, but may be vulcanised subsequently.

D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER CO., LTD., C. HAYES, E. W. MADGE, and F. H. LANE (B.P. 330,536, 8.3.29).—The addition of excess of formaldehyde, to natural or artificial latex of rubber or similar materials, in excess of that necessary to neutralise the free alkalinity such as ammonia, leads to the formation of a continuous coagulum or gel in a definite and controllable time. The higher the ammonia content of the latex the greater is the necessary excess of formaldehyde to produce a continuous coagulum in a given time at room temperature. For treatment of a non-compounded latex containing more than 0.05% of ammonia and concentrated by a centrifuging process, it is necessary also to introduce a small proportion of a sensitising agent such as ammonium sulphate, zinc sulphate, or an ammonium salt of an organic acid, *e.g.*, oxalic, tartaric, or citric acid. Emulsions or dispersions so treated are applied to manufacturing purposes by such processes as spreading, dipping, spraying, or moulding.

D. F. TWISS.

Manufacture of rubber-like products. I. G. FARBERIND. A.-G. (B.P. 307,937—8, 15.3.29. Ger., [A], 16.3.28, [B] 17.3.28).—(A) Rubber-like products are obtained by polymerisation of diolefines in the form of an emulsion which has previously been forced through a homogeniser. (B) An aqueous solution of a material promoting emulsification, *e.g.*, sodium oleate, and a solution in a diolefine of an emulsion-promoting material, *e.g.*, magnesium oleate, are emulsified together. Emulsification is thereby facilitated and the yield of rubber-like product improved.

D. F. TWISS.

Polymerisation of hydrocarbons of the butadiene series. I. G. FARBERIND. A.-G. (B.P. 315,356, 1.7.29. Ger., 12.7.28).—Polymerisation of butadiene hydrocarbons with the aid of sodium hydride is slower than with metallic sodium, which sometimes induces polymerisation with almost explosive violence, and the product is more homogeneous. The process is conveniently performed in the presence of an indifferent solvent, such as benzene, with agitation.

D. F. TWISS.

Improving synthetic rubber-like substances. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 330,922, 16.3.29).—Polymerisation of butadiene, its analogues or homologues sometimes leads to rubber-like products which, however, are insoluble in benzene and do not readily become plastic when milled. These undesirable features can be removed by treating with an organic nitro-compound, *e.g.*, mono-, di-, or tri-nitrobenzene, advantageously in the presence of a solvent or diluent, such as benzene and at an elevated temperature, *e.g.*, 140–150°.

D. F. TWISS.

Manufacture of rubber solutions. F. KEMPTER

(B.P. 330,953, 22.2.29).—The duration of the process of preparing rubber solution is shortened by stirring the rubber with a solvent in a trough having an irregularly surfaced wall and one or more stirrers, desirably also provided with irregularities.

D. F. TWISS.

Vulcanisation of rubber. RUBBER SERVICE LABS. Co., Assees. of W. A. MOORE (B.P. 330,981, 22.3.29. U.S., 12.11.28).—Rubber stock containing all the desired compounding ingredients with the exception of the vulcanisation accelerator is vulcanised by immersion in a hot aqueous dispersion (*e.g.*, at 95–100°) of a water-insoluble ultra-accelerator, such as the carbon disulphide derivative of the reaction product of piperidine and formaldehyde, the dispersion being stabilised by the presence of an alkali salt or "triethanolamine" salt of a fatty acid, gum arabic, starch, or saponin.

D. F. TWISS.

Manufacture of resistant porous sheets. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 331,197 and 331,227, 24.1.29).—(A) Chemically-resistant porous sheets suitable for diaphragms in electrolytic cells are produced by incorporating a non-distillable polymerisation product of butadiene, or a mixture thereof with india-rubber, with a porous substance such as coarse kieselguhr; compounding ingredients of the customary types, including fillers, vulcanising agents, and anti-oxidants, may also be added, and the sheets may be vulcanised. If desired, the sheets may be reinforced, *e.g.*, with porous fabrics or metal gauze. (B) Instead of using a porous substance as in (A), a substance capable of forming pores by dissociation into gases or vapours, *e.g.*, ammonium carbonate, or by dissolution in an inert solvent, *e.g.*, common salt, is incorporated in the mixture, the treatment otherwise being as described.

D. F. TWISS.

Manufacture of articles from aqueous dispersions containing rubber, gutta-percha, balata, and similar resins. DUNLOP RUBBER CO., LTD., G. W. TROBRIDGE, and E. A. MURPHY (B.P. 330,970, 21.3.29).—In the manufacture of articles from aqueous dispersions of rubber and similar materials by such processes as dipping, spreading, or spraying, removal of the product from the mould or former is facilitated by providing the surface of the latter with a film of a dehydrating and setting liquid (cf. B.P. 303,544; B., 1929, 220), prior to introduction into or contacting with the aqueous dispersions. The process of dipping in the dispersions may be repeated, if desired, with alternate dipping into the dehydrating and setting fluid. Alternatively, or finally, the deposit or deposits may be treated to develop a wrinkled or roughened surface, or may be simply dried and vulcanised.

D. F. TWISS.

Electrodeposition of rubber. W. A. WILLIAMS (U.S.P. 1,769,659, 1.7.30. Appl., 12.5.27. U.K., 28.5.27).—See B.P. 293,095; B., 1928, 649.

Thickening and treatment of latex. S. D. SUTTON, Asst. to VEEDIP, LTD. (U.S.P. 1,770,092, 8.7.30. Appl., 27.3.28. U.K., 2.1.28).—See B.P. 312,947; B., 1929, 652.

Mercaptobenzthiazoles (B.P. 306,492).—See III. Coated paper (U.S.P. 1,746,888).—See V.

XV.—LEATHER; GLUE.

Influence of neutral salts on [raw] hide. M. BERGMANN (J. Soc. Leather Trades' Chem., 1930, 14, 307—312).—The volume of water forced through raw skin in unit time under a pressure of 1 atm. was determined and is termed the "primary water permeability." Salt solutions of different concentrations (0.01—0.4*N*) were similarly forced through the same pieces of skin to determine the "salt permeability," after which water was again forced through and the "secondary water permeability" determined. The salt and secondary water permeabilities expressed as percentages of the primary water permeability are found to depend on the salt concentration used. The salt permeability was less than the primary water permeability, due to the shrinkage of the pores by the action of the salt. Except for 0.01*N*-sodium chloride, the secondary water permeabilities were less than the primary. Similar tests with other chlorides did not show any gradation in accordance with the Hofmeister series. Tests with sodium salts of different anions showed diminished permeabilities which did not recover on elimination of the salt from the pelt. The original permeability of pelt which had been treated with 0.00625*N*-solutions was restored by eliminating the salt. It was found that the permeability of salt solutions decreased much more when the salt was forced through from the grain than from the flesh. The reverse effect was obtained for potassium chloride. This effect was not due to the grain layer, since it was repeated on pelt from which the grain layer had been removed.

D. WOODROFFE.

Qualitative analysis of tannins. L. POLLAK (J. Soc. Leather Trades' Chem., 1930, 14, 299—307).—Solutions of tanning extracts are prepared in ammonia and ammonium sulphhydrate of definite concentrations and examined spectroscopically with the instrument arranged vertically over a 50-c.p. Philips lamp. A cell containing the tannin solution is fixed between the instrument and the lamp and wave-length measurements are taken at every 5 mm. in depth until total extinction is obtained. The wave-length-depth of liquid curves obtained are characteristic for the different vegetable tanning materials. Magnesium sulphate must be removed from extracts before treating them with ammonia, but they cannot be examined in ammonium sulphhydrate solution. It has been shown that a mixture of a pyrocatechol tannin and 25% of sulphited quebracho extract or 75% of sulphite-cellulose extract fails to give a definite reaction in the Stiasny formaldehyde-hydrochloric acid test. By adding 1 g. of urea, however, a definite reaction was obtained for pyrocatechol tannins even in the presence of 10% of sulphited quebracho extract or 90% of sulphite-cellulose extract. A high percentage of precipitate was obtained when this modified formaldehyde-hydrochloric acid test was applied to Neradol D, but not with other synthetic tannins. D. WOODROFFE.

Measurement of colour of tanning extracts. II. Photocolorimeter method. A. DE LA BRUÈRE (J. Soc. Leather Trades' Chem., 1930, 14, 315—316; cf. B., 1929, 140).—In using the Toussaint photoelectric colorimeter, not only the voltage supplied to the lamp but

that of the accumulators connected to the photoelectric cell must be kept constant. Electrical leaks must be avoided. Photocolorimetric measurements of different tannin extracts are recorded. The error caused by calculating tintometer measurements of tannin solutions of analytical concentration to 0.5% of tannin has been confirmed.

D. WOODROFFE.

Effect of the particle size of kaolin and P.H.A.S. on the filtration of tannin solutions by the contact method. A. DE LA BRUÈRE (J. Soc. Leather Trades' Chem., 1930, 14, 313—314).—The insoluble matter and soluble tans were determined in a number of different extracts, using kaolins and "P.H.A.S." of different particle size, respectively. No variation in the results was observed, thus disagreeing with Jamet and Girard's results (B., 1928, 721).

D. WOODROFFE.

Water-resisting properties of glue and casein glue. I, II. T. ARAKI and T. MATSUNO (J. Soc. Chem. Ind., Japan, 1929, 32, 183—184 B, 184 B).—I. An automatic recording device has been employed for determining the time after which a glued joint yields when immersed in water at constant temperature (about 20°) and subjected to a constant load. In general, glues of good quality are more resistant to the action of water than are commercial casein glues, although the results obtained for the former group vary more widely among themselves; liquid glues are considerably inferior as regards their power of resisting the action of water.

