

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

NOV. 23, 1928.

### I.—GENERAL ; PLANT ; MACHINERY.

**By-products of chemical warfare.** A. A. FRIES (Ind. Eng. Chem., 1928, 20, 1079—1084).—Subsidiary activities of the U.S.A. chemical warfare service have included the following. The protection of piling against marine borers by diphenylaminechloroarsine in creosote or fuel oil was studied, long-period test pieces having been put down in 1924. The toxicity of sodium fluosilicate and a special calcium arsenate (20%  $As_2O_3$ ) to the cotton boll weevil has been investigated. These are equal weight for weight to ordinary calcium arsenate. Paints for the protection of ships' bottoms have been studied, and the oxides of copper and mercury are recommended as toxic agents with rosin modified by coal tar or synthetic resins as vehicle. A mixture of cyanogen chloride with hydrogen cyanide has been introduced as a fumigant, the lachrymatory effect of the former rendering the mixture much safer than hydrogen cyanide alone. Masks for protection against this mixture, against ammonia, and against carbon monoxide have been designed. Iodine pentoxide is used as a filling in those of the last type and also as a carbon monoxide detector. C. IRWIN.

**Technique of mechanical production of highly disperse solutions of solid substances in the ball-mill.** K. BERGL and J. REITSTÖTTER (Kolloid-Z., 1928, 46, 53—55).—The mechanics of the colloid mill are discussed and improvements are suggested. E. S. HEDGES.

**Adsorption from solutions and examination of the adsorptive power of medicinal charcoal.** T. SABALITSCHKA and K. OEHLKE (Pharm. Zentr., 1928, 69, 629—639, 645—650).—The partial and total adsorptions of methylene-blue from 0.3% aqueous solution, mercuric chloride from aqueous, 90% alcoholic, and 0.1*N*-hydrochloric acid solutions, iodine from 0.025*N*-solution in 50% alcohol, phenol from 0.15% aqueous solution, and nicotine from 0.05*N*-aqueous solution by purified and unpurified, dried and undried commercial samples of beechwood, lindenwood, sponge, bone, blood, and medicinal charcoals, "Carbovent," kaolin, and aluminium hydroxide have been examined. Adsorption curves are given and the effects of the removal of impurities from the adsorbents and of drying and sieving are discussed. A modification of the German Pharmacopœia VI method for determining the suitability of charcoals for medicinal purposes is proposed. E. H. SHARPLES.

**Technical sedimentation analysis. III.** F. V. VON HAHN and A. F. THÖLCKE (Kolloid-Z., 1928, 46, 44—52; cf. B., 1922, 839 A; 1923, 121 A).—The existing methods of preparation of disperse material for sedi-

mentation analysis are criticised. With tooth pastes dispersoid fractions of all particle sizes between  $40\mu$  and molecular dimensions are obtained. A single method for obtaining a paste in its primary particles has not been discovered. In many cases the best way is to make the substance into a paste with a soap solution, and in others by Soxhlet extraction with ether, or by boiling with alcohol followed by suspension with water, or by a combination of these methods. The ease with which tooth paste can be suspended in distilled water is related to the surface activity of the paste. E. S. HEDGES.

**Dehumidification of air.** C. S. KEEVIL and W. K. LEWIS (Ind. Eng. Chem., 1928, 20, 1058—1060).—It is possible partially to dry unsaturated air by water-cooling without bringing the whole of the air to its saturation point. This fact is discussed with consideration of the temperature and humidity gradients through the surface film, and it is shown that at low humidity the direction of change in the condition of air in contact with water, as shown in the humidity diagram, is towards the point on the saturation curve corresponding to the surface-water temperature. This conclusion was confirmed by passing unsaturated air through a column the walls of which were wetted with water at a temperature below the dew point of the air. The entrance and exit humidities were plotted against the temperatures. For nearly saturated air the curves no longer intersect the saturation curve—an effect which is attributed to condensation in the surface film. The latent heat of condensation in these cases is liberated at the interface and not in the main body of the air. C. IRWIN.

**Waste-heat drying system.** WINDSOR and WESTEN-DICK. **Dorr classifiers for clay.** ANABLE.—See VIII.

### PATENTS.

**[Tunnel] kiln.** H. M. ROBERTSON (U.S.P. 1,683,807, 11.9.28. Appl., 28.1.28).—The kiln is provided with a settling chamber (having a clean-out port at one end) which connects a fuel port with a longitudinal combustion chamber. H. ROYAL-DAWSON.

**Apparatus for removing large-sized particles and aggregates from certain finely-divided powders etc.** G. GALLIE and B. D. PORRITT, ASSIS. to RES. ASSOC. OF BRIT. RUBBER & TYRE MANUFRS. (U.S.P. 1,688,307, 16.10.28. Appl., 20.3.26. U.K., 7.10.25).—See B.P. 260,741; B., 1927, 32.

**Emulsification apparatus.** G. C. HURRELL (U.S.P. 1,685,424, 25.9.28. Appl., 12.11.27. U.K., 13.11.26).—See B.P. 285,159; B., 1928, 320.

**[Firing of powdered-fuel] furnaces or combustion chambers.** E. COCKIN (B.P. 297,345, 16.3.27).

**Burners for pulverised fuel.** STURTEVANT ENGINEERING Co., LTD., and F. W. R. WILLIAMS (B.P. 297,545, 25.7.27).

**Production of fire-extinguishing foam or foam-producing liquids.** EXCELSIOR FEUERLÖSCHGERÄTE A.-G., and H. BURMEISTER (B.P. 276,696, 29.8.27. Ger., 28.8.26).

**Process and apparatus for producing foam.** R. SCHNABEL, J. JACOBSEN, and EXCELSIOR FEUERLÖSCHGERÄTE A.-G. (B.P. 297,782, 24.5.27).

**Absorption refrigerating apparatus.** ELECTROLUX, LTD., ASSEES of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 290,207 and 275,188, 30.6.27. Ger., [A] 26.7.26, [B] 31.7.26).

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Low-temperature carbonisation.** [Illingworth plant.] D. MUIR (Gas J., 1928, 184, 30—34).—The Illingworth low-temperature plant at Treforest is described. Each retort consists of 18 H-section cast-iron conductors, each 14 in. wide and 12 ft. long, and is slightly tapered from top to bottom (cf. B., 1926, 228, 428). The capacity of each retort is 30 cwt. The flues are of high-silica fireclay. The temperature in the combustion chambers is maintained at about 700°, falling to 650° in the top section. After carbonisation the coke is dropped into a drum containing wet washed smalls, the drum is rotated, and the contents are then screened. This operation serves to cool the coke, dry the coal, and provide an automatic blend suitable for charging into the retorts. The coke forms a good smokeless fuel suitable for either domestic or commercial purposes. From a coal containing 9.6% of moisture, 35.0% of volatile matter, and 4.6% of ash, 15 cwt. of coke and 17.5 gals. of tar per ton of dry coal were obtained. A balance sheet based on the carbonisation of 220 tons of coal per 24 hrs. for 300 working days per year shows a profit of 6s. 2d. per ton.

A. B. MANNING.

**Chemistry of coal. V. Maturing of coal considered from the point of view of its benzene-pressure extraction.** W. A. BONE, L. HORTON, and L. J. TEI (Proc. Roy. Soc., 1928, A, 120, 523—545; cf. Bone and Quarendon, B., 1926, 305).—Recent criticisms by Colman (J.S.C.I., 1925, 44, 298), by Fischer (B., 1926, 393, 858), and by Cockram and Wheeler (B., 1927, 401, 802) of the benzene-pressure extraction process are discussed. It can be shown experimentally that, contrary to the opinion of Colman, the substances removed by benzene-pressure extraction were not produced by thermal decomposition, in the sense of any real breakdown of the coal substance, but were pre-existent in it, either as such or more probably in some loose molecular association with the coal complex. A comparison between the extraction methods used by Fischer and by the authors indicates that Fischer's method leads to less complete extraction, especially in regard to fraction IV. It is found that in the case of some bituminous coals, fraction II may contribute to the binding properties as well as fractions III and IV. No case has been found in which fraction I has any influence on the coking propensities, nor has it been

found possible to correlate the two in any way. With the object of discovering how the various typical substances comprised in the benzene-pressure extract from a bituminous coal have originated and been developed, an examination has been made of the Morwell brown coal and a number of coals from the Western Canadian coalfield, including a Saskatchewan brown lignite, a series of four black lignites, and a non-coking, semi-bituminous coal. Both fractions I and II of the benzene extract from bituminous and sub-bituminous coals have their counterparts in corresponding fractions obtained from both brown coals and lignites, but although fractions III and IV could also be traced through their counterparts in the laminated black lignites examined, nothing similar to them was found in either the Morwell brown coal or the Saskatchewan brown lignite. In the brown coal and lignite the places of fractions III and IV of the maturer coals were taken by phenolic esters and phenols respectively. It is therefore considered probable that fractions III and IV have originated in the phenols and phenolic esters found in the brown coals. It appears that the general effect of maturing is progressively to diminish the oxygen contents of the substances comprised in the various fractions of the benzene extracts, as well as of the benzene-extracted residue.

L. L. BIRCUMSHAW.

**Reactivity of coke.** F. J. DENT (Gas J., 1928, 184, 199—200).—Methods for determining the reactivity of cokes in steam, carbon dioxide, and oxygen are described (cf. B., 1926, 970; 1927, 833). Further experiments showed that on blowing air through a column of hot graded coke the highest temperatures are attained by cokes unreactive to carbon dioxide. Ignition-point determinations indicate that the carbonisation temperature is an important factor, high-temperature cokes having high ignition points, and conversely. The chemical properties desirable in cokes for various purposes, viz., for the cupola, blast furnace, producer, water-gas generator, and the domestic grate, are discussed in the light of these experiments.

F. J. DENT.

**Coagulation of active charcoal.** O. SPENGLER and E. LANDT (Z. Ver. deut. Zucker-Ind., 1928, 549—565).—Suspensions of the charcoal Supranorit 2X are stabilised [peptised] by caustic soda at a concentration of 0.0001N or less, but coagulated at about 0.1N. Coagulation is also brought about by hydrochloric acid and by salts. 1 g. of charcoal shaken with 120 c.c. of liquid is coagulated by the presence of 15 c.c. of 0.1M-sodium chloride, 10 c.c. of 0.1M-ammonium chloride, 10 c.c. of 0.01M-calcium chloride, or 5 c.c. of 0.01M-zinc sulphate. By the use of a polarisation photometer it is possible to follow quantitatively the course of sedimentation. The cations tried exhibit the following relative activity: methylene-blue > H > K > NH<sub>4</sub> > Na > Li; Cu > Pb > Zn; Ba > Sr, Ca > Mg > H; NHEt<sub>3</sub> > NH<sub>2</sub>Et<sub>2</sub> > NH<sub>3</sub>Et > NH<sub>4</sub>. The bivalent ions are about 30 times as active as the univalent ions, and the introduction of each ethyl group into the ammonium radical increases the activity about 1.5 times.

F. E. DAY.

**Temperatures of welding flames.** WEHRMANN (Gas- u. Wasserfach, 1928, 71, 950—952).—The factors

controlling the theoretical flame temperatures of oxy-acetylene and oxy-illuminating gas flames are discussed. The temperature distribution in such flames has been determined by introducing gradually along the axis of the flame cones of refractory material of known m.p., and observing the point at which they melt. Although an oxy-illuminating gas flame develops a slightly lower maximum temperature than an oxy-acetylene flame of the same listed power, the greater efficiency of the latter is not due to this difference, but is due to the greater total quantity of heat produced per unit time.

A. B. MANNING.

**Analysis of gas oils and hydrocarbon oils from tars.** R. H. GRIFFITH (J.S.C.I., 1928, 47, 21—24 r).—For the determination of different classes of hydrocarbons present in gas oils or tars, a 200 c.c. sample is first distilled from a 250 c.c. flask to give a b.p. curve; 50 c.c. of the distillate are then shaken for 15 min. with 80% sulphuric acid, and the remaining oil, after washing, is distilled to the same end-point. Loss is due to removal of unsaturated substances. Further treatment of the residual oil with 96% sulphuric acid removes aromatic hydrocarbons, and determination of the aniline solution point of this final residue enables the naphthenes to be determined by reference to a standard curve. Figures are also given which show the relation between the aniline solution point of a paraffin hydrocarbon and its b.p. or mol. wt. Examples are included which show the accuracy of the method when applied by independent observers, and demonstrating the results given by a wide range of hydrocarbon oils.

**Chemical composition of Rumanian crude [petroleum] oils according to their origin and geological occurrence.** N. DANAILA and (Miss) V. STOENESCU (Petroleum, 1928, 24, 1303—1309).—The composition (% of aromatic, olefine, cycloparaffin, and paraffin hydrocarbons) and physical properties are given of 18 Rumanian crude oils and of their fractions, b.p. 65—95°, 95—115°, 115—145°, and 145—300°. Of these oils four come from the Oligocene, ten from the Miocene, and four from the Dacian formation. The composition varies not only from one formation to another (even within the same locality), but also within the same formation. Hence it is impossible to correlate geological origin and chemical composition. The following classification of Rumanian petroleum is based on the content of volatile aromatic hydrocarbons: (1) oil from the Miocene at Moreni or the Dacian at Baicoiu, 4% of aromatics; (2) oil from the Dacian at Moreni or Guraoncitei or from the Miocene at Arbanasi, 2½%; (3) from the Oligocene at Solont, Moinesti, Zemes, or Bustenari, or from the Miocene of Bustenari-Gropi or Runcu, 1—1½% of light aromatic hydrocarbons.

W. S. NORRIS.

**Artificial ageing of mineral oils.** II. F. EVERS and R. SCHMIDT (Wiss. Veröff. Siemens-Konz., 1928, 7, (i), 343—371; cf. B., 1927, 98).—Artificial ageing tests of oils for insulating purposes are carried out by heating the oil at 120° in a closed tube filled with oxygen after previously mixing the oil with a quantity of silica gel impregnated with a catalyst. The catalyst is made by stirring 60 g. of silica gel with 44.48 g. of ferrous sulphate crystals dissolved in water and, after oxidation has taken

place, soaking the mass in a solution of 14.13 g. of ammonium heptamolybdate in 100 c.c. of water, drying, and heating at 410° until ammonia and sulphuric acid are completely expelled. This quantity of catalyst suffices for 15 g. of oil. Curves obtained for American and Russian white oils are of the form  $y = c \log x + k$  when the oxygen absorption is plotted against the time; the values of the constants  $c$  and  $k$  vary with the temperature and with the composition, physical character, and proportion of catalyst used. The oxidation that takes place in the test is practically confined to the constituents with a double linking, e.g., cetene, which become converted into fatty acids with the liberation of hydrogen, carbon monoxide, and carbon dioxide; the presence of unsaturated fatty acids appears to have no influence on the rate of oxidation.

A. R. POWELL.

**Determination of the calorific value of Diesel oil.** F. V. WARNOCK (Engineering, 1928, 126, 418).—When a bomb calorimeter is not available the calorific value of a fuel oil can be determined within 2—3% by means of a Darling calorimeter. A mixture of the oil (0.75—1.5 g.) with about twice its weight of calcined kieselguhr is loosely packed into a crucible, then covered with a layer of the plain kieselguhr and fired with an iron wire fuse. Heat losses from the instrument may be determined by tests with an oil of known calorific value.

R. H. GRIFFITH.

**Pressure of paraffin wax and other oil products at various temperatures and constant volume.** L. SELSKI (Azerbeid. Neft. Choz., 1928, No. 4, 69—73).

**Adsorption from solutions.** SABALITSCHKA and OEHLKE.—See I. **Synthetic hydrocarbons from methane.** FISCHER and others. **Benzene and toluene from xylene etc.** JUCHNOVSKI.—See III. **Solid carbon dioxide.** HOWE.—See VII. **Road surfacing materials.** BUTTERFIELD.—See IX. **Coke-oven gas for Siemens-Martin furnaces.** BULLE.—See X. **Carbon blacks and rubber.** COX and PARK.—See XIV.

#### PATENTS.

**Apparatus for treatment of carbonaceous matter to obtain products therefrom.** P. DVORKOVITZ (B.P. 296,793, 8.3.27. Cf. B.P. 192,816; B., 1923, 301 A).—The retorts are arranged in pairs, one above the other, in such a way that after treatment in the upper retort the material can be discharged therefrom into the lower retort and subjected therein to further treatment. Each retort is provided with a rotatable screw or other means of stirring the charge. The retorts are heated externally by the hot combustion products from a furnace, while at the same time a current of preheated neutral gas is passed downward through the material in order to carry off the oils. The first stage of the treatment yields lighter oils, and the second stage heavier oils of the paraffin series.

A. B. MANNING.

**Production of gases.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,713 and 296,751, 2.4.27. Addns. to B.P. 214,544; B., 1924, 549).—(A) A producer using granular fuels, as described in the main patent, is provided with a grate composed of two layers of stiffened grate bars arranged in such a way that the slots of the lower sets of bars are masked by the bars of the upper set, the bars being so spaced that neither

the fuel nor the ashes can fall through the grate. The bars slope longitudinally towards clinker ports or pockets. The grate may be of the travelling type. (B) Means are provided for supplying air and steam at a number of different levels in the producer as well as below the grate. This permits complete combustion of the fine dust and some control over the composition of the gas produced. The storage vessel is kept under the same pressure as the air supplied to the producer.

A. B. MANNING.

**Purifying gases and gaseous mixtures.** J. H. BRÉGEAT (B.P. 296,925, 24.11.27).—The gases are scrubbed with the hydrogenated derivatives of naphthalene, hydrogenated cresols, or hydrogenated terpenes. Naphthalene may be removed from coal gas by scrubbing with tetralin, and may then be recovered in a saleable crystalline form by cooling the solution. The tetralin vaporised by the gas will partly condense in the cooler parts of the distributing pipes, thus aiding in the removal of previously formed deposits of naphthalene; the condensed tetralin containing naphthalene in solution can be collected from siphons attached to the distributing pipes.

A. B. MANNING.

**Oxidation of hydrogen sulphide in gas mixtures to sulphur.** I. G. FARBENIND. A.-G., Asses. of A. ENGELHARDT and O. MOTSCHMANN (G.P. 447,757, 30.9.25).—In the oxidation of hydrogen sulphide by means of an active carbon or other large-surface catalyst the gases are kept so moist as to be nearly at the dew-point.

C. HOLLINS.

**Treatment and fractionation of cracking-plant vapours.** E. H. LESLIE and E. M. BAKER (U.S.P. 1,684,771, 18.9.28. Appl., 11.5.23).—Superheated hydrocarbon vapours from a vapour-phase cracking plant are passed up a fractionating column, and a considerable portion of the liquid obtained by condensing the vapour escaping from the top of the column is returned at the top to scrub the ascending vapours.

L. A. COLES.

**Apparatus for fractional extraction of petroleum hydrocarbons with alcohol.** T. A. WERKENTHIN, Assr. to SOLAR REFINING Co. (U.S.P. 1,680,353, 14.8.28. Appl., 4.11.25).—Petroleum, freed from gasoline and kerosene, is extracted with anhydrous alcohol introduced through pipes at the bottom of the oil tank. The extract overflows through a settler into a still where alcohol is recovered and a cracking oil (35% of the petroleum) is left. The residual oil undissolved by the alcohol is run into a second alcohol-extraction tank where light lubricating oil (10%) is dissolved out. A third (35%) and fourth (10%) extractions give heavier lubricating oils and the residue is asphalt (10%).

C. HOLLINS.

**Manufacture of hydrocarbons.** I. G. FARBENIND. A.-G. (B.P. 271,452, 9.5.27. Ger., 22.5.26).—The catalytic reduction of the oxides of carbon by hydrogen to produce liquid hydrocarbons and their oxygen derivatives is carried out by passing the mixed gases over catalysts of iron, cobalt, or molybdenum, formed by the decomposition of the corresponding metal carbonyls.

A. B. MANNING.

**Manufacture of hydrocarbons.** C. ARNOLD. From STANDARD DEVELOPMENT Co. (B.P. 296,536, 8.8.27).—

Finely-divided solid fuel is treated with a hydrocarbon oil and an aqueous liquid in order to separate the bulk of the ash with the latter. The solid fuel moistened with oil is then treated with hydrogen at an elevated temperature and under high pressure, if necessary in the presence of a catalyst. The product is subsequently distilled.

A. B. MANNING.

**Cracking of hydrocarbons.** E. W. ISOM and C. L. PARMELEE, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,675,558, 3.7.28. Appl., 3.3.23).—In a cracking process deposition of carbon is minimised by adding a solvent oil to the partly cracked oil so as to keep the tar in solution. A suitable plant is described.

C. HOLLINS.

**Apparatus for cracking hydrocarbons.** E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,683,184, 4.9.28. Appl., 11.6.27).—The hydrocarbons are distilled under pressure by circulation through heating coils to and from a horizontal cylindrical drum having a centrally disposed bed of a filtering or absorbent medium. The oil enters the coils from beneath this bed and the cracked oil is discharged into the space above the bed, whence the vapours are also withdrawn for refluxing and condensation.

C. O. HARVEY.

**Method of distilling hydrocarbons.** F. E. WELLMAN, Assr. to KANSAS CITY GASOLINE Co. (U.S.P. 1,680,580, 14.8.28. Appl., 3.6.22).—Cracking stock is distilled at high cracking temperature and pressure in the first zone, and the vapour produced is fractionated in the second zone, uncondensed vapour from which is conducted off and condensed; the heavier condensed portion is redistilled in a third zone at a temperature lower than that in the first cracking zone, the vapour produced being fractionated in a fourth zone. The light vapour from the fourth zone is removed and condensed but the heavier fraction from this zone is returned to the third zone. All the heavier fractions from the third zone are returned in liquid phase to the first zone.

W. S. NORRIS.

**Fractional distillation [of hydrocarbons].** J. W. LEWIS, JUN., Assr. to ATLANTIC REFINING Co. (U.S.P. 1,680,421, 14.8.28. Appl., 25.3.26).—The vapour of the oil is passed counter-current to and in contact with reflux oil, part of which is withdrawn from an intermediate point in the fractionating column and is passed counter-current to and in contact with steam, whereby low components are removed, thus yielding an intermediate fraction of higher flash point; the other part of the reflux oil is continued counter-current to the vapour in an earlier stage of the fractionating system.

W. S. NORRIS.

**Cracking and fractionation of hydrocarbon mixtures and apparatus therefor.** C. ARNOLD. From STANDARD DEVELOPMENT Co. (B.P. 296,919, 22.10.27).—A stream of a hydrocarbon oil is heated to the cracking temperature, maintained there for a sufficient time to permit any desired degree of cracking, and then passed to a fractionating tower provided with a controlled reflux, in which fractions heavier than kerosene are separated. The uncondensed vapours are passed about midway into another fractionating tower in which the kerosene and gasoline fractions are separated.

A. B. MANNING.

**Conversion of hydrocarbons of high b.p. into compounds of lower b.p.** I. G. FARBENIND. A.-G. (B.P. 270,313, 28.4.27. Ger., 1.5.26. Addn. to B.P. 268,796; B., 1928, 663).—The following metals may also be employed in the process: titanium, zirconium, niobium, tantalum, thorium, uranium, copper, or thallium, or the metalloids boron and silicon or their alloys. F. G. CROSSE.

**Working with hydrocarbons at high temperatures.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,683, 296,693—4, and 296,752—3, Appl., [A—E] 3.3.27).—In order to prevent or to diminish the deposition of carbon or the formation of coke in certain processes in which hydrocarbons are subjected to high temperatures, the surfaces which come into contact with the hydrocarbons at these temperatures are (A) made of a noble metal, ferrosilicon, alloys of chromium, manganese, molybdenum, or cobalt, or steels containing chromium, manganese, or tungsten; (B) made of aluminium; (C) coated with lustrous carbon (cf. K. A. and U. Hofmann, B., 1926, 802); (D) made of metals containing, or having deposited thereon, small quantities of boron, arsenic, antimony, bismuth, phosphorus, selenium or a compound thereof, or silicon; (E) made of molybdenum, tantalum, or chromium. A. B. MANNING.

**Producing diolefines from naphtha, naphtha fractions, and naphtha residues.** B. W. BYSOW (B.P. 297,231, 20.10.27).—The materials, preheated to 300—400°, are decomposed in a short zone at 750—1000° in the presence or absence of catalysts, and the products are allowed to expand and to be cooled as rapidly as possible, the whole process being conducted under reduced pressure. The gases are subsequently condensed and fractionated. The pyrogenesis may be carried out in the presence of inert gases. W. S. NORRIS.

**Treating oil shales and other bituminous materials.** W. W. BLAISDELL (U.S.P. 1,684,007, 11.9.28. Appl., 3.5.23).—Before removal of the hydrocarbons by distillation, the material is preheated in a separate chamber by means of the residual gases remaining after condensation of the hydrocarbons. Air is mixed with the residual gases to cool them before they are introduced into the pretreatment chamber, and vapour is not allowed to escape while the material is being transferred from the pretreatment chamber, in which it is agitated, to the distillation chamber. F. G. CLARKE.

**Refining or fractionating mineral oils and mineral oil products.** A.-G. F. KOHLENSÄURE-IND., and E. B. AUERBACH (B.P. 277,946, 23.8.27. Ger., 25.9.26).—The materials are treated at ordinary temperature in a steel container with liquid carbon dioxide, and the oil-carbon dioxide solution after being separated is freed from the solvent, which is re-liquefied and returned to the process. F. G. CROSSE.

**Removal of paraffins from fluid hydrocarbons.** AKTIEBOLAGET SEPARATOR-NOBEL and K. G. MALM (B.P. 296,805, 7.6.27).—The paraffins are precipitated, partly at least in the crystalline state, by cooling, and are then separated from the liquid hydrocarbons after dilution of the latter with a solvent which does not dissolve the paraffins at low temperatures. The

centrifugal separator is provided with mechanical means for carrying away the paraffins. Certain products may be first distilled, and fractions thereby separated from which the paraffins are easily recovered by filtration, the residue being then treated as above.

A. B. MANNING.

**Compound of kerosene and alcohol.** W. HUESSEY (B.P. 267,530, 10.3.27. Ger., 10.3.26).—Motor fuel, for addition to petrol, is prepared by preheating at 160° and 10 atm. a mixture of kerosene and commercial alcohol (containing 5—50% of water), passing the vapours produced through molten lead maintained at 400—500°, and condensing the issuing vapour. The kerosene-alcohol ratio may be, for example, 90:10, and known substances, e.g., bismuth or cadmium, may be used to lower the m.p. of the lead.

W. S. NORRIS.

**Lubricants.** STANDARD OIL DEVELOPMENT Co., and C. ARNOLD (B.P. 297,068, 9.5.27. Addn. to B.P. 260,602).—In a modification of U.S.P. 1,628,646 (B., 1927, 516) the soap may be formed in the body of the oil by adding the desired base and fatty acid in substantially combining proportions (or by adding the base and an excess of a saponifiable oil, e.g., maize oil) and heating the mixture to 176—190°. When the already formed soap is to be added to the oil, it may be added as an aqueous solution, e.g., a 10% solution. Alternatively, a concentrated solution or gel of soap in oil may be prepared, and this then mixed with more oil in the absence of air (to prevent the formation of an emulsion). W. S. NORRIS.

**Means for preventing sludging in transformer and like oils maintained at high temperatures.** BRIT. THOMSON-HOUSTON Co., LTD., Asses. of D. C. COX (B.P. 278,365, 28.9.27. U.S., 28.9.26).—Sludging of the oil in, for example, a transformer is reduced and its useful life prolonged by maintaining a quantity of fuller's earth in contact with the oil. About 6% (by vol.) of fuller's earth is said to increase the useful life of the oil 2—3 times. W. S. NORRIS.

**Method of disposing of acid sludge and spent clay in oil-refining plants.** J. M. CORY and F. H. BUNKE, Assrs. to SOLAR REFINING Co. (U.S.P. 1,682,369, 28.8.28. Appl., 27.1.26).—The sludge is agitated with steam, allowed to settle, and the separated acid removed. Spent clay, fuller's earth, coal dust, or other spent oil-purifying material is then added, together with sufficient fuel oil, tank bottoms, or similar oil of low sp. gr. to give a liquid product suitable for use in liquid-fuel burners, and the whole is agitated with steam. The spent purifying material may be added suspended in oil, tar, etc. F. G. CLARKE.

**Distillation of coal and similar carbonaceous substances.** C. H. PARKER (U.S.P. 1,687,989—1,687,991, 16.10.28. Appl., 27.1.28. U.K., 25.9.26).—See B.P. 287,584—7; B., 1928, 394.

**Stable suspension and paste of coal.** R. GRIESSBACH and J. EISELE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,681,335, 21.8.28. Appl., 23.3.27. Ger., 24.3.26).—See B.P. 292,217; B., 1928, 593.

**Producing dry gaseous fuel.** E. R. GODWARD. ASSR. to ECLIPSE PETROL ECONOMISER SYSTEM Co., LTD,

(U.S.P. 1,686,610, 9.10.28. Appl., 23.10.25).—See B.P. 258,222; B., 1927, 930.

