

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 5, 1927.

I.—GENERAL; PLANT; MACHINERY.

Theory of fine grinding. IV. Air analysis of large quantities of crushed sand. G. MARTIN and W. WATSON. V. Existence and preparation of statistically homogeneous grades of crushed sand. G. MARTIN, E. A. BOWES, E. H. COLEMAN, and T. H. LITTLEWOOD (Trans. Ceram. Soc., 1926, 25, 226—239, 240—257; cf. B., 1926, 903).—IV. Apparatus for elutriating large quantities of crushed sand, with air velocities varying from 2 in. to 20 ft. per sec., is described. The compound interest law connecting particle size and number was confirmed, $\log W/X^3$ plotted against X giving a straight line, where W is the weight of a grade, and X the average arithmetical diameter of particles in a grade. The grading velocities and particle size of various grades of sand were determined. V. By repeated air elutriation, crushed sand can be separated into "homogeneous" grades, in which the average arithmetical diameter of the particles cannot be altered by further fractional elutriation. The shape of the distribution curve obtained by plotting the frequency number of the particles against their diameters also remains unchanged. The average frequency curve obtained by plotting the frequencies against the diameters of the particles in a homogeneous grade is a probability curve. F. SALT.

Particle size and volume of a powder. H. WOLFF (Farben-Ztg., 1927, 32, 1667—1669).—The author corrects the misleading assumption that the particle size of a powder varies inversely as the volume occupied by the powder in close packing. The volume occupied by spheres and cubes of different sizes is mathematically treated from first principles, and it is shown that shape and not size of particle influences the bulk of a closely packed powder, as well as its rate of settling.

S. S. WOOLF.

Technical uses of X-rays. A. ST. JOHN (Ind. Eng. Chem., 1927, 19, 339—342).—Applications of X-rays to industrial problems, such as detection of flaws in engineering materials or of ash in coal, and the determination of atomic structure in carbons, are described.

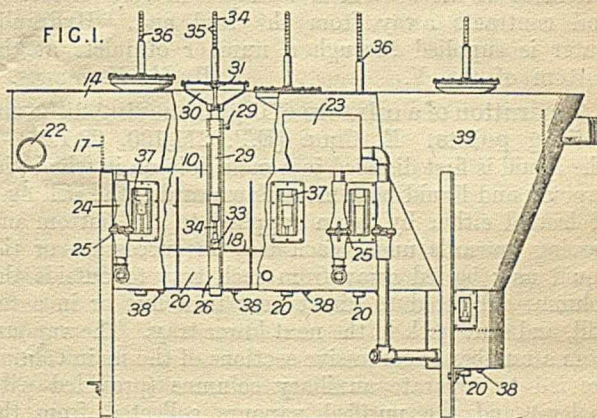
L. M. CLARK.

Colour measurement. T. ORYNG (Physikal. Z., 1927, 28, 298—300).—A review of methods and replies to criticisms. The only unequivocal method of colour measurement depends on troublesome investigations of absorption spectra. Two alternative methods, those of Ostwald and Hübl, are available, but it is concluded that further research is necessary to decide between them.

R. A. MORTON.

PATENTS.

[Fahrenwald] hydraulic classifiers. E. C. R. MARKS. From DORR Co. (E.P. 268,663, 4.10.26).—The aim of this invention is the discharge of the coarse or heavy solids at exactly the same rate as they are collected, so that there is always a bed of settled pulp round the outlet valve—settled, but not compacted, because the upward current or hydraulic water passes



through it. Several classifying compartments may preferably be combined in one structure as shown in the diagram, where the main flow of pulp is from left to right in a longitudinal upper passage and hydraulic water is supplied through the adjacent passage, 23, and pinch-cocks, 25, to chamber, 20 (reverting to the singular), beneath the classifying compartment, 10, the division, 18, between these two compartments being perforated with a number of holes of which the spacing is large compared with their diameter. The discharge valve, 33, admits the settled solids to a pipe, 26, which passes through the hydraulic water chamber. The operation of the valve is controlled by the balance of a longer, clear water column within the pipe, 29, against a shorter pulp column in settling chamber, 10, and the main supply channel. Should the solids accumulate, the pulp column becomes heavier and causes the clear water column to rise; this creates pressure upwards on the flexible diaphragm, 31, of the control chamber, 30, and opens the valve, 33, by means of its long stem, 34, and *vice versa*. The whole control unit from outlet pipe, 26, to valve stem, 34, is quickly detachable, and, to compensate for the fact that, in general, settled pulp of finer material in later compartments is less dense than that of coarser material, the height of the diaphragm, 31, is adjustable by sliding the pipe, 29, and securing

it by a set screw, corresponding adjustments to the length of valve stem being effected by pin, 36, in holes, 35. A bleeder, 38, is provided to take care of stray solid matter passing the perforated plate, 18. The final compartment of the series of classifiers shown includes a large conical settling tank, 39, so that very fine solids may be collected. The upper edge of the nearly stationary settled pulp may be observed through the windows, 37; the stream of thick pulp from outlets, 26, also gives visual indication that the apparatus is working properly.

B. M. VENABLES.

Wet-separation of the constituents of mineral and other pulp. T. G. MARTYN (E.P. 264,128, 30.8.26. Conv., 7.1.26).—The pulp is stratified by upward-current hydraulic water, and as the pulp as a whole flows substantially horizontally the various layers are cut off and collected in turn by partitions depending from the closed top of the apparatus, there being a draw-off pipe at the apex between each partition. The stratifying compartment at the bottom is V-shaped in vertical section and contracts away from the feed end. Hydraulic water is supplied through a number of inlets at the bottom of the V.