II. The resistance of glue to water is considerably increased by treatment with a mixture of paraldehyde and oxalic acid, or if the material to be joined is first treated with formalin; casein glues are improved in like manner by admixture with a small quantity of a solution of a copper salt.

H. F. GILLBE.

Diffusion of oakwood and chestnut extracts into a gelatin jelly. A. DE LA BRUÈRE (J. Soc. Leather Trades' Chem., 1930, 14, 296—298).—Test-tubes partially filled with a 2% gelatin jelly were covered with the solution of the extract and kept at 15°. The chestnut extracts penetrated further than did the oakwood, and showed a marbled effect, whilst the oakwood diffused with a brownish appearance. Diffusion tests made on solutions at different p_H values showed that the marbled effect was produced with oakwood extracts at low p_H values, and was no longer shown by chestnut extracts at higher p_H values.

D. WOODROFFE.

Wetting etc. agents (B.P. 308,824). Water-soluble substances (B.P. 329,622).—See III. **Azo dyes for chrome leather (B.P. 313,927).**—See IV. **Solutions of animal proteins (B.P. 306,464).**—See V.

XVI.—AGRICULTURE.

Properties of organic hardpan soils with special reference to their formation. L. A. RICHARDSON (Soil Sci., 1930, 29, 481—488).—Less clay and silt occurs in hardpan soils than in non-hardpan soils. The reactions were almost identical. In the hardpan horizon there is a definite accumulation of aluminium, but not of calcium or iron. The C/N ratio in non-hardpan soils decreased from the surface downward, but was almost constant in the hardpan layer. In

Florida the formation of an organic hardpan is favoured by a loose sandy soil, low in silt and clay and free of electrolytes, and with a water-table near the surface during part of the year. A. G. POLLARD.

Podsols and moor soils of Russia and the Baltic states. H. VON RATHLEF (Z. Pflanz. Düng., 1930, 17A, 201—226).—The formation, flora, and chemical and physical properties of certain Russian soils are described. A. G. POLLARD.

Correlations between the specific conductivities of soil extracts, nitric nitrogen, and soluble calcium. C. H. WRIGHT (8th Ann. Bull. Agric. Dept., Nigeria, 1929, 104—109).—Periodical sampling and analysis of cropped soils show that the calcium and nitrate contents of 1 : 5 soil-water extracts are closely correlated with their specific conductivities. Increased conductivity is evidence of biological activity in the soil. A. G. POLLARD.

X-Ray and microscopical examinations of soil colloids. S. B. HENDRICKS and W. H. FRY (Soil Sci., 1930, 29, 457—479).—The finer fractions of soils contain crystalline materials. Samples give characteristic powder diffraction patterns which can be identified as arising from one of the clay minerals. Montmorillonite-beidellite, Ordovician bentonite, and halloysite are common constituents of soil colloids. Bauxite was also found. The finer fractions from specific soil types in different localities give the same type of diffraction pattern. A. G. POLLARD.

Can the laboratory examination of a soil give sufficiently definite information as to its lime requirement? A. GEHRING (Z. Pflanz. Düng., 1930, 9B, 241—245).—Laboratory determinations of the lime requirement of soils give satisfactory information for practical purposes and, moreover, save much time. Mitscherlich's plea for field trials is criticised. A. G. POLLARD.

Equilibrium between soil and electrolytes and its influence on some lime requirement methods. N. A. CLARK and E. R. COLLINS (Soil Sci., 1930, 29, 417—427).—Comparative electrometric titrations were made of soil suspensions in water and in calcium chloride solution with lime water and in sodium chloride solutions with sodium hydroxide. Equilibrium is attained after varying periods with different soils, but little change occurs after 90 hrs. In general, equilibrium is reached more rapidly in the salt solutions. The lime requirement measured after 90 hrs. is much higher in salt suspensions (2—5 times) than in aqueous suspensions. The bearing of these results on Christensen and Jensen's measurements (lime requirement = $3 \times$ direct lime titration; cf. B., 1927, 309) is discussed. The suggested instantaneous neutralisation occurring in lime titrations (cf. Saint, B., 1927, 309) is not confirmed. A. G. POLLARD.

Chemistry of mineral soil acidity. II. Chemistry of exchange acidity in soils. III. M. TRÉNEL and J. WUNSCHIK (Z. Pflanz. Düng., 1930, 17A, 257—296, 296—305; cf. B., 1930, 254).—II. Permutit was treated with a number of acids of varying concentration and the extent of the removal of bases and of the decomposition of the permutit was examined. With the progressive

removal of bases the permutit is gradually decomposed into its constituents. Probably no "permutit acid" exists. When bases are partially removed, permutit behaves as a mixed gel of the hydrates of the sesquioxides and of silica. Silicic acid remains after complete removal of the bases. The action of acids on permutit depends on the nature of the acid, its concentration, and the temperature; 0.1N-inorganic acids completely decompose permutit. With lower concentrations decomposition of the permutit begins when about half of the bases have been removed. Solutions of aluminium chloride and acetic acid remove bases from permutit to a similar extent to inorganic acids of corresponding concentrations, but differ in having much weaker decomposing effects. Oxalic acid is distinguished by its ability to remove large amounts of silica from permutit. Water containing carbon dioxide removes bases from permutit, but cannot decompose it. During electrodialysis permutit is similarly decomposed and an acid-reacting silicic acid sol appears in the middle chamber. Exchange acidity depends on the degree of decomposition of the permutit, on the dryness of the residual substance, and on the temperature and concentration of the neutral salt solution. It is apparent in *N*-potassium chloride at room temperature when about half of the bases have been removed. Completely decomposed permutit (essentially silica) shows small exchange acidity. At higher temperatures (85°) the acidity is increased and the silica and aluminium hydroxide are peptised. Drying the gel mixture at 150° after removal of bases largely reduces its reactivity. Methyl-red indicates smaller acidity values than phenolphthalein, either because two dissociating substances are present in permutit, or because exchange acidity is not a single-factor phenomenon. Probably peptised silica is titrated when phenolphthalein is used. The amount of iron and aluminium oxides in potassium chloride extracts of permutit is practically the same as that in acid extracts. Potassium chloride appears to peptise the gel. The term "exchange" acidity is inappropriate since the phenomenon is not a direct exchange either of hydrogen or of aluminium ions. Exhausted permutit residues have no absorbed acids to which "exchange" acidity might be attributed. Examination of soil gave results similar to the above. The acidity of potassium chloride extracts is always associated with the presence of silicic acid and sesquioxides.

III. The observed acidity produced in potassium chloride solutions by silica gel depends on the peptisation of the gel which does not exhibit any "neutral salt decomposition." Alumina and ferric oxide gels, after purification by electrodialysis, produce an alkaline reaction in potassium or ammonium chloride solutions. This alkalinity decreases with rising temperature, decreases in the presence of silica gel, and in the latter case is uninfluenced by rise of temperature. The mixed gel of the "silicates" of iron and aluminium produced by precipitation with water-glass exhibits towards potassium chloride solutions exchange acidity depending to some extent on the concentration of the salt solution and the temperature. The gel produced by the removal of bases from sodium-permutit exhibits similar properties. In both cases potassium chloride

causes dissolution of silica and sesquioxides, but there is no equivalence between the titratable acidity and the amount of alumina in solution. The increased peptisation of silica produced by warming causes higher "phenolphthalein" acidity. Exchange acidity represents equilibrium points of the reaction $\text{Al}(\text{OH})_3 + 3\text{KCl} \rightleftharpoons \text{AlCl}_3 + 3\text{KOH}$. "Mineral" acidity in soils is explained on similar lines.

A. G. POLLARD.

To what extent can ammonium salts cause acidity in soils, by the preferential absorption of ammonium cations compared with anions? G. HAGER and W. STOLLENWERK (*Z. Pflanz. Düng.*, 1930, 17A, 129—165).—The physiological acidity of ammonium salts is discussed in relation to ion hydration and the permeability of root-cell membranes. Examination of plant growth in sand cultures shows that the physiological acidity of the ammonium salts increases in the order of the Hofmeister series: nitrate, chloride, sulphate, phosphate. With the sulphate and chloride the majority of the ammonia enters the plant as free ammonia. From the nitrate in faintly acid media the NH_4 ion is absorbed to a greater extent than the NO_3 ion. In very acid media the proportion of NO_3 absorbed is increased. The indirect acidity of the ammonium salts varies in the same order as above. Soil acidity results not only from the actual nitrification of the ammonium salts, but also from the preferential absorption of non-hydrated NH_4OH molecules as compared with that of the anions associated with them.

A. G. POLLARD.

Effect and velocity of action of calcium carbonate in the field on the indexes of acidity in soils. S. GOY [with P. MÜLLER and O. ROOS] (*Z. Pflanz. Düng.*, 1930, 17A, 165—176; cf. B., 1930, 254).—The action of marl and of chalk on soils differs widely with the soil type. The rate of reaction of chalk with a particular soil differs from that of quicklime. Chalk does not completely neutralise soil acidity, even when applied in large excess, presumably because the residual carbon dioxide gives indications of weak hydrolytic acidity. An increased calcium content in the soil solution after liming is of great significance. A general standard of liming, based only on laboratory determinations, is not recommended. Correct applications of lime can only be determined with reference to soil conditions. The authors' system of soil classification affords a satisfactory basis of consideration for this purpose.