**Manufacture of fuel [water-]gas.** F. WINKLER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,687,118, 9.10.28. Appl., 27.9.23. Ger., 27.9.22).—See G.P. 437,970; B., 1927, 548.

**Resolving emulsions of tar or oil.** H. W. ROBINSON and D. W. PARKES (U.S.P. 1,687,314, 9.10.28. Appl., 23.2.27. U.K., 30.3.26).—See B.P. 268,547; B., 1927, 469.

**Constructive conversion of heavy into light hydrocarbons.** W. E. SHORE (U.S.P. 1,687,595, 16.10.28. Appl., 26.12.23).—See B.P. 231,625; B., 1925, 492.

**Plant for washing coal and other minerals.** A. FRANCE (B.P. 281,605, 26.8.27. Belg., 4.12.26).

**Atmospheric gas burners.** J. & C. G. BOLINDERS MEKANISKA VERKSTADS AKTIEBOLAG, and E. K. BOLINDER (B.P. 297,647, 26.3.28).

**Petroleum-like liquid** (Addn. F.P. 31,758). **Phenols and cresols from tar** (G.P. 446,783). **Phenols of coal tar** (G.P., 447, 540).—See III. **Curing cementing materials** (B.P. 291,784).—See IX. **Soaps from mineral oils** (U.S.P. 1,681,237).—See XII. **Plastic material from peat** (U.S.P. 1,681,155).—See XIII.

### III.—ORGANIC INTERMEDIATES.

**Synthesis of benzene hydrocarbons from methane under ordinary pressures and without catalysts.** F. FISCHER [with H. PICHLER, K. MEYER, and H. KOCH] (Brennstoff-Chem., 1928, 9, 309—316).—The yields of tar and light oil formed when methane is passed through a heated tube have been determined. A relatively high temperature (above 1000°) and a high gas velocity are necessary to obtain satisfactory yields of liquid products. The formation of free carbon increases rapidly with rise of temperature, but can be prevented or diminished by correspondingly increasing the gas velocity. Quartz and porcelain are the most suitable materials for the tube; iron and copper favour the separation of carbon. Up to 12.9% of the methane has been converted into light oil and tar by one passage through a heated quartz tube. If the methane is diluted with other gases a higher reaction temperature is necessary in order to bring about the same percentage conversion. The tar contains considerable quantities of naphthalene; the presence of anthracene and phenanthrene has also been established. The light oil consists principally of benzene and toluene. A. B. MANNING.

**Preparation of benzene and toluene from technical xylene and from solvent naphtha.** G. L. JUCHNOVSKI (Ukraine Chem. J., 1928, 4, [Tech.], 65—87).—Solvent naphtha or technical xylene mixed with hydrogen is passed through metallic tubes heated at 700—800°, and the action of the substance of which the tube is composed, and of various catalysts placed within it, on the yield of benzene or toluene is studied. An unlined iron tube has too intense a catalytic action, less than 54% yields of products boiling below 130° being obtained whilst much soot is deposited. Even lower yields

(43%) are obtained when the tube is packed with iron filings, or with nickel (50%). 64% yields are obtained with iron tubes lined and packed with ferrous sulphide, no soot being deposited, whilst the use of nickel sulphide leads to only 47% yields. Only 43% is given by iron tubes packed with copper turnings; this yield is raised to 56% by lining the tube with ferrous sulphide, whilst replacing it with a copper tube gives a yield of only 2% more. Where sulphide-lined iron tubes packed with alumina catalyst are used, the yields are at first low, owing to oxidation of hydrocarbons, but after using the same catalyst eight times up to 74% yields of products boiling below 135° are obtained, and with this catalyst, which appears to be the best for technical use, no soot formation occurs. The use of magnesia leads to yields of less than 50%. Below 775° no benzene is produced, whilst at 815° the product contains about 43% of benzene. The optimum temperature for this reaction is, however, 775°, as above this naphthalene and gaseous products are formed in increasingly great quantities. The use of coal gas for reduction gives tarry products, difficult to purify, and the same effect is given by substituting nitrogen for hydrogen.

R. TRUSZKOWSKI.

**Butyl alcohol.** GABRIEL.—See XVIII.

### PATENTS.

**Conversion of methane into a petroleum-like liquid.** A. GOUDET (Addn. 31,758, 9.12.25, to F.P. 613,146; cf. B.P. 255,493; B., 1927, 868).—The fission of the methane into olefines and hydrogen is facilitated by passing the gas at the higher temperatures over a layer of calcium fluoride, to which may be added sulphides, selenides, or tellurides. C. HOLLINS.

**Chlorination of acetylene.** HOLZVERKOHLUNGS-IND. A.-G. (Austr. P. 106,813, 16.8.23. Addn. to Austr. P. 98,699. Cf. B.P. 174,635; B., 1922, 692 A).—The proportion of chlorine in the prior process is increased to 1.8 vols. to 1 vol. of acetylene with production of dichloroethylene, or to more than 2 vols. with formation of tetrachloroethane. To avoid explosions at least 1—2 vols. of steam must be present for each vol. of chlorine. C. HOLLINS.

**Preparation of a catalyst for synthesis of methyl alcohol.** SOC. NAT. DE RECHERCHES SUR LE TRAITEMENT DES COMBUSTIBLES (F.P. 610,649, 5.2.26).—An intimate mixture of copper oxide (4 pts.) and zinc oxide (96 pts.) is prepared by precipitation of the mixed salts with boiling alkali; the precipitate is dried and reduced at lowest possible temperature with hydrogen or carbon monoxide. 200—250 g. of methyl alcohol per hr. per dm.<sup>3</sup> of this catalyst are produced from hydrogen and carbon monoxide at 150 atm. C. HOLLINS.

**Catalytic production of methyl alcohol.** COMMERCIAL SOLVENTS CORP., Asses. of J. C. WOODRUFF, G. BLOOMFIELD, and W. J. BANNISTER (B.P. 279,377, 28.3.27. U.S., 25.10.26).—For the hydrogenation of carbon monoxide or dioxide to methyl alcohol a nickel catalyst prepared by reduction at 300—950°, preferably 550°, is used. A gas mixture containing 84% H<sub>2</sub> and 16% CO passed at 20 litres per hr. over 10 c.c. of nickel at 400° under 3000 lb./in.<sup>2</sup> pressure gave 6 c.c. of 50%

methyl alcohol per hr.; at 3.5 litres per hr. 1.1 c.c. of 82% methyl alcohol were obtained per hr.

C. HOLLINS.

**Sulphonation of fatty acids and their esters.**

H. T. BÖHME A.-G. (B.P. 263,117, 27.11.26. Ger., 16.12.25. Addn. to B.P. 261,385; B., 1928, 418).—By increasing the proportion of acid anhydride (or chloride) used in the sulphonation process of the prior patent more highly sulphonated products are obtained. These have excellent wetting-out and foaming properties even in acid solution, are not salted out by salt or concentrated alkali, and are useful additions to mercerisation liquors. A good product is obtained by sulphonation of a mixture of equal weights of castor oil and acetic anhydride.

C. HOLLINS.

**Rendering higher alcohols soluble in aqueous media.** H. T. BÖHME A.-G., and H. BERTSCH (B.P. 272,919, 15.6.27. Ger., 15.6.26).—Butyl and higher alcohols give clear solutions in water, alkali, or dilute (carbonising) acid when mixed with the sulphonates prepared according to B.P. 261,385 or 263,117 (B., 1928, 418, and preceding).

C. HOLLINS.

**Manufacture of formaldehyde from methylene dichloride.** E. KRAUSE and K. RÓKA, Assrs. to HOLZVERKOHLUNGS-IND. A.-G. (U.S.P. 1,679,673, 7.8.28. Appl., 23.10.24. Austr., 17.9.21).—Methylene dichloride is heated at 130–150° with 5% aqueous ammonia in a closed vessel, the ammonia being added gradually to avoid strong alkaline reaction.

C. HOLLINS.

**Manufacture of paraformaldehyde.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,847, 1.7.27).—Further to B.P. 267,768 (B., 1927, 428), the reaction gases from the catalytic oxidation of methyl alcohol vapour are cooled to 50° and washed with 60% formaldehyde solution at 50°. Water and formaldehyde condense, but the wash liquors remain at about the same concentration; excess liquor is run off continuously and on keeping changes to a tallow-like paste of paraformaldehyde.

C. HOLLINS.

**Manufacture of monoaryldiguanidine salts of substituted dithiocarbamic acids.** I. G. FARBENIND. A.-G., Asses. of E. SÖRENSEN (G.P. 448,631, 24.3.25).—A mixture of a monoaryldiguanidine and a primary or secondary amine is treated with carbon disulphide. Phenyl- and *o*-tolyl-diguanidine phenylethylthiocarbamates and *o*-tolyl-diguanidine phenyldithiocarbamate are described. If commercial ethylaniline is used, the diethylaniline present is recovered.

C. HOLLINS.

**Manufacture of heavy-metal salts of disubstituted dithiocarbamic acid.** I. G. FARBENIND. A.-G., Asses. of E. SÖRENSEN (G.P. 448,632, 14.3.25).—An ammoniacal solution of zinc chloride, copper sulphate, or nickel sulphate is stirred with carbon disulphide and a secondary amine, e.g., methyl- or ethyl-aniline, or ethyl-*o*-toluidine; the technical mixture of secondary and tertiary amines is used with advantage. No cooling is needed, since no heat is evolved. Yields are 75–95%.

C. HOLLINS.

**Manufacture of new compounds from indene and phenols [disinfectants; resins for lacquers].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,075, 14.6.27).—Indene (or solvent naphtha, b.p.

160–190°) condenses with phenol in presence of hydrochloric acid to give an oil, b.p. above 180°/9 mm., which on dissolution in cyclohexane deposits 3-*p*-hydroxyphenylhydrindene, m.p. 88° (compound with hexamethylenetetramine, m.p. 112°), a non-toxic disinfectant. Crude cresol gives an oil, b.p. 180–230°/9 mm., and a spirit-soluble resin; *o*-cresol yields 3-(6-hydroxy-*m*-tolyl)hydrindene, m.p. 63°, b.p. 200–230°/10 mm., and a viscous oil. The product from resorcinol distils at 220–260°/2 mm. and sets to a glassy mass. 1-Xylylindene, obtained from indene and crude methylbenzyl chloride, condenses with phenol or cresols to give an alkali-insoluble, viscous oil, b.p. 200–250°/10 mm.

C. HOLLINS.

**Manufacture of unsaturated hydrocarbons, esters, and salts [camphene, isobornyl acetate, sodium acetate].** M. ALLINGER (F.P. 583,341, 28.9.23).—Cuprous oxide or a cuprous salt (or a cupric salt and a reducing agent) is used to remove halogen from organic hydrohalides or from inorganic halides, particularly in presence of acetic acid to form acetic esters or inorganic acetates. Pinene hydrochloride heated with cuprous oxide gives camphene, or, in presence of sulphuric and acetic acids, isobornyl acetate. Sodium chloride, cuprous oxide, and acetic acid, stirred for 15 min. with exclusion of air, give sodium acetate.

C. HOLLINS.

**Manufacture of highly chlorinated perylenes.** A. ZINKE and K. FUNKE, Assrs. to F. BENSA (U.S.P. 1,681,077, 14.8.28. Appl., 7.6.27. Austr., 2.4.27).—7 to 12 atoms of chlorine may be introduced into perylene by chlorination in nitrobenzene suspension in presence of aluminium chloride or antimony pentachloride.

C. HOLLINS.

**Treatment of partial oxidation products.** J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,681,185, 21.8.28. Appl., 5.11.19).—Acids are separated from aldehydes etc. in oxidised paraffins and converted into salts.

C. HOLLINS.

**Purification of partial oxidation products.** J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,681,238, 21.8.28. Appl., 14.12.25).—Unsaturated constituents of oxidised liquid hydrocarbons are hydrogenated or otherwise converted into saturated products.

C. HOLLINS.

**Conservation of food, provisions, technical and pharmaceutical preparations, etc. and manufacture of disinfecting preparations.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,074, 14.6.27).—Phenol ethers containing no free hydroxyl group have greater antiseptic power than the free phenols, are non-corrosive, do not precipitate proteins, and are insensitive to alkali. Examples are veratrol, anisic acid, and acetanisidide. Reference is made to Public Health (Preservatives etc. in Food) Regulations, 1925.

C. HOLLINS.

**Manufacture of benzene and its homologues from mixtures of oxides of carbon and hydrogen.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,179, 13.7.27).—Water-gas at 50 atm. is passed at 40 litres per hr. per litre of catalyst over an iron-silver catalyst at 370°, and the resulting mixture of olefines and paraffins, after being cooled in a heat-exchanger and washed at 30 atm. with water, is passed at ordinary

pressure through a copper tube filled with iron-free copper turnings at 800° at a speed of 60 litres per hr. per litre of tube. The gas after the first stage before washing contains 13% of olefines, 10% of ethane, and 4% of methane; in the washed gas these figures are increased to 32%, 25%, and 10%, and this mixture yields an equal weight of 80% benzene in the second stage. C. HOLLINS.

**Recovery of phenols and cresols from crude tar.** GELSENKIRCHENER BERGWERKS A.-G., and F. SCHÜTZ (G.P. 446,783, 30.9.23).—Crude tar of b.p. 170–360° is agitated with a dilute solution of sodium hydroxide containing just sufficient alkali to dissolve the phenols with low b.p. The aqueous layer is separated, filtered through sand or coke, and treated with a stream of carbon dioxide on the counter-current principle. The precipitated phenols are then separated by fractional distillation. A. R. POWELL.

**Isolation of *p*-xylenol and *m*-4-xylenol from phenols of coal tar.** GES. F. TEERERWERTUNG M.B.H., Assees. of E. MOEHRLE (G.P. 447,540, 27.2.26).—The tar-xylenol fraction, b.p. 207–211°, is sulphonated as usual and the *p*-xylenolsulphonic acid which separates on dilution and keeping is hydrolysed to *p*-xylenol with concentrated sulphuric acid and superheated steam. The sulphonation mother-liquor is added to a cold saturated solution of potassium chloride, whereby potassium *m*-4-xylenolsulphonate is at once precipitated. This re-dissolves on warming and crystallises out as the solution cools. The sulphonate is hydrolysed to give *m*-4-xylenol, b.p. 208.5–210°, as an oil which sets readily when seeded. The yields are 15–17% of *p*- and 25–30% of *m*-xylenol. C. HOLLINS.

**Manufacture of *p*-methylaminophenol.** K. FRICKER (G.P. 449,047, 3.10.23).—*p*-Aminophenol is methylated by boiling with 1 mol. of methyl sodium sulphate in water for 6–8 hrs. C. HOLLINS.

**Production of aromatic amines.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,212, 10.9.27).—Aromatic nitro-compounds are reduced to amines by hydrogen under 140–200 atm. pressure in presence of a metal catalyst (iron powder, spongy iron, nickel deposited on zeolite). Catalyst poisons (*e.g.*, sulphur) must be present in the nitro-compound and/or the reducing gas to diminish the activity of the catalyst. Reaction begins at about 100°; 150–200° is recommended. C. HOLLINS.

**Manufacture of formyl compounds of [amines of] the aromatic series.** I. G. FARBENIND. A.-G., Assees. of R. WIETZEL (G.P. 449,112, 12.3.25).—Formanilide (*etc.*) is obtained by the interaction of formamide and aniline (*etc.*) at reduced pressure (*e.g.*, 10–30 mm.) to facilitate removal of ammonia. High temperatures are unnecessary. Reaction is facilitated by presence of water (added continuously up to a total of 10–20%), or formic acid, or by a stream of inert gas. The formanilide produced is technically pure, and may be used directly for manufacture of *p*-nitroaniline. C. HOLLINS.

**Intermediate compounds for preparation of dyes.** BRIT. DYESTUFFS CORP., LTD., J. B. PAYMAN, and

E. G. BAINBRIDGE (B.P. 296,458, 2.5.27).—1:8-Naphthasultone-3-sulphonic acid is converted by phosphorus pentachloride at 140° into the 3-sulphonyl chloride, which reacts with dry ammonia to give the 3-sulphonamide, with aniline to form the 3-sulphonanilide or with cold aqueous ammonia to give  $\alpha$ -naphthol-3:8-disulphonamide. The 3-sulphonanilide is converted by aqueous sodium hydroxide into 8-sulpho- $\alpha$ -naphthol-3-sulphonanilide or by ammonia into 8-aminosulpho- $\alpha$ -naphthol-3-sulphonanilide. C. HOLLINS.

**Manufacture of amines of the hydroaromatic-aliphatic series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,484, 23.5.27).—Ethylene oxide reacts with cyclohexylamine in water at 0° to give cyclohexyl- $\beta$ -aminoethyl alcohol, m.p. 50°, b.p. 123–127°/14 mm., and cyclohexyldi-( $\beta$ -hydroxyethyl)-amine, b.p. 180–184°/14 mm. The secondary amine is also obtained from cyclohexylamine and ethylene chlorohydrin, or by catalytic hydrogenation, in presence of nickel at 130–140° and 35 atm., of a mixture of cyclohexanone and alcoholic  $\beta$ -aminoethyl alcohol. Similar products are obtained from homologous cyclic ketones and aminoalcohols, or by condensing alicyclene oxides with aminoalcohols. The products are insecticides and bactericides, and their acyl derivatives are plasticisers. C. HOLLINS.

**Purification of naphthalene.** COMP. DE PROD. CHIM. & ÉLECTROMÉTALLURGIQUES ALAIS, FROGES, & CAMARGUE (F.P. 602,408, 26.11.24).—Crude naphthalene is heated at 100° with 7% of the chloride of sulphochloroacetic acid (obtained from trichloroethylene and 100% sulphuric acid). Sodium hydroxide is added and an easily hydrogenated, pure naphthalene is distilled off in steam, leaving the impurities as a resinous, non-volatile residue. C. HOLLINS.

**Manufacture of sulphonic acids of aralkylated polynuclear aromatic or hydroaromatic hydrocarbons.** I. G. FARBENIND. A.-G., Assees. of K. DAIMLER and G. BALLE (G.P. 449,113, 23.7.24).—Saturated or unsaturated alcohols are condensed with the products from aralkyl halides and polynuclear aromatic or hydroaromatic hydrocarbons, before, during, or after sulphonation, to give compounds with improved frothing, wetting-out, and emulsifying properties for application in the dyeing and textile industries. Examples are: benzylated naphthalene treated with *n*-butyl alcohol and oleum and chlorosulphonic acid at 70–80°; naphthalene- $\beta$ -sulphonic acid with benzyl chloride, *n*-butyl alcohol, and concentrated sulphuric acid at 100–120°; benzylated naphthalene sulphonated at 60–100° with 20% oleum and chlorosulphonic acid, and the reaction mixture treated with cyclohexanol at 80°; benzylated tetrahydronaphthalene sulphonated and condensed with isobutyl alcohol. C. HOLLINS.

**Manufacture of hydrogenated naphthastyril** I. G. FARBENIND. A.-G. (B.P. 281,257, 9.11.27. Ger., 25.11.26).—Naphthastyril is hydrogenated in presence of nickel at 100–150° and 20 atm. to give 10:3:4:5-tetrahydronaphthastyril (N = 1), m.p. 125–126°. C. HOLLINS.

**Manufacture of 7-acylamino-1:4-naphthoquinones.** I. G. FARBENIND. A.-G. (B.P. 291,340,



29.5.28. Ger., 28.5.27).—7-Acetamido- $\alpha$ -naphthol is oxidised with chromic-acetic acid below 0° to 7-acetamido-1 : 4-naphthaquinone, decomp. 228—230°. The 7-benzamido-compound, decomp. 232°, is similarly prepared. C. HOLLINS.

**Manufacture of 4-nitro-2-amino-1-benzoic acid [4-nitroanthranilic acid].** I. G. FARBENIND. A.-G. (B.P. 285,877, 23.2.28. Ger., 24.2.27).—2-Chloro-4-nitrobenzoic acid is heated with 33% aqueous ammonia and copper powder at 120° in a closed vessel to give 4-nitroanthranilic acid, m.p. 279°. C. HOLLINS.

**Manufacture of derivatives and condensation products of anthraquinones [benzanthrones].** W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,129, 12.5.27).—Anthrones are condensed in sulphuric acid with cinnamaldehyde, and the products are converted by aluminium chloride or heat into benzanthrones. Cinnamylideneanthrone, heated with aluminium chloride at 100—105°, gives benzanthrone, but when heated alone or with 1-chloronaphthalene, *p*-phenetidine, sodium acetate, phosphoryl chloride, etc. it is converted into 3-phenylbenzanthrone, m.p. 181°, which is also obtainable in smaller yield by heating together cinnamaldehyde, anthrone, and the solvent or condensing agent. Cinnamylidene derivatives of the anthrones from 1-hydroxy- and 2-chloro-anthraquinones similarly yield substituted 3-phenylbenzanthrones. Treatment of 3-phenylbenzanthrones with aluminium chloride causes migration of the phenyl group to the 2-position (2-phenylbenzanthrone, m.p. 199—200°). C. HOLLINS.

**Manufacture of new condensation products of the benzanthrone series.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 296,490, 9.6.27).—3-Nitro-4-methylbenzanthrone, m.p. 243°, is converted by 60% oleum, or by a mixture of 70% oleum and chlorosulphonic acid, or by aluminium chloride in trichlorobenzene into grey to black vat dyes, together with the lactam of 3-aminobenzanthrone-4-carboxylic acid, m.p. 320°, which may be removed by extraction with acetic acid, and yields on hydrolysis the 3-amino-4-carboxylic acid, m.p. 278°. C. HOLLINS.

**Manufacture of anthrahydroquinone [dihydroanthraquinone] derivatives.** O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 297,042, 13.4.27).—2-Acylaminoanthraquinones are reduced by hydrogen at 20 atm. in presence of nickel in a solvent (pyridine, chlorobenzene) or in alcoholic suspension, or in aqueous alkali to give 2-acylamino-*ms*-dihydroxyanthracenes, which are converted by the usual methods into ethers or esters, with or without removal of the acyl group. 2-Acetamido-9 : 10-dihydroxyanthracene (dimethyl ether, m.p. 253°) gives with pyridine and chlorosulphonic acid the dihydrogen disulphate, from which the acetyl group may be removed by boiling with alkali; the dihydroxy-compound condenses with chloroacetic acid to give the glycollic acid [di(carboxymethyl) ether], m.p. 240°, from which the acetyl group may similarly be removed. C. HOLLINS.

**Production of dye intermediates [pyrazolanthrone].** I. B. ANDERSON, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 297,366, 18.3. and

25.6.27).—An  $\alpha$ -chloroanthraquinone is condensed with hydrazine sulphate in presence of calcium oxide or hydroxide, sodium carbonate, or sodium acetate at 180—200° in a closed or open vessel. With small amounts of the alkaline agent (3 mols.) an anthraquinonylhydrazine is formed, but with excess the product is the pyrazolanthrone. C. HOLLINS.

**Manufacture of acetaldehyde.** H. S. REID and W. C. HOVEY, Assrs. to CANADIAN ELECTRO PRODUCTS Co., LTD. (U.S.P. 1,687,228, 9.10.28. Appl., 21.12.22).—See B.P. 208,689; B., 1925, 114.

**Dispersing agent [for dyes] and its manufacture.** F. FELIX, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,680,778, 14.8.28. Appl., 23.5.27. Switz., 29.5.26).—See B.P. 271,898; B., 1928, 781.

**Manufacture of aromatic amino-sulphocyanoderivatives.** O. SPENGLER and W. MÜLLER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,687,596, 16.10.28. Appl., 28.4.26. Ger., 24.9.24).—See B.P. 240,420; B., 1925, 1012.

**Preparation of alkyl ethers of 3'-nitro-4'-hydroxy-*o*-benzoylbenzoic acid.** NEWPORT Co. (B.P. 273,342, 28.6.27. U.S., 28.6.26).—See U.S.P. 1,665,541; B., 1928, 474.

**Manufacture of *N*-dihydro-1 : 2 : 1' : 2' - anthraquinoneazine.** F. B. DEHN. From NEWPORT Co. (B.P. 297,692, 25.4.27).—See U.S.P. 1,634,473; B., 1927, 771.

**Manufacture of carbamide (G.P. 448,200). Catalyst for methyl alcohol and formaldehyde synthesis (F.P. 606,596).**—See VII. Sulphonic acids (B.P. 288,612).—See XII.

#### IV.—DYESTUFFS.

##### PATENTS.

**Manufacture of water-soluble condensation products from amines or acid amides and acetaldehydedisulphonic acid.** I. G. FARBENIND. A.-G., Asses. of T. MARIAM and W. BUTTENSCHÖN (G.P. 448,738, 8.4.25).—Bases are obtained in water-soluble form, from which the bases are readily regenerated by hydrolysis in presence of acid, by condensation with acetaldehydedisulphonic acid. Examples are methylamine, carbamide, *m*-aminophenol, dianisidine, chrysoidine. The chrysoidine compound dyes acetate silk reddish-yellow from a neutral bath. C. HOLLINS.

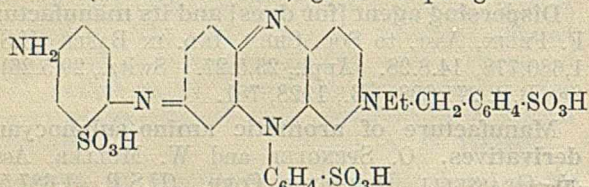
**Manufacture of dyes containing chromium.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,616, 11.1.28).—Dyes formed by coupling a diazotised *o*-aminophenol, e.g., 4-nitro- or 4-chloro-*o*-aminophenol-6-sulphonic acid, with pyrazolones, e.g., 1-phenyl-3-methylpyrazolone, are heated under pressure with aqueous chromium formate at 120°. C. HOLLINS.

**Manufacture of after-chromable dyes.** I. G. FARBENIND. A.-G., Asses. of H. GROTHOWSKY (G.P. 447,754, 14.7.25).—An aromatic aldehyde is condensed with an equimolecular mixture of an *o*-hydroxycarboxylic acid and a heterocyclic compound containing a reactive CH<sub>2</sub> or CH group, and the product is oxidised. 2 : 6-Dichlorobenzaldehyde with *o*-cresotic acid and

dihydroxythiazole or phenylmethylpyrazolone gives a chrome-blue; benzaldehyde-*o*-sulphonic acid with *o*-cresotic acid and barbituric acid a chrome red-blue.

C. HOLLINS.

**Manufacture of new direct dyes for cotton and for regenerated cellulose materials.** BRIT. DYESTUFFS CORP., LTD., and W. A. SILVESTER (B.P. 296,803, 7.6.27).—Acid dyes of the azine type, containing an external amino-group and preferably three sulphonic groups, are converted by phosgenation into direct dyes for cotton and viscose silks. Thus the dye of B.P. 13,406 of 1907 (annexed formula) gives on phosgenation a



bright blue dye; similarly, acid azine dyes made from 4-nitro-4'-aminodiphenylamine-2 : 3'-disulphonic acid, or 4-nitro-4'-amino-3-methyldiphenylamine-2'-sulphonic acid, may be phosgenated.

C. HOLLINS.

**Manufacture of dyes of the anthracene series.**

I. G. FARBENIND. A.-G. (B.P. 272,924, and Addn. B.P. 273,299 and 279,401, 15.6., 21.6., and 26.8.27. Ger., 15.6., 24.6., and 21.10.26).—(A) The sulphuric ester of leuco- $\beta$ -aminoanthraquinone, prepared by reduction of 2-acetamidanthraquinone, treatment with chlorosulphonic acid and pyridine, and hydrolysis with alkali, is oxidised with alkaline permanganate to a violet acid wool dye, which is further oxidised with nitrous acid to indanthrone. (B) Oxidation of the leuco-ester with acid ferric chloride solution gives indanthrone directly. (C) The dimethyl ether of leuco- $\beta$ -aminoanthraquinone is prepared by reduction of 2-acetamidanthraquinone with hydrogen and nickel, treatment with methyl sulphate and alkali, and hydrolysis with alcoholic hydrochloric acid of the 2-acetamido-meso-dimethoxyanthracene, m.p. 253°, so obtained. The ether is oxidised by acid ferric chloride solution to a compound, m.p. above 290°, from which by removal of methoxy-groups with concentrated sulphuric acid at 90–100° indanthrone is obtained.

C. HOLLINS.

**Manufacture of condensation products of anthraquinone.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 296,817, 10.6.27).—Glyoxal, or a compound furnishing glyoxal, is condensed in acetic acid with anthrone or  $\alpha$ -chloroanthrone to give glyoxylidenebisanthrones, which are yellow vat dyes.

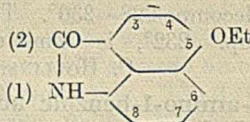
C. HOLLINS.

**Manufacture of dye preparations and their application.** SOC. CHEM. IND. IN BASLE (B.P. 293,795, 4.7.28. Switz., 12.7.27).—Bright blue-violet shades are obtained by using a mixture of *isodibenzanthrone* (brown-violet) and its dichloro-compound (red-violet). The dyes may be mixed in the form of powders, leuco-compounds, or leuco-ester salts.