B. M. VENABLES.

Separation of a mixture of liquids having different boiling points. E. CHUR (E.P. 271,130, 22.1.26).—The liquid is first distilled in a main column in which the vapours and liquid both move downwards; the still may be heated either by steam supplied to the bottom and passing upwards under each tray in succession, or the liquid may be led away from each tray to outside the column, subjected to direct heat in a flue or in a tar still, and sent back to the next lower tray. The vapours from a number of successive sections of the main column are led to separate auxiliary columns (provided with coolers) and the purified vapours collected from the latter. The liquid from one auxiliary column passes to the next auxiliary column in the same order as the original vapours were taken off the main column.

B. M. VENABLES.

Removal or separation from gaseous fluid of material suspended therein. R. S. PORTHAM, and TANGENTIAL DRYERS, LTD. (E.P. 271,545, 17.2. and 12.11.26).—The dusty gas is caused to impinge on a device having a number of points, *e.g.*, a bundle of pointed rods, through the interstices of which the collected dust can flow away, and is then given a circular motion by means of vanes, and while rotating, caused to impinge on a number of bars, or the circular motion may be produced by scrolls which divide the stream of gas into thin laminae.

B. M. VENABLES.

Manufacture of adsorbents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 271,564, 25.2.26).—Silica or metal oxides, *e.g.*, alumina, are obtained in an active condition from mineral jellies by removing part of the impurities by washing, then drying above 120°, and removing the remaining impurities from the shrunk mass by boiling with acids, and then with water, finally drying above 120°.

W. G. CAREY.

Manufacture of porous artificial [filter] stones. SCHUMACHER'SCHE FABR. G.M.B.H. (E.P. 251,964—5, 24.4.26. Conv., 8.5.25).—(A) A mixture of a synthetic resin with a stony raw material of a definite, substantially

uniform granular size is moulded under pressure and heated at 100–200°, forming a porous stone of a predetermined degree of porosity for use in filtering. The granulated material may be fluorspar for a stone proof against hydrofluoric acid, or coke for a stone proof against caustic solutions. (B) A stony raw material is mixed with organic substances, *e.g.*, a phenolic substance and an aldehyde, which form hard insoluble resinous condensation products when heated, with or without the addition of a catalyst, the mixture being moulded under pressure and heated.

B. W. CLARKE.

Low freezing-point solutions. PYRENE CO., LTD., ASSEES. OF A. Z. PEDERSEN (E.P. 267,561, 14.3.27. Conv., 27.5.26).—A solution suitable for motor-car radiators etc. is made by dissolving 35 pts. by wt. of calcium chloride in water, and then mixing with a solution containing 1 pt. by wt. of sodium chromate and neutralising agent, *e.g.*, chromic acid.

W. G. CAREY.

Refrigerating machine. H. SCHIER (E.P. 271,358, 30.12.26).—In a continuously operating absorption refrigerating system the energy of the cooling water delivered under pressure is used to operate the circulating pump for refrigerant, and pressure-controlling devices are arranged between the high- and low-pressure sides of the system to prevent the difference in pressure from rising so far as to stop the circulation.

B. M. VENABLES.

Absorption refrigerating apparatus. D. W. BERLIN (E.P. 271,634, 18.5.26).—In an absorption refrigerator where one vessel acts alternately as boiler and absorber and another vessel as condenser and then as evaporator or refrigerator proper, the two vessels are rigidly connected by twin conduits which are in close relation to each other, and also lead to and from the nearest points on the two vessels, and are so arranged that the whole liquid contents of the refrigerator can be run back into the boiler on tilting the vessels. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 234,110, 16,293 of 1888, and 3153 of 1877.]

B. M. VENABLES.

Kiln. E. L. JOHNSON (E.P. 271,291, 7.8.26).—A shaft kiln is provided with a hearth for solid fuel situated in a tunnel placed diametrically across the bottom of the kiln. Outlets for products of combustion are formed through the walls of the tunnel in such a manner, *e.g.*, inclined first upwards then downwards, that the material in the kiln cannot enter the combustion chamber.

B. M. VENABLES.

Annular ovens or furnaces with rotating hearths for treating pulverulent, granular, or like materials. TROCKNUNGS-VERSCHWELUNGS- u. VERGASUNGS-G.M.B.H., L. HONIGMANN, and F. BARTLING (E.P. 271,707, 2.10.26. Addn. to E.P. 259,389; B., 1926, 999).—Modifications to the oven described in the original patent are described, comprising means for depositing a uniform layer of charged material, and a jet and collecting hood for discharging the material.

B. M. VENABLES.

Method and apparatus for annealing metals, ceramic and other materials. A. BESTA (E.P. 248,394, 27.2.26. Conv., 28.2.25).—The articles to be heat-treated are placed in interchangeable boxes or

containers and pushed in succession up one side of a tower, across the top, and down the other side. Strong heating is confined to the upper sections of the tower, and may be effected by reheating a portion of the gases circulating round the upper part only while the other portion of the gases is brought down to the bottom of the tower and recirculated. B. M. VENABLES.

Grinding mill. P. HÄNEL (E.P. 271,780, 10.2.27).—A roller grinding mill is provided with four or any greater even number of rolls so placed that any roll co-acts with two others, forming a number of grinding nips equal to the number of rolls. The rolls rotate successively in opposite directions, and at a speed increasing from the first to the last, the material progressing in the same order. Alternate rolls are provided with adjustable bearings, so that the grinding gaps may be varied two at a time. B. M. VENABLES.

Machines for crushing ores, stone, rock