A. G. POLLARD.

Effect of different fertilisers on the reaction of strongly acid soils. W. SCHMIDT (*Z. Pflanz. Düng.*, 1930, 9B, 272—275).—Field trials are recorded, which serve to emphasise the importance of considerations of soil reaction and optimum condition of growth for individual crops, in relation to fertiliser practice.

A. G. POLLARD.

Fixation of phosphoric acid in soils. T. GAARDER, (*Medd. Vestlandets Forstl. Forsøksstat.*, No. 14, 1930, 140 pp.).—The solubility of the phosphates of iron, aluminium, calcium, and magnesium in dilute solutions of varying reaction and base content was examined. For each base there is a definite p_{H} range within which the phosphates are relatively insoluble. The variable

effects of soil treatments on the solubility of phosphates are attributed to differences in the quantity and nature of the bases present, either in the adsorbed or soluble condition. Within the reaction range of normal soils the phosphates of iron and aluminium are of prime importance in controlling phosphate solubility. The two maxima observed in p_{H} -growth curves of plants (Arrhenius) may be explained by variations in the solubility of phosphates with soil reaction. In soils rich in "active" sesquioxides, within the entire range p_{H} 4.0—7.5, the phosphate is largely insoluble and liming does not always effect an improvement. In humid and semi-humid soils containing humus the solubility of the phosphates depends mainly on the relative proportions of "active" sesquioxides and humus present, the former tending to decrease and the latter to increase their solubility. From such soils having only moderate sesquioxide contents and p_{H} 4.5—6.5 organically combined phosphorus may be leached sufficiently rapidly to bring about a phosphate deficiency. Where lime did not increase the solubility of phosphates in soils rich in sesquioxides, treatment with sodium silicate proved effective, presumably by the precipitation or "deactivation" of the sesquioxides.

A. G. POLLARD.

Determination of plant-assimilable nutrients in soils. M. VON WRANGELL (*Landw. Jahrb.*, 1930, 71, 149—169).—To determine the assimilable phosphate content, 1 g. of soil is shaken with 100 c.c. of water for 5 hrs. and the suspension is centrifuged. The residue is similarly treated with a further 100 c.c. of water. The total assimilable phosphate is calculated as $a^2/(a-b)$, where a and b are the phosphate contents of the first and second extracts, respectively. Results agree well both with Neubauer values and field trials. The method is suitable for determining the relative availability of phosphatic fertilisers. A similar process is used for determining assimilable potash in soils, but the exact significance of the results is less clearly defined. In water-culture experiments the use of very dilute solutions corresponding in concentration to soil solutions is preferable to the common introduction of relatively concentrated solutions.

A. G. POLLARD.

Home management [of soils] in agriculture. H. KAPPEN (*Z. Pflanz. Düng.*, 1930, 9B, 245—249).—A criticism of Mitscherlich's advice to farmers to carry out their own examination of lime and fertiliser requirements.

A. G. POLLARD.

Effect on crops and acidity condition of soils of amounts of lime calculated from the exchange acidity and from titration curves. L. FRESenius (*Z. Pflanz. Düng.*, 1930, 17A, 176—193).—Applications of lime calculated by Daikuhara's method or from titration curves were insufficient to prevent injury to barley in acid soils. The grain : straw ratio increased with the amount of lime used. Corresponding trials with oats gave similar relative crop yields and grain : straw ratios. Under acid conditions oats utilised existing nutrients more easily than did barley. A particular degree of acidity affected plant growth to a greater extent on sandy soils than on clays. Lime requirements calculated by the two methods examined showed no proportionality for soils of varying types. Plant injury due to excessive

liming occurred only on light sandy soils, and did not persist for a second season. In comparative tests of ammonium sulphate and nitrate on variously limed soils, crop yields with the nitrate were consistently higher than with the sulphate, the differences decreasing with increasing amounts of lime used. Differences in the effects of the two fertilisers were greater in sandy than in clay soils. These crop yields were paralleled by differences in p_H values of the soils, except in light soils treated with ammonium sulphate, where, in some cases, the fertilising effect predominated over the ill effects of increased acidity. A. G. POLLARD.

Yield law and *Aspergillus niger*. R. MEYER (Z. Pflanz. Düng., 1930, 17A, 199—201).—A discussion of some aspects of the yield-law theory based on observations with *A. niger*. A. G. POLLARD.

Nature and course of the nutrient intake of various vegetables. T. REMY and F. WEISKE (Landw. Jahrb., 1930, 71, 315—331).—Cultural trials are recorded designed to show the rate of intake of nutrients by a number of vegetables. The significance of this and of the length of the growing period in the consideration of appropriate fertiliser treatment is discussed. A. G. POLLARD.

Fixation of the nitrogen of liquid manure by lignin and humus-lignite. F. HONCAMP and H. WIESSMANN (Z. Pflanz. Düng., 1930, 17A, 194—199).—Liquid manure absorbed by lignin is an effective nitrogenous fertiliser. Lignin alone has no effect on plant growth, and the efficiency of liquid manure use alone was small. Used in a similar manner, a humus-lignite proved slightly better than lignin. A. G. POLLARD.

Action and value of hot-fermented stall manure. F. LÖHNIS (Z. Pflanz. Düng., 1930, 9B, 268—272).—A reply to recent criticisms (Gerlach, B., 1930, 73; Ehrenberg, *ibid.*, 209). A. G. POLLARD.

Boron as an essential element for healthy growth of citrus. A. R. C. HAAS (Bot. Gaz., 1930, 84, 410—413).—Citrus plants grown in culture solutions from which boron was carefully excluded gave every indication of malnutrition. Addition of borax produced normal healthy growth within a week. Plants appear to obtain sufficient boron for normal needs from the glaze of earthenware culture vessels. A. G. POLLARD.

Control of ragwort and other weeds by spraying. J. W. DEEM (N. Zealand J. Agric., 1930, 40, 291—294).—Sodium and calcium chlorates killed ragwort in full flower and also other weeds. Spraying with 2½—5% solutions is recommended, and quantities ranging from 2 lb. per acre (for scattered plants) up to 22 lb. per acre were used. Little damage was done to grass by 5% chlorate solutions. Dry sodium chlorate was equally successful if applied directly to the weeds, but such applications are more costly. A. G. POLLARD.

Insecticidal action of some derivatives of pyridine and pyrrolidine and of some aliphatic amines. C. H. RICHARDSON and H. H. SHEPARD (J. Agric. Res., 1930, 40, 1007—1015).—Some 33 nitrogenous organic

compounds, mostly related structurally to nicotine, have been tested as contact insecticides against *Aphis rumicis*, L., colonised on nasturtium plants, with the object of finding a product which could be synthesised to replace nicotine. Metanictine and nicotyrine were the only substances tested which showed a toxicity of the same order as that of nicotine. Benzylpyridine appears to show promise of useful application where its oily properties are advantageous. E. HOLMES.

Test to determine toxicity of pyrethrum vapours to honeybees. J. M. GINSBURG (J. Agric. Res., 1930, 40, 1053—1057).—The essential oils of pyrethrum either exerted no toxic effect on bees or were not sufficiently volatile to produce a lethal concentration in the present experiments. It appears that the toxicity of ground pyrethrum flowers is primarily due to the non-volatile substances, pyrethrins I and II. E. HOLMES.

See also A., July, 872, **Utilisation of potassium phosphates** (ASKENASY and NESSLER). 881, **Determination of calcium in the presence of iron and aluminium** (WILEY). 964, **Soil reaction and growth** (REIFENBERG and FRANKENTHAL). 965, **Rôle of magnesium in the ageing of plants** (LUTMAN and WALBRIDGE). **Selection characters as correlated with percentage of sucrose, weight, and sucrose content of sugar beet** (PACK). 967, **Action of ultra-violet rays on germination of seeds and growth of plants** (MEZZADROLI and VARETON).

Iodine content of waste water from sewage. KÖHLER.—See XXIII.

PATENTS.

Manufacture of organic mercury compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 329,987, 26.2. and 22.7.29).—Organic mercuri-hydroxides are more reactive than the mercuri-halides and may be condensed with phenols, thiophenols, or mercaptans to give compounds, $Ar \cdot Hg \cdot O$ (or S) $\cdot R$, useful for seed disinfection. Phenylmercuri-hydroxide [hydroxymercuri-benzene], m.p. 216°, reacts in hot water or alcohol with salicylic acid (product, m.p. 159°), α -hydroxynaphthoic acid (product, m.p. 171°), *o*-chlorophenol (product, m.p. 104°), *m*-hydroxybenzaldehyde (product, m.p. 159°), thiosalicylic acid [product, m.p. 226° (decomp.)], thio-*p*-cresol (product, m.p. 107°), thiophenol (product, m.p. 101.5°), thioglycolic acid [product, m.p. 114° (decomp.)], and 2-thiolbenzthiazole (product, m.p. 147°); ethylmercuri-hydroxide [hydroxymercuriethane] with thio-*p*-cresol (product, m.p. 162°), and 2-thiolbenzthiazole (product, m.p. 86.5°). C. HOLLINS.

Preparation of organic mercury compounds and dust disinfectants containing them. (SIR) G. C. MARKS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 329,672, 19.1.29).—Organo-derivatives of metals replaceable by mercury by double decomposition, e.g., the, alkyl, aryl, or aralkyl compounds of lead, tin, bismuth, or magnesium, are treated with a mercury salt (chloride, acetate, etc.) in absence of liquid media. E.g., lead tetraethyl is milled with mercuric acetate and kaolin

to give directly a dusting powder for use as a seed disinfectant. A water-soluble diluent (sodium sulphate) may be used in place of kaolin. C. HOLLINS.