C. HOLLINS.

**Manufacture of new derivatives of anthranthone.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,234, 25.10.27).—Hydroxy- and alkoxy-1 : 1'-dinaphthyl-8 : 8'-dicarboxylic acids, obtained from the corresponding naphthastyrils, are cyclised by means of

sulphuric acid or zinc chloride to give vat dyes. 5-Ethoxy-naphthastyril (annexed formula) is boiled with alkali



and the resulting amino-acid is converted by diazotisation into 5 : 5'-diethoxy-1 : 1'-dinaphthyl-8 : 8'-dicarboxylic acid (cf. B.P. 276,126 and 278,100; B., 1927,

808, 902), which when treated with concentrated sulphuric acid yields 3 : 9-diethoxyanthrone, a reddish-orange vat dye; 3 : 9- and 2 : 8-dimethoxyanthrones (orange) are similarly obtained from 5- and 4-methoxynaphthastyrils, respectively. Naphthastyril-6-sulphonic acid is hydrolysed and diazotised to give 4 : 4'-disulpho-1 : 1'-dinaphthyl-8 : 8'-dicarboxylic acid, which is converted by alkaline fusion into the 4 : 4'-dihydroxy-compound; cyclisation of this with zinc chloride in acetic anhydride yields 4 : 10-dihydroxyanthrone; the dimethyl ether is a violet vat dye.

C. HOLLINS.

**Manufacture of vat dyes.** I. G. FARBENIND. A.-G., Assees. of H. WOLFF (G.P. 447,556, 25.7.25).—Red-violet vat dyes are obtained by the action of chlorosulphonic acid on *isodibenzanthrones* in presence of selenium or its compounds.

C. HOLLINS.

**Manufacture of new [yellow] vat dyes.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,386, 30.5.27).—Yellow vat dyes are obtained by alkaline fusion of anthrapyrimidines or anthrapyrimidone [pyrimidanthrones or ketopyrimidanthrone]; the pyrimidone products are subsequently alkylated, e.g., with methyl *p*-toluenesulphonate. 2-Methylpyrimidanthrone is prepared from 2-amino-1-hydroxyanthraquinone by conversion first into the oxazole with acetic anhydride, and thence by the action of ammonia into 4-amino-2-methylpyrimidanthrone, and finally removing the amino-group.

C. HOLLINS.

**Manufacture of [vat] dyes from dinaphthylene oxide.** I. G. FARBENIND. A.-G., Assees. of R. PUMMERER and E. PRELL (G.P. 449,121, 3.1.25).—Dinaphthylene oxide is oxidised in nitrobenzene and acetic acid with lead peroxide to a violet-brown vat dye, or in nitrobenzene with benzoyl peroxide to a grey (cotton) to black (wool) vat dye. Neutral oxidation with benzoyl peroxide gives a yellow compound which is converted by sulphuric acid into a brown-red vat dye. Oxidation with iron alum also gives a brown-red vat dye, whilst ozonisation in carbon tetrachloride leads to a red dye.

C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone series.** I. G. FARBENIND. A.-G., Assees. of O. BRAUNSDORF, P. NAWIASKY, and E. HOLZAPFEL (G.P. 448,262, 21.10.24).—Benzanthronyl thio-ethers are heated with alkali. 3 : 3'-Dibenzanthronyl sulphide or *p*-tolyl 3-benzanthronyl sulphide yields *isodibenzanthrone*; 2-methoxy-3 : 3'-dibenzanthronyl sulphide, m.p. 338–340°, gives methoxy-*isodibenzanthrone*, a blue-violet vat dye; from 2 : 2'-dimethoxy-3 : 3'-dibenzanthronyl sulphide a blue vat dye results. Violet vat dyes are obtained by alkaline fusion of 8-amino-3 : 3'-dibenzanthronyl sulphide, the 8 : 8'-diamino-compound, mono- or dibrominated 3 : 3'-dibenzanthronyl sulphide.

C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone series.** A. CARPMAEL. From I. G. FARBENIND. A.-G.

(B.P. 297,133, 11.6.27).—Aluminium chloride combines with pyridine to form a well-defined compound, soluble in excess of pyridine, which is an excellent condensing agent; less aluminium chloride is used, and reaction proceeds more smoothly at lower temperatures and without frothing. Other tertiary bases may be employed in place of pyridine. 4-Benzamido-1:1'-dianthraquinonylamine, heated at 125° with aluminium chloride (1.6 pts.) and pyridine (5 pts.), gives a brown vat dye. Similarly 1:2-dianthraquinonylamine gives an orange-yellow, 6- $\alpha$ -anthraquinonylamino-*N*-methylpyridoneanthrone a brownish-orange, 1:4-di( $\alpha$ -anthraquinonylamino)anthraquinone a reddish-brown, and 2-aminoanthraquinone flavanthrone. (Cf. B.P. 278,771 and 288,291; B., 1928, 9.)  
C. HOLLINS.

**Manufacture of indigoid vat dyes.** I. G. FARBENIND. A.-G. (B.P. 282,805, 28.12.27. Ger., 24.12.26).—5-Chloro-7-methylisatin- $\alpha$ -5-chloro-*o*-tolylimine, prepared from 5-chloro-*o*-toluidine, is condensed with 5-chloro-7-methylthioindoxyl (bluish-violet), with 5:6:7-trichlorothioindoxyl (violet), with 5:7-dichloro-4-methylthioindoxyl (violet-blue), with 6-chloro-4-methylthioindoxyl (violet), or with 5-chloro-4:7-dimethylthioindoxyl (bluish-violet).  
C. HOLLINS.

**Manufacture of [indigoid] vat dyes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,376, 20.6.27).—Clear fast dyes, including navy-blue and red shades, are made by condensing a dihalogenomethylisatin, or its  $\alpha$ -chloride etc., with naphthols, anthrols, indoxyls, thioindoxyls, etc. Examples are: 6-chloro-5-bromo-7-methylisatin  $\alpha$ -chloride with 4-chloro- $\alpha$ -naphthol (navy-blue); 4-chloro-5-bromo-7-methylisatin  $\alpha$ -chloride with  $\alpha$ -anthrol (greenish-blue); 5:7-dichloro-6-methylisatin  $\alpha$ -chloride with 6-chloro-4-methylthioindoxyl (reddish-violet), or reduced with hydrogen sulphide to 5:5':7:7'-tetrachloro-6:6'-dimethylindigo (reddish-blue); 5:7-dichloro-6-methylisatin with thioindoxylic acid (red); 5:7-dichloro-4:6-dimethylisatin  $\alpha$ -chloride with 4-chloro- $\alpha$ -naphthol (navy-blue).  
C. HOLLINS.

**Manufacture of [thioindigoid] vat dyes.** I. G. FARBENIND. A.-G. (B.P. 271,906, 30.5.27. Ger., 29.5.26).—5:5'-Dichloro-4:4':6:6'-tetramethylthioindigo, from 2-chloro-*m*-4-xylylidine, m.p. 59–60°, 6:6'-dichloro-4:4':5:5'-tetramethylthioindigo, from *o*-3-xylylidine by way of 5-chloro-2-cyano-3:4-dimethylphenylthioglycollic acid, and the condensation products of the corresponding thioindoxyls with isatins, thioisatins, acenaphthaquinone, etc. (especially with the *p*-dimethylaminoanils of 5:6-dichloro-4-methyl- and 6-chloro-4-methyl-thioisatins) are very fast to light and to kier-boiling.  
C. HOLLINS.

**Manufacture of azo dyes.** O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 296,473, 1.6.27).—2:6-Dichloro-*p*-xylylidine, m.p. 47–48°, or 5:6-dichloro-*p*-xylylidine, m.p. 78–79°, obtained by chlorination of *p*-xylylidine sulphate, are coupled in substance or on the fibre with 2:3-hydroxynaphthoic or acetoacetic arylamides. The former, e.g., 2:3-hydroxynaphthoic 5-chloro-*o*-toluidide or  $\beta$ -naphthylamide, give dyeings of good fastness to chlorine and kier-boiling.  
C. HOLLINS.

**Production of metal compounds of azo dyes.** I. G. FARBENIND. A.-G. (B.P. 272,908, 13.6.27. Ger., 15.6.26).—Water-insoluble *o*-hydroxyazo dyes are converted smoothly into their chromium or copper complex compounds if a large proportion of a solvent for the dye is added to the metal formate solution. Thus the dye from *p*-chloro-*o*-aminophenol and  $\beta$ -naphthol or phenylmethylpyrazolone is heated in an autoclave with about 4 pts. of aqueous chromium formate solution and about 10–11 pts. of ethyl alcohol. Other suitable solvents are acetone, glycol, ethylene chlorohydrin, polyglycol, etc.  
C. HOLLINS.

**Manufacture of complex metal compounds of *o*-hydroxymonoazo dyes.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,819, 20.5.27).—Monoazo dyes from diazotised *o*-aminophenol ethers are boiled with chromium or copper formate under pressure at 125–130° so that the alkoxy group is hydrolysed to hydroxyl. Examples are *o*-anisidine  $\rightarrow$   $\beta$ -naphthol; 4-chloro-*o*-anisidine  $\rightarrow$  F-acid; 4-nitro-*o*-anisidine  $\rightarrow$  F-acid; *o*-anisidine-4-sulphonic acid  $\rightarrow$   $\beta$ -naphthol; *o*-phenetidine  $\rightarrow$  Laurent acid.  
C. HOLLINS.

**Production of azo dyes containing copper and chromium.** I. G. FARBENIND. A.-G. (B.P. 272,949, 17.6.27. Ger., 17.6.26).—Chromium or copper compounds of azo dyes containing less than saturation amounts of the metal are treated with copper or chromium salts to produce complexes containing both metals; or the azo dye may be treated with a mixture of chromium and copper salts. Thus the dye, 4-nitro-2-aminophenol-6-sulphonic acid  $\rightarrow$   $\beta$ -naphthol, gives with chromium salts alone a black, but with chromium and copper salts a bright violet.  
C. HOLLINS.

**Stable diazo preparations.** I. G. FARBENIND. A.-G., Assees. of K. SCHNITZSPAHN (G.P. 442,075 and 443,284, 19.6.25. Addns. to G.P. 426,033; B., 1926, 781).—Nitroarylamines, containing no sulphonic or carboxylic groups, in the form of their mineral acid salts (A) or free bases (B), are mixed dry with sodium nitrite and solid mineral acids or acid salts capable of liberating nitrous acid from nitrites. Partly dehydrated aluminium sulphate etc. or stabilising compounds such as sodium naphthalenedisulphonates may be added. Addition of sodium *isopropyl*naphthalenesulphonate improves the wetting properties.  
C. HOLLINS.

**Stable diazo preparations.** I. G. FARBENIND. A.-G., Assees. of K. SCHNITZSPAHN (G.P. 447,069, 28.12.24).—A nitroarylamine hydrochloride is mixed with sodium 1:6-naphthalenedisulphonate and sodium nitrite.  
C. HOLLINS.

**Manufacture of dicyclic derivatives of pentamethylene.** [1-Aryl-3:4-trimethylene-5-pyrazolone.] C. MANNICH (U.S.P. 1,685,407, 25.9.28. Appl., 15.10.26. Ger., 27.10.25).—See B.P. 260,577; B., 1927, 869.

**Glyoxaldianthraquinone compounds.** R. BERLINER, B. STEIN, and W. TRAUTNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,646,782, 25.10.27. Appl., 16.9.26. Ger., 30.10.25).—See B.P. 296,817, preceding.

**Diazotisable dyes.** M. BOENIGER, Assr. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,686,947, 9.10.28.

Appl., 15.10.25. Ger., 28.3.25).—See B.P. 252,957; B., 1926, 657.

**Benanthrone condensation products.** (B.P. 296,490).—See III. Lakes (B.P. 275,943).—See XIII. Photographic plates etc. (B.P. 292,140).—See XXI.

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

**Behaviour in polarised light of threads from silk coagula of various stages of dehydration and age.**

P. P. VON WEIMARN (Kolloid-Z., 1928, 46, 36—38).—The author has studied the microstructure of a silk coagulum and its behaviour in polarised light during the alteration of its consistency and elastic properties with age, and the phenomena are compared with the reverse process, *i.e.*, the swelling of natural silk threads. When natural silk threads swell in hot, concentrated sodium iodide solution the fibrils become better delineated and assume a spiral form, eventually lying perpendicular to the axis of the thread. Ultimately the fibrils liquefy to drops, which are at first spiral in form, and the mass becomes syrupy. In polarised light, the bright interference colours of the silk threads gradually disappear during swelling. To study the reverse process, a solution of silk in aqueous sodium iodide solution was coagulated by addition of concentrated sodium citrate solution to a syrupy consistency. On keeping, spiral forms can be observed under the microscope and later a thread-like structure appears; simultaneously, interference colours begin to be shown in polarised light, and these increase in brightness with age. Silk threads which have been prepared artificially in this way, and have been kept for 2 years, are not essentially different in properties from natural silk threads. E. S. HEDGES.

**Physico-chemical nature of natural silk solutions in neutral, acid, and alkaline solvents.** P. P. VON WEIMARN (Kolloid-Z., 1928, 46, 40—43).—The action of aqueous solutions of neutral salts on natural silk is compared with the effect of an excess of potassium iodide or silver nitrate on silver iodide. In the latter case a small excess of potassium iodide or silver nitrate peptises silver iodide to a sol, the particles of which dissolve in a large excess of the reagent to give a complex salt; the latter is decomposed on addition of water with formation of a sol once more. Precisely similar relations hold in the action of dilute and concentrated solutions of neutral salts on natural silk. The rôle of hydration in these phenomena is discussed, and a distinction is made between capacity and intensity of hydration. In solutions of acids or alkalis, the behaviour of silk is similar to that in neutral salt solutions only when the temperature is kept low; otherwise decomposition of the fibroin occurs and the regenerated substance differs from the original in composition. E. S. HEDGES.

**Causes of the rubber-like state of matter.**

P. P. VON WEIMARN (Kolloid-Z., 1928, 46, 38—40).—Silk coagula with elastic properties can be obtained, and examination of these has shown that they are characterised by a spiral formation of the fibrils, whilst the highly viscous liquid between the fibrils can act in the manner of a lubricating oil. Only under these conditions is elasticity observed in silk coagula, and when with age the spiral fibrils unite to form larger threads the

coagulum assumes plastic properties. By stretching, or through the syneresis which develops in time, the fibrils become oriented parallel to the axis of the threads and then the substance takes on solid properties. Threads of caoutchouc also possess the spiral form described above. When the caoutchouc has been subjected to stretching several hundreds of times in succession, the fibrils become oriented parallel to each other, but revert to the spiral form after resting.

E. S. HEDGES.

**Microbiology of wool.** R. BURGESS (J. Text. Inst., 1928, 19, 315—322 T).—The essential properties of an efficient antiseptic for the prevention of mildew in woollen goods are described. Whilst many substances such as sodium fluoride, chloramine-T, chlorinated phenols, etc. do not inhibit the growth of fungi and bacilli, others such as mercuric chloride, salicylic acid, thallium carbonate, etc., and notably sodium silicofluoride, give better protection. The protective effect of dyes is discussed, and chrome dyeing is found to afford considerable protection. The conditions of testing antiseptics, the effect of the alkalinity or acidity of the cloth under examination, and methods of detecting damaged fibres by staining tests are discussed.

B. P. RIDGE.

**Determination of china clay in sized cotton goods.** G. SMITH (J. Text. Inst., 1928, 19, 323—328 T).—A simple, direct method is described which depends on the fact that when sized cotton material is heated with hydrochloric acid (*d* 1.1), practically the whole of the inorganic material of the cotton is rendered soluble in water, whilst china clay is not appreciably attacked, and soluble salts of zinc, magnesium, calcium, and sodium present in the size are not affected. In order to allow for loss of combined water and of acid-soluble matter from the clay, a correction factor is applied to the weight of ignited clay found. The terms "natural," "dry," and "ignited" clay are defined, the possible sources of error in the method are discussed, and the conditions necessary for obtaining representative values are detailed. B. P. RIDGE.

**New reagent for detection of oxycellulose.** W. F. A. ERMEN (J. Soc. Dyers and Col., 1928, 44, 303—305).—Oxidised cellulose may be detected by staining blue with ferric ferrocyanide when immersed for 1 min. in a boiling solution of ferric ferrocyanide consisting of 250 c.c. of water and 5 c.c. each of solutions containing (per 100 c.c.) 20 g. of ferric sulphate and 25 g. of ammonium sulphate, and 33 g. of potassium ferrocyanide. Pure bleached cotton reacts slightly, and unbleached cotton more so with this reagent, but not sufficiently to interfere with the detection of oxidised cellulose. The reagent is readily reduced by sodium hyposulphite, phenol, aniline, tannic acid, wool, but not silk. Cotton may be dyed with Prussian Blue by first mordanting with tannic acid and tartar emetic, and then immersing in the cold reagent, whereas wool may be dyed directly. A suitable method for dyeing rich black shades free from brown on fur consists of mordanting the fur with Prussian Blue by treatment with the reagent and then dyeing it with *p*-phenylenediamine in the presence of an oxidising agent. Sulphur-black dyeings may be topped with Prussian Blue by

treatment with the reagent; similarly, sulphur-blues may be brightened and sulphur-yellows toned to bright greens. When treated together with the reagent, wood pulp becomes stained deep blue and cotton or linen paper remains almost unaffected. A. J. HALL.

**X-Ray methods used in determining the structure of cellulose fibres.** O. L. SPONSLER (Ind. Eng. Chem., 1928, 20, 1060—1062).—To avoid complications resulting from the presence of the central canal in cellulose fibres, small blocks of practically parallel fibres are prepared for section cutting. These may be rotated in the X-ray beam to secure longitudinal or transverse passage of the rays. A. G. POLLARD.

**Decomposition of pine wood with sulphites.** C. G. SCHWALBE and W. LANGE (Papier-Fabr., 1928, 26, 641—644).—The action of sodium sulphite solution on pine wood at 5—5½ atm. pressure continues for a period of 48 hrs. Preliminary soaking with sodium bisulphite decreased the necessary cooking period subsequently. Digestion of the wood shavings even at 170° after preliminary soaking still produced a harsh pulp. The preliminary soaking, whilst not incurring any serious chemical changes, is considered of importance in loosening incrusting material. Comparison of steam-digestion processes following preliminary impregnation with the bisulphites of sodium, calcium, and magnesium showed the last-named to be the most effective. In the production of wet-digested pulp, the best quality half-stuff was obtained by a short preliminary soaking in sodium bisulphite, followed by pressure-digestion with sodium sulphite for 8½ hrs. at 170°. In the steam-digestion trials the fibre was softened, but incrusting material was not satisfactorily removed; the pulp was characterised by high lignin content. A. G. POLLARD.

**Influence of metallic salts on the tensile strength of artificial silk.** J. EGGERT (Chem.-Ztg., 1928, 52, 794—795).—The general effects of the presence of metallic salts in bleaching and finishing baths for spun filaments are outlined and discussed. In particular, copper chloride increases the elasticity and decreases the tensile strength of both wet and dry filaments. This effect is not the result of over-bleaching produced by the activation of hypochlorite by the copper, but may be due to changes within the cellulose molecule. The importance of the prevention of contamination of bleaching baths by contact with metallic machine parts is emphasised. A. G. POLLARD.

**Turbidity and gelatinisation of viscose caused by addition of chemicals.** T. MUKOYAMA (J. Cellulose Inst., Tokyo, 1928, 4, 170—177).—A viscose solution (4.1% of cellulose and 3.4% of alkali) was treated with various salt solutions, and changes in turbidity and rate of gelatinisation were examined. The ammonium ion tends to accelerate turbidity formation and gelatinisation proportionally to the concentration used. Sodium chloride, sodium sulphate, and glycerin are much slower in their action, and their effect is less sensitive to changes in concentration. Sulphate and sulphite ions restrain the gelatinising effect of salts, especially when the sodium salts are used. A. G. POLLARD.

**Paper pulp from logging waste in the Douglas fir region.** A. H. HODGSON (Ind. Eng. Chem., 1928,

20, 819—821).—About 20% of the timber in this region is being left in the woods as waste; of this two fifths in the form of western hemlock, white fir, and Sitka spruce can be used for sulphite and mechanical pulp, whilst the remainder, Douglas fir, cedar, and pine, which has inferior pulping qualities, is best suited for lumber. F. R. ENNOS.

**Importance of hydrogen-ion concentration in the sizing of paper.** H. ROSCHER (Pulp and Paper Mag., 1928, 26, 639—640, 679—682, 715—717, 745—748).—Experiments are described which show that under ordinary manufacturing conditions engine-sized paper contains both aluminium resinate and free resin (either of which is of itself an effective sizing agent) in relative amounts depending on the  $p_H$  of the stock. Thus, at  $p_H$  3.7—3.9 the amount of aluminium resinate formed is comparatively small, whilst at  $p_H$  4.5—5.0 the combined resin in the size is converted mainly into aluminium resinate. In the first case the mechanism of sizing is probably as follows: the fibre immediately fixes any aluminium resinate that is formed and in addition adsorbs aluminium ions, the positive charge resulting therefrom enabling it to attract and fix the negatively-charged free resin. Under these conditions a large proportion of the alum is carried away in the backwater. In the second case, *i.e.*, at  $p_H$  4.5—5, the aluminium resinate is fixed by the fibre, whilst the excess of alum is hydrolysed to aluminium hydroxide in association with a small quantity of sulphuric acid; aluminium hydroxide being positively charged adsorbs the negatively-charged free resin giving an adsorption complex of the same sign which in turn is adsorbed by the negatively-charged fibres. Under these conditions very little alum appears in the backwater. D. J. NORMAN.

**Dispersion of rosin in paper sizing.** P. DELCROIX (Pulp and Paper Mag., 1928, 26, 1049—1051).—In the preparation of rosin size by passing caustic soda solution through a tower containing lump rosin, the optimum concentration of the soda solution is 5.2 g./litre. At this concentration variations in the temperature and rate of flow of the solution through the tower do not materially influence the rosin content (40 g./litre) of the resulting size, provided that the temperature does not fall below 15°, and the rate of flow does not exceed a certain maximum value, approximately 15 litres per day per litre capacity of the apparatus. D. J. NORMAN.

**Effect of beating on the sizing resistance of paper.** J. BERGER (Papier-Fabr., 1928, 37, 561—565).—The sizing effect resulting solely from beating has been investigated by preparing a range of bleached sulphite-wood papers beaten for varying periods of time in a Lampén mill and comparing them as regards their sizing resistance (measured by various flotation tests) with resin-sized printing papers of varying degrees of hardness. The results indicate that sizing resistance increases slowly with the degree of beating (wetness) of the pulp up to a point and then rapidly increases with further beating, so that it is possible to prepare beaten sheets which, though containing no added sizing agent, show a better resistance to penetration by aqueous liquids than do fully resin-sized printing papers of the same

weight. In the flotation tests for sizing resistance the most satisfactory results were obtained with an ink bath or with a bath containing 0.1% caustic soda solution, using phenolphthalein in admixture with 9 times its weight of powdered sugar as a dry indicator. For soft-sized papers the caustic soda bath gave the more reliable results, but for hard-sized papers an ink bath was preferred owing to the action of the caustic soda on the sizing agent after prolonged contact.

D. J. NORMAN.

#### Measurement of sizing strength of paper.

B. SCHAPIRA (Papier-Fabr., 1928, 26, 609—617).—Methods of determining the sizing strength of paper are collected, described, and classified according to the factor on which the method depends, thus, (1) absorption of ink, (2) absorption of interacting liquids, such as, *e.g.*, ferric chloride and tannin solutions, (3) electrolytic conductivity, and (4) the action of water, *e.g.*, in capillary rise or in causing the curling up of floating specimens of the paper. Distinction is made between surface and interior sizing strength, and the methods of determining the latter are criticised. The best relative values are given by the dry indicator method of the Bureau of Standards, although, with few exceptions, good agreement is obtained between the results of this and of the curling method. The method of Stöckigt gives fairly uniform results, but its error increases in magnitude with increasing sizing strength on account of selective adsorption. The electrolytic and ink-floating methods are too unreliable.

B. P. RIDGE.

**Determination of  $p_H$  value of paper.** B. SCHÜLZE (Papier-Fabr., 1928, 26, 625—629).—The significance of hydrogen-ion concentration in determining acidity or alkalinity is discussed and general methods of determining  $p_H$  values are described. The  $p_H$  value of paper may be found either colorimetrically by the use of indicator papers, graded with respect to their  $p_H$  range, or of the Wulff colorimeter, or electrolytically by means of *E.M.F.* measurements using a quinhydrone electrode. For samples other than raw or parchment papers the colorimeter method is recommended, using an aqueous extract of the paper, whilst for the exceptions named either the electrolytic or the indicator paper method may be used on the paper itself, without extraction. Parchment papers have  $p_H$  3—4.5, whilst other papers have  $p_H$  4.5—9 according to the conditions of manufacture; reasonable agreement is shown between results obtained by the different methods used.

B. P. RIDGE.

**Bisulphite liquors.** STEWART.—See VII.

#### PATENTS.

**Cotton fabric and its manufacture.** E. GMINDER (U.S.P. 1,679,767, 7.8.28. Appl., 23.12.25. Ger., 17.9.24).—Cotton goods are carded to produce a slight nap, and are then treated with sodium hydroxide solution (*d* 1.11 or stronger) to curl the nap, which is finally roughened and oiled to produce a woolly appearance.

C. HOLLINS.

**Manufacture of artificial silk.** BRYLSILKA, LTD., and F. W. SCHUBERT (B.P. 293,977 and 296,856, 15.7.27).—Difficulties attending the production of threads of uni-

form denier in the stretch-spinning of cuprammonium silk are obviated by placing in the spinning bath: (A) a volute taper funnel comprising a strip wound in the form of a volute spring so that the friction caused by the passage of the thread through the funnel causes fresh liquor to be drawn in through the space between the successive convolutions of the spring; (B) a spinning funnel wider at the bottom than at the top, slightly flared at the bottom, and, when two-liquor spinning is used, preferably provided at the top with an inverted, truncated conical member.

D. J. NORMAN.

**Production of artificial silk and apparatus therefor.** J. P. BEMBERG A.-G. (B.P. 275,637, 4.8.27. Ger., 5.8.26).—Strong uniform threads are obtained in the stretch-spinning of, *e.g.*, cuprammonium hydroxide solutions of cellulose if two or more groups of filaments are produced and caused to travel through the coagulating bath adjacent to but separated from each other until they have lost their tackiness, after which the groups of filaments are united into a single thread. As many funnels as groups of filaments may be provided in the spinning bath, in which case the funnels lead to a common outlet tube. Various types of apparatus are described.

D. J. NORMAN.

**Production of artificial silk or films.** S. OHSAKA (B.P. 297,047, 13.6.27).—A solution of cellulose in sodium hydroxide and carbon disulphide is mixed with not more than 20% of a protein or albumin solution associated with fatty oils. After ripening, the solution is spun into a coagulating bath, heated to 70—100°, and dried. The product has increased tenacity and elasticity both in the dry and wet state. [Stat. ref.]

F. R. ENNOS.

**Manufacture of artificial threads by the bobbin process.** O. SINDL (B.P. 297,618, 10.1.28. Ger., 15.11.27).—After leaving the coagulating bath the thread is wound on to a rotating bobbin, which moves vertically in and out of a washing bath. In its upward movement the bobbin, owing to its rotation, throws off the diluted coagulating liquid adhering to it, which is collected by a protective sheath and prevented from returning to the washing bath.

F. R. ENNOS.

**Manufacture of artificial silk threads.** ACETA GES.M.B.H. (B.P. 286,608, 5.3.28. Ger., 3.3.27).—In the dry-spinning process the threads are made to pass downwards in an ascending current of warm air in a cell subdivided by an adjustable constriction, both parts being independently connected at their upper ends to the suction pipe. The main portion of the drying air and solvent vapour is thus withdrawn at the upper end of the lower subdivision of the cell, so that only a comparatively small volume of air and vapour passes near the spinning nozzle.

F. R. ENNOS.

**Artificial silk.** E. HEYMANN (B.P. 297,364, 15.3.27).—To produce a thread of subdued lustre, a mixture of a non-volatile substance, *e.g.*, mineral, animal, or vegetable oils, aniline, etc., with a volatile substance, *e.g.*, benzene, is finely distributed throughout the spinning solution, and the resultant thread is treated for the removal of a part of the added substances by evaporation, in some cases *in vacuo*, or by means of solvents.

F. R. ENNOS.

**Manufacture of viscose silk.** I. G. FARBENIND. A.-G. (B.P. 290,231, 10.5.28. Ger., 10.5.27).—Viscose is spun into a coagulating bath containing not more than 0.1% of a zinc salt (zinc sulphate) whereby threads having finely-channelled surfaces are formed, which enhance the optical covering power and give the product a silvery, opaque appearance. F. R. ENNOS.

**Manufacture of viscose.** DU PONT RAYON Co., Assees. of G. W. BLANCO and C. HENNINGSSEN (B.P. 272,475, 23.5.27. U.S., 12.6.26).—Sheets of raw cellulose are steeped in caustic alkali solution while being held out of contact with each other as far as possible by suitable corrugations thereon, so as to permit free passage of the steeping liquor to all parts. The excess liquor is removed by pressure and re-used to steep other raw material, the presence of a relatively high percentage of hemicelluloses having no deleterious effect, but actually serving to accelerate the ripening operation. F. R. ENNOS.

**Production of viscose.** R. SAJITZ and E. POTT [CHEM. FABR. POTT & Co.] (B.P. 281,679, 30.11.27. Ger., 30.11.26).—Considerable latitude in the temperature and duration of ripening of the alkali-cellulose and of the composition of the precipitating bath is permissible if addition is made in one or more of the successive stages of the manufacture of viscose of small quantities of the following: salts of aromatic sulphonic acids; naphthalenesulphonic acids alkylated in the nucleus, their salts and other derivatives; sulphonates of hydroaromatic compounds, especially of the phenanthrene and anthracene series. F. R. ENNOS.

**Preparation of viscose cellulose compounds.** J. M. BERNARD (B.P. 273,280, 14.6.27).—Cellulose is mixed in a closed vessel with carbon disulphide, the excess of which is removed and recovered by draining and evacuation. After treatment of the product with sufficient sodium hydroxide solution to form cellulose xanthate, the residual carbon disulphide is removed by a further evacuation, and the product is dissolved in dilute sodium hydroxide solution. F. R. ENNOS.

**Imparting a silk lustre to bodies produced from viscose and other aqueous cellulose solutions.** WOLFF & Co., E. CZAPEK, and R. WEINGAND (B.P. 274,054, 21.6.27. Ger., 8.7.26).—Finely-powdered asbestos or asbestine is mixed with the cellulose solution, which is then worked up in the usual manner. F. R. ENNOS.

**Manufacture of cellulose esters.** I. G. FARBENIND. A.-G. (B.P. 291,360, 30.5.28. Ger., 30.5.27).—Cellulose is treated with a halide of a lower fatty acid, *e.g.*, acetyl chloride, in the presence of an organic base, *e.g.*, pyridine, and an indifferent organic medium, *e.g.*, chlorobenzene, which dissolves, at least partly, the reaction product of the acyl halide with the organic base formed during the esterification process, but which is not a solvent for the cellulose ester itself. F. R. ENNOS.

**Manufacture of sulphonated cellulose derivatives.** I. G. FARBENIND. A.-G. (B.P. 277,317, 29.8.27. Ger., 10.9.26).—Sodium-cellulose reacts with *o*- or *p*-sulphobenzyl chloride to give a water-soluble sulphobenzyl ether which may be isolated as sparingly soluble calcium or barium salt, or by performing the reaction in alcohol,

and evaporating the alcohol. The products are non-inflammable, and textiles may be fire-proofed by impregnation with the aqueous solution and precipitation of the calcium, barium, or aluminium salt within the fibre. C. HOLLINS.

**Manufacture of thermoplastic materials.** BRIT. CELANESE, LTD. (B.P. 282,723, 23.11.27. U.S., 29.12.26).—Cellulose acetate or other cellulose ester or ether, finely ground so that 80% passes 80-mesh, is mixed with one or more plasticisers and/or softening agents, *e.g.*, ethyl phthalate and/or triacetin, in the absence of volatile solvents. Colouring and filling materials may be subsequently added. F. R. ENNOS.

**Method of washing cooked straw.** DE W. C. AMERINE (U.S.P. 1,678,718, 31.7.28. Appl., 9.6.26).—Straw is cooked under pressure with lime and sulphur in a revolving spherical digester. When digestion is complete water is forced into the digester against the steam pressure through a tube running through a trunnion and extending one quarter round the inside of the digester. This tube is fixed to and rotates with the digester; the outlet pipe, running through the other trunnion, remains stationary. D. J. NORMAN.

**Chemically treating and washing pulp.** G. A. RICHTER and M. O. SCHUR, ASSRS. to BROWN Co. (U.S.P. 1,683,262, 4.9.28. Appl., 19.4.27).—Cellulose pulp is digested in an alkaline liquor, bleached in hypochlorite, and superbleached in chlorine water. The pulp is washed after each treatment, fresh water being used for the superbleached pulp whilst the effluent wash-waters from the superbleached and bleached pulp are used for washing the bleached pulp and alkali-digested pulp respectively, the first contaminated portions of each washing being discarded if desired. F. R. ENNOS.

**Producing a substance for obtaining a high gloss and pure white colour on paper and the like.** SIEBENHIRTNER CHEM. IND.-WERK GES.M.B.H. (B.P. 287,559, 15.3.28. Austr., 24.3.27).—A pasty mixture of saponified stearin and heavy-metal salts, preferably zinc salts, is added to the paper pulp. F. R. ENNOS.

**De-inking of paper.** A. R. LUKENS, ASSR. to RICHARDSON Co. (U.S.P. 1,680,949, 14.8.28. Appl., 22.6.25).—Old newspapers etc. are treated with warm 3% sodium carbonate solution for 30 min., and a sodium salt of a sulphonated hydrocarbon oil (0.5%) is added to facilitate the washing-out of the inky matter. C. HOLLINS.

**Fulling wool and the like.** J. NÜSSLEIN, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,685,680, 25.9.28. Appl., 13.8.26. Ger., 21.1.25).—See B.P. 270,333; B., 1927, 599.

**Manufacture of cellulose compounds.** L. LILLENFELD (U.S.P. 1,674,405, 19.6.28. Appl., 23.7.24. Austr., 4.4.24).—See B.P. 231,802; B., 1925, 913.

**Manufacture of artificial materials [from cellulose derivatives].** L. LILLENFELD (U.S.P. 1,674,403, 19.6.28. Appl., 23.7.24. Austr., 4.4.24).—See B.P. 231,808; B., 1925, 914.

**Manufacture of artificial materials [from *N*-substituted cellulose urethanes].** L. LILLENFELD (U.S.P. 1,674,404, 19.6.28. Appl., 22.5.26. Austr., 30.5.25).—See B.P. 248,994; B., 1926, 532.

**Manufacture of cellulose thiourethane esters and ethers.** L. LILLENFELD (U.S.P. 1,674,402, 19.6.28. Appl., 17.9.25. Austr., 11.10.24.)—See B.P. 241,149; B., 1926, 47.

**Apparatus for filtering solutions used in manufacture of artificial silk etc.** W. P. DREAPER (U.S.P. 1,685,775, 2.10.28. Appl., 2.6.23. U.K., 22.6.22.)—See B.P. 207,214; B., 1924, 52.

**Treatment of fabrics [containing cellulose esters or ethers].** G. H. ELLIS, ASST. to CELANESE CORP. OF AMERICA (U.S.P. 1,686,149, 2.10.28. Appl., 3.12.26. U.K., 27.7.26.)—See B.P. 280,989; B., 1928, 85.

**[Vibration-damping] bearings, and applications thereof [for textile spindles].** BRIT. CELANESE, LTD., E. C. COPE, and E. KINSELLA (B.P. 297,875, 30.6.27).

**Process and apparatus for dressing webs of fabric.** H. MALY (B.P. 290,217, 19.4.28. Ger., 10.5.27).

**Drying apparatus for laundry and dry-cleaning purposes.** BRITISH-AMERICAN LAUNDRY MACHINERY CO., LTD. FROM AMER. LAUNDRY MACHINERY CO. (B.P. 297,515, 2.7.27).

**Electric centrifuge for spinning artificial silk.** BERGMANN ELEKTRICITÄTS-WERKE A.G., ASSEES. OF F. BEIER (B.P. 285,011, 6.2.28. Ger., 8.2.27).

**Method of treating cakes of artificial silk manufactured by the centrifuge-spinning process.** (Sir) G. C. MARKS. FROM F. KÜTTNER A.-G. (B.P. 287,097, 14.3.28).

**Oxygen-containing compounds** (U.S.P. 1,681,156).—See VII. **Livering of cellulose lacquers** (B.P. 278,696).—See XIII. **Disinfectant** (G.P. 448,705).—See XXIII.

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

**Valuation of protective agents [in wet processes for wool].** H. BLACKSHAW (J. Soc. Dyers and Col., 1928, 44, 297—300).—Three methods are described for the valuation of agents used for protecting wool from the deleterious action of alkaline solutions. In a quantitative method, wool is treated under conditions comparable with those used on a large scale, and the physical properties of the treated wool are determined by suitable mechanical apparatus such as a Goodbrand ballistic machine. In two qualitative methods wool may be subjected to an exaggerated treatment such that the effect of a protective agent is more definitely shown by mechanical tests, or loose wool is treated under normal large-scale conditions, and the damage to the wool determined by the resulting increase in its affinity for Acid Scarlet 4R extra. Typical results obtained with Protectol are given. A. J. HALL.

**Influence of heat on the affinity of cotton for dyes.** C. K. PATEL (J. Soc. Dyers and Col., 1928, 44, 301—303).—The affinity of cotton for Chlorazol Sky Blue FF, Benzopurpurine 4B, and Methylene Blue was determined after heating in an air oven at 150—285° for various periods of time, it being observed that the heated cotton has a smaller affinity for both direct and basic dyes. The loss of affinity for direct dyes is proportional to the duration and temperature of heating,

but for basic dyes the initial loss of affinity is large compared with further losses. It is suggested that part of the lustre of pile fabrics which have been singed is due to the comparatively small absorption of dye by the tips of the fibres which have been most affected by the heat of singeing. A. J. HALL.

**Method for increasing the affinity of cotton and other fibres for colouring matters.** G. E. HOLDEN (J. Soc. Dyers and Col., 1928, 44, 305—307).—Cotton acquires an increased affinity for direct, sulphur, vat, water-solubilised vat, and basic (after mordanting with tannic acid or Katanol O) dyes when impregnated with a 5—20% solution of gelatin, then steamed for about 1 hr. under 5 lb. pressure, and afterwards freed by washing from the 82—83% of gelatin not thereby rendered insoluble (cf. Holden, B., 1927, 565). The increase of affinity is greater than that produced by mercerisation, and is directly proportional to the amount of gelatin fixed on the cotton; the resulting dyeings are better penetrated, faster to washing, and equal in fastness to light to those produced on cotton not prepared with gelatin. Multi-tone effects may be obtained by dyeing cotton fabric printed with solutions of gelatin of different concentrations. The insoluble gelatin in cotton prepared with gelatin is fast to soap, alkalis, acids, and sodium hyposulphite, but is removed by treatment with solutions containing free chlorine; fabric prepared with gelatin may be discharged by printing with a thickened 10% solution of chloramine-T and then steaming. Glue is not a satisfactory substitute for gelatin, but isinglass is superior. Harshness in handle of cotton materials prepared with gelatin may be removed by soaping at 80°. A. J. HALL.

**Effect of light on coloured [cotton] fabric. II.** (Miss) E. HIBBERT (J. Soc. Dyers and Col., 1928, 44, 300—301; cf. B., 1927, 840).—Cotton fabric, particularly that having a pile surface, dyed with *o*-tolueneazo- $\beta$ -naphthol became covered with bright lustrous crystals after exposure for 3 min. to light from a fadeometer (the temperature of the air surrounding the fabric was about 45°); the crystals were shown to be capable of further sublimation, and when recrystallised from chloroform were obtained as crystals having a different character but the same m.p. (133°) as that of *o*-tolueneazo- $\beta$ -naphthol. A thin layer of powdered *o*-tolueneazo- $\beta$ -naphthol sublimed only to a very small degree when heated to 45°, and it was not possible to obtain crystals as in the case of dyed fabric. When faded by exposure in a fadeometer, cotton fabric dyed with *o*-tolueneazo- $\beta$ -naphthol suffers a loss of depth of shade due to sublimation, but its colour does not change or become dull as when exposed to sunlight. Sublimation effects were observed with cotton dyed with *o*-tolueneazo- $\alpha$ -naphthol and with combinations of Naphthol AS-BO and AS-RL with Fast Orange G base, but no crystals were formed on the surface of the fabric. A. J. HALL.

**Reagent for oxycellulose.** ERMEN.—See V.

## PATENTS.

**Printing with developing dyes, especially on acetate silk.** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 297,374, 20.6.27).—Acetate silk is printed with a solution of the dye components in thiodiglycol, tri-



( $\beta$ -hydroxyethyl)amine, cyclohexyldi-( $\beta$ -hydroxyethyl)amine (B.P. 297,484; B., 1928, 846), or other suitable solvent, with thickening, and developed in an acidified nitrite bath with or without previous steaming; or nitrite may be added to the printing paste and the colour developed by acid alone. Examples of printing pastes are:  $\beta$ -naphthylamine, resorcinol, and nitrite in thiodiglycol (orange); 2-chloro-*p*-anisidine, 2:3-hydroxynaphthoic acid, and nitrite in cyclohexyldi-( $\beta$ -hydroxyethyl)amine (bluish-red); dianisidine and 2:3-hydroxynaphthoic acid in cyclohexyldi-( $\beta$ -hydroxyethyl)amine (blue-black). C. HOLLINS.

**Oxidation process in dyeing or printing of textiles [with aniline black].** MANCHESTER OXIDE CO., LTD., R. H. CLAYTON, and F. SCHOLEFIELD (B.P. 296,530, 4.8.27).—In dyeing and printing with aniline black the ferrocyanide oxygen carrier is replaced by sodium carbonylferrocyanide. Greater stability at 15° is claimed, with increased activity at 100°; premature oxidation is thus minimised. C. HOLLINS.

**Dyeing and printing of vegetable and animal fibres with basic dyes.** E. I. DU PONT DE NEMOURS & Co. (F.P. 620,746, 6.8.26).—Dyes which are fixed by the fibre without steaming or mordants are obtained by condensing basic dyes (*e.g.*, methylene-blue) in the form of free colour base with aromatic hydroxy-acids (tannin,  $\beta$ -resorcylic acid, salicylic acid) in presence of mineral acids, such as phosphoric, hydrochloric, sulphuric, and boric acids. The condensation products are salted out from the neutralised reaction mixture. C. HOLLINS.

**Treatment of filaments, threads, yarns, and fabrics containing them of artificial silk having for basis cellulose esters or ethers [to produce discharge effects].** G. B. ELLIS. From Soc. FABR. DE LA SOIE "RHODIASETA" (B.P. 297,186, 26.7. and 31.10.27).—Dyes (except developed and ice colours) on acetate silk are discharged by application of adsorbent substances, such as animal charcoal, kaolin, fuller's earth, or infusorial earth, either in aqueous suspension or particularly in printing pastes. The action is enhanced by addition of swelling agents and by heat (*e.g.*, by steaming after printing). C. HOLLINS.

**Production of dyes or coloured substrates.** I. G. FARBENIND. A.-G., Assees. of E. MÜNCH (G.P. 448,909, 24.4.25).—Indigoid dyes are regenerated by hydrolysis of the bisulphite compounds of their *N*-acetyl derivatives in presence or absence of a substrate. Cotton, *e.g.*, is padded with a solution of *NN'*-diacetylindigo bisulphite compound and developed in an alkaline bath containing a little hydrogen peroxide. Wool is padded with the 4:4'-dichloro-compound in presence of acetic acid and sodium sulphate, and developed with dilute sodium hydroxide or ammonia with addition of hydrogen peroxide and sodium sulphate. Wool, padded with the bisulphite compound of *N*-acetyl-3-indole-2-thionaphthenindigo, is developed with dilute sulphuric acid and nitrite. By addition of sodium aluminate solution and sulphite-cellulose liquor to the bisulphite compound of *NN'*-diacetylindigo, colloidal indigo is precipitated, and a blue lake is obtained on acidification with acetic acid. The process may also be applied to printing. C. HOLLINS.

**Dyeing [regenerated cellulose material].** BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, R. BRIGHTMAN, and P. CHORLEY (B.P. 296,845, 7.6.27).—2:2'-Disubstituted benzidines (other than 2:2'-dinitro-) are tetrazotised and coupled with 2 mols. of a coupling component or of two different coupling components. Examples are: 2:2'-dichlorobenzidine with salicylic acid and  $\gamma$ -acid (orange-brown) or with 2 mols. of  $\gamma$ -acid (maroon); *m*-tolidine with salicylic acid and  $\gamma$ -acid (maroon) or phenyl- $\gamma$ -acid (brown); benzidine-2:2'-dicarboxylic acid with 1-*p*-sulphophenyl-3-methylpyrazolone and  $\beta$ -naphthol (orange); benzidine-2:2'-disulphonic acid with 2 mols. of 1-phenyl-3-methylpyrazolone (yellow). Not more than two sulphonic groups should be present in the finished dye. C. HOLLINS.

**Reserves on silk.** I. G. FARBENIND. A.-G., Assees. of W. SCHLEGEL (G.P. 448,847, 6.12.23. Addn. to G.P. 432,111; B., 1927, 214).—The sulphurised phenols (syntans) applied in the prior patent for reserves on wool are now used, with or without addition of tin salts, for reserving silk, which remains white (or coloured) when admixed cotton or artificial silk is dyed with direct or basic colours. C. HOLLINS.

**Reserve dyeing on vegetable fibres.** H. LEEMANN and G. TAGLIANI, Assis. to MUNITEX CORP. (U.S.P. 1,679,477—1,679,481, 7.8.28. Appl., 20.5.26. Ger., 7.5.24).—Cotton is locally reserved against direct dyes by printing with alkali and treating with an acid chloride (toluene-*p*-sulphonyl chloride). The reserved parts may be dyed (A) with a gallocyanine, (B) with acid dyes, (c) with acid dyes in the same bath as direct dyes for the unreserved parts, (D) with an arylamine which is subsequently diazotised and coupled with a developer, (E) in batik effects by means of a wax covering and basic and direct dyes. C. HOLLINS.

**Production of yellow, orange, and brown coloured patterns on cotton or silk materials with the aid of wax reserves.** I. G. FARBENIND. A.-G., Assees. of H. EICHWEDE and C. ZECHENTMAYER (G.P. 446,542, 19.12.24).—The wax-patterned material is padded with phosgenated 1-*p*-aminophenyl-3-methylpyrazolone, drained, and coupled with diazotised nitroaniline for yellow. Padding with the dye, benzidine-3:3'-disulphonic acid = 2 mols. of 1-*p*-aminophenyl-3-methylpyrazolone, and developing with diazotised *o*-nitroaniline gives an orange, which may be diazotised on the fibre and coupled with phenylmethylpyrazolone (orange). For orange-brown the material is padded with the dye, salicylic acid  $\leftarrow$  benzidine-3-sulphonic acid  $\rightarrow$  1-*p*-aminophenyl-3-methylpyrazolone, and developed with diazotised *o*-nitroaniline. All the couplings proceed smoothly in the cold. C. HOLLINS.

**Modifying the capacity of cellulose-containing materials for taking up colours.** HERMINGHAUS & Co., G.M.B.H., and K. L. WENZ (B.P. 281,696, 1.12.27).—The successive treatment of cellulose fibres with a concentrated caustic alkali and an inorganic oxychloride in an inert solvent for conferring on such fibres an increased affinity for basic dyes and a decreased affinity for direct dyes (cf. B.P. 192,173; B., 1923, 304 A) is modified in so far as the concentrated alkali is replaced by alkali solutions not exceeding 3%; the

fibres do not suffer appreciable shrinkage under these conditions.

A. J. HALL.

**Production of azo dyes on the fibre.** I. G. FARBENIND. A.-G. (B.P. 292,100, 5.6.28. Ger., 13.6.27).—The diazo component is applied in the form of a diazonium fluoborate. Enhanced brilliance is claimed.

C. HOLLINS.

**Dyeing.** P. MIJER, Assr. to TWO-TONE CORP. (U.S.P. 1,683,687, 11.9.28. Appl., 14.2.24).—The liquid colour is atomised and deposited as a cloud in minute non-spotting particles on material placed below.

F. G. CLARKE.

**Method of ornamenting textile pile fabrics.** H. LIVSEY, G. E. HOLDEN, and J. & J. M. WORRALL, LTD. (B.P. 297,607, 17.12.27).—Pile fabrics are printed with solutions of cellulose, such as viscose and cuprammonium solutions, then dried, steamed, and washed to regenerate the cellulose.

A. J. HALL.

**Treating and wetting-out fibrous material. Carbonising woollen fibres.** R. H. POTT, Assr. to CHEM. FABR. POTT & Co. (U.S.P. 1,686,836—7, 9.10.28. Appl., [A] 23.9.25, [B] 26.7.24. Ger., [A, B] 10.7.23).—See B.P. 248,814; B., 1926, 403.

**Production of fast dyeings on the fibre.** T. KIRCHEISEN and J. GÜRTLER, Assrs. to GRASSELLI DYE-STUFF CORP. (U.S.P. 1,680,791, 14.8.28. Appl., 21.1.27. Ger., 23.1.26).—See B.P. 262,537; B., 1927, 104.

[Apparatus for] dyeing textile goods. I. G. FARBENIND. A.-G. (B.P. 270,307, 27.4.27. Ger., 27.4.26).

**Apparatus for the dyeing or treating of hanks.** K. S. MILLS (B.P. 297,755, 22.3.27).

**Dye preparations** (B.P. 293,795).—See IV.

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

**Evaporating system for salt.** A. MITTENBERG (Ukraine Chem. J., 1928, 4, [Tech.], 43—64).—A detailed analysis of the thermal economy of brine evaporating plants is given.

R. TRUSZKOWSKI.

**Determination of iron carbonyl.** R. H. GRIFFITH and G. C. HOLLIDAY (J.S.C.I., 1928, 47, 311—312 T).—Gases containing iron pentacarbonyl are passed through strong sulphuric acid, which is then evaporated to dryness, the residue redissolved, and the iron determined as Prussian-blue by a colour-density method. If the gas contains unsaturated hydrocarbons, sulphuric acid cannot be used, and a method of determination by absorption in active charcoal is described. Examples of the carbonyl content of various gases are given.

**Preparation of calcium and magnesium bisulphite liquors.** D. W. STEWART (Pulp and Paper Mag., 1928, 26, 1013—1016).—The difficulties associated with the preparation of bisulphite liquor containing both lime and magnesia by the ordinary tower system can be obviated by adding pulverised calcined magnesite (temperature of calcination about 900°) to the water-feed at the top of an absorption tower containing limestone. By regulating the addition of the calcine, liquors are readily obtained with a lime-magnesia ratio as high as 0.64 : 1, owing to the solubility of the pulverised calcine.

D. J. NORMAN.

**Manufacture of [solid] carbon dioxide.** H. E. HOWE (Ind. Eng. Chem., 1928, 20, 1091—1094).—The production of gases of combustion containing 16—18% CO<sub>2</sub> from coke involves heavy renewal of boiler settings. The gases are scrubbed with water in steel towers packed with limestone and absorbed by 2.2*N*-sodium carbonate in high coke towers. The exit gases still contain 8% CO<sub>2</sub>. The towers operate at 49° and the lye boilers at 115°. These boilers work at 7—8 lb. pressure, as fixed by the efficiency of the heat exchangers. The gas evolved is cooled and, after separation of oil and water, is compressed in three stages to 1100 lb./in.<sup>2</sup> The compressed gas is passed through permanganate and cooled to 21°, when it liquefies. The liquid is expanded to a pressure of 15 lb., from 50 to 60% evaporates and is returned, and the remainder solidifies. It is moulded, pressed, and wrapped in heavy paper.

C. IRWIN.

**Determination of small quantities of carbonate in presence of excess of sulphide and chloride, with particular reference to the analysis of metallic corrosion products.** W. H. J. VERNON and L. WHITBY (J.S.C.I., 1928, 47, 255—259 T).—The ordinary gravimetric method for determination of carbonate is critically examined. Errors due to presence of sulphide or chloride are eliminated by interposing powdered copper or *p*-nitrosodimethylaniline respectively in the absorption train. By means of these reagents a large excess of hydrogen sulphide or hydrogen chloride may be quantitatively separated from very small quantities of carbon dioxide; it is shown that the reagents commonly recommended for the purpose are quite inadequate. The copper powder should precede the *p*-nitrosodimethylaniline; to ensure freedom from action on carbon dioxide it should not have been prepared by reduction with hydrogen. Phosphoric acid is used for decomposing the material in the evolution flask. "Sofnolite" is used for absorption of carbon dioxide. The accuracy obtainable is such that the observed weight-increment due to carbon dioxide is within 0.1 mg. of the calculated value, even when sulphide and chloride are simultaneously present in large excess. Small quantities of sulphide are determined by evolution as hydrogen sulphide, absorption in cadmium acetate solution, and titration with iodine and thiosulphate; to obtain a quantitative yield of hydrogen sulphide in presence of excess of copper salts, stannous chloride is added to the mixture in the evolution flask. The hygroscopicity of corrosion products is referred to; a curve is given showing the rate of removal of hygroscopic moisture from a sample of corrosion product (60 : 40 brass) on heating at 103°.

**Detection of traces of carbonate.** J. RAE (Pharm. J., 1928, 121, 315).—The substance is treated with dilute sulphuric acid and 2 c.c. of chloroform in a small flask, fitted with a rubber stopper carrying a thistle funnel with a two-bulb trap in which are placed 2—3 c.c. of lime water. Heating on the water bath causes the chloroform to distil, bringing with it the carbon dioxide; a turbidity in the lime water is apparent if the sample contains 1 mg. of sodium carbonate or its equivalent.

A. R. POWELL.

**Fermentation carbon dioxide.** WANDERSCHECK.  
—See XVIII.

## PATENTS.

**Production of hydrocyanic acid.** M. J. BROWN and E. A. HARDING, ASSTS. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,680,662, 14.8.28. Appl., 19.10.25).—Sodium cyanide, or crude material containing it, is stirred with a solution of sodium hydrogen carbonate and vacuum is applied so that the water is vaporised at 35–45°. Hydrogen cyanide is carried away with the steam and is separated by known means.

C. HOLLINS.

**Manufacture of oxygen-containing compounds of the aliphatic series [oxalic acid].** I. S. MELLANOV, ASSR. to KEMIKAL, INC. (U.S.P. 1,681,156, 14.8.28. Appl., 13.3.28).—Waste paper etc. is pulped and heated to 200° with caustic alkali, air being injected into the mass. Paraffin is added, and the temperature, after being raised to 210°, is finally maintained for some hours at 150° without air-injection. Sodium oxalate is produced.

C. HOLLINS.

**Manufacture of pure tartaric acid and tartrates.** SOC. ENO-TARTARICA ITALIANA (F.P. 624,540, 13.11.26. Italy, 17.11.25).—To tartar and aluminium hydroxide hydrochloric acid is added in such amount that the alumina is retained in solution on boiling. Pure potassium hydrogen tartrate crystallises out when cold. Calcium hypochlorite and aluminium sulphate are added to decolorise the solution. The potassium hydrogen tartrate is heated with water and sulphuric acid and potassium removed as potash alum by addition of alum. The centrifuged solution is evaporated *in vacuo* to *d* 1.374 and tartaric acid crystallises out.

C. HOLLINS.

**Manufacture of alkali and ammonium phosphate.** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 297,546, 27.7.27).—Dicalcium phosphate is treated with alkali fluoride or ammonium fluoride solution, the resulting calcium fluoride is removed by filtration, and the solution is concentrated or cooled after the addition, if desired, of phosphoric acid or fixed or volatile alkali. The mother-liquor is returned to the process. Alternatively, precipitation of the ammonium phosphate from the above filtrate may be effected by means of methyl alcohol.

W. G. CAREY.

**Cyanides and [their manufacture from] cyanamide salts.** FABR. DE PROD. CHIM. DE THANN ET DE MULHOUSE (Addn. F.P. 31,723, 26.11.25. to F.P. 586,110. Cf. B.P. 225,160; B., 1925, 98).—In the prior process the alkaline-earth carbide may be replaced by carbides of other metals (*e.g.*, aluminium) or by alkali- or alkaline-earth metals, or magnesium, aluminium, or zinc. Certain oxides such as litharge or antimony trioxide, or certain alkali salts, such as sodium chloride, may be used in place of alkali carbonates.

C. HOLLINS.

**Drying of gases containing ammonia and carbon dioxide, especially of gas mixtures circulating in the manufacture of carbamide.** I. G. FARBENIND. A.-G.; ASSEES. of R. GRIESSBACH and M. SCHMIHING (G.P. 448,200, 3.2.25).—Alumina gel, prepared according to G.P. 436,671 (B., 1927, 482) and dried at red heat, absorbs water from mixtures of ammonia and carbon

dioxide. The mixture of carbon dioxide, ammonia, and water, obtained by heating ammonium hydrogen carbonate, is passed, for example, through a tower filled with alumina gel, and yields 80–90% of the ammonia as carbonate. The alumina may be replaced by a strongly heated mixture of bauxite and active silicic acid or base-exchanging substances. The process is of special importance for the drying of the wet gases from the working-up of the carbamide melt before returning them to the cycle. It is also applicable in the manufacture of methylamine from carbon dioxide, ammonia, and hydrogen. The drying agent may be regenerated after use by heating in a current of dry air or dry waste gases.