XVII.—SUGARS; STARCHES; GUMS.

Scholler's process for the conversion of cellulose into sugar. H. LÜERS (Z. angew. Chem., 1930, 43, 455—458).—The mathematical principles governing the reaction and the development of the process during the last six years are reviewed. A small-scale plant has been in operation for some months with satisfactory results; the cellulose, as wood shavings, is heated in a series of vertical tubes while a countercurrent stream of 1% sulphuric acid under pressure at 185° is passed through the tubes, the end tube of the series being emptied, refilled, and made the first of the series after each leach. The lignin residue obtained is about 30% of the weight of wood used, the total solid in the extract 57%, reducing sugars amount to 46%, and fermentable sugars to 38%. The solution obtained contains about 4% of reducing sugars, but only 0.003% N, and processes have been devised for the manufacture of alcohols and various organic acids by suitable fermentation of this liquor. A. R. POWELL.

Determination of the tenacity of starch. SCHULZ and PARLOW (Z. Spiritusind., 1930, 53, 186—187).—The tenacity is deduced from the viscosity at 20° of a solution of the starch prepared by mixing 3 g. of starch with sufficient distilled water to bring the total weight of the solution to 500 g., and warming the mixture on a steam-bath until the mucilage is formed. The viscosity is determined in a Parlow viscosimeter which has a special arrangement at the exit end of the capillary to exclude errors due to surface tension. The moisture content of the starch must also be determined. A determination is made of the concentration of the aqueous glycerin mixture which has the same time of flow as that of the solution of unknown starch, and the amount of Wolff's standard starch to which the unknown starch is equivalent is calculated from a table giving the relative values of Wolff's standard starch and of the aqueous glycerin mixtures. C. RANKEN.

See also A., July, **Inulin** (PRINGSHEIM and HENSEL). **Purification of polysaccharides** (REILLY and MCSWEENEY). 923, **Sweetening power of 5-benzyl-furfuraldoxime** (GILMAN and DICKEY). 965, **Selection characters as correlated with percentage of sucrose, weight, and sucrose content of sugar beet** (PACK).

PATENTS.

Manufacture of dextrin. K. PERL, and F. STEINITZER, CHEM. TECH. INST. (G.P. 456,841, 6.7.26).—A white dextrin of good solubility and low acidity is obtained by heating starchy matter with acids to which has been added a little manganous chloride, aluminium chloride, cobaltous chloride, platonic chloride, or other metallic chloride or nitrate. C. HOLLINS.

Nitrated carbohydrates (U.S.P. 1,750,948, 1,751,063—4, 1,750,949, 1,751,367, 1,751,377, 1,751,436—8).—See XXII.

XVIII.—FERMENTATION INDUSTRIES.

Yeast testing. W. H. COOK and J. G. MALLOCH (Cereal Chem., 1930, 7, 133—142).—The method consists in measuring the carbon dioxide produced from an 8% maltose solution by a definite quantity of yeast at 28° in presence of sodium monohydrogen phosphate and of potassium dihydrogen phosphate. Using the above medium, the initial p_H of 5.6 falls to 5.2 in 1 hr. and to 4.7 in 2 hrs. The gas produced in 1 hr. is almost exactly half that produced in 2 hrs.; that produced during the first 30 min. is less than that produced during the second, the subsequent period showing an intermediate value. Within wide limits the volume of gas produced is proportional to the concentration of the yeast. Baking tests show that the relative efficiency in dough of two strains of yeast is not necessarily the same as their relative gas production in the above medium, possibly owing to the stimulative effect of the phosphate.

W. J. BOYD.

Solubility of maize proteins in mashing. II. R. H. HOPKINS (J. Inst. Brew., 1930, 36, 296—304; cf. B., 1930, 212).—The permanently soluble nitrogen in wort which is apparently derived from flaked maize when mashed with malt is not appreciably assimilated and removed by the yeast in a primary fermentation. The nitrogen is ultimately assimilable if the alcohol is boiled off after the first fermentation and the de-alcoholised wort seeded with fresh yeast. C. RANKEN.

Nitrogen balance in brewery mashes and spent washes. I. Electrolyte precipitation of protein substances in brewery mashes. B. BLEYER and W. DIEMAIR [with K. SICHERT] (Landw. Jahrb., 1930, 71, 1—16).—Examination is made of the changes occurring in the nitrogenous matter during the customary processes of preparation and fermentation of potato mash. The Scherning method for the fractional precipitation of proteins is successfully adapted to the elucidation of protein precipitation during steaming, the activity of proteolytic enzymes during mashing, and the further precipitation and decomposition of proteins during fermentation. A. G. POLLARD.

Brewing value of hop tannin. A. A. D. COMRIE (J. Inst. Brew., 1930, 36, 307—311).—From a review of the experimental evidence on the brewing value of tannin it is deduced that, although the tannin precipitates very little more of the nitrogen in a wort than does mere boiling alone, it accelerates the precipitation to a marked degree, and in order that the precipitate should be as complete and permanent as possible, the tannin must be converted into phlobaphen. Unchanged tannin by remaining soluble tends to hinder fermentation by clogging the surface of the yeast and to give rise to two kinds of haze in the beer. The first type is due to the slow conversion of the soluble tannin compounds into insoluble phlobaphen compounds, a change accelerated by rise in temperature such as in pasteurisation; the second is due to reduction of the solubility of the tannin compounds by fall in temperature such as occurs in the process of beer-chilling. C. RANKEN.

Sterilisation of beer by filtration. F. HARDER (Woch. Brau., 1930, 47, 305—309).—Yeast and bacteria

are removed from beer by the E.K. filter, which is built up of from 2 to 80 filter-plates each 30 cm. in diam., and the filtering material of which consists chiefly of asbestos. A pressure of 1.5–3 atm. is required, and from a filter containing 40 plates, 35 hectolitres of beer can be obtained in 6 hrs., provided the beer undergoes a normal filtration before entering the filter. The flavour of the beer is improved slightly, the proteins are not altered, and the colour decreases only at the beginning of filtration. Protective colloids are apparently removed, since the filtered beer is more sensitive to cold after pasteurisation than is the normally filtered beer. A haze appears at -1.5° compared with -3° to -4° with the normally filtered beer. Head retention is affected equally by both types of filtration. The stability of all kinds of beer is improved after being passed through the E.K. filter, provided the beer is filled into specially sterile bottles by a sterile bottling machine. With bottles cleansed in the ordinary manner, the E.K.-filtered beer is almost always less stable than that obtained by normal filtration. This follows from a greater sensitiveness to infection, which is probably due to the removal of antiseptic or protective substances.

C. RANKEN.

Protein-tannin combination [in beer]. B. D. HARTONG (Woch. Brau., 1930, 47, 295–297).—When beers are treated with adsorbents, *e.g.*, norit, hide powder, or alumina, the proportion of protein to tannin in the adsorbate is constant for any one beer although the total amounts adsorbed differ widely with different adsorbents. There are indications that the proportion of silicic acid is also constant. This is considered to indicate the presence of a complex colloid containing these substances, the coagulation of which may be a frequent source of turbidity (cf. B., 1929, 833).

F. E. DAY.

Reduction in white wines. J. DUBAQUIÉ (Ann. Falsif., 1930, 23, 295–301; cf. B., 1925, 821).—The gradual appearance of a turbidity, and finally of a brownish-red precipitate, in wines after bottling is discussed. This precipitate consists of organic matter, together with ferrous or cuprous oxides, or complex basic sulphites of these metals. The affected wines can be cleared by aeration. This reduction to cuprous salts is not due to dextrose, but is caused by the presence of reductases in the wines; it occurs mostly in wines which have not remained long enough in barrels, and the reductase can be destroyed by pasteurisation at 75° , after which the wine will remain clear.

B. W. TOWN.

See also A., July, 1958, **Action of poisons on living yeast, dried yeast, and macerated juice** (KOSTYTSCHEV and BERG). **Effects of monochromatic light on growth of yeast** (HUTCHINSON and NEWTON). 1959, **Butyric acid fermentation** (ELIASBERG).

PATENTS.

Manufacture of a spirit beverage [apple gin]. W. B. MELROSE, and MELROSE-DROVER, LTD. (B.P. 330,496, 9.2.29).—Ethyl alcohol, which is obtained by the rectification of diluted grain whisky, is diluted to 34% with water and is distilled with various berries,

herbs, roots, and seeds. A tincture of apples in ethyl alcohol is then added together with citric acid to the distillate.

C. RANKEN.

Treatment of molasses [for use in manufacture of yeast]. R. HAMBURGER and S. KAESZ, Assrs. to STANDARD BRANDS, INC. (U.S.P. 1,770,402, 15.7.30. Appl., 12.10.23. Czechoslov., 23.12.22).—See B.P. 209,034; B., 1924, 844.

Drying of pulp (B.P. 330,663).—See I. Salad cream (B.P. 330,708).—See XIX.

XIX.—FOODS.

Determination of ash [of flour] by the direct-weight method. E. O. KLOPFSTEIN (Cereal Chem., 1930, 7, 189–191).—The sample (5 g.) is weighed into an aluminium scoop, and transferred to a crucible which is placed in an electric muffle at 593° , the door being left open. When the flame has died out the furnace is allowed to cool to 420° with the door still open. The door is then closed and the temperature raised slowly to 579° and kept at this till 3 hrs. have elapsed from the beginning of incineration. The temperature is further raised to 593° and kept at this for 1 hr. The crucible is cooled in a desiccator till just warm to the touch, and then the ash is transferred to a counterpoised watch-glass on the balance pan. Vitreosil crucibles of 15-c.c. capacity, 41 mm. in diam., and 25 mm. high are used. The method is found to be quicker and more reliable than the usual method of weighing in the crucible.