C. HOLLINS.

**Preparation of a copper catalyst for synthesis of methyl alcohol and formaldehyde from carbon monoxide and hydrogen.** SOC. NAT. DE RECHERCHES SUR LE TRAITEMENT DES COMBUSTIBLES (F.P. 606,596, 27.2.25).—A boiling solution of copper sulphate or nitrate, free from impurities (especially iron), is treated with caustic alkali, and the black hydrated oxide, washed free from alkali and dried in a vacuum at 50°, is mixed with 10–20% of the violet modification of metallic copper (Sabatier) and reduced at the ordinary pressure by hydrogen or carbon monoxide in a slow stream or diluted with nitrogen or methane not above 130–140°. The resulting porous, fine-grained, pyrophoric catalyst converts hydrogen and carbon monoxide at 275–300° and 100–200 atm. into methyl alcohol and formaldehyde.

C. HOLLINS.

**Production of aluminium compounds from raw materials.** CLAY REDUCTION Co. (B.P. 279,515, 25.10.27. Norw., 25.10.26).—Material containing oxygen compounds of aluminium and silicon, *e.g.*, clay, is treated with a solution of ammonium fluoride above 34°, the water is evaporated, and the mixture heated at about 300° to remove volatile compounds of ammonium and silicon with fluorine, which are treated with ammonia and water to form silica gel and ammonium fluoride. The residue of complex metallic fluorides, after reduction of the iron with producer gas, is heated at 400–500° with dry hydrochloric acid and ammonia to convert the fluorides into chlorides, with formation of aluminium chloride and ammonium fluoride which are volatilised and separated by fractional condensation.

W. G. CAREY.

**Refining of crude arsenic trioxide.** C. L. READ, ASSR. to AMER. SMELTING & REFINING Co. (U.S.P. 1,681,496, 21.8.28. Appl., 14.4.26).—The volatilised arsenic trioxide is passed through baffled flues, maintained at such a temperature that arsenic trioxide is in part deposited in the vitreous condition. The latter absorbs the antimony present in the fume as impurity, and the pure arsenic trioxide passes on and is condensed. The volume of air passing through the furnace and flue is regulated in order to control the temperature and time of contact of the fume with the vitreous oxide.

F. G. CLARKE.

**Recovery of hafnium.** SIEMENS & HALSKE A.-G., ASSEES. of B. FETKENHEUER (G.P. 446,489, 26.3.26).—The hafnium-rich mother-liquors obtained in the fractionation of double fluorides of zirconium and hafnium are treated with methyl or ethyl alcohol to

precipitate the double fluorides present, and the residual solution is neutralised with alkali or treated with further quantities of alkali fluoride to precipitate the remainder of the hafnium.

A. R. POWELL.

**Manufacture of hydrogen.** I. G. FARBENIND. A.-G. (B.P. 267,535, 10.3.27. Ger., 10.3.26).—Hydrocarbons, alone or mixed with other gases which do not contain free oxygen, are treated with steam at 300–600° in the presence of nickel, cobalt, or iron activated with chromium, vanadium, potassium, magnesium, aluminium, or alkaline-earth metals, the resulting carbon dioxide then being removed. The reaction may be performed in stages by transforming some of the hydrocarbons, removing the carbon dioxide, and repeating the succession of operations with the hydrocarbon–hydrogen mixture until all the hydrocarbons are converted.

W. G. CAREY.

**Production of hydrogen.** M. CASALE-SACCHI (B.P. 297,135, 13.6.27).—A mixture of oxygen and carbon dioxide, with or without nitrogen, is passed over a carbonaceous fuel heated at above 1000°, readily soluble or absorbable catalyst poisons are removed, and the resulting carbon monoxide is treated with steam in the presence of a catalyst, *e.g.*, a metal of the iron group, and the carbon dioxide is separated from the hydrogen, part of the carbon dioxide being used for further admixture with oxygen.

W. G. CAREY.

**Treatment of sulphur.** J. W. SCHWAB, Assr. to TEXAS GULF SULPHUR CO. (U.S.P. 1,683,731, 11.9.28. Appl., 27.12.26).—Abnormally coloured sulphur has its normal colour restored by treatment of the molten material with finely-divided activated carbon, from which it is afterwards separated.

H. ROYAL-DAWSON.

**Purification of gases [in synthesis of ammonia].** G. F. UHDE (U.S.P. 1,685,733, 25.9.28. Appl., 15.4.26. Ger., 9.2.25).—See B.P. 247,226; B., 1926, 979.

**Synthetic production of ammonia.** G. F. UHDE (U.S.P. 1,685,734, 25.9.28. Appl., 28.5.26. Ger., 4.6.25).—See B.P. 253,122; B., 1927, 481.

**Apparatus for production of synthetic ammonia.** G. FAUSER (U.S.P. 1,686,371, 2.10.28. Appl., 21.8.25. Italy, 24.9.24).—See B.P. 240,436; B., 1926, 979.

**Apparatus for synthesis of ammonia.** G. CLAUDE, Assr. to LAZOTE, INC. (U.S.P. 1,686,799, 9.10.28. Appl., 22.12.21. Renewed 17.12.26).—See B.P. 174,041; B., 1923, 144 A.

**Carrying-out [exothermic] gaseous catalytic reactions [for synthesis of ammonia].** R. E. SLADE, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,686,349, 2.10.28. Appl., 1.5.26. U.K., 4.5.25).—See B.P. 255,964; B., 1926, 822.

**Precipitating heavy metals from ammoniacal solutions.** C. MÜLLER, L. SCHLECHT, and W. SCHUBARDT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,686,391, 2.10.28. Appl., 11.3.27. Ger., 9.4.26).—See B.P. 269,164; B., 1927, 907.

**Methods of obtaining zirconium compounds.** TITANIUM ALLOY MANUF. CO., Asses. of C. J. KINZIE

(B.P. 271,873, 23.5.27. U.S., 27.5.26).—See U.S.P. 1,658,807; B., 1928, 297.

**Apparatus for utilising impure gases or exhaust gases containing carbon dioxide.** F. RIEDEL, Assr. to RIEDEL FERTILISING PROCESS CO. (U.S.P. 1,687,229, 9.10.28. Appl., 8.3.22. Ger., 14.3.16).—See G.P. 315,019; B., 1920, 489 A.

**Sodium acetate** (F.P. 583,341).—See III. **Anti-corrosive silver alloys** (B.P. 297,165).—See X. **White lead** (B.P. 273,287).—See XIII. **Solidified iodine solutions** (B.P. 277,953).—See XX.

## VIII.—GLASS; CERAMICS.

**Heat balance of a glass tank furnace.** R. D. PIKE and G. H. WEST (J. Amer. Ceram. Soc., 1928, 11, 734–744).—A method is described for determining the thermal balance of an oil-fired, continuous, regenerative, glass tank furnace. Means for reducing the fuel consumption by 14% are indicated. Considerable loss of heat through the furnace brickwork was discovered.

F. SALT.

**Waste-heat drying system involving recuperation.** W. T. WINDSOR and F. C. WESTENDICK (J. Amer. Ceram. Soc., 1928, 11, 730–733).—In order to eliminate smoke and utilise waste heat from a brick kiln for drying, a “double-deck” system of ducts was arranged leading to the kiln outlet. By means of dampers the upper duct was made to take the waste heat from cooling kilns, and the lower duct to take the combustion gases. The waste heat was discharged by a fan into a main duct connecting with each tunnel.

F. SALT.

**Dorr classifiers for clay-washing.** A. ANABLE (J. Amer. Ceram. Soc., 1928, 11, 791–794).—The Dorr bowl classifier is described, and its advantages over the old method of using settling channels in washing clays, particularly primary and secondary kaolins, are pointed out.

F. SALT.

**Vacuum treatment of clay slips and bodies.** H. M. KRANER and A. H. FESSLER (J. Amer. Ceram. Soc., 1928, 11, 725–729).—Simple apparatus is described for determining the amount of gas removable from ceramic slips by subjecting them to reduced pressure. In a factory porcelain slip from 1 to 1.3% of air by vol. was found. The removal of this adsorbed air from the slip had practically no effect on the dielectric and mechanical strength of the fired porcelain.

F. SALT.

**Firing terra cotta.** S. J. McDOWELL and R. M. MURPHY (J. Amer. Ceram. Soc., 1928, 11, 745–752).—Tests were made on a 14 ft. periodic, coal-fired muffle kiln having no centre stack. Temperature and draught readings were taken, and gas analyses were made. A marked difference in the top and bottom temperatures in the muffle was observed; this was reduced from 300° to 100° maximum by the construction of a stack, 18 in. in diam., within the muffle. Kiln and operating data are given.

F. SALT.

**Pores in bricks.** G. J. EASTER (J. Amer. Ceram. Soc., 1928, 11, 764–768).—A number of standard refractory materials were tested for air permeability in a “permeameter” (*cf.* Westman and Pfeiffer, B., 1926, 917). The pores are arbitrarily considered as replaced

by parallel capillaries extending through the brick, of equal volume and of such size and number as to offer equal resistance to air flow. The dimensions of the capillaries are then computed. The method is limited to materials having open pores. F. SALT.

**Vapour absorption of a fired earthenware body.** C. L. DEEDS (J. Amer. Ceram. Soc., 1928, 11, 769—770).—Three specimens of an earthenware body, fired to different temperatures, were placed over 10% sulphuric acid for 36 days and the increase in weight was noted at intervals. Within limits, the amount of vapour absorption was proportional to the porosity, but the most porous specimen (20.25% absorption) showed a higher rate of vapour absorption and also greater variation. F. SALT.

**Silica dust in lungs.** HEFFERNAN and GREEN.—See XXIII.

#### PATENTS.

**Heat-resistant borosilicate glass.** L. HOCHSTEIN, Assr. to E. T. BROWN (U.S.P. 1,676,331, 10.7.28. Appl., 10.4.25. Ger., 15.3.25).—The glass consists of silica, boric oxide, alumina, and zirconia; its coefficient of expansion is about 0.000017. H. ROYAL-DAWSON.

**Manufacture of abrasive articles.** D. E. WEBSTER, Assr. to NORTON Co. (U.S.P. 1,681,891, 21.8.28. Appl., 26.10.26).—Abrasive material and a vulcanising agent are added to rubber latex (30% or over), the resulting paste being shaped as desired, dried, and vulcanised. C. HOLINS.

**Manufacture of glass, sand, or flint paper, emery cloth, and like abrasives.** C. A. KLEIN and R. S. BROWN (U.S.P. 1,687,453—4, 9.10.28. Appl., 28.10.25. U.K., 26.8.25).—See B.P. 258,412 and 260,704; B., 1926, 948; 1927, 44.

**Glass furnaces.** UNITED GLASS BOTTLE MANUFACTURERS, LTD., and T. C. MOORSHEAD (B.P. 297,856, 1.6.27).

**Glass-annealing lehrs.** BRIT. HARTFORD-FAIRMONT SYND., LTD., T. WARDLEY, and H. V. E. M. RENN (B.P. 297,865, 29.6.27).

### IX.—BUILDING MATERIALS.

**Road-surfacing materials.** W. J. A. BUTTERFIELD (J.S.C.I., 1928, 47, 293—309t).—A review of the development of bituminous materials for surfacing roads in non-urban areas. Bitumen, according to the definition adopted in June, 1928, by a Committee instituted by the International Road Congress of 1926, comprises:—"Mixtures of natural and pyrogenous hydrocarbons and of their non-metallic derivatives, which may be gaseous, liquid, viscous, or solid, but must be completely soluble in carbon disulphide." Road tar is thus recognised as containing a large proportion of bitumen, and when it is necessary to distinguish the bitumen of asphalt from that in tar the former must, in future, be termed "asphaltic bitumen." The provisions of successive standard specifications for tar for surface dressing, and for tar for making tar macadam, are given. Recent specifications increase the consistency of the tar used for both purposes. Methods of testing the consistency of road tars are described, and comparative figures given for the Hutchinson viscosimeter and the modified Redwood instrument adopted by the British Road Tar

Association. The former requires 15 to 20 times as much tar as the latter for a test, and is otherwise less convenient in use. Good road tars are characterised by a moderate content of "free carbon" and of high-boiling viscous oils, as well as of naphthalene and phenols, both of which are indicative of the relative stability of other components. Tar has constituents which correspond to the vehicle of paint, which cause it to set and dry, whereas asphaltic bitumen ordinarily is wholly stable, and the necessary fluidity for application is secured solely by heating it. Both tar and asphaltic bitumen are, however, often made up into emulsions for ease of application. The stability of the constituents of asphaltic bitumen delays the ultimate disintegration of its coating as compared with tar, and efforts are now directed to rendering tar more nearly asphaltic constitutionally. Some, but not all, tars blend with asphaltic bitumen, and the resultant mixtures combine good features of both materials. Blinding or gritting materials need careful selection and screening in order that they shall both prevent the bituminous material adhering to tyres, and preclude slipperiness, whether the road surface is dry, damp, or wet. Fuller investigation of the action of dust and mud from different gritting materials, as dry and wet lubricants, is called for. The effect of the dust and mud resulting from the wear of bituminous surfaces is discussed in relation to roadside vegetation and rain washings. The silt conveyed by the latter is detrimental to fish life, owing to the fineness and colloidal character of the particles. The toxicity to fish of solutions of many tar products is set out for different months of the year. Trout are killed in 1 hr. by solutions of 1 pt. (in August) to 5 pts. (in December) of phenol or mixed cresols per 100,000. The silt from bituminous surfaces may be dangerous to fish even if it is free from phenols or other distinctive tar products. A comparison is made of three trials in which the whole of the rain washings from trafficked roadways were passed for about 6 months into small ponds containing trout. The surfacing materials were:—(1) a thin tar, with a large content of naphthalene, on granite; (2) an asphaltic bitumen on old tarred macadam made up with fresh granite, and (3) "Tarvia" (mainly coal tar of high consistency) on granite. No trout were killed at any time by the washings from (2) or (3), and from (1) only a few days after application, and after the lapse of 4 months, when the coating had been broken up by wear. In the absence of special channels for the collection and delivery of the road washings into a small pond or stream, the danger to fish from tar or other bituminous road surfacings is stated to be remote, and to be altogether negligible if the washings are aerated or filtered by passage over pasture or other land. Cement surfaces, both bare and dressed with tar or asphaltic bitumen, are briefly discussed.

**Analysis of portland cement for factory purposes.** W. J. PITT (Chem. Eng. and Min. Rev., 1928, 20, 402—406).—The method of analysis follows very closely the ordinary procedure of that of silicates. Full manipulative details are given for determination of silica, alumina, ferric oxide, calcium oxide, magnesium oxide, sulphuric anhydride, alkalis, and insoluble matter. C. A. KING.

## PATENTS.

**Curing cementing materials.** BARBER ASPHALT Co., Assees. of F. W. McRAE (B.P. 291,784, 23.4.28. U.S., 10.6.27).—Concrete etc. after it has dried on the surface but before it has set, *i.e.*, after about 24 hrs., is sprayed or painted with a waterproof coating consisting, *e.g.*, of a solution of asphaltic material in a petroleum distillate, *d* 1.44—1.56. L. A. COLES.

**Apparatus for making a spumous mass of cementitious material.** G. B. HINTON (B.P. 297,638, 27.2.28).—Apparatus for carrying-out the process of U.S.P. 1,657,716 (B., 1928, 266) is described.

F. G. CROSSE.

**Manufacture of cellular cementitious materials.** G. B. HINTON (U.S.P. 1,687,067, 9.10.28. Appl., 2.2.28).—See B.P. 297,638; preceding.

**Floor covering** (B.P. 275,949).—See XIII.

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

**Mechanism of the blast furnace [smelting of iron ores] with reference to sintering and the precipitation of carbon.** B. OSANN (Arch. Eisenhüttenw., 1928—9, 2, 137—143; Stahl u. Eisen, 1928, 48, 1402—1403).—By smelting a mixture of small pieces of iron ore, marble, and coke in a crucible in a coke fire, and blowing a blast of air through the mixture the mechanism of the blast-furnace smelting of iron ores may be followed if the process be interrupted after varying periods. In this way it has been shown that, in contact with iron ore, carbon monoxide decomposes at 300° and the carbon liberated is deposited in a finely-divided state in the pores of the ore, thus causing progressive breaking up of the ore lumps. This is followed by the reduction of the iron oxide by the carbon on the surface; in the hot zone of the blast furnace this reaction takes place simultaneously with the deposition of the carbon and thus produces a highly carburised iron, whereas in the cooler zones the carbon dust tends to accumulate faster than it is used up. The reduction process is accompanied by sintering of the ore particles to the fluxes and, if the correct quantity of lime is not present, this is followed by fusion caused by ferrous oxide entering the slag. The ferruginous slag then acts as a decarburising agent on the iron first formed. The carburisation of the iron in the blast furnace is dependent on the fineness of the charge, too many fines hindering the diffusion of gases and thereby reducing the amount of carbon deposited. Many other phenomena observed in blast-furnace working may be explained by the results obtained by the crucible method outlined above.

A. R. POWELL.

**Coke-oven gas as a fuel for the Siemens-Martin furnace.** G. BULLE (Stahl u. Eisen, 1928, 48, 1353—1362).—A summarised review of modern practice in Germany describing the methods employed in operating open-hearth steel furnaces with coke-oven gas and with mixtures of this gas with flue gases and with producer and flue gases. The composition and calorific

value of the gas mixtures, the effect of pre-heating, and the types of gas burners used are briefly discussed.

A. R. POWELL.

**Effect of cold-rolling and annealing at different temperatures on the tensile properties and structure of high-grade soft iron sheets.** E. MARKE (Arch. Eisenhüttenw., 1928—9, 2, 177—184; Stahl u. Eisen, 1928, 48, 1404—1405).—If soft iron sheets are annealed at 650—850° after cold-rolling to reduce the cross-sectional area by 5—20% a coarsely crystalline structure is obtained, but at 900° complete recrystallisation into a fine-grained structure is obtained. If this operation is carried out in annealing boxes, however, the sheets are liable to adhere to one another. With a reduction in area of less than 5% or more than 20%, annealing at 650—930° relieves the strain induced by cold-working without appreciably affecting the mechanical properties, but, for obtaining a very fine-grained structure, a prolonged anneal at 580° is recommended after a reduction in area of more than 20% by cold-rolling.

A. R. POWELL.

**Evaluation of case-hardening materials [for iron].** F. HEBLER (Chem.-Ztg., 1928, 52, 775—776).—For temporary case-hardening of iron, no better results are obtained with wood charcoal than with other carbonaceous materials, such as coke, lignite, etc. The degree of case-hardening is scarcely affected by the addition of chalk to the carbon, but a progressive increase in the thickness of the hardened layer is obtained by the use of the carbonates of sodium, strontium, and barium.

F. R. ENNOS.

**Causes of failure of wrought iron chain and cable.** H. J. GOUGH and A. J. MURPHY (Proc. Inst. Mech. Eng., 1928, 293—326).—Apart from obvious causes, failure of wrought iron chain may be due to overheating or burning the metal during manufacture or to progressive deterioration of the weld by reason of service shocks and strains. An important cause of brittleness is associated with the use of chain, *e.g.*, snatch loading, rattling through hawse pipes, cleaning in rumbling machines, which destroys the ductility of the surface layer and results in cracking in later use. The customary reannealing of iron chains effectually restores the ductility.

C. A. KING.

**Practical corrosion research [applied to iron and steel].** E. H. SCHULZ (Stahl u. Eisen, 1928, 48, 1393—1401).—A review of recent work on the corrosion of iron and steel and on the development of corrosion-resisting steels in which the action of various corroding media is discussed, together with the influence of copper and silicon in reducing the rate of corrosion of constructional steels.

A. R. POWELL.

**Flocks in chromium-steel.** W. AICHHOLZER (Stahl u. Eisen, 1928, 48, 1332—1334).—Steels containing 0.8—1.2% C and 1—3% Cr frequently contain glistening nodular "flocks" in the inside of large ingots. Microscopic and macroscopic examination shows the existence of hair cracks between the surfaces of the flocks and the remainder of the ingot. The microstructure of the steel round these cracks reveals the presence of a carbide-rich constituent containing about 1.5—2% C and 13%

Cr, as shown by its behaviour during heat treatment. This constituent suffers only a very small increase in volume during hardening, whereas the remainder of the ingot increases 0.13% in length during this treatment. The strains set up, therefore, by heat treatment produce hair-cracks along the zones in which the harder constituent has segregated, and the characteristic bright crystalline appearance of the surface of these cracks gives rise to these flocks. A. R. POWELL.

**Electrochemical behaviour and tendency to rusting of chromium steels.** O. MEYER and K. ROESCH (Arch. Eisenhüttenw., 1928—9, 2, 121—127; Stahl u. Eisen, 1928, 48, 1372—1373).—Measurements of the potential of a 13% chromium steel in various salt solutions, together with corrosion tests, show that rusting commences whenever the potential falls below the value  $\epsilon_R = -0.015$  to  $-0.010$  volt. In acid solutions the potential of chromium steels becomes more positive and the rate of corrosion decreases with an increase in the quenching temperature. The higher the carbon content of the steel the higher is the temperature from which the steel must be quenched in order to prevent rusting, as the more nearly the structure approaches a single solid solution the greater is the chemical resistance; in this sense, therefore, carbon must be considered as an objectionable impurity in rust-resisting steels. Other things being equal a highly polished surface resists corroding media to a greater extent than does a rough surface, and the difference between the behaviour of rough and smooth surfaces is more marked in hardened steel than in forged or annealed steel. A. R. POWELL.

**Determination of vanadium in steel.**—F. IBBOTSON (Analyst, 1928, 53, 531).—Evans and Clarke's method (B., 1928, 786) for the determination of vanadium was found very satisfactory for an alloy steel containing also chromium, molybdenum, tungsten, nickel, and cobalt, but the glyoxime precipitate formed during the removal of nickel was found untractable, and for a steel containing about 0.5% each of nickel and vanadium, together with other metals, the nickel was not removed and the filtration from the two ferrocyanides was less difficult than from vanadium ferrocyanide alone. Two Munktell 15 cm. filters on a ribbed funnel were used by the author instead of a pulp filter. D. G. HEWER.

**Citric acid solubility and hardness of basic slag.** H. WEISS (Arch. Eisenhüttenw., 1928—9, 2, 81—85; Stahl u. Eisen, 1928, 48, 1403—1404).—The percentage of phosphoric acid soluble in citric acid in basic slag increases with the silicification factor to a maximum between 0.42 and 0.45. For pig iron low in silicon the necessary silica to obtain this factor should be added in the form of sand to the converter  $\frac{1}{2}$ —1 min. before the end of the blow. Slags with a low lime content and a high content of phosphoric acid often have a good citric acid solubility in spite of a low silicification factor owing to the high temperature required in the converter and consequent greater fluidity of the slag. On the other hand very basic slags, e.g., with 60% of lime, having a high silicification factor yield only 85—90% of their phosphoric acid to citric acid owing to their greater viscosity when molten. The citric acid solubility is

greater with soft slags than with hard slags that contain a large percentage of ferrous or manganous oxides which, by combining with the silica, reduce the amount available for the formation of the soluble silicophosphate.

A. R. POWELL.

**Examination of the structure of slags.** O. GLASER (Arch. Eisenhüttenw., 1928—9, 2, 73—79; Stahl u. Eisen, 1928, 48, 1373—1374).—A review of the various physical, optical, and thermochemical methods used in examining the constitution and structure of slags, with especial reference to those produced in smelting iron ores and in the manufacture of steel. A. R. POWELL.

**Manufacture of copper castings with a high electrical conductivity.** G. MASING and C. HAASE (Wiss. Veröff. Siemens-Konz., 1928, 7, 321—334).—The electrical conductivity and the density of sand castings of copper have been determined after deoxidation with phosphorus, lithium, beryllium, magnesium, calcium, aluminium, silicon, and boron suboxide. The use of phosphorus as a deoxidiser results in a gradual increase in the density as the amount of phosphorus retained by the casting increases, but the electrical conductivity is rapidly decreased. Lithium, added in the form of a 10% alloy with copper, effects complete removal of oxygen and sulphur from copper; 0.02% Li in copper ensures sound, dense castings of very high sp. gr. and electrical conductivity, but the melting must be carried out under a flux of soda-glass to obtain a smooth surface to the casting free from the corrugations which are otherwise obtained. Less than 0.04% of beryllium added to molten copper efficiently removes oxygen, but sulphur is retained in the form of minute thread-like inclusions, which, however, do not affect the density, conductivity, or mechanical properties. Magnesium reduces cuprous oxide and sulphide to metal, but the magnesium compounds formed are not readily removed from the metal, so that the surface of the castings shows irregularities due to nests of oxide inclusions; the density and conductivity of the metal are, however, satisfactory. Similar results are obtained with calcium and aluminium as deoxidisers, whereas silicon tends to lower the conductivity below the standard value. Efficient deoxidation is obtained with boron suboxide only when the metal is heated at 1300°; simple heating at 1400° without a deoxidiser also yields castings of good quality, owing to the rapidity with which occluded gas is expelled at this temperature, and to the interaction of sulphide and oxide inclusions. The good results obtained by the use of beryllium for producing high-conductivity copper castings are easily reproducible on a large scale using 0.3 g./kg. of beryllium in the form of a 10% alloy with copper. A. R. POWELL.

**Anomalies of annealing after cold-beating of copper and brasses.** EUGÈNE (Compt. rend., 1928, 187, 378—380).—The beginning and end of the zone of germination of the annealing of pure copper are marked by two anomalies on the temperature-hardness curves, the latter being the more marked, and corresponding with the most favourable annealing temperature for working copper. The addition of zinc increases and diminishes the first and second anomalies, respectively. The anomalies obey the law of displaced equilibria,

although they accompany an irreversible change of state in which there is no thermal reaction.

J. GRANT.

**Condenser materials and blue powder in zinc smelting.** R. W. MILLAR (*Min. and Met.*, 1928, 9, 395).—At 850° mixtures of zinc vapour and carbon monoxide react very slowly in a silica tube, whereas from thermodynamic considerations reaction should take place according to the equation:  $Zn(gas) + CO = ZnO + C$ . The rate of reaction is not affected by the presence of zinc oxide, alumina, or clay from which the iron oxide has been removed; ferruginous clay is, however, an active catalyst as it catalyses the reaction  $2CO = CO_2 + C$ , and the carbon dioxide formed then rapidly oxidises the zinc vapour. It is suggested, therefore, that the presence of iron oxide in the clay from which condensers are made may be the cause or one of the causes of the formation of blue powder in zinc smelting.

A. R. POWELL.

**Assay of low-grade tin ores and tailings.** G. E. GABRIEL (*Chem. Eng. and Min. Rev.*, 1928, 20, 407).—1 g. of the finely-ground sample is added to a cooled melt of 8 g. of sodium hydroxide and a small quantity of charcoal. If sulphides are present, the ore is first oxidised with aqua regia. After fusion, the cooled cake is dissolved in 50 c.c. of water and 50 c.c. of hydrochloric acid and transferred to a conical flask fitted with a stopper and tube which dips into a solution of sodium carbonate. Reduction to stannous chloride is effected by means of powdered antimony in an atmosphere of carbon dioxide and, while cooling, sodium carbonate is drawn into the flask to maintain a neutral atmosphere. The stannous chloride is titrated cold with iodine solution.

C. A. KING.

**Electrodeposition of aluminium.** D. B. KEYES, S. SWANN, JUN., W. KLABUNDE, and S. T. SCHICKTANZ (*Ind. Eng. Chem.*, 1928, 20, 1068—1069).—An adherent coating of aluminium was obtained on a copper cathode by the electrolysis of the reaction products of aluminium and ethyl iodide (cf. Grignard and Jenkins, *A.*, 1924, i, 951) treated with dry ether. A less successful result was obtained with tetraethylammonium bromide mixed with aluminium bromide. Aluminium electrolyte would find application in economiser tubes, gas cylinders, cracking stills, etc.

C. IRWIN.

**Influence of composition and cold-working on corrosion and increase of grain size of aluminium.** L. GUILLET and BALLAY (*Compt. rend.*, 1928, 187, 585—587).—Pure aluminium corrodes less than if only 98.81% pure in aqueous hydrochloric acid, pure nitric acid, and 3% sodium chloride solution. Both metals behave alike in 5% nitric acid and sulphuric acid solutions. The influence of cold-working is much greater in the case of the impure aluminium, both in increasing the corrosion and enlarging the grain size.

C. W. GIBBY.

**The Madsenell process [for the degasification of metal surfaces before plating].** F. M. DORSEY (*Ind. Eng. Chem.*, 1928, 20, 1094—1099).—The work is transferred along a line of tanks by an overhead crane. Oil is removed in a bath of potassium hydroxide solution with whale oil soap, working with a 6-volt

direct current. The article is rinsed, pickled, again rinsed, and is then ready for degasification. This is carried out in 95% sulphuric acid with a 12 volts *E.M.F.* The current rises to 50 amp. and then falls to zero when gas ceases to be evolved. Embedded oil and grease are removed as well as occluded gases. The preparation of a nickel plating bath and its control by means of bromothymol-blue are described. After 48 hrs. the bath must be filtered and its composition adjusted. Polarisation is minimised by working as near the neutral point as possible ( $p_H$  4—6), by addition of hydrogen peroxide, and by superimposing an alternating current. A continuous plant for the treatment of wire etc. is described. The product is characterised by perfect adherence in any thickness, a maximum corrosion resistance, and a bond which allows of bending, welding, soldering, etc.

C. IRWIN.

**Determination of small quantities of carbonate.** VERNON and WHITEBY.—See VII.

#### PATENTS.

**Treatment of sulphide ores or sulphidic metallurgical products.** F. KRUPP GRUSONWERK A.-G. (B.P. 297,597, 24.11.27. Addn. to B.P. 234,826; B., 1925, 676).—The material, contained in, *e.g.*, a rotary tubular furnace, is first roasted in an oxidising atmosphere which may contain air, in order to remove a portion only of the sulphur as sulphur dioxide. The product, mixed with reducing and sulphur-binding agents, such as alkaline-earth compounds, is then subjected to a combined reducing, roasting, and distilling treatment in a volatilisation operation so as to liberate a distillation product which is made to react with the gases in the atmosphere of the preliminary roasting zone or of the volatilisation zone to form metallic sulphates. The zinc sulphate so formed is extracted by lixiviation, any lead sulphate associated with it remaining behind as an insoluble residue.

M. E. NOTTAGE.

**Treatment of ores.** Q. BENT, E. BARNHART, and F. W. WOOD, Asses. to BETHLEHEM STEEL Co. (U.S.P. 1,684,006, 11.9.28. Appl., 4.10.20).—In their passage through a moderately heated, slightly inclined, horizontal, rotary kiln having longitudinal ribs, wet plastic ores are partially dried and formed into masses of convenient size and in a condition suitable for subsequent furnacing.

F. G. CLARKE.

**Purification of pig iron.** F. WÜST (B.P. 274,438, 17.6.27. Ger., 17.7.26).—Molten pig iron is brought into contact with a dephosphorising and desulphurising slag in a rotary furnace, and on saturation of the slag the metal is purified finally by the introduction of a further quantity of fresh slag, which is afterwards used for the preliminary treatment of a later quantity of iron direct from the blast furnace.

C. A. KING.

**Wrought articles of iron-chromium-nickel alloy.** H. E. POTTS. From ELECTRO METALLURGICAL Co. (B.P. 297,045, 9.6.27).—An alloy of iron containing 15—40% Cr, 2—15% Ni, 0.7—3% Si, 0.7—3% Mn, and 1% C, or less, is rolled into plates and formed into articles by welding; the articles having a high resistance to corrosion. [Stat. ref.]

C. A. KING.



**Ferrous alloy.** P. A. E. ARMSTRONG (U.S.P. 1,687,486, 16.10.28. Appl., 14.8.25).—The alloy contains approx. 9% Cr, 3.5% Si, 3% Ta, and 0.5% C.

F. G. CROSSE.

**Alloy.** G. H. BENDER (U.S.P. 1,685,975, 2.10.28. Appl., 4.4.27).—Copper (70.3 pts.) is alloyed with tin (24.53 pts.) and lead (5.2 pts.).

F. G. CROSSE.

**Heat-treating copper-nickel-beryllium alloys.** M. G. CORSON (B.P. 279,425, 3.10.27. U.S., 20.10.26).—An alloy containing copper, 0.1—2% (0.3%) Be, and up to 40% (4%) Ni is worked, heated to 900°, and either cooled slowly or quenched and reheated to 350—700°. [Stat. ref.]

C. A. KING.

**Alloy and its manufacture.** A. L. FEILD, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,684,696, 18.9.28. Appl., 25.10.24).—In order to combine zirconium with copper, an alloy of aluminium and zirconium is introduced into a superheated bath of copper.

F. G. CROSSE.

**[Nickel] alloy.** R. FRANKS and B. E. FIELD, Assrs. to HAYNES STELLITE Co. (U.S.P. 1,684,131, 11.9.28. Appl., 5.3.26).—The alloy claimed in U.S.P. 1,675,798 (B., 1928, 609) is modified by substituting titanium for zirconium.

F. G. CROSSE.

**[Silver] alloys resistant to chemical action.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,165, 30.6.27).—Silver alloys resistant to tarnishing by sulphides and to corrosion by hydrochloric acid contain at least 78% Ag and up to 22% of a mixture of aluminium with one or more of the elements thallium, magnesium, zinc, arsenic, antimony, bismuth, tin, or silicon. The alloy containing 78% Ag, 5% Al, and 17% Tl is claimed to resist almost completely the action of concentrated hydrochloric acid and to be suitable for the manufacture of chemical apparatus for handling this acid.

A. R. POWELL.

**Recovery of tin from tin scrap or cuttings.** T. TWYNAM (B.P. 297,528, 11.7.27).—Tin scrap is immersed in commercial hydrochloric acid diluted with 3—4 times its volume of water, or in the acid liquor from galvanising pickling operations, the temperature being slowly raised to 80—90° so as preferentially to dissolve the tin, which is subsequently precipitated from solution by means of zinc.

M. E. NOTTAGE.

**Fluxes for use in melting aluminium or other non-ferrous metals.** G. W. WARDLE (B.P. 297,635, 17.2.28).—Cryolite is crushed, fused at a temperature considerably above the m.p. of aluminium, and formed into blocks, which will float on the surface of the molten metal.

M. E. NOTTAGE.

**Polishing of chromium.** METALS PROTECTION CORP., Assees. of J. C. PATTEN (B.P. 277,296, 24.6.27. U.S. 9.9.26).—Finely-divided, partly calcined hydrated alumina or bauxite, made by heating precipitated aluminium hydroxide at 600—1000°, is used either alone or mixed with stearic acid, a mixture of stearic acid and beeswax, or with jellied soap.

M. E. NOTTAGE.

**Manufacture of magnesium[chromium] alloy.** W. R. VEAZEY, Assr. to DOW CHEM. Co. (U.S.P. 1,685,653, 25.9.28. Appl., 19.12.24).—Magnesium is alloyed with 0.01—0.1% of chromium.

F. G. CROSSE.

**Making synthetic pig iron.** E. EDWIN, Assr. to A./S. NORSK STAAL (ELEKTRISK-GAS-REDUKTION) (U.S.P. 1,686,075, 2.10.28. Appl., 25.1.27. Norw., 10.12.25).—See B.P. 286,067; B., 1928, 372.

**Production of wrought iron with increased tensile properties.** G. G. GEDDA (U.S.P. 1,685,602, 25.9.28. Appl., 9.12.25. Swed., 17.7.25).—See B.P. 260,789; B., 1927, 46.

**Refining of lead bullion containing other metals.** G. K. WILLIAMS (U.S.P. 1,687,187—8, 9.10.28. Appl., 9.2.27. Austral., 6.3.26).—See B.P. 267,104; B., 1928, 270.

**Producing metals or alloys low in carbon directly from ore etc. Direct-reduction process for producing carbon-binding metals or alloys.** H. G. FLODIN and E. G. T. GUSTAFSSON, Assrs. [B] to H. G. E. CORNELIUS (U.S.P. 1,686,206—7, 2.10.28. Appl., [A] 17.12.24, [B] 7.4.25. Swed., [A] 12.1.24, [B] 27.11.24).—See B.P. 227,435 and 243,743; B., 1925, 766; 1927, 337.

**Manufacture of sand mould for casting magnesium.** G. MICHEL (U.S.P. 1,685,553, 25.9.28. Appl., 5.2.25. Fr., 4.10.24).—See B.P. 254,159; B., 1926, 755.

**Impregnation of metal layers.** N. MEURER, Assr. to METALLOGEN GES.M.B.H. (U.S.P. 1,688,127, 16.10.28. Appl., 20.12.22. Ger., 28.1.22).—See B.P. 211,181; B., 1924, 428.

**Alloy and its application to the manufacture of electrical conductors.** W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (U.S.P. 1,688,247, 16.10.28. Appl., 6.1.27. U.K., 10.7.26).—See B.P. 278,454; B., 1927, 912.

**Electrolytically separating alloys of silver with other precious or base metals.** R. CARL (U.S.P. 1,687,056, 9.10.28. Appl., 27.7.23. Austr., 24.11.22).—See G.P. 377,144; B., 1923, 936 A.

**Placer mining by direct leaching with cyanide or other solvents.** B. STOCES (B.P. 296,813, 9.6.27).

**Process and apparatus for welding.** W. E. BEATTY. From BELL TELEPHONE LABS., INC. (B.P. 297,493, 23.6.27).

## XI.—ELECTROTECHNICS.

**Apparatus for electrolysis with high current densities.** E. DUHME and H. GERDIEN (Wiss. Veröff. Siemens-Konz., 1928, 7, 304—306).—The apparatus comprises a lower electrode consisting of a solid copper cylinder inside a glass mantle covered with a quartz plate, and an upper electrode consisting of a smaller, hollow, cylindrical electrode of platinum or other suitable metal. The copper cylinder has a vertical hole through the middle lined internally with a platinum tube, cone-shaped at its upper end, which terminates 2 m. above the copper and is directly below a hole in the centre of the quartz plate. The liquid to be electrolysed is passed downwards through a vertical tube in the centre of the upper electrode directly on to the hole in the lower electrode, so that 90% passes through the hole and 10% circulates back to the upper electrode.

A. R. POWELL.

**Electrochemical behaviour of chromium steel.** MEYER and ROESCH. **Copper castings.** MASING and HAASE. **Electrodeposition of aluminium.** KEYES and others. **Madsenell process.** DORSEY.—See X. **Conductivity of soil extracts.** SEN.—See XVI. **Determining the  $p_H$  values of beers etc.** STOCKHAUSEN and ROTHENBACH.—See XVIII.

## PATENTS.

**Dry cell.** L. MELLERSH-JACKSON. From C. F. BURGESS LABS., INC. (B.P. 297,400, 13.6.27).—A perforated anode in the form of a shallow pan is provided with gelatinisable electrolyte on one side and an expansion chamber on the other. Opposed electrodes are spaced apart by a collar of fibrous material impregnated with polymerised tung oil. J. S. G. THOMAS.

**Manufacture of accumulator plates.** I. G. FARBEIND. A.-G. (B.P. 290,193, 2.4.28. Ger., 9.5.27. Addn. to B.P. 284,352; B., 1928, 717).—Prisms of lead alloy are inserted or pressed into cells in a lead skeleton structure, and the whole is pressed to form a massive block or plate, from which the metal alloyed with the lead is subsequently removed. J. S. G. THOMAS.

**[Positive plates for] alkaline storage batteries.** J. F. MONNOT (B.P. 297,996, 4.11.27).—A perforated metallic tube, *e.g.*, of steel, of substantially circular cross-section is filled with alternate layers of perforated nickel foil and nickel hydroxide, the foil employed being greater in size than the internal cross-sectional area of the tube so that it makes good contact with the wall of the tube. J. S. G. THOMAS.

**Battery composition.** P. J. KELLEHER (U.S.P. 1,685,674, 25.9.28. Appl., 16.3.26).—A composition capable of preventing "sulphation" in storage batteries consists of 84% of magnesium sulphate, 3% of ammonium sulphate, 6% of potassium sulphate, and 7% of aluminium sulphate. F. G. CROSSE.

**Electrolyte for storage batteries.** A. B. WERBY, Assr. to AMER. AUTOMOTIVE CORP. (U.S.P. 1,684,852, 18.9.28. Appl., 5.6.28).—Dilute sulphuric acid containing ammonium acetate is used. F. C. CROSSE.

**Electrolyte for lead accumulators.** M. KUGEL (B.P. 280,197, 28.10.27. Ger., 6.11.26).—Sulphation in lead accumulators is minimised by adding a small quantity (0—20 g./litre) of phosphoric acid to the dilute sulphuric acid usually present. H. ROYAL-DAWSON.

**Electrolyte for accumulators.** M. KUGEL (B.P. 291,020, 28.10.27. Ger., 24.5.27. Addn. to B.P. 280,197; B., 1928, preceding).—Sulphation is avoided by adding fresh phosphoric acid from time to time to the electrolyte to replace loss due to wastage.

H. ROYAL-DAWSON.

**Anodes for gas-discharge vessels.** SIEMENS & HALSKE A.-G. (B.P. 279,501, 22.10.27. Ger., 23.10.26).—The anode is made of conducting refractory material, *e.g.*, tantalum or molybdenum, which absorbs gases at its normal working temperature. J. S. G. THOMAS.

**Electrolytic rectifier.** W. C. READ, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,684,684, 18.9.28. Appl., 8.3.27).—In a rectifier consisting of an anode of lead composition and an electrolyte of 10—40% sulphuric

acid containing the sulphate of a metal, the cathode is composed of silicon alloyed with 10—40% of a metal of the titanium group. F. G. CROSSE.

**Measuring the intensity of radiation especially of sources of ultra-violet light.** J. Y. JOHNSON. From I. G. FARBEIND. A.-G. (B.P. 296,198, 8.9.27).—The intensity of ultra-violet radiation of 210—350 $\mu\mu$  is determined by exposing solutions of phototropic compounds, *e.g.*, leucocyanines, carbinols, or sulphurous compounds of crystal-violet, malachite-green, fuchsin, etc., containing small quantities of substances counteracting the photochemical colour change, *e.g.*, potassium cyanide, potassium hydroxide, and sulphurous acid, respectively, to the action of the rays, and comparing with standard solutions of the dyes themselves the intensity of the coloration produced.

L. A. COLES.

**Self-baking electrodes [for electric furnaces].** C. W. SÖDERBERG, Assr. to NORSKE A./S. FOR ELEKTROKEM. IND. (U.S.P. 1,686,474, 2.10.28. Appl., 18.9.26. Norw., 19.9.25).—See B.P. 258,560; B., 1927, 257.

**Electric battery of the Leclanché type.** G. N. ANTONOFF, Assr. to M. A. ADAM (U.S.P. 1,687,051, 9.10.28. Appl., 26.5.24. U.K., 8.6.23).—See B.P. 225,889; B., 1925, 105.

**[Electrodes for] batteries, electric accumulators, and electrolysing apparatus.** R. OPPENHEIM, Assr. to SOC. ANON. LE CARBONE (U.S.P. 1,687,307, 9.10.28. Appl., 30.9.25. Fr., 10.12.24).—See B.P. 244,417; B., 1927, 17.

**Sludging of transformer oils (B.P. 278,365).—**See II.

## XII.—FATS; OILS; WAXES.

**Fatty acids of Egyptian butter fats.** H. ATKINSON (Analyst, 1928, 53, 520—530).—An analytical examination of the butter of the gamoos, cow, goat, and Syrian sann shows that the usual methods are insufficient to determine adulteration when the origin of the butter is unknown. The variations in the Polenske and Reichert-Meissl values allow of additions of appreciable proportions of coconut or palm-kernel oils, so that the methods of Kirschner, Blichfeldt, Ewers, Cassal, and Gerrans and Gilmour fail. Since the acids differentiating the fats of sheep and goats from those of cows and gamoos (octoic and deoic) are soluble in aqueous alcohol, Shrewsbury and Knapp's method is of little use. If, however, 1 g. of saponified fat is distilled with 210 c.c. of water, the titration value of the non-volatile acids expressed as mg. of potassium hydroxide obtained by subtracting that of the volatile acids from the saponification value is, in general, lower in the sheep and goat butters. Further, the sum of oleic acid and acids insoluble in 62% aqueous alcohol, subtracted from the total non-volatile acids, is, within reasonable limits, a constant for each fat, and this constant is increased by coconut or palm-kernel oil practically in proportion to the amount present, and the residual acids are increased by about 12 units for every 10% of fat. These latter are decreased by beef fat by about 2 units for every 10% of fat. Having thus ascertained the purity of a butter fat,

the origin may be deduced from the Polenske-Reichert-Meissl ratio or the "insoluble silver" value. Tables of figures for various types of butter from cows, gamoos, goat, and Syrian samn are given, both pure and with addition of varying proportions of palm-kernel oil, coconut oil, and beef fat, also a chart comparing the acids of Egyptian butter fats with those of coconut and palm-kernel oils. D. G. HEWER.

**Duclaux method for determination of volatile fatty acids, and its application to the determination of butter fat in margarine.** A. KNETEMANN (Rec. trav. chim., 1928, 47, 950—970).—In the Duclaux method for the determination of volatile fatty acids (A., 1896, ii, 504), the relation between the concentration of the acid in the vapour and the liquid (distillation value = *C*) is constant at each stage of the distillation and varies with the acid used. This value has been determined experimentally for formic, acetic, propionic, butyric, valeric, hexoic, and octoic acids, and it increases regularly with increase of the carbon chain. Distillation curves are plotted for the above acids and for benzoic and salicylic acids. The constants deduced by Duclaux and by Boekhout and de Vries (A., 1917, ii, 50) are shown to be erroneous. For the determination of butter fat in margarine the fat is hydrolysed with concentrated potassium hydroxide solution in presence of glycerol at 135° (higher temperatures must be avoided as volatile acids are produced), and after dilution and acidification the solution is extracted with light petroleum. The filtered aqueous solution is distilled and two consecutive portions (100 and 200 c.c.) of the distillate are titrated with standard alkali. Substitution of these titration values in a formula deduced experimentally from the results obtained using artificial mixtures of butter fat, oleo oil, and coconut oil, gives the percentage value. The results appear to be trustworthy even when abnormal coconut oil is present. H. BURTON.

**Detection and determination of arachis oil in olive oil.** E. JAFFE (Annali Chim. Appl., 1928, 18, 368—386).—The lithium soaps of arachis oil, either pure or mixed with olive oil, are precipitated from their alcoholic solution at a higher temperature than the corresponding soaps of pure olive oil. Moreover, the fatty acids obtained by decomposing the lithium salts collected at 17—18° are precipitated from their solution in 90% alcohol only if arachidic acid is present. The details of the procedure to be followed are given. From the results obtained the proportion of arachis oil present may be determined by means of a table giving various temperatures of precipitation of the lithium salts of the fatty acids (ranging from 24° to 57°) and temperatures of crystallisation of the crude arachidic acid from 90% alcohol (over the range 15—43°). T. H. POPE.

**Determination of oil in seeds, etc.** F. GOGOLEV (Masloboino-Shirowoje Djelo, 1927, No. 5, 20—21).—It is recommended that seeds should be ground in a mortar, oil-cake in a mortar or mill, and kept for 12 hrs. in the solvent before extraction (5—6 hrs. extraction for seeds, 2—3 hrs. for oil-cake); if this is omitted, an extra hour's extraction must be given. C. HOLLINS.

**Margarine.** VITOUX.—See XIX.

## PATENTS.

**Separation of butter fat from buttermilk and buttermilk whey. Recovering butter from buttermilk.** A. L. RUSHTON, Assr. to M. H. LANE (U.S.P. 1,683,728—9, 11.9.28. Appl., [A] 16.2.27, [B] 29.9.27).—(A) Sour cream buttermilk is treated, e.g., by heating or adding an acid or alkali, so that the buttermilk having a heavy casein content settles from the clear whey. The latter is then passed slowly through a cream separator. (B) Buttermilk whey is passed through a separator at the usual rate for milk, the recovered "buttermilk cream" is then passed through the separator at a slower rate; the buttermilk cream is finally churned. F. G. CLARKE.

**Stabilisation of fats, oils and waxes.** J. Y. HUBER, JUN. (U.S.P. 1,680,047, 7.8.28. Appl., 4.2.24).—0.01% of furfuraldehyde added to animal or vegetable oils or fats retards the development of rancidity. C. HOLLINS.

**Manufacture of an insoluble soap from aldehyde-fatty acid mixtures [catalytic oxidation products] of mineral oil.** J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,681,237, 21.8.28. Appl., 7.3.19).—The mixture obtained by catalytic oxidation of petroleum hydrocarbons contains  $\omega$ -aldehyde-derivatives of acids C<sub>12</sub> to C<sub>15</sub>, together with aldehydes and hydrocarbons. The former are converted into soaps by treating with caustic alkali and separate as gels. C. HOLLINS.

**Manufacture of sulphonic acids derived from non-aromatic carboxylic acids.** I. G. FARBENIND. A.-G. (B.P. 288,612, 21.6.27. Ger., 21.6.26. Cf. B.P. 272,967; B., 1928, 703).—Non-aromatic carboxylic acids containing more than eight carbon atoms in the molecule are sulphonated by strong sulphonating agents (e.g., sulphur trioxide, chlorosulphonic acid) at above 35° (e.g., 100°). Neutral media, unacted on in the working conditions, e.g., carbon tetrachloride, and/or catalysts may be employed. E. LEWKOWITSCH.

**Manufacture of a finely-divided dry soap product.** INDUSTRIAL SPRAY-DRYING CORP., Asses. of R. L. HOLLIDAY (B.P. 269,506, 21.3.27. U.S., 19.4.26).—See U.S.P. 1,621,506; B., 1927, 339.

**Purification of oils and fats.** W. GENSECKE, Assr. to AMER. LURGI CORP. (U.S.P. 1,685,195, 25.9.28. Appl., 5.2.25. Italy, 15.2.24).—See B.P. 229,283; B., 1925, 889.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Unreliability of visual inspection of exposure tests of paints.** P. H. WALKER and E. F. HICKSON (Ind. Eng. Chem., 1928, 20, 997).—After three years' exposure of two coats of red lead paint on steel, visual inspection indicated an advanced state of decay of the coating, although no serious corrosion had occurred. On vigorous washing with soap and water the paint was found to be quite intact and in excellent condition. This was confirmed by the electrical conductivity method (B., 1928, 578). Deposits of dirt, especially on red lead paint, frequently have the appearance of iron rust. S. S. WOOLF.

**Analysis of Bordeaux spirits of turpentine by rise of temperature on mixing with sulphuric acid.**

Mlle.) M. BARRAUD (Bull. Inst. Pin, 1928, 47, 73—76).—The rise of temperature observed when freshly prepared spirits of turpentine is mixed with sulphuric acid ( $d^{15} 1.72$ ) is constant for products of the same origin, but increases rapidly with ageing of the turpentine. The first 20% of the distillate gives fairly constant values whether the sample is oxidised or not; hence the addition of 2.5% of white spirit can be detected. The rise of temperature for turpentines of different origins varied from 71.5° to 116.5°.

CHEMICAL ABSTRACTS.

**Inhalation experiments with certain lacquer solvents.** H. F. SMYTH and H. F. SMYTH, JUN. (J. Ind. Hygiene, 1928, 10, 261—271).—The toxicity of lacquer solvents, with the exception of benzene, has been studied by means of gassing tests on guinea-pigs; the concentrations employed were such as might be expected to occur in practice, depending on the volatility and percentage of the solvent in the lacquer. Ethyl and amyl acetate, and possibly butyl acetate, are usable with safety in the concentrations generally employed, and possibly in somewhat higher concentrations; gasoline, turpentine, and xylene are possibly harmful in concentrations materially greater than those in present use; the proportions of toluene and butyl alcohol now employed represent very nearly the upper limit of safety. With suitable exhaust ventilation these limits may be exceeded with impunity. F. R. ENNOS.

**False equilibria, with special reference to rosin solutions and gold size.** R. P. L. BRITTON (J. Oil and Colour Chem. Assoc., 1928, 11, 323—330).—A general discussion on "false equilibria" is illustrated by the behaviour of solutions of rosin in methylated spirit etc. and of gold size (considered as a combination of a varnish and a strong solution of drier). The author indicates the possibility of a mobile equilibrium between the super-fused and the crystalline varieties of varnish material, *e.g.*, gums, linoleates, etc. The use of protective agents or stabilisers to control such equilibria is of importance in the varnish industry, the superiority of turpentine over white (petroleum) spirit as a solvent being an example of this type of protection. S. S. WOOLF.

**Rosin size.** DELCROIX.—See V.

## PATENTS.

**Anti-corrosive heat-resisting paint or composition.** J. C. CARROLL (B.P. 297,342, 18.6.27).—A powdered and dried titanium ore, *e.g.*, ilmenite, ground in raw linseed oil and thinned with dark boiled oil, varnish, and white (petroleum) spirit is claimed. S. S. WOOLF.

**Fatty essence [oxidised turpentine oil].** L. DESALBRES and J. DUBOURG (F.P. 620,462, 19.8.26).—Turpentine etc. is oxidised with air or oxygen in presence of a catalyst. C. HOLLINS.

**Production of white lead.** F. T. BAILEY and W. AUSTIN (B.P. 273,287, 16.6.27. U.S. 22.6.26).—A mixture of dilute acetic acid and powdered lead oxide is repeatedly sprayed through a current of carbon dioxide in suitable apparatus until the oxide is completely converted into white lead. As hydrating agent or catalyst, ammonium nitrate, nitric acid, etc. may replace acetic acid. Conversion into white lead is direct, the intermediate formation of lead acetate being eliminated. S. S. WOOLF.

**Manufacture of coloured compounds [lakes].** I. G. FARBENIND. A.-G. (B.P. 275,943, 21.6.27. Ger., 12.8.26).—Compounds which are not basic dyes and are free from acid groups, but contain amino-groups, are treated in acid solution with sodium phosphotungstomolybdate or other similar salt. Complex phosphotungstomolybdates of 4:4'-tetramethyldiamino-diphenylmethane, -benzophenone, -benzhydrol, and *p*-aminobenzaldehyde are described. C. HOLLINS.

**Production of colouring materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,076, 14.3.27. Cf. B.P. 267,924; B., 1928, 708).—For the dispersion of colouring matters (*e.g.*, soot, umber) these are mixed with sulphonated mineral oil etc. as used in the prior patent for treatment of textiles. C. HOLLINS.

**Manufacture of transparent lacquers.** CHEM. FABR. DR. K. ALBERT G.M.B.H. (G.P. 448,297, 11.6.25).—The free acid of natural resins, with or without admixture with synthetic resins, is partly or wholly converted into lithium salts (for spirit lacquers) or cadmium salts (for oil lacquers). *E.g.*, molten French colophony is treated at 200—250° with 1% of cadmium oxide, which rapidly dissolves, and then with successive small amounts of calcium hydroxide; American colophony is melted with cadmium resinate and esterified with glycerol; a phenol-acetone-formaldehyde resin is melted with cadmium oleate and colophony. The products yield lacquers which remain transparent for long periods. C. HOLLINS.

**Prevention of livering in pigmented carbohydrate-compound compositions [cellulose esters and ethers].** E. I. DU PONT DE NEMOURS & Co., Asses. of J. D. McBURNEY and E. H. NOLLAU (B.P. 278,696, 26.9.27. U.S., 5.10.26).—The livering is prevented or removed by the addition of not more than 5% of a compound having a dissociation constant of 1.0—4.7, *e.g.*, citric, malic, or tartaric acid. L. A. COLES.

**Manufacturing floor covering etc.** ARMSTRONG CORK Co. (B.P. 275,949, 4.7.27. U.S., 13.8.26).—Hard floor-covering materials are surfaced with pigmented nitrocellulose lacquers applied in a block printing machine, the atmosphere around which is cooled and saturated with lacquer solvents, thus precluding evaporation of solvents from the lacquer. The surfaced material is subsequently dried in a curing chamber from which solvent vapours are withdrawn. S. S. WOOLF.

**Preparation of derivatives of resinous phenol-aldehyde condensation products.** I. G. FARBENIND. A.-G. (G.P. 449,276, 14.1.20. Addn. to G.P. 386,733; B., 1924, 604).—Fusible phenol-aldehyde resins are treated with halogenated aliphatic acids, *e.g.*, mono- or di-chloroacetic,  $\beta$ -bromopropionic, or chloromalonic acids. The products are fusible and are soluble in alkali. C. HOLLINS.

**[Resinous] condensation product and its manufacture.** A. L. BROWN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,680,408, 14.8.28. Appl., 23.2.22).—Phenol is condensed with formaldehyde and tung oil. C. HOLLINS.

**Plastic material from peat, and its manufacture.** I. S. MELLANOV, Assr. to KEMIKAL, INC. (U.S.P. 1,681,155, 14.8.28. Appl., 23.2.28).—A solution of peat

in hot aqueous sodium hydroxide is treated with phenol and formaldehyde to give a brown to black plastic mass.

C. HOLLINS.

#### Preparation of resinous condensation products.

BRIT. THOMSON-HOUSTON CO., LTD., Assees. of [A] L. V. ADAMS and [B] R. H. KIENLE (B.P. 273,748 and 284,349, [A] 1.7.27, [B] 28.1.28. U.S., [A] 1.7.26, [B] 29.1.27).—(A) The heat-hardening of "glyptal" resins is rapidly carried to the completely condensed or C-stage, regardless of the production of cavities. The pumice-like mass is then ground and extracted in a closed container with suitable solvents, *e.g.*, glycol diacetate, ethyl phthalate, acetone oils, toluidine, benzyl alcohol, cresol, indene, etc. (B) Polyhydric alcohols and polybasic aromatic acids are condensed with oxidisable unsaturated fatty acids (*e.g.*, acids derived from drying oils), in the absence of inorganic acids, *e.g.*, sulphuric acid. The products are soluble in drying oils at ordinary temperatures, and can also be fully hardened without baking. S. S. WOOLF.