W. J. BOYD.

Rapid and simple carbide method for determining moisture in flour. M. J. BLISH and B. D. HILES (Cereal Chem., 1930, 7, 99–107).—The flour (1 g.) is weighed into a dry flask fitted with a rubber stopper carrying a 2-way stopcock, through which the flask can be connected to the air or to an open mercury manometer through a T-piece which carries a second stopcock to give communication between the outside air and the manometer. A bent metal spoon, the end of which is forced into the rubber stopper, hangs down inside the flask. Freshly powdered calcium carbide (2–3 g.) is placed in the bowl of the spoon, the stopper is inserted, and the flask connected with the manometer. The stopcock on the T-piece is opened for an instant, then closed, and the flask is tipped and shaken so as to spill the carbide gradually on the flour. Vigorous shaking at intervals of 1 min. (usually for 3–4 min.) is continued until the manometer reading is constant. The apparatus is calibrated by means of repeated determinations, using weighed quantities of water from 0.01 to 0.1 g., and a graph is constructed from which the weight of water corresponding to a given pressure can be read. All the moisture in the flour does not react with the calcium carbide, but, by comparative determinations on many samples, using the carbide and the oven methods, it was found that the residual moisture was reasonably constant for flours of different types and moisture contents. A correction of 0.0455 g. is therefore added to the value obtained from the manometer reading. Hence the method is not applicable to flours containing less than 4.5% of moisture.

W. J. BOYD.

F.p. of milk as a means of detecting added water. G. D. ELSDON and J. R. STUBBS (Analyst, 1930, 55, 423—432).—Hortvet's cryoscopic method (B., 1921, 274 A) has been used for determining the f.p. of milk. Duplicate readings agreeing within 0.002° are readily obtained, the usual deviation being less than this and almost entirely due to errors of parallax. Tables of f.p. depressions obtained by other workers and by the authors are given. An average of 0.54 is taken for the purpose of calculating added water, but no milk is considered as watered on the evidence of the f.p. of a single sample unless the depression falls below 0.53 . The method is applicable only to fresh milk or to slightly sour samples after correction. D. G. HEWER.

Routine detection of nitrates in milk. A. F. LERRIGO (Analyst, 1930, 55, 433—435).—Nitrates may be detected in milk by adding 6—7 drops of mercury reagent (a solution containing 20% of mercuric chloride, 5% of ammonium chloride, and 20 vol.-% of concentrated hydrochloric acid) to 4 or 5 c.c. of milk in a test-tube previously washed with milk, shaking occasionally for 2 min., and filtering into 2 c.c. of diphenylamine reagent (cf. Elsdon and Sutcliffe; A., 1913, ii, 979). With normal milk the line of demarcation is colourless; with fairly large quantities of nitrate it is dark blue, and with quantities of the order of 0.1 pt. of nitric nitrogen per 100,000 or less the blue colour appears at the bottom of the serum on gently agitating. Reagents should not be stored in the same room as nitric acid. The test applied to all samples of milk containing less than 8.5% of solids-not-fat (102 out of 1172) gave a positive reaction for 50.

D. G. HEWER.

[Effect of] adding sodium bicarbonate to milk. P. CAZENEUVE (Ann. Falsif., 1930, 23, 303—305).—This practice, though common, is to be deprecated as it greatly favours the development of pathogenic bacteria. The adulterant can be detected on the effervescence on acidification, and can be determined by ashing a given volume of milk and titrating the aqueous extract with standard acid, using first phenolphthalein and then helianthin as indicators. The second titration gives a figure for the sodium phosphate which arises by double decomposition from the calcium phosphate of the milk.

B. W. TOWN.

Content of salt in cheese. F. MÜNCHBERG (Milch. Zentr., 1930, 59, 201—202).—The cheese (1—2 g.) is boiled with 10 c.c. of halogen-free nitric acid (d 1.40) and 10 c.c. of 0.1N-silver nitrate, and 10% potassium permanganate solution is gradually added till the liquid is clear. The solution is cooled by addition of 150 c.c. of distilled water, and, after further addition of 2 c.c. of saturated iron ammonium alum solution, excess of silver is titrated with 0.1N-ammonium thiocyanate. (1 c.c. of 0.1N-silver nitrate = 0.005856 g. NaCl.) As the salt content should be reckoned on the dry weight of the cheese, the moisture content is determined by mixing 2—3 g. of sample with sand and drying it to constant weight at 105° with frequent stirring. Values found for the moisture and salt contents of various kinds of cheese are given.

W. J. BOYD.

Determination of egg in ice-cream. N. C. SMITH

(J. Assoc. Off. Agric. Chem., 1930, 13, 272—291).—A method for the determination of egg yolk in ice-cream has been worked out and is based on the lipid phosphorus content. The average lipid phosphorus content of a typical egg-free mixture has been calculated to be 2.49 mg. per 100 g., and this value agrees well with the average found for 21 commercial samples, viz., 2.56 mg. per 100 g. Special mixes containing a high proportion of cream have higher lipid phosphorus contents, approximately proportional to the fat content. Analyses of egg products made by Hertwig (cf. B., 1923, 1040 A) showed the average value of the lipid phosphorus of water-free, commercial egg yolk to be 777.7 mg. per 100 g., and this value has been confirmed by the author. The percentage of egg yolk in ice-cream is then given by $100(A - 2.56)/(777.7 - 2.56)$ or $(A - 2.56) \times 0.129$, where A is the phosphorus content of the sample expressed in mg. per 100 g. To calculate the percentage of whole egg the factor 0.129 becomes 0.166, and for fresh egg yolk the dry egg yolk percentage is doubled (total solids in fresh egg yolk = 50%). The Röse-Gottlieb method for extracting the lipoids was found satisfactory, and the phosphorus content of the residue was determined colorimetrically by a modification of the Briggs method (cf. A., 1922, ii, 718), using a solution of potassium dihydrogen phosphate (0.2193 g./litre) as colour standard. By applying these methods and the formula to egg-free mixtures to which known amounts of egg yolk had subsequently been added, an average recovery of 95.5% of the lipid phosphorus was obtained. The general applicability of the formula has been tested on samples of ice-cream without preservative, stored in a refrigerator for periods up to 1 month, on samples containing formaldehyde as preservative stored at room temperature for 10 days, and on samples pasteurised for 40 min. at 63° . The changes in the phosphorus content were insignificant.

H. J. DOWDEN.

Relative merits of sucrose, dextrose, and lævulose as used in the preservation of eggs by freezing. O. M. URBAIN and J. N. MILLER (Ind. Eng. Chem., 1930, 22, 355—356).—The white of egg was found to undergo no change when frozen at -15° , maintained at this temperature for 72 hrs., and then thawed, but the yolk treated similarly became ropy. The cause of this ropiness was found to be the dehydration and coagulation of a lecitho-protein, and could be prevented most efficaciously by the addition of dextrose or lævulose, in the presence of which less than 1% of the lecithin was coagulated, whilst in the presence of sucrose 98% was coagulated. Dextrose and lævulose were also found to prevent the fermentation and bacterial composition of the batters more efficiently than did sucrose. It is suggested that the differences are due to the greater osmotic pressure of a monosaccharide solution compared with a disaccharide solution of equal concentration. A. SHORE.

Use of lead acetate in the determination of the acidity of fruit products. B. G. HARTMANN and F. HILLIG (J. Assoc. Off. Agric. Chem., 1930, 13, 259—265).—The total free acidity of phosphates is not indicated by direct titration using phenolphthalein as indicator, but when lead acetate is added an equivalent

amount of acetic acid is liberated which gives a true measure of the acidity. In the examination of fruit products, 200 c.c. of the solution (or 25 c.c. of fruit juice diluted to 200 c.c.) are mixed with 20 c.c. of dilute nitric acid (15 c.c. in 1 litre), and to the mixture are added 20 c.c. of lead acetate solution (100 g. in 300 c.c. of water containing 10 c.c. of glacial acetic acid diluted to 1 litre and filtered). After shaking, diluting to 250 c.c., and filtering, lead is removed by adding solid potassium oxalate in slight excess, and after refiltering, the filtrate is titrated with 0.1N-caustic soda against phenolphthalein. The solutions are partially decolorised by precipitation of the lead and a sharp end-point was always obtainable. The results given by the method checked excellently with materials of known composition, but were always higher than those given by direct titration and by electrometric titration. H. J. DOWDEN.

Determination of caffeine in decaffeinated coffee. W. F. ALLEN (J. Assoc. Off. Agric. Chem., 1930, 13, 265—272).—In the official (Power-Chesnut) and the tentative (Fendler-Stüber) A.O.A.C. methods of analysis, the caffeine content is based on the weight of the residue after removal of the chloroform, and may be verified by nitrogen determinations on the residue (factor $\times 3.464$). Two commercial brands of decaffeinated coffee were examined by each method, and the purity of the residues was tested by nitrogen determinations and by sublimation at 180—190° for 10 hrs. The results showed that values based on the weight of the residues are quite erroneous since only 20—40% is pure caffeine. Satisfactory results are given by nitrogen determinations either by micro- or macro-Kjeldahl methods (provided the dilute solutions of the former are used for the latter method), and these results are in close agreement with those obtained by sublimation. The average values of 15 samples examined by the Power-Chesnut and Fendler-Stüber methods were: 0.0569 and 0.0487% of caffeine, respectively, by direct weighing; 0.0203 and 0.0183% by nitrogen determination; 0.0208 and 0.0174% by sublimation; and 0.0127 and 0.0151% by nitrogen determination after sublimation.

H. J. DOWDEN.

Vitamins. I. Vitamin content of foods and delicacies. II. Vitamin content of preparations manufactured from fresh vegetables [fruit]. F. V. VON HAHN (Z. Unters. Lebensm., 1930, 59, 4—18, 18—31).—I. A discussion of technique.

II. Results of the examination of commercial preparations from oranges and lemons and of lemon substitutes are detailed.

H. J. DOWDEN.

Control of mineral waters and their salts under the Federal Food and Drugs Act. J. W. SALE (Ind. Eng. Chem., 1930, 22, 332—335).—The application of the Federal Food and Drugs Act to bottled mineral waters is discussed, and suggested methods of analysis are outlined.

A. SHORE.

See also A., July, 872, **Photochemical oxidation of food materials by air** (PALIT and DHAR). 963, **Colorimetric determination of the anti-beri-beri vitamin content of rice** (SPRUYT). **Fat-soluble vitamins of**

milk (CRAWFORD and others). 964, **Biochemistry and histochemistry of fruits and seeds** (NIETHAMMER).

Disintegration. CHWALA.—See I. **Yeast testing.** COOK and MALLOCH.—See XVIII. **Fumigation with ethylene oxide.** RUSS.—See XXIII.

PATENTS.

Deodorising and similar treatment of milk, cream, and other liquids containing butter fat. A. E. WHITE. From PFAUDLER Co. (B.P. 330,662, 19.3.29).—In an apparatus described, the milk etc. heated to above 38° is subjected in a fine state of division to aeration under reduced pressure. By this means the objectionable natural flavours sometimes present in milk and cream may be removed. E. B. HUGHES.

Apparatus for pasteurising milk or other liquids. AKTIEB. RUDELIUS & BOKLUND (B.P. 330,867, 14.11.29. Swed., 28.11.28).—Closed containers for pasteurising milk by the "holding" process are described in which the temperature of the surface of the milk is maintained by heating-tubes just below and also above the surface of the liquid. E. B. HUGHES.

Continuously producing ice-cream and other plastic substances. C. W. VOGT, and VOGT INSTANT FREEZERS, INC. (B.P. 330,887, 18.1.29).—The material, such as ice-cream or vegetable or animal oils, with or without air or other gas, is forced under pressure through a cylindrical space wherein the material is kept in constant agitation. Means are provided for cooling or heating the material during agitation and, in order to maintain a constant plasticity, the effect of the cooling or heating medium is controlled by the consistency of the material passing through. E. B. HUGHES.

Manufacture of salad cream. CROSSE & BLACKWELL, LTD., and W. CLAYTON (B.P. 330,708, 29.4.29).—Salad creams or dressings may be prepared by emulsifying a vegetable oil (olive oil) and milk with egg-yolk, sugar, starch, or other suitable nidus for bacterial growth. Edible organic acids (acetic and lactic) may be added if desired. After emulsifying, a bacterial culture of *Streptococcus lacticus* or *Hollandicus* or *B. Bulgaricus* is added which matures the cream in the bottle, imparting definite aroma and flavour.

E. B. HUGHES.

Preservation of gherkins and the like. F. TARNAWSKI (B.P. 331,161, 17.10.29. Czechoslov., 18.10.28).—Such materials are fermented in 10% salt solution, soaked in water, spices and other vegetables and a 2% unsweetened vinegar solution added, and the product is sterilised in sealed vessels for 5—10 min.

E. B. HUGHES.

Process and apparatus for expressing material, e.g., of the consistency of dough or pasty material. O. KREMMLING (B.P. 331,489, 28.3.29).

Machines for treating fish. GEN. FOODS Co., Assees. of C. BIRDSEYE (B.P. 309,018, 7.1.29. U.S., 3.4.28).

Treatment of non-fibrous material (B.P. 330,649).—See V. **Edible fatty products** (B.P. 301,750).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Porphyroxine test for Indian opium. F. BAMFORD (Analyst, 1930, 55, 445—446).—The purple colour produced in the porphyroxine test for opium has been regarded as characteristic of Indian opium and as a means for differentiating the latter from Turkish or Smyrna opium, but a sample recently seized in the Egyptian Custom House, and known to be Turkish, gave a positive reaction.

D. G. HEWER.

Methylene-blue, U.S.P., as precipitant of Irish moss. G. E. EWE (J. Amer. Pharm. Assoc., 1930, 19, 568—570).—Zinc-free methylene-blue is almost quantitatively precipitated from aqueous solution by an extract of Irish moss, 1 g. of dry extractive matter being equivalent to 1.17 g. of dye at the ordinary temperature and to 1.30 g. at 100°. A precipitate is not obtained from methylene-blue with the mineral constituents of the moss, or with other gums, nor from moss extract with 15 other typical dyes.

H. E. F. NOTTON.

See also A., July, 897, Local anæsthetics from dialkylaminopropanediols (RIDER). 916, Reactions of 4-hydroxy-3-ethoxybenzaldehyde (KLOTZ). 921, Essential oil of *Backhousia angustifolia* (GIBSON and others). 926, Synthesis of 2-methylquinuclidine (WINTERFELD). 935, Potentiometric measurements of certain homologues of ephedrine (KRAUTZ and HARTUNG). Amine oxides of alkaloids (POLONOVSKI and POLONOVSKI). 936, Strychnine alkaloids (WIELAND and MÜNSTER; LEUCHS and KRÖHNKE). Tetrachloriodides of alkaloids (CHATTAWAY and PARKES). 937, New reaction for codeine and dionine (DE HAAS). Neoarsphenamine (JURIST and CHRISTIANSEN). Arsenic derivatives of pyridine (BINZ and others). Quinoline compounds containing arsenic (SLATER). 938, 10-Chloro-5:10-dihydrophenarsazine and its derivatives (GIBSON and JOHNSON). 960, Purification of anticarbuncle serum (MODERN and WERNICKE). Germicidal power of colloidal silver and of ionic silver on *B. typhosus* (SAMAAN). 961, Bio-assay of adrenaline-procaine mixtures (MUNCH and DECKERT). Preparation of insulin (KAULBERSZ). 966, Non-volatile constituents of *Mentha piperita* (BRAUN). Essential oil of green violet leaves. Mignonette oil (WALBAUM and ROSENTHAL). Volatile oils in plant economy (MARSH and MAUS). 968, Testing of ampoule glass for alkalinity (STICH).

Aluminium sulphate. KULLERUD and HØRLÜCK.—See VII. Toxicity of pyrethrum vapours. GINSBURG.—See XVI. Control of mineral waters. SALE.—See XIX.

PATENTS.

Treatment of tobacco. DEVELOPMENT OF INDUSTRIES, LTD., and V. A. GAVRILOVITCH (B.P. 331,026, 25.4.29).—Tobacco is subjected to the effects of high-frequency electricity, with or without ultra-violet rays, ozone, and electric sparks.

E. H. SHARPLES.

Ephedrine-aldehyde and -ketone compounds and solutions, and their preparation. E. H. STUART, Assr. to E. LILLY & Co. (U.S.P. 1,749,361

and 1,749,452, 4.3.30. Appl., [A] 3.5.28, [B] 20.8.28. Renewed [B] 8.1.30).—(A) An aldehyde is added to an ethereal solution of ephedrine. The products from *l*-ephedrine with acetaldehyde (b.p. 239°), furfuraldehyde (m.p. 48°), citral, heptaldehyde, benzaldehyde (m.p. 73°), anisaldehyde (m.p. 89.5°), *o*-methoxybenzaldehyde (m.p. 103.5°), salicylaldehyde (m.p. 117°), veratraldehyde (m.p. 97°), vanillin, and cinnamaldehyde (m.p. 92.5°), and from *d*- ψ -ephedrine with cinnamaldehyde (m.p. 75°), benzaldehyde (m.p. 67°), salicylaldehyde (m.p. 79—80°), and vanillin (m.p. 124.5—125°) are described. These are soluble in paraffin oil and are used for nasal sprays. (B) Similarly applicable oil-soluble products are made by dissolving ephedrine in liquid (or molten) ketones, reaction being complete in several days or weeks. Compounds described are: from *l*-ephedrine with acetone (m.p. 46°), acetophenone (m.p. 34°), methyl ethyl ketone, menthone (m.p. 35°), and camphor; *d*- ψ -ephedrine with acetone (b.p. 152.5°/12 mm.), acetophenone (m.p. 115°), methyl ethyl ketone, ethyl propyl ketone, and camphor (m.p. 87°).

C. HOLLINS.

Preparation of α -*p*-hydroxyphenyl- β -methylaminopropanol [β -methylamino- α -*p*-hydroxyphenylpropyl alcohol]. I. G. FARBENIND, A.-G. (B.P. 314,448, 27.6.29. Ger., 27.6.28).—This therapeutic compound, m.p. 152—154° (hydrochloride, m.p. 209—211°; oxalate, m.p. 216—218°), is obtained by hydrogenation of the corresponding propiophenone or its *N*-benzyl derivative in presence of a nickel catalyst at 100° or palladium at 15°, with hydrogen at 40 atm.