**Manufacture of artificial resins.** I. G. FARBENIND. A.-G., Assees. of M. PAQUIN, A. VOSS, and H. WOHLERS (G.P. 448,427, 5.8.24).—The resins obtained by condensation of acetaldehyde, crotonaldehyde, aldol, etc. are suspended or dissolved in inert media (*e.g.*, alcohol, water) and treated with chlorine or with potassium chlorate and hydrochloric acid. The products are suitable for production of water- and light-fast polishes.

C. HOLLINS.

**Preparation of spirit-soluble resins.** I. G. FARBENIND. A.-G., Assees. of R. LEOPOLD and A. MICHAEL (G.P. 449,115, 5.5.25).—Transparent yellow to red resins, suitable for spirit varnishes and polishes, are obtained by condensing a vinyl ester (*e.g.*, acetate) with aliphatic aldehydes or substances generating them; *e.g.*, paraformaldehyde at 150° and 20 atm. in absence of oxygen gives a yellow fusible resin. Acetaldehyde, *n*-butaldehyde, paralal, crotonaldehyde, ethylidene diacetate, etc. are also suitable.

C. HOLLINS.

#### Removing liquid from resinous products.

NAUGATUCK CHEM. CO., Assees. of M. G. SHEPARD (B.P. 276,627, 20.6.27. U.S., 25.8.26).—Mechanically retained liquid, formed or used in the preparation of resinous aldehyde-amine condensation products, is removed by melting the latter with a fluxing agent, heating, and agitating until the liquid is eliminated. The liquid-free product is drawn off, cooled, and solidified. As fluxing agents, materials, *e.g.*, stearic acid, compatible with the further use of the resinous material, *e.g.*, in rubber compounding, must be used.

S. S. WOOLF.

**Manufacture of shellac-like masses.** SIEMENS & HALSKE A.-G. (G.P. 449,275, 29.10.22).—Resins closely resembling shellac in nature and properties are obtained by heating together aliphatic and hydroaromatic hydroxylated acids. For the aleuritic acid of shellac are used lactic, glyceric, dihydroxystearic (from oleic acid), trihydroxystearic (from ricinoleic acid), or tetrahydroxystearic (sativic) acid etc. or the oxidation products from linseed oil acids, and as second component in place of shelloleic acid *cyclohexanolcarboxylic acids*,

tetrahydronaphthoic acids or their hydroxy-derivatives etc. Examples are: lactic acid and methylcyclohexan-4-ol-2-carboxylic acid heated at 120° in a vacuum for 3 hrs. and then with dihydroxystearic acid at 140° for a further 3 hrs.; trihydroxystearic acid heated at 120°, then dissolved in alcohol and saturated with hydrogen chloride, finally heated with trihydroxypalmitic and  $\beta$ -hydroxycamphoric acid at 150° and mixed with beeswax and carnauba wax; sativic acid mixed with hydroxyabietic and lactic acids and melted under reduced pressure with zinc chloride, wax being added after 5–6 hrs.

C. HOLLINS.

**Manufacture of urea-formaldehyde condensation products.** K. RIPPER, Assr. to F. POLLAK (U.S.P. 1,687,312, 9.10.28. Appl., 20.3.24. Austr., 31.3.23).—See B.P. 213,567; B., 1925, 681.

**Removal of scale from boilers** (B.P. 281,598).—See XXIII.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Reinforcement of rubber by fillers.** J. T. BLAKE (Ind. Eng. Chem., 1928, 20, 1084–1088).—Ingredients which form with the rubber a bond which is stronger than the matrix are classed as reinforcing agents; those giving a bond weaker than the matrix are diluents. Regarding the tensile strength, calculated on the cross-section at break, as a measure of the tensile strength of the matrix, a reinforcing agent such as carbon black does not increase the strength of the matrix; the apparent increase in tensile strength is due to the reduction in elongation. When the proportion of reinforcing agent is increased beyond the stage of reinforcement, perfect dispersion is no longer possible and rupture occurs at the bond between filler and rubber and the elongation is disproportionately reduced. The controlling factor in reinforcement is not particle size, but the surface energy in the filler-rubber bond. An intimate relationship between the natural fatty acids of rubber and the dispersion of a filler such as carbon black is indicated by the fact that the reinforcing effect of carbon black in rubber ceases near the proportion of 30 vols. to 100 of rubber, and that this is in approximate agreement with the maximum proportion of dispersed carbon black over which the natural fatty acids of the rubber could provide a unimolecular film. This result probably explains the improved dispersion of carbon black obtainable by the additional incorporation of stearic acid, pine tar, or reclaimed rubber.

D. F. TWISS.

**Dispersion of "pigments" in rubber. I. Microscopical studies of agglomeration and flocculation.** E. A. GRENQUIST (Ind. Eng. Chem., 1928, 20, 1073–1078).—The particle size of carbon black in rubber is 40–50 $\mu$ . It is difficult to count the particles in the microscope, but from the degree of blackness of mixtures containing 0.5% it is possible to discriminate between various types of amorphous carbon. In a 1% mixture in rubber, zinc oxide shows an average particle size of 0.49 $\mu$ . Vulcanisation of mixtures of rubber with sulphur and other ingredients appears to induce an initial improvement in dispersion, followed by flocculation, this being most pronounced with carbon black,

lamp black and thermatomic black exhibiting flocculation to a less degree. In a mixture containing carbon black (2 pts.) and zinc oxide (30 pts.) the latter seems to check the dispersion of the former. Agglomeration of particles of ingredients on the mixing mills usually occurs around any larger nuclei, *e.g.*, of foreign matter, in the rubber. D. F. TWISS.

**Light absorption of stretched and unstretched rubber and of isoprene.** M. KRÖGER and H. STAUDE (*Gummi-Ztg.*, 1928, 43, 22—24).—In a quartz spectrograph, isoprene and various samples of rubber exhibit only continuous absorption. Synthetic rubber made from isoprene is more transparent than isoprene itself at the same thickness. The transparency of synthetic and also of natural rubber increases with stretching, presumably on account of some molecular change. Rubber vulcanised with sulphur chloride is much less transparent. The relative transparency of stretched rubber to ultra-violet light might lend itself to the production of lenses consisting of spherical membranes of rubber distended with an optically transparent liquid. D. F. TWISS.

**Carbon blacks and their use in rubber. III. Ageing effects.** L. B. COX and C. R. PARK (*Ind. Eng. Chem.*, 1928, 20, 1088—1091; cf. Goodwin and Park, *B.*, 1928, 579, 649).—Vulcanised mixtures containing various proportions of different types of carbon black have been examined as to the alteration in their physical characteristics after dark storage for 18 months at 18° in moving air. The samples show slight hardening, and the stress-strain curves become straighter. The decrease in tensile strength is distinctly greater for typical carbon blacks than for those, *e.g.*, thermatomic black, made by processes involving thermal decomposition; the ageing of the mixtures vulcanised with the aid of mercaptobenzthiazole or ethylideneaniline also is superior to those containing hexamethylenetetramine or diphenylguanidine. The relative alteration of the samples with respect to loss in tensile strength and in abrasion resistance and to increase in weight is fairly consistent. The additional presence of stearic acid does not affect the ageing properties of the "compounds," nor does the presence of acidity in the sulphur used, the zinc oxide also present presumably exerting a sufficient neutralising effect. A close connexion appears to exist between the ageing influences of the various blacks and their adsorptive character, possibly because of the different proportions of oxygen thereby introduced into the rubber. D. F. TWISS.

**Rubber mixtures containing carbon black.** W. ESCH (*Gummi-Ztg.*, 1928, 43, 75—78).—Attention is drawn to the need for care in discriminating between carbon blacks made by the incomplete combustion of natural gas, special blacks made from natural gas by thermal decomposition, and lamp blacks made by the incomplete combustion of oil. There are marked distinctive features; *e.g.*, carbon blacks give rubber mixtures with lower rate of vulcanisation and with higher tensile strength than lamp blacks. D. F. TWISS.

**Jelutong.** C. D. V. GEORGI (*Malayan Agric. J.*, 1928, 16, 204—211; cf. *B.*, 1928, 309).—Introduction of

soluble iron or copper salts into latex causes a tendency to oxidation in the extracted jelutong; soaking of fresh coagulum in solutions of these salts or of caustic alkali has no such effect. Manganese salts do not induce oxidation, but favour mould development. The type of deterioration suffered by jelutong is influenced by the locality of its origin. D. F. TWISS.

**Valuation of jelutong.** C. D. V. GEORGI (*Malayan Agric. J.*, 1928, 16, 220—233).—The irregularity of distribution of moisture in a block of jelutong and variation between the blocks themselves render trustworthy evaluation difficult. It is desirable that the weight and proportion of samples to be taken and the actual method of sampling and testing be standardised. D. F. TWISS.

**Rubber-like state of matter.** VON WEIMARN.—See V.

#### PATENTS.

**Treatment of rubber latex and manufacture of rubber articles from latex.** S. M. CADWELL, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,680,856—8, 14.8.28. Appl., [A] 28.8.25, [B, C] 22.9.25. Renewed [B] 19.6.28).—(A) Rubber latex, particularly ammonia-preserved 33% latex which has been twice treated with pectin and creamed, is heated with zinc oxide (3%), sodium acetate (3%), and glue (1%) at 65°. It deposits rubber rapidly on porous articles by the dipping process. In place of sodium acetate a thio-acid salt (*e.g.*, sodium dithiobenzoate) may be used, and the latex vulcanised with sulphur before deposition. (B) A vulcanised latex is obtained by adding sulphur, zinc oxide, and tetramethylthiuram disulphide (sufficient to give a hard rubber below 100°) to a latex which already contains sulphur, zinc oxide, and a non-nitrogenous carbon disulphide accelerator sufficient to give a soft elastic product. (C) A latex treated as in (A) with zinc oxide, sulphur, and sodium dithiobenzoate, with or without glue, is vulcanised at 65°, and rubber articles are made by a dipping process. C. HOLLINS.

**Treatment of rubber, and product thereof.** H. E. CUDE, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,680,862, 14.8.28. Appl., 27.5.27).—Rubber, raw or vulcanised, is incorporated with an organic acid capable of forming a soluble soap (*e.g.*, oleic or stearic acid, sulphonated oils, etc.), and residual acidity is neutralised with alkali. A reclaim rubber is obtained as a plastic mass, or may be emulsified in the usual manner. C. HOLLINS.

**Treatment of rubber-fibre waste material.** R. P. ROSE, Assr. to MECHANICAL RUBBER CO. (U.S.P. 1,680,915, 14.8.28. Appl., 7.1.28).—The broken-up material is worked with a soap-forming fatty acid to a putty-like consistency, and then with aqueous sodium hydroxide. The rubber is finally recovered as dispersion by beating the product with aqueous alkali and a protective colloid. C. HOLLINS.

**Vulcanisation of rubber.** R. V. HEUSER, Assr. to A. C. BURRAGE, JUN. (U.S.P. 1,681,806, 21.8.28. Appl., 27.1.21).—Tri-*o*-tolylguanidine is used as accelerator. C. HOLLINS.

**Production of coherent caoutchouc or the like products.** ANODE RUBBER CO., LTD. From P. KLEIN,

F. GÁBOR, and A. SZEGVÁRI (B.P. 297,127, 16.3.27).—Two or more pieces of material obtained directly as deposition strata from aqueous dispersions, *e.g.*, by electrophoresis, dipping, spreading, or spraying, when brought together in a moist condition, with or without pressure, and then dried, become united and inseparable. The process can be applied to the production of hollow rubber articles from latex and to the attachment of valve patches to air-tubes formed by electrophoretic deposition from aqueous rubber dispersions.

D. F. TWISS.

**Vulcanisation of rubber.** C. J. T. CRONSHAW and W. J. S. NAUNTON, Asses. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,687,861, 16.10.28. Appl., 4.1.26. U.K., 12.3.25).—See B.P. 253,197; B., 1926, 682.

**Abrasive articles** (U.S.P. 1,681,891).—See VIII.

## XV.—LEATHER; GLUE.

**Action of acids on vegetable-tanned leathers.** D. WOODROFFE (J. Soc. Leather Trades' Chem., 1928, 12, 385—389; cf. B., 1927, 757, 790).—A 10% solution of oxalic acid destroyed vegetable-tanned leather soaked therein for 2 months; similarly a 5% solution considerably weakened it. The effect of other acids depended on the  $p_H$  value of their solutions, the lower the  $p_H$  value, the greater is the weakening effect. The leather was not weakened by immersion in warm solutions of acids (5% or 10%) for  $\frac{1}{2}$  hr., washing, and drying out. Vegetable-tanned skivers, East Indian sheep, basils, kip splits, and shoulders, respectively, were treated with *N*-solutions of oxalic acid, ammonia-ammonium chloride, sulphuric acid, and with 1% sulphuric acid for  $\frac{1}{2}$  hr., then dried, and kept for 6 months. The thinner the leather the greater was the relative weakening effect of both the acids. The moderate use of sulphuric or oxalic acids, such as might be required in leather dressing, does not damage vegetable-tanned leathers, except skivers.

D. WOODROFFE.

**Utilisation of tanning materials containing a large amount of soluble non-tans.** V. S. SADIKOV and P. A. YAKIMOV (Trans. State Inst. Appl. Chem. Moscow, 1927, No. 6, 40—63).—The finely-powdered bark of *Saxifraga* (*Bergenia crassifolia* (badan) is treated first with calcium hydroxide, then with cold water, and finally with hot water and oxalic acid or sodium hydrogen sulphate. The first (cold) extract contains 30%, and the second (hot) extract contains 60%, of the tannins. The method has been applied to other Russian tanning materials.

CHEMICAL ABSTRACTS.

### PATENTS.

**Leather substitute.** E. I. DU PONT DE NEMOURS & Co., Asses. of J. D. MCBURNEY and E. H. NOLLAU (B.P. 273,324, 24.6.27. U.S., 24.6.26).—Textile fabric coated with a cellulose ester, *e.g.*, pyroxylin, is provided with a flexible, waterproof surface comprising casein and glycerol to allow adhesives to spread over and adhere to it.

L. A. COLES.

**Manufacture of artificial [horn-like] masses containing casein.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,483, 23.5.27).—Solid or

liquid hydroaromatic or aliphatic-hydroaromatic amines or aliphatic amines containing hydroxyl groups, or their salts or derivatives, are incorporated with casein.

L. A. COLES.

**Mineral tanning and products obtained thereby.** F. W. WEBER (B.P. 297,754, 21.3.27).—See U.S.P. 1,642,054; B., 1927, 917.

## XVI.—AGRICULTURE.

**Soils. III. Absorbing power of soils for ammonia.** J. CLARENS and (Mme.) PÉRON (Bull. Soc. chim., 1928, [iv], 43, 962—969; cf. B., 1927, 950).—The absorption of ammonium salts by a clay soil containing considerable quantities of lime and a small amount of humus is investigated. True adsorption does not occur, since the absorption takes place in stages. The order of absorbability is hydroxide > phosphate > sulphate > chloride. The bearing of these results on the artificial manuring of the soils is discussed. O. J. WALKER.

**Losses and gains of nitrogen in an Indian soil.** H. E. ANNETT, A. R. P. AIYER, and R. N. KAYASTH (Mem. Dept. Agric. India, 1928, 9, 155—232).—Six plots on the black cotton soil of Nagpur were sampled at weekly intervals for two years. Determinations of nitrate on these samples were made by the phenol-disulphonic acid method. A rapid accumulation of nitrates took place during the June and July rains. Heavy rains in August washed these nitrates through the soil, and the nitrate content of the soil then remained at a low level till January. The cultivation of cold-weather crops was then accompanied by a further rise in the nitrate content of the soil. Although these losses of nitrates were accompanied by rises in the nitrate content of drainage and well waters, it is concluded that some denitrification must take place in order to account for the large amounts of nitrates lost. In spite of this heavy loss of nitrogen, the total nitrogen content of the soil now is the same as it was 25 years ago. Considerable fixation of nitrogen must therefore have taken place.

H. J. G. HINES.

**Forms and properties of water-soluble phosphorus in soils.** F. W. PARKER (Science, 1928, 67, 402—403).—The presence of at least two forms of organic phosphate in soil extracts and solutions is indicated; the amounts of the two forms are usually approximately equal. Soil extracts and solutions can be boiled for 2 hrs. under reflux without materially increasing the inorganic phosphate content.

A. A. ELDRIDGE.

**Electrodialysis of soil.** K. N. TARANOV (Ukraine Chem. J., 1928, 4, [Tech.], 89—112).—Various Russian soils were subjected to electrodialysis, which is shown to be a more efficient means of separating inorganic anions and cations than extraction with ammonium chloride solution. The time necessary to complete the process varied from 19 hrs. for one soil to 152 hrs. for Ukrainian black earth. The acidity of the soil rose considerably after electrodialysis, in one case from 15 to 174 c.c. of 0.1*N*-acid. This increase in acidity applies both to total acidity as measured by extraction with *N*-sodium acetate, and to replaceable acidity as measured by titration of the potassium chloride extract with 0.1*N*-

alkali. This suggests the presence of two different types of substance in the soil, the relative ratio of which determines its eventual acidity. The soil residue after electro-dialysis was practically non-hygroscopic, pointing to an aggregation of colloidal particles under the influence of this process.  
R. TRUSZKOWSKI.

**Determination of the electrical conductivity of the aqueous extract of soil as a rapid means of detecting its probable fertility.** A. SEN (Mem. Dept. Agric. India, 1928, 9, 247—254).—The sieved air-dry sample is placed in a tube, 5 pts. of conductivity water are added, and the tube is closed by a paraffined cork carrying a dip electrode. The tube is then shaken and placed in a thermostat at 30°. After 2 hrs. the conductivity is measured. Similar measurements are made at intervals up to 9 days. Fertile soils show a rapid increase in the conductivity of the extract; infertile soils show little or no change. The comparison is, of course, limited to soils from adjacent areas.  
H. J. G. HINES.

**Law of crop yield.** A. RIPPEL (Z. Pflanz. Düng., 1928, 12A, 38—55).—Oats, mustard, and sunflowers were grown simultaneously in pots and fertilised with increasing amounts of nitrogen and potash. On applying the Mitscherlich formula to the crop yields it was shown that the nutrient constant varied from crop to crop. It is concluded that the factors influencing crop yield cannot be combined in a single simple expression of the Mitscherlich type.  
H. J. G. HINES.

**The hot-fermentation process [of manure] and "edelmist."** G. RUSCHMANN (Z. Spiritusind., 1928, 51, 258—259, 266—267, 275—276, 284).—The Krantz process for the hot fermentation of manure is described. The humification process in hot-fermented stacks is more complete than in ordinary yard stacks, there being practically no fibrous matter remaining. Comparison of the carbon dioxide production in soil composts with manure stacked and fermented in different ways indicates that the ageing of "edelmist" does not result in appreciable losses of easily oxidisable carbonaceous matter. The importance of close adherence to the Krantz procedure is emphasised. Unless the initial fermentation temperature of 60—70° is reached, the second stage or ripening of the stack involves loss of both organic matter and nutrient material.  
A. G. POLLARD.

**Artificial farmyard manure.** W. R. GREENSTREET (Malayan Agric. J., 1928, 16, 194—203).—Under the conditions obtaining in Malay, vegetable matter will rot easily and quickly provided that acidity is neutralised, a free supply of air is given, and the moisture content maintained at 75%. Although the claims made for the "Adco" process are substantiated, the addition of "Adco" reagents to the materials tested would appear to be superfluous.  
H. J. G. HINES.

**Basic slag.** WEISS.—See X. **Fruit of *Schinus molle*.** CREMONINI.—See XX.

## PATENTS.

**Mixed fertilisers.** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 297,960, 15.9.27).—The fertilisers comprise potassium nitrate and diammonium phosphate.  
L. A. COLES.

**Dry fungicide.** E. MOLZ, Asss. to CHEM. FABR. L. MEYER (U.S.P. 1,685,715, 25.9.28. Appl., 8.9.25. Ger., 24.9.24).—A mixture of mercuric chloride and alkali iodide.  
F. G. CROSSE.

**Insecticide and fungicide.** F. M. ROGERS and C. P. McNEIL, Asss. to STANDARD OIL CO. (U.S.P. 1,679,919, 7.8.28. Appl., 3.5.26).—Paraffin oil, an alkali salt of sulphonated mineral oil ("mahogany soap"), and a copper naphthenate or mineral-oil sulphonate are emulsified in water with addition of a little alcohol, oleic acid, and alkali if desired.  
C. HOLLINS.

**Protection of plants from animal pests.** CHEM. FABR. DR. H. STOLTZENBERG, Asses. of R. JANISCH (G.P. 448,446, 18.4.25).—Furfuraldehyde, solubilised with glue, is sprayed in the form of aqueous solution containing also contact or respiratory poisons.  
C. HOLLINS.

**Amines of hydroaromatic-aliphatic series** (B.P. 297,484).—See III.

## XVII.—SUGARS; STARCHES; GUMS.

[Liming of diffusion juice.] H. CLAASSEN (Z. Ver. deut. Zucker-Ind., 1928, 543—548).—The analytical results from a number of molasses are calculated to a basis of non-sugar constituents. The results indicate a lower lime content and colour when dry-liming is practised.  
F. E. DAY.

## XVIII.—FERMENTATION INDUSTRIES.

**Kilning of malt.** R. SCHLENK (Woch. Brau., 1928, 45, 415—418, 426—430, 436—440, 451—456).—A mathematical treatment of kilning conditions, based on Mollier's diagrammatic representation of the properties of air at varying temperatures and moisture content. From the data given the relations between rate of drying and air and malt temperatures and moisture contents are deduced. Since the evaporation cools the malt its temperature is much below that of the air in the earlier stages. As the moisture in the malt falls below 25% the hygroscopic effect becomes increasingly important. Neither this effect nor that due to varying surface is constant for different malts.  
F. E. DAY.

**Nitrogenous constituents of wort assimilable by yeast.** W. WINDISCH, P. KOLBACH, and E. HENNECKE (Woch. Brau., 1928, 45, 389—393, 399—406, 409—415, 421—426, 431—435).—If wort is diluted till it contains about 2.7% of solids, inoculated with a trace of yeast, and after 48 hrs. at room temperature incubated for 7 hrs. at 30° with aeration, almost all the assimilable nitrogen is removed. When the amino-nitrogen in worts before and after fermentation is determined by the Van Slyke and Sørensen's formol titration methods, the latter gives higher values in both cases. The decrease in amino-nitrogen during fermentation as determined by these methods accounts for about 60% of the nitrogen assimilated by the yeast. Since by adsorption on asbestos-kaolin or by ultrafiltration, as much as 25% of the total but no assimilable nitrogen is removed, the more complex proteins are not assimilable, neither are peptones, since the action of papain or pepsin does not increase the amount of nitrogen assimilated. The action of trypsin, the proteolytic enzyme of malt, and of acid



hydrolysis increases proportionally the amounts of both assimilable and amino-nitrogen, and the maximum action of each agent is shown by both after equal times of digestion. Formol titration in stages indicates that the ratio of polypeptides to amino-acids is about the same before and after fermentation, hence both are assimilable.

F. E. DAY.

**Influence of various yeast-races and temperatures on fermentation and quality of beer.**

R. HORCH and SCHULTEIS (Woch. Brau., 1928, 45, 394—398).

—Similar worts were fermented with three different bottom-fermentation yeasts, two of which flocculated normally, and the other settled in a powdery form ("Staubhefe"). The fermentations were repeated, employing pitching temperatures of 3.5°, 4.5°, and 5.6°, and maximum temperatures of 6.5°, 7.5°, and 9° respectively. The temperature, saccharometer readings, and  $p_H$  values are recorded for each fermentation, and from these and the character of the resulting beers it is concluded that lower temperatures are favourable to the quality of the beer, the best pitching temperature being about 4°, the maximum during fermentation 7—8°. Since a higher degree of fermentation is given by the "Staubhefe" than by the others, it is suggested that the condition and stability of beers might be improved by fermenting as usual with a flocculating yeast and later adding a yeast of higher fermenting power to induce a vigorous secondary fermentation.

F. E. DAY.

**Comparative determinations of hydrogen-ion concentration of worts and beers by the hydrogen and quinhydrone electrodes.**

F. STOCKHAUSEN and E. F. ROTHENBACH (Woch. Brau., 1928, 45, 459—462).

The hydrogen electrodes employed were those of Michaelis and of Lüers and gave results in good agreement on a series of worts and beers. The quinhydrone electrodes of Trénel and of Lüers gave results which differed from them in some cases by as much as  $p_H$  0.23, generally towards the acid side. It is concluded that though the quinhydrone electrode is sufficiently accurate for brewery routine purposes, if due regard is paid to the fact that its readings in different liquids are not strictly comparable, it should not be used with worts or beers if scientifically accurate results are required.

F. E. DAY.

**Formol titration [of beers etc.].**

P. KOLBACH (Woch. Brau., 1928, 45, 446—451).

—In carrying out the formol titration on beers and worts, the phosphates are first removed by precipitation with barium chloride in alkaline solution. The  $p_H$  of the filtered liquid is first adjusted to neutrality against a phosphate buffer by employing neutral-red as indicator in a Lüers acidimeter. After addition of formaldehyde, it is titrated to  $p_H$  9, against a borate buffer, using phenolphthalein as indicator. Precautions are taken to maintain correct concentrations of wort, indicators, and water at the comparison points.

F. E. DAY.

**Volatility in steam of certain oenologic acids.**

P. JAULMES (Ann. Falsif., 1928, 21, 384—390).

—After passing 100 c.c. of water as steam through 10 c.c. of wine, 87% of the benzoic acid and 50% of the salicylic acid present were carried over, the volume of the wine being kept constant. If the wine was benzoated to the

extent of 1% the correction was 0.26 g. (when sulphuric acid had been added and the technique of Blarez followed), and for the addition of 0.5% of salicylic acid the correction was 0.09. The effect of lactic acid produced during "malo-lactic retrogradation" is insignificant. If distillation by Duclaux's method is used, the correction for benzoic acid is 0.151 and for salicylic acid 0.037. The conclusion of Fonzes-Diacon (B., 1928, 686) that salicylic acid should be used as stabiliser for wines is confirmed.

D. G. HEWER.

**Butanol [butyl alcohol] fermentation process.**

G. L. GABRIEL (Ind. Eng. Chem., 1928, 20, 1063—

1067).—A general description of the factory of the Commercial Solvents Corporation of Maryland is given, together with a survey of the development of the butyl alcohol industry as an offshoot from the original acetone production.

A. G. POLLARD.

**Hydrogen sulphide in fermentation carbon dioxide.**

WANDERSCHECK (Woch. Brau., 1928, 45, 441—

446, 463—468).—The hydrogen sulphide liberated by yeast during fermentation is determined by absorption in lead nitrate solution, dissolving the precipitate in aqua regia, and colorimetric determination of the lead as sulphide. A series of worts, containing about 11% of solids, fermented at 25° for 5 days, gave the following results. The amount of hydrogen sulphide varies very widely, even with worts from similar malts; thus unhopped Pilsener worts yielded 7.03—16.52 c.c. and hopped worts 8.37—16.26 c.c. per hectolitre. Highly attenuative yeasts yield more than yeasts of low attenuative power, but the proportions from different worts are constant. No appreciable amount of hydrogen sulphide is produced during yeast autolysis. Though the malt is the chief source of the hydrogen sulphide, none is evolved during germination. Part of the sulphur compounds of malt are precipitated during boiling, hence addition of the deposit produced to a subsequent fermentation raises the amount of hydrogen sulphide, whilst the addition of hops to the boil reduces it. This latter effect is masked if sulphured hops are used. The addition of sodium, magnesium, and calcium sulphates lowers, leaves unchanged, and raises the amounts of sulphide, respectively. The amount is also increased by the addition of free sulphur or of sulphurous acid, also by high temperature and acidity; low temperature and alkalinity have the reverse effect. The amount of yeast added is without influence, but yeasts cultivated at any temperature do not reach their maximum hydrogen sulphide production at another temperature until after several cultivations. Experiments in the brewery give results comparable with those obtained in the laboratory with the same worts, and indicate that (in bottom-fermentation) hydrogen sulphide begins to be evolved during the third day, and that the amount increases up to about the sixth day, or frothing stage, after which it decreases rapidly, but is continued slowly during the after-fermentation. The bearing of the results on brewing practice is fully discussed.

F. E. DAY.

**The Gutzeit test [for arsenic in brewing materials].**

A. D. COMRIE and T. J. WARD (J. Inst. Brew.,

1928, 34, 530—533).—The test may be applied to the

examination of brewing materials with equal accuracy to the Marsh-Berzelius method, and without susceptibility to many disturbing factors possessed by the latter. Since the organic matter present in malt is more or less constant in composition, its destruction prior to the determination of arsenic by the Gutzeit method may be omitted. The arsenic is determined in the untreated malt and the content so obtained corrected by multiplying by the factor 1.3. On the other hand beer, hops, grains, etc., owing to the retarding effect of their constituents on the evolution of the gas, as a preliminary to the Gutzeit test, should be evaporated to dryness with a 20% solution of magnesium nitrate, and the residue ignited until free from carbon. C. RANKEN.