C. HOLLINS.

Manufacture of therapeutical agents. I. G. FARBENIND, A.-G. (B.P. 306,905, 26.2.29. Ger., 27.2.28).—Salts of ethers of 6:8-dihydroxyquinoline with 5:5-disubstituted barbituric acids are made by double decomposition, e.g., from sodium 5-phenyl-5-ethylbarbiturate and 6:8-diethoxyquinoline hydrochloride, or sodium 5-cyclohexenyl-5-ethylbarbiturate and 6:8-dimethoxyquinoline hydrochloride. They are tasteless and have hypnotic and antispasmodic properties.

C. HOLLINS.

Mercury-bromine derivatives of fluorescein. W. L. VOGEL, Assr. to P. W. PRUTZMAN (U.S.P. 1,749,201, 4.3.30. Appl., 28.5.28).—Fluorescein is monobrominated in acetic acid. The product, m.p. 110°, is boiled with mercuric acetate solution, and the precipitated mercury compound is treated with the calculated amount of alkali to give $C_{20}H_5O_5Br(HgOH)_2Na_4$, a non-irritant antiseptic.

C. HOLLINS.

Increasing the therapeutic action and lowering the toxicity of arsenic-containing organic therapeutic substances. J. I. STEINFELD (B.P. 308,600, 19.3.29. Ger., 24.3.28).—Arsenicals such as atoxyl, salvarsan, neosalvarsan, myosalvarsan, etc. are treated with phosphatides (e.g., lecithin, cephalin, and cerebroside), or with extracts of animal organs rich in phosphatides, such as brain, liver, etc., or suspensions prepared from such organs. Adsorption compounds are obtained which, although having a very small arsenic content, possess the same therapeutic action

as that possessed by the arsenic compounds alone. The preparations are more resistant to atmospheric oxidation and the increased therapeutic action is an inherent property of the adsorption compound and is not obtained by the separate incorporation of the two substances.

E. H. SHARPLES.

Preparation of compressed iodine in the form of rods, pencils, etc. O. and I. BRAUN (B.P. 331,118, 19.7.29. Ger., 18.4.29).—Iodine is mixed with an iodine compound, such as potassium iodide, and small quantities of water, and the resulting mass is converted into a paste with binding agents such as bolus or kaolin and a large proportion of lactose. The plastic masses are dried at a low temperature, mixed with sufficient quantities of talc, and compressed into pencils, rods, etc.

E. H. SHARPLES.

Obtaining gland extracts. PARKE, DAVIS & Co. (B.P. 303,362, 21.12.28. U.S., 31.12.27).—Hormones are separated from glandular substances by treating the material with a lower fatty acid in relatively high concentration (98–100% acetic, 95% propionic, 90% butyric acids, etc.) followed by treatment of the acid extract, which contains the hormones in solution but little foreign protein, with organic solvents such as acetone, sulphuric ether, and light petroleum. The active principles are precipitated. Precipitation may be fractional and fractions having various degrees of physiological activity obtained. The most active precipitates can be redissolved in fatty acid and reprecipitated with solvents. By extraction of the posterior lobe of the pituitary gland, or active extract thereof, with the concentrated fatty acid followed by precipitation with sulphuric ether, a fraction high in the pressor principle (*b*-hypophamine) and low in oxytocic principle (*a*-hypophamine) is obtained. From the filtrate, by addition of light petroleum, a precipitate having high oxytocic and low pressor activity is formed. The fatty acid extract may be treated with acetone to remove foreign proteins prior to the above fractional precipitation. By repeated fractionation, the two principles can be obtained substantially free from each other; pressor and oxytocic principles are thereby obtainable having a potency 80 and 150 times as great, respectively, as that of the U.S.P. standards.

E. H. SHARPLES.

Manufacture of sexual hormones. SCHERING-KAHLBAUM A.-G. (B.P. 307,844, 13.3.29. Ger., 14.3.28).—The faeces of mammals is extracted with either a water-soluble organic solvent, the hormone being separated in the customary manner, or with a water-insoluble solvent, the hormone being isolated from the unsaponifiable portion of the extract. The starting material may be dried, or mixed with water and alkali, or with an alkaline earth, or an indifferent porous material may be added previous to extraction.

E. H. SHARPLES.

Medicinal articles. [Coated dried fruit carrying a medicine at the core.] C. W. WALLIS (B.P. 331,321, 7.5.29).

Alkylene-substituted phenols (B.P. 304,727).
Amino-alcohols (B.P. 307,304). **o-Hydroxycarbox-**

ylic acids of fluorene (B.P. 330,305).—See III.
Organic mercury compounds (B.P. 329,672).—See XVI.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Light filters for the isolation of narrow spectral regions. L. A. JONES (Phot. J., 1930, 70, 337–346).—A series of eight filters of good stability suitable for the isolation of spectral bands in the infra-red, ultra-violet, and visible spectra have been constructed from a combination of Wratten dyed-gelatin filters, various grades of Corning glass, aqueous solutions of nickel and copper sulphates, and distilled water. The components and transmission characteristics obtained by spectrophotometric methods are given for each filter.

J. W. GLASSETT.

See also A., July, 871, **Photochemistry of silver nitrate** (BELENKY and JUSE). **Microtome studies of photographic layers** (SCHAUM and KLEIN). **Photosensitisation by ammonia** (TAYLOR and EMELEUS).

Heat expansion of solids. COHN.—See I.

PATENTS.

Photographic and kinematographic films. K. BRATRING (B.P. 307,431, 6.3.29. Ger., 7.3.28).—The inflammability of films is greatly reduced by suitably securing to the picture side only or to both sides of the finished film, which may be very thin, a thin layer of a non-inflammable cellulose derivative, such as the hydrate or acetate.

J. W. GLASSETT.

Apparatus and developer for developing [diazotype] photographic prints. F. VAN DER GRINTEN (B.P. 330,625, 13.3.29. Holl., 8.12.28).—Apparatus for developing diazo-types with a solution containing alkaline substances, azo dye components, buffer salts, and reducing agents is claimed.

J. W. GLASSETT.

Photographic printing by imbibition. F. J. TRITTON, and COLOUR SNAPSHOTS (1928), LTD. (B.P. 330,199, 12.12.28).—A process is developed for photographic printing on a layer of silver halide-gelatin emulsion containing an orange or yellow dye, developing, fixing, and tanning the layer, so as to produce the gradations in the image in relief in the gelatin. The latter is dyed and printing carried out by pressing on a layer of gelatin, which takes up the dye by imbibition.

J. W. SMITH.

Colour photography. C. L. FINLAY, and FINLAY PHOTOGRAPHIC PROCESSES, LTD. (B.P. 329,018, 9.2.29).—In the preparation and reproduction of colour transparencies, the colour record is obtained in intimate optical contact with the colour viewing screen by coating the latter with a gelatin layer, which is sensitised with a solution of alkaline potassium dichromate, dried, and exposed, after colour relation has been adjusted, behind a positive colour-record transparency. The screen is removed, bathed in water, and the differentially hardened image is treated with a solution of a black dye (nigrosine), which is absorbed in the unhardened parts, washed, and dried. Any brown or blue tinge must be

neutralised by the addition to the bath of a small quantity of blue or brown dye, respectively, of similar character.

J. W. GLASSETT.

Films for colour photography. KODAK, LTD., Assees. of J. G. CAPSTAFF (B.P. 312,992, 4.5.29. U.S., 4.6.28).—To lessen halation and the consequent degradation of colour values, a dye, preferably bleachable by prolonged exposure to light, is incorporated or applied in a thin layer over the lenticulated support in sufficient concentration to absorb about 20% of the incident light. Kryptocyanine dissolved in a 1:2 mixture of ethyl acetate and ethyl alcohol is recommended for the purpose.

J. W. GLASSETT.

Production of multicoloured transparencies or diapositives, especially applicable to kinematograph films. K. WAHL (B.P. 306,026, 18.1.29. Ger., 14.2.28).—A cellulose film is coated on both sides with a chromated gelatin solution capable of giving differentially hardened images. One side is exposed under a negative and the other, after alignment, under a positive; alternatively, both may be exposed under a negative. The images so formed are then dyed in different colours by imbibition or by tinting after the unhardened gelatin has been removed by hot water.

J. W. GLASSETT.

Production of multicolour pictures. JOS-PE FARBENPHOTO-GES.M.B.H. (B.P. 330,997, 27.3.29. Ger., 28.2.29).—Very flat gelatin reliefs corresponding to the colour-record images are produced by heavily tinting the silver halide emulsion with a colour complementary to the exposure light, exposing through the support, treating after development with metallic salts to give differential hardening, and, finally, after fixation, removing the unhardened gelatin with warm water. These reliefs are then dyed with the appropriate colours, and applied successively in register to a gelatin-coated support.

J. W. GLASSETT.

Recovery of celluloid and silver from scrap films. H. PICK (B.P. 331,006, 8.4.29).—The scrap film is treated first with a warm (30–50°) 1% solution of caustic soda, which serves to free the silver-gelatin layer, and then for at least 2 hrs. with cold *N*-caustic soda, which removes the gelatin substratum. The gelatin and silver are then precipitated by coagulation at the b.p. with aluminium sulphate.

J. W. GLASSETT.

Production of negative or positive printing plates. MEISENBACH, RIFFARTH, & Co. A.-G. (B.P. 310,500, 15.12.28. Ger., 27.4.28. Addn. to B.P. 302,887; B., 1930, 350).—The dried collodion plates may be etched by the process of the prior patent if treated first with a swelling agent (ether) and then with the hygroscopic agent (*e.g.*, glycerin) and the protective varnish.

J. W. GLASSETT.

Manufacture of light-sensitive films. U. DIEM-BERNET (U.S.P. 1,769,940, 8.7.30. Appl., 17.5.27. Switz., 21.5.26).—See B.P. 271,507; B., 1928, 625.