**Hot fermentation of manure.** RUSCHMANN.—See XVI.

#### PATENTS.

**Isolation of nucleic acids from yeast.** EINSIEDLER BRAUHAUS, Asses. of H. HAEHN (G.P. 448,947, 12.10.24).—Pressed yeast is stirred with hot concentrated sodium hydroxide solution, and glacial acetic acid is added until faintly acid ( $p_H$  5–6). The yeast is removed by centrifuging and nucleic acids are precipitated as ferric salts. These are treated with 10% sodium acetate, ferric hydroxide is removed by filtration, and nucleic acids are precipitated from the filtrate by addition of alcohol and acid. C. HOLLINS.

**Production of lipase.** S. YAMAMOTO (U.S.P. 1,687,050, 9.10.28. Appl., 9.6.25. Japan, 16.8.24).—See B.P. 238,507; B., 1925, 890.

**Tartaric acid and salts** (F.P. 624,540).—See VII.

### XIX.—FOODS.

**Composition of sheep's milk.** R. MARTIN (Ann. Falsif., 1928, 21, 390–397).—Analytical figures for a series of morning and evening milks from individual Larzac sheep from Roquefort show that the C.M.S. ("simplified molecular constant") is higher (a mean of 80.5 for 97 milks) than that of cows' milk (with a mean of 73–74), owing particularly to the greater proportion of chlorides present, and the freezing point is correspondingly lower (for cows' milk between  $-0.55^\circ$  and  $-0.56^\circ$  and for sheep's milk between  $-0.58^\circ$  and  $-0.611^\circ$ ). The original acidity of sheep's milk is higher than that of cows' milk, since the proportion of casein is higher. With certain exceptions, the serum of the milk and the blood of a female in milk are distinctly isotonic, and differences of the same order are present in the blood sera of sheep and cattle as in the milk sera. D. G. HEWER.

**Use of starch for characterisation of margarine.** E. VITTOUX (Ann. Falsif., 1928, 21, 420–424).—The starch used (e.g., potato or rice starch) is best incorporated in the margarine in the mixture of fats. In order to determine the amount of starch present, light petroleum is added to the sample of margarine, and the water layer is separated, deprived of all fat, and the invert sugar present determined. The aqueous extract from another sample is made up to known volume, acidified with sulphuric acid, boiled under reflux, neutralised, 60% methyl alcohol is added, and the mixture is cleared, made up to volume, and titrated with Fehling's solution.

The content of invert sugar is deducted from the total, and the difference multiplied by 0.9 gives the amount of starch added. In order to detect starch in butter to which margarine containing 0.2% of starch has been added, the water from a 50 g. sample is separated by centrifuging, deprived of all fat, and, after addition of 60% methyl alcohol, centrifuged for 15 min. The casein holding the starch is thus agglomerated, and, after pouring off the clear liquid, 5 c.c. of 95% alcohol are added to dehydrate the casein and allow it to be completely extracted by ether. It is then suspended in warm water, dissolved by addition of 1 c.c. of ammonia, and, after centrifuging, the starch from the original 50 g. is left at the bottom of the tube mixed with a little casein, which is removed by further washings with ammonia solution. The starch is examined microscopically and the addition of 1% of margarine to butter may thus be detected. D. G. HEWER.

**The formol tests for distinguishing artificial from natural foodstuffs.** H. E. HILL (Chem. Eng. and Min. Rev., 1928, 20, 401–402).—An adaptation of Sörensön's formol titration for proteins in which solutions of foodstuffs and formaldehyde, both carefully neutralised with sodium hydroxide to a defined pink coloration of phenolphthalein, are mixed, the degree of resultant acidity being determined (cf. B., 1927, 456), was used in respect to fruit juices in beverages. Juices of various fruits gave titrations which are of the same order in the different species, and the method appears to be capable of considerable applications. When applied to vinegars the results correlated closely with the nitrogen and phosphorus contents of the samples. C. A. KING.

**Butter fats.** ATKINSON. **Butter fat in margarine.** KNETEMANN.—See XII.

#### PATENTS.

**Neutralisation of acidity or alkalinity of decomposition products [foods] by glutamic acid or its salts.** M. FUJII (U.S.P. 1,680,865, 14.8.28. Appl., 3.8.26. Japan, 7.8.25).—To food products prepared by alkaline treatment of proteins, carbohydrates, etc., glutamic acid, its hydrochloride, or calcium glutamate is added to neutralise free alkali, the sodium glutamate formed being pleasant-tasting and nutritious. C. HOLLINS.

**Manufacture of cattle foodstuffs.** W. ELLIS (B.P. 297,235, 26.10.27).—A mixture of substances insoluble in water or animal saliva, e.g., calcium phosphate, with others which are soluble, e.g., sodium chloride, sugar, or glucose, is compressed into a form suitable for consumption by chewing or licking. F. R. ENNOS.

**Preparation of a foodstuff for feeding animals.** A. EHRENREICH (B.P. 284,339, 27.1.28. Belg., 28.1.27).—The body or cuttings of fish such as *Chondropterygii selachii* or more particularly the plagiostomi such as the shark are disintegrated, and the pulp is desiccated *in vacuo* at  $50$ – $60^\circ$ . F. R. ENNOS.

**Manufacture of feeding cakes.** C. N. KJAERGAARD (B.P. 297,596, 23.11.27).—The organic waste materials, which may contain adhesive substances, are dried at  $55^\circ$  to a water content of not more than 16% and then compressed at 350 atm. H. ROYAL-DAWSON.

Conservation of food etc. (B.P. 297,074).—See III.  
Butter from buttermilk (U.S.P. 1,683,728—9).—See XII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Physical determinations of some iodine preparations used in pharmacy. E. CANALS and P. SUIFFET (J. Pharm. Chim., 1928, [viii], 8, 308—313).—The Tyndall effect, Brownian movement, ionisation factor, electric charge, and behaviour on dialysis and ultrafiltration of the following preparations, each containing 0.005% I, have been examined: iodine in aqueous potassium iodide, a mixture of tincture of iodine and water, iodised water without potassium iodide, iodised tannin, iodine gum, iodine jelly, iodised gelatin, "sol lumière," and "sol gautrelet." With the possible exceptions of iodised water and the tincture of iodine and water mixture, the iodine was not present in the colloidal state. E. H. SHARPLES.

Rapid method for differentiating tartaric and citric acids. L. ROSSI (Annali Chim. Appl., 1928, 18, 366—368).—A drop of the freshly prepared aqueous solution of the acid to be tested is added to 0.1—0.2 g. of solid ammonium metavanadate. On stirring, an orange-red polyvanadate is formed in the case of tartaric acid, whilst with citric acid the liquid becomes yellow. If excess of either tartaric or citric acid solution is heated with 1 c.c. of cold saturated vanadic acid solution, the blue colour of the vanadyl cation appears; this reaction proceeds also in the cold, but more slowly. T. H. POPE.

Antipyrinum coffeinocitricum. G. MIKÓ (Ber. ungar. pharm. Ges., 1926, 2, 88—96, 216—217; Chem. Zentr., 1928, i, 1563).—For the identification of the caffeine by the murexide test, less than 25% of antipyrine must be present. The separation is effected by shaking with 96% alcohol or, better, with amyl alcohol; the residue is washed with ether and oxidised with 3% hydrogen peroxide solution and a few drops of 5*N*-hydrochloric acid. A. A. ELDRIDGE.

Evaluation of theobrominum natriosalicylicum. G. MIKÓ (Ber. ungar. pharm. Ges., 1927, 3, 127—129).—An aqueous solution of the material is treated with a mixture of alcohol (96%) or alcohol and ether with hydrochloric acid, the precipitated theobromine being washed and weighed. For the residue the murexide test is employed. A. A. ELDRIDGE.

Evaluation of ergot of rye. P. LIPTÁK (Ber. ungar. pharm. Ges., 1926, 2, 211—216; 1927, 3, 426—430; Chem. Zentr., 1928, I, 1562—1563).—Determinations were made of the acid value, activity, and alkaloid content. If the acid value is greater than 2 the activity is subnormal. A. A. ELDRIDGE.

Evaluation of ergot of rye. B. GAAL (Ber. ungar. pharm. Ges., 1928, 4, 32—42; Chem. Zentr., 1928, I, 1563).—A precipitate, the origin of which was not discovered, was obtained when the ethereal acid extract of physiologically inactive samples was rendered alkaline. A. A. ELDRIDGE.

Evaluation of guarana. G. and I. MIKÓ (Ber. ungar. pharm. Ges., 1926, 2, 8—13; Chem. Zentr., 1928,

I, 1560—1561).—The caffeine content is determined by finding the smallest quantity of substance which, after extraction with ammonia and chloroform and oxidation of the residue (obtained by evaporation of the filtered solution) with hydrochloric acid and hydrogen peroxide, yields an orange-yellow coloration. A. A. ELDRIDGE.

Fruit of *Schinus mollis*. A. CREMONINI (Annali Chim. Appl., 1928, 18, 361—365).—As was stated by Spica and Arata (Gazzetta, 1884, 14, 199), the ripe fruit of *Schinus mollis* contains no piperine, although various text-books still contain statements to the contrary. The fruit contains also a small proportion of a very bitter compound of unknown composition, and a glucoside, to be investigated further. The ethereal oil contains  $\beta$ -phellandrene, pinene, carvacrol, and possibly limonene and dipentene, but not thymol. The presence of the ferrous ion in considerable amount indicates that the fruit may contain aero-oxydase. T. H. POPE.

Medicinal charcoal. SABALITSCHKA and OEHLKE. Sedimentation analysis. VON HAHN and THÖLCKE.—See I.

### PATENTS.

Manufacture of solid alcoholic solutions of free iodine. K. JUNGSMANN and O. KOLBERT (B.P. 277,953, 7.9.27. Ger., 21.9.26).—"Solid" alcoholic iodine solutions consisting, e.g., of a solution of iodine in alcohol solidified with sodium stearate or palmitate, are stabilised by the presence of iodides (e.g., sodium iodide).

B. FULLMAN.

Production of inactive menthol (the racemate of natural menthol). RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 285,403, 4.4.27. Ger., 15.2.27; cf. B.P. 285,394; B., 1928, 691).—Inactive *neo*- and *neoiso*-menthols, obtained by the process of the prior patent, are isomerised by the action of hydrogen and nickel or by heating the sodium (etc.) salts, or are oxidised to inactive menthone or *isomenthone* which is reduced with hydrogen and nickel. *dl*-Menthol, m.p. 34—36°, is isolated from the products by the methods of B.P. 289,125 (B., 1928, 547). C. HOLLINS.

Preparation of aromatic [hydr]oxyaldehydes [vanillin]. J. D. RIEDEL A.-G. (B.P. 285,451, 20.12.27. Ger., 17.2.27).—Alkali salts of *isoeugenol* are oxidised by nitrobenzene at 150° to vanillin, which, after removal of the aniline formed, is separated as bisulphite compound and distilled. 4-Hydroxy-3-ethoxypropenylbenzene, *isochavibetol*, 4-hydroxy-3-(methoxymethoxy)propenylbenzene, and other mono-ethers of 3:4-dihydroxypropenylbenzene may be similarly oxidised. C. HOLLINS.

Manufacture of urethanes [of nuclear-substituted benzyl alcohols]. I. G. FARBEININD, A.-G. (G.P. 448,694, 25.3.25).—*o*- and *p*-Methylbenzyl alcohols are converted by carbonyl chloride in benzene at 0° in presence of dimethylaniline into the corresponding *chloroformates*, from which by the action of ice and ammonia *o*-methylbenzylurethane, m.p. 86—87°, and *p*-methylbenzylurethane, m.p. 122—123°, are obtained. These are almost tasteless and have much stronger antispasmodic action than benzylurethane, and have none of the toxic effects of side-chain-substituted benzylurethanes. C. HOLLINS.

**Manufacture of terpin [hydrate] from nopinene.** G. AUSTERWEIL (Swiss P. 117,777, 18.12.25).—Nopinene ( $\beta$ -pinene) is treated with an equal weight of 45% sulphuric acid; preferably concentrated acid is added to nopinene and water. After 5–6 hrs. the mixture is poured into ice-water, and terpin hydrate is removed after keeping 10 hrs. C. HOLLINS.

**Manufacture of camphene and derivatives.** L. PEUFAILLIT and G. AUSTERWEIL (F.P. 563,208, 20.5.22).—Pinene or turpentine is heated at ordinary or increased pressure with weak acids, such as abietic acid, colophony, or other resin acids, in presence or absence of an inert solvent (benzene or xylene). An unstable compound is formed which readily splits into camphene and abietic acid. The camphene may be frozen out or converted into bornyl and isobornyl acetates. C. HOLLINS.

**Manufacture of chloroiodo-compounds of the quinoline series.** CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of M. DOHRN and A. THIELE (G.P. 447,538, 2.7.24).—Quinolinecarboxylic acids or esters are stirred with iodine mono- or tri-chloride in acetic acid at 50–70°. Atophan forms a *chloroiodide*, m.p. 237–238°; *chloroiodides* are also obtained from amyl 2-phenylquinoline-4-carboxylate and quinaldine-4-carboxylic acid, m.p. 133–135° and 147–148°, respectively. The compounds lose both halogens when boiled with water. C. HOLLINS.

**Manufacture of *N*-acyl derivatives of 3-amino-4-hydroxybenzene-1-arsenoxide and dichloroarsines.** POULENC FRÈRES, E. FOURNEAU, and J. TREFOUËL (F.P. 606,238, 19.2.25).—*N*-Acyl derivatives of 3-amino-4-hydroxybenzenearsinic acid are converted by sulphur dioxide and iodine in presence of concentrated hydrochloric acid into the corresponding phenyl-dichloroarsines, and these by treatment with ammonium carbonate give the arsenoxides. Formyl, carbethoxy-, and acetyl derivatives are described. C. HOLLINS.

**Manufacture of an organic bismuth-arsenic compound.** C. LEVADITI (F.P. 609,146, 31.10.24).—A 15% aqueous solution of sodium 3-acetamido-4-hydroxybenzenearsinate is added gradually at 80–90° to a solution of sodium or potassium hydrogen tartrate containing 30% Bi. Bismuth 3-acetamido-4-hydroxybenzenearsinate is precipitated. C. HOLLINS.

**Extracting the cardio-active substance of *Bulbus scillæ*.** CHEM. FABR. VORM. SANDOZ (B.P. 287,147, 16.3.28. Ger., 16.3.27).—Fresh squill or its juice is mixed with a readily soluble salt (ammonium or magnesium sulphate) and extracted with an organic solvent immiscible with water (methyl or ethyl acetate) which may contain an aromatic carboxylic acid and/or a small amount of a water-miscible solvent. Dried squill is treated with water and then as above. The active glucoside is recovered by almost complete evaporation of the solution and precipitation with ether. Part of the product is crystallisable, and the rest is freed from tannin-like impurities by treatment with tannin-precipitating agents. B. FULLMAN.

**Manufacture of a difficultly water-soluble glucoside of *Adonis vernalis*.** F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 288,129, 5.1.28. Ger., 1.4.27. Addn. to B.P. 265,941; B., 1927, 797).—The dark-brown oil obtained in the process of the prior patent is treated with water and with ether to remove the easily soluble glucoside etc. The residue is extracted with chloroform, and the chloroform solution shaken out with water, concentrated, and poured with stirring into light petroleum. A difficultly water-soluble glucoside is precipitated, which is purified by dissolution in 50% alcohol and precipitation of impurities from the solution with lead acetate. The solution is treated with sodium phosphate to remove excess of lead, and the alcohol removed *in vacuo*. The residual oil is dissolved in chloroform and the solution added to light petroleum, when the glucoside is obtained as a yellowish powder. B. FULLMAN.

**Treatment of animal glands etc. to increase the activity of the hormones.** G. EICHELBAUM and J. ALTENBURG (B.P. 275,183, 21.6.27. Ger., 28.7.26).—Glands and other animal organs having internal secretions are treated (preferably after comminution or reduction to a dry powder and possibly admixture with, e.g., kieselguhr) with ultra-violet light, with movement or stirring, and with or without the use of a heat filter. The substances and their extracts acquire increased efficiency. [Stat. ref.] B. FULLMAN.

**Preparation of vaccines from bacterial poisons.** I. G. FARBENIND. A.-G., Assees. of R. BIELING and K. JOSEPH (G.P. 447,813, 16.9.24).—The total bacterial poisons, precipitated from the culture in known manner, are subjected to a 30% formaldehyde treatment or a heat treatment or both. This has the advantage of eliminating other poisons, such as toxones, from the specific toxin, and enables a much more rapid preparation of the vaccine to be made. Thus a beef broth containing 1% of peptone is inoculated with diphtheria bacilli. After 7 days 1 c.c. of a 1% dilution of the culture kills a 250 g. guinea-pig in 4 days. The alcohol precipitate of the culture has the same activity at 0.01% dilution. The precipitate in 0.1% solution is treated at 37° with 0.3% of 30% formaldehyde. 1 c.c. of the liquid after 3 days is easily tolerated by guinea-pigs. C. HOLLINS.

**Isolation of alcohols and phenols from mixtures.** A. DEPPE SÖHNE, and O. ZEITSCHEL (G.P. 448,419, 14.12.24. Addn. to G.P. 444,640. Cf. B.P. 252,570; B., 1926, 691).—Further to the B.P., crude Japanese peppermint oil containing 44.5% of free alcohols (as menthol), warmed with boric anhydride at 80–100° under reduced pressure, gives an alcohol, m.p. 15°,  $d_{20}^{25}$  0.903,  $\alpha_D^{20}$  –35.8° in 1 dm. tube. Thyme oil containing 25% of phenols gives a mixture of thymol and carvacrol with 30% of borneol and linalool. C. HOLLINS.

**Manufacture of cosmetic preparations and the like.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 249,130, 16.3.27; cf. B.P. 292,655; B., 1928, 681).—Cosmetic preparations comprising the customary constituents are improved by the inclusion of sorbitol

or its dehydration products or of ether or ester derivatives of either. D. F. TWISS.

**Mono[hydr]oxyphenylalkyl ketone derivatives.** H. LEGERLOTZ (B.P. 297,756, 22.4.27).—See U.S.P. 1,680,055; B., 1928, 857.

**Substituted 4 : 4'-dihydroxybisacylaminoarsenobenzenes [3 : 3'-diacylamino-4 : 4'-dihydroxyarsenobenzenes].** L. BENDA, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,680,845, 14.8.28. Appl., 23.5.27. Ger., 28.6.26).—See B.P. 296,327; B., 1928, 857.

**Unsaturated hydrocarbons (F.P. 583,341). Pharmaceutical preparations (B.P. 297,074). Naphthastyril (B.P. 281,257).**—See III. **Nucleic acids from yeast (G.P. 448,947).**—See XVIII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Sensitising ripening nuclei of gelatin.** A. STEIGMANN (Kolloid-Z., 1928, 46, 57—59).—Observations are described which show that Sheppard's silver sulphide nucleus theory of photographic sensitisation is not sufficiently comprehensive. E. S. HEDGES.

**Tartrates and borotartrates as [photographic] restrainers.** L. LOBEL (Bull. Soc. franc. Phot., 1928, 15, 167—169).—The addition of tartaric acid and borotartaric acid, prepared by admixture of equimolecular quantities of tartaric and boric acids, to a metol-quinol developer caused retrogression of inertia and apparent decrease in sensitivity resembling the effect of addition of alkali bromide. J. W. GLASSETT.

**Measurement of rates of detonation of explosives.** JONES.—See XXII.

### PATENTS.

**Desensitising photographic plates or films.** I. G. FARBENIND. A.-G. (B.P. 292,140, 23.5.28. Ger., 15.6.27).—*iso*Phenosafranines, *e.g.*, 1 : 3-diaminophenylphenazonium chloride or 2 : 3-diaminophenylphenazonium nitrate, at 0.02% concentration, are used for the desensitising of plates and films before development. The dye can be washed out of the gelatin and does not stain the hands. C. HOLLINS.

## XXII.—EXPLOSIVES; MATCHES.

**Photographic study of detonation in solid explosives.** I. Development of a photographic method for measuring rates of detonation. E. JONES (Proc. Roy. Soc., 1928, A, 120, 603—620).—The explosive is fired behind a narrow slit in a steel screen, so that the cartridge is obscured with the exception of a narrow central strip of which the width is adjusted so as to give an image about 0.007 cm. thick. In this way the width of the image can be made as small as desired without limiting any dimension of the cartridge, and, as the explosive can be fired much closer to the lens and camera than in previous methods, there is a corresponding increase in the intensity of an image of similar magnifica-

tion. In a cylindrical cartridge of uniform composition, detonating axially, the detonation front is symmetrical about the axis of the cartridge, so that the progress of detonation along any narrow longitudinal strip of the cartridge correctly reproduces that along the whole surface. The effect of the surrounding medium is considered in detail. It is found that, under certain conditions and in certain directions, the disturbance outside a cartridge of solid explosive detonated in a gaseous medium can have a considerably higher speed than that of the detonation zone in the cartridge itself, and in this case the photographic record of the detonation phenomenon may be distorted. A method for overcoming this effect is described. The contour of the disturbed region surrounding a particular cartridge of explosive has been approximately determined at definite intervals from the moment of initiation of the disturbance. The values for the velocity of detonation obtained by the photographic method are in substantial agreement with those from the Dautriche method. Results are tabulated to illustrate the two velocities at which detonation is capable of propagation in the gelatinous explosives and the change in velocity on storing; also the effect of the nitroglycerin content of a powder explosive on its detonation velocity. A method is given for determining the approximate duration of the detonation phenomenon over a transverse section of a cartridge. The stable form assumed by the detonation front inside a cartridge (7.6 cm. diam.) of a modified Glasgow dynamite has been determined. L. L. BIRCUMSHAW.

### PATENTS.

**Finely-divided nitroguanidine and its manufacture.** A. P. TANBERG and R. L. KRAMER, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,679,752, 7.8.28. Appl., 6.4.22).—Nitroguanidine, free from sulphuric acid and in a form suitable for propellant powders, is obtained by saturating water at 140—160° with recrystallised nitroguanidine and discharging the solution into ice and water. Water dissolves 9.09% of nitroguanidine at 100°, 60% at 146°, 96.5% at 158°. C. HOLLINS.

**Explosives.** IMPERIAL CHEMICAL INDUSTRIES, LTD., and T. J. R. ALEXANDER (B.P. 297,375, 20.6.27).—A mixture of ammonium perchlorate and sodium nitrate is prepared by the double decomposition of concentrated solutions of sodium perchlorate and ammonium nitrate at 140°. If less concentrated solutions are used a lower temperature will suffice. S. BINNING.

**Explosive compositions for blasting fuses.** J. P. NIENHUIS (B.P. 297,365, 17.3.27).—A slow-burning fuse powder contains saltpetre, sulphur, and oil pitch or asphalt pitch, with or without charcoal. A suitable composition is saltpetre 73%, sulphur 15%, and pitch 12%. S. BINNING.

**Cleansing agent for fire-arms.** J. WAGNER (B.P. 296,550, 29.8.27).—A mixture of esters of hydroxy-fatty acids, particularly ethyl lactate, with cleansing agents known to remove acid residues will remove both acid and lead residues. S. BINNING.

**Manufacture of detonators for blasting and industrial purposes and for shells, mines, etc.** NOBEL'S EXPLOSIVES CO., LTD., and G. MORRIS (B.P. 297,853, 1.4.27).—The main charge is loaded in a molten condition into the detonation casing and allowed to set, or it may be loaded as a powder and then melted. The main charge may be a single substance, *e.g.*, erythritol tetranitrate, or a mixture of at least two substances, *e.g.*, tetryl and trinitrotoluene.

S. BINNING.

### XXIII.—SANITATION; WATER PURIFICATION.

**Method of action of silica dust in the lungs.** P. HEFFERNAN and A. T. GREEN (J. Ind. Hygiene, 1928, 10, 272—278).—Pulmonary silicosis is not due to direct injury by sharp insoluble particles, nor to the toxic action of the silica ion, but rather to the colloidal action of hydrated silica formed from minute particles of silica dust. The activity of colloidal silica in the lungs may be prevented by such substances as clay, carbon, shale dust, etc., which form a protective coating on the silica dust and coagulate any colloidal silica formed.

F. R. ENNOS.

**Determination of dust inhaled by workmen.** A. I. BURSTEIN (J. Ind. Hygiene, 1928, 10, 279—291).—The amount of dust drawn into any type of air filter depends on the negative pressure developed at the intake of the filter, and on the direction and position of the intake; it is therefore not necessarily an accurate measure of the amount of dust per unit volume of air in a room. The term "dust coefficient" is adopted to signify the average amount of dust inhaled by a workman during 1 hr. in a workroom. Two types of apparatus for determination of the "dust coefficient" are described: (a) the koniogravimeter, in which air is inspired through the filter by the workman, and (b) consisting of a pump and bellows suitably designed to imitate the process of human respiration.

F. R. ENNOS.

**Treatment of boiler-feed waters of low incrustant content.** S. C. JOHNSON (Ind. Eng. Chem., 1928, 20, 1071—1072).—It has been found that in locomotive practice the use of soda ash and/or sodium aluminate is advantageous even with feed waters of 34—86 p.p.m. of total hardness. Systematic blowing down is also essential.

C. IRWIN.

**Zeolite softening of lime-treated water at Columbus, Ohio, Water Softening and Purification Works.** C. P. HOOVER, V. L. HANSLEY, and C. Q. SHEELY (Ind. Eng. Chem., 1928, 20, 1102—1105).—An investigation was made of the operating costs of zeolite softening in conjunction with lime treatment to remove temporary hardness, in place of the use of soda ash. Two small-scale softeners were used, one charged with greensand and one with a synthetic zeolite, and the following data were obtained. Greensand removed 2500 grains of hardness per cub. ft. between regenerations, the synthetic zeolite 5000 grains. Salt consumption was 0.337 lb. per 1000 grains of hardness. It is more economical to use too little salt than too much. Water losses were 2.4—4.6%. Cost analyses

showed a considerable saving by the use of zeolite, but no account was taken of loss of material by disintegration. The water used contained 70—100 p.p.m. of permanent hardness.

C. IRWIN.

**Road-surfacing materials.** BUTTERFIELD.—See IX.  
**Toxicity of lacquer solvents.** SMYTH and SMYTH, JUN.—See XIII.

#### PATENTS.

**Treatment of garbage etc.** R. P. KINKEL (U.S.P. 1,684,448, 18.9.28. Appl., 21.3.25).—Garbage, after being dried in a current of hot gases with recovery of ammonia, is utilised as a source of heat for the continuance of the process.

C. JEFSON.

**Manufacture of a disinfectant.** KONIGSBERGER ZELLSTOFF-FABR. U. CHEM. WERKE KOHOLY A.-G. (G.P. 448,705, 2.8.24).—The product (26% Cl) from chlorination of sulphite-cellulose liquor is mixed with an equal weight of unchlorinated liquor, and after a short time forms a solution which can be diluted with water. The chlorinated compound may also be compressed with an equal weight of sulphite-cellulose pitch into soluble pastilles.

C. HOLLINS.

**Sterilising water.** O. and R. ADLER (B.P. 292,099, 31.5.28. Czechoslov., 13.6.27. Addn. to B.P. 286,338; B., 1928, 350).—Iron, manganese, and other heavy-metal salts are removed in the prior process by passing the water, before it is treated with material containing carbon, over insoluble material resistant to the action of free or active chlorine and capable of precipitating the salts, *e.g.*, manganese dioxide, ferric oxide, etc., alone or mixed with acid-binding agents, *e.g.*, limestone, chalk, bauxite, etc. The material may also serve to obstruct the passage of the bacteria, or may be mixed with the material used for this purpose.

L. A. COLES.

**Preparation of a scale-removing and -preventing substance for use in steam boilers.** SOC. ANON. SUBOX (B.P. 281,598, 20.5.27. Fr., 4.12.26).—A natural resin, *e.g.*, colophony, is saponified with hot dilute sodium carbonate solution, and hydrochloric acid is added to the cooled solution to precipitate the resin acids in a hydrated state. After excess of water has been removed by pressure, protective colloids, *e.g.*, gum arabic, tragacanth, may be added to the paste-like product together with tannin, dextrin, etc. if desired.

W. G. CAREY.

**Increasing the efficiency of zeolites in treatment of water.** W. C. FOSTER and A. C. GARRETT (U.S.P. 1,683,967, 11.9.28. Appl., 17.1.24).—Water having low alkalinity is treated with an alkaline salt and, before the alkalinity of the water diminishes, is passed through the zeolitic bed.

F. G. CLARKE.

**Filtration of alkaline waters.** R. E. HALL, Assr. to J. M. HOPWOOD (U.S.P. 1,686,558, 9.10.28. Appl., 3.5.24).—See B.P. 291,970; B., 1928, 626.

**Compounds from indene and phenols (B.P. 297,075). Disinfectants (B.P. 297,074). Amines of hydroaromatic-aliphatic series (B.P. 297,484).—See III.**