Manufacture of diazo-type sensitive papers and the like. H. D. MURRAY, Assr. to NORTON & GREGORY, LTD. (U.S.P. 1,770,352, 8.7.30. Appl., 28.9.29. U.K., 14.6.28).—See B.P. 320,395; B., 1930, 218.

XXII.—EXPLOSIVES; MATCHES.

Comparison between calculated and experimental explosion pressures. H. MURAOUR and G. AUNIS (Compt. rend., 1930, 190, 1547–1549; cf. A., 1925, ii, 319; 1926, 480).—The pressures attained on explosion of a powder consisting of 52% of guncotton (12.2% N) and 48% of nitroglycerin in tube form (8.80 × 4.25), at five densities of loading (0.062–0.285), as measured by crusher, and corrected by two different methods for the cooling effect of the walls, are compared with calculated results. These assume a temperature of explosion of 3600°, and are based on an extrapolation of the Nernst-Wohl curves for sp. heat, and on gaseous compositions, taking into account the dissociation at different pressures. Agreement is (with one exception) within 3.5%. It is inferred that the correction for cooling at the lower pressures is doubtful.

C. A. SILBERRAD.

Coloured-light recipes. L. VANINO (J. pr. Chem., 1930, [ii], 127, 16–19).—The following coloured phosphorescent mixtures are produced by heating the constituents together for 1 hr., the number of parts being in parenthesis. *Violet-blue*: quicklime (15.0), strontium hydroxide (5.0), sulphur (8.0), magnesia (1.0), sodium carbonate (3.0), lithium sulphate (1.0), and 6 c.c. of 0.3% aqueous colloidal bismuth solution; or, alternatively, quicklime (17.0), strontium hydroxide (5.0), sulphur (8.0), lithium fluoride (1.0), magnesia (1.0), 1.0 c.c. of 1% rubidium nitrate solution, and 6 c.c. of 0.3% colloidal bismuth solution. *Violet*: quicklime (20.0), barium hydroxide (10.0), sulphur (9.0), sodium sulphate (1.0), potassium sulphate (1.0), lithium sulphate (1.5), 2 c.c. of 0.5% alcoholic bismuth nitrate solution, 1 c.c. of 0.5% aqueous thallium sulphate solution, and 0.5 c.c. of 0.5% aqueous thorium sulphate solution (the nitrates may be substituted for thallium and thorium sulphates). *Blue*: strontium hydroxide (15.0), quicklime (5.0), sulphur (8.0), magnesia (1.0), sodium carbonate (3.0), lithium sulphate (1.0), and 6 c.c. of 0.3% colloidal bismuth solution. *Green*: strontium hydroxide (20.0), sulphur (8.0), magnesia (1.0), sodium carbonate (3.0), lithium sulphate (1.0), and 6 c.c. of 0.3% colloidal bismuth solution.

A. I. VOGEL.

See also A., July, 880, **Quantitative analysis of azides** (REITH and BOUWMAN).

Cellulose benzoate nitrates. SENDO and KONDO.—See V.

PATENTS.

Initially detonating explosives and manufacture of detonating caps. E. VON HERZ (B.P. 331,162, 17.10.29. Ger., 18.10.28).—In detonators the main charge consists of a nitric ester of the general formula $C_nH_{n+2}(NO_3)_n$, such as hexanitromannitol or its molecular mixture with nitropentaerythritol. This is covered by a perforated cap and then pressed at 100–400 kg./cm.² The priming charge may consist of a mixture of an oxygen carrier, such as barium nitrate, and aluminium or magnesium powder, together with lead hypophosphite, if desired, and is pressed at the same pressure as the main charge.

W. J. WRIGHT.

Manufacture of nitrated sugar explosives. P. G. WRIGHTSMAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,750,948 and 1,751,063, 18.3.30. Appl., [A] 17.9.27, [B] 7.12.27).—(A) Sugar is nitrated in the form of a suspension in a mixture of glycerin and ethylene glycol, the sugar particles being of such fineness as to pass 100-mesh, and the amount of sugar being greater than 25%. (B) A mixture containing more than 5% of a monosaccharide and about 25% of a disaccharide is dissolved in a polyhydric alcohol, or mixture of polyhydric alcohols of lower viscosity than that of glycerin, and nitrated. A suitable composition consists of dextrose 10–30%, sucrose 20–25%, and ethylene glycol 45–70% (or ethylene glycol 20–60% with glycerin 20–50%). W. J. WRIGHT.

Manufacture of nitrated carbohydrate explosive. P. G. WRIGHTSMAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,750,949 and 1,751,377, 18.3.30. Appl., [A] 17.8.28, [B] 7.11.27).—(A) Mixtures of carbohydrates and polyhydric alcohols having 4–6 carbon atoms are nitrated in a liquid polyhydric alcohol medium, the maximum amount of carbohydrate being thus held in solution. A suitable mixture consists of sucrose 20–25% and sorbitol 10–50%, the remainder being a mixture of glycerin and ethylene glycol. (B) Sugar is nitrated at 10° in suspension in monochlorohydrin, or in a mixture of monochlorohydrin and glycerin, the particles of sugar being of such fineness as to pass 100-mesh, and the amount of sugar in suspension being 25–50%. W. J. WRIGHT.

Manufacture of explosives. C. C. AHLUM, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,751,064, 18.3.30. Appl., 20.4.28).—A liquid explosive of sufficiently low viscosity to permit of its being readily stabilised is produced by nitrating a solution of a polyhydric alcohol having 4–6 carbon atoms, such as sorbitol, in a liquid polyhydric alcohol, such as glycerin, ethylene glycol, or a mixture of these. Alternatively, a mixture of the solid and liquid polyhydric alcohols may be nitrated, or the alcohols may be introduced separately into the nitrator and then nitrated.

W. J. WRIGHT.

Treatment of nitrated bodies. J. V. SKOGLUND, Assr. to TROJAN POWDER Co. (U.S.P. 1,751,367, 18.3.30. Appl., 8.10.21).—To remove residual acid from nitrostarch, the material is placed on a filter plate in a displacement vessel and subjected to suction or pressure. Sulphuric acid of the same sp. gr. as that of the mixed acid is then caused to flow over the material to displace the mixed acid, under suction or pressure as before, the nitrostarch, saturated with sulphuric acid, being finally removed and "drowned" in water. Alternatively, the sulphuric acid may be removed from the nitrated material by displacement with water instead of by "drowning."

W. J. WRIGHT.

Manufacture of (A) blasting explosive, (B) gelatinised explosive, (C) nitrated sorbitol. F. H. BERGEIM, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,751,436–8, 18.3.30. Appl., [A] 3.5.28, [B] 27.6.28, [C] 6.10.28).—(A) A non-freezing dynamite contains erythritol tetranitrate, an inorganic nitrate, and a

carbohydrate, together with a solid explosive, such as nitrostarch, if desired. (B) A gelatinised explosive is prepared by mixing nitrated sorbitol, together with another polyhydric alcohol having 4–6 carbon atoms, if desired, with nitrocellulose and inorganic oxidising salts and combustibles. (C) An explosive of low m.p. and high power, suitable for use in bombs and high-explosive shells or as a component of dynamites, is obtained by nitrating sorbitol, dissolving the hexanitrate formed in alcohol, and precipitating with water. The product contains 17–18.48% N and has m.p. 44–54°. W. J. WRIGHT.

Lachrymators (U.S.P. 1,750,101).—See III.

XXIII.—SANITATION; WATER PURIFICATION.

Iodine content of waste water [from town sewage]. R. KÖHLER (Z. angew. Chem., 1930, 43, 503–507).—Analyses of effluent water before and after filtration at various German sewage farms show that it contains appreciable quantities of iodine, much of which remains in the slime. During the course of purification of the water the iodine decreases with decreasing content of organic matter; most of this loss of iodine escapes into the air, especially during filtration through the sand filters. The use of the slime as a fertiliser results in an appreciable increase in the iodine content of the crop, especially in the case of rye.

A. R. POWELL.

Fumigation with ethylene oxide. J. M. RUSS, JUN. (Ind. Eng. Chem., 1930, 22, 328–332).—The most efficient methods of fumigating grain, store rooms, and refrigerator cars with ethylene oxide are described. Tests show that a mixture of ethylene oxide and carbon dioxide is a more efficient fumigant for the destruction of insect pests than the former substance alone. For the fumigation of grain in enclosed bins a dosage of 2 lb. of ethylene oxide and 14 lb. of solid carbon dioxide per 1000 bushels of grain, introduced gradually into the grain as it enters the bin, is recommended. This fumigant has no deleterious effect on any of the materials on which it has been tested (grain, cotton, dried fruit, nuts, etc.); it does not leave an odour and is non-inflammable. A mixture of carbon dioxide and ethylene oxide ("Carboxide") is supplied in cylinders and can be used for house fumigation. The dosages and times of contact for various types of fumigation are given.

A. SHORE.

See also A., July, 884, **Distillation apparatus for water** (FRIEDRICHS).

Disintegration. CHWALA.—See I.

PATENTS.

Insecticides. I. G. FARBENIND. A.-G. (B.P. 308,661, 25.3.29. Ger., 24.3.28).—A mixture comprising ethylene oxide and carbon tetrachloride is claimed.

H. ROYAL-DAWSON.

Fumigating apparatus. H. A. SMITH, and LAWES' CHEM. MANURE Co., LTD. (B.P. 331,192, 23.3.29).

Alkylene-substituted phenols (B.P. 304,727).—See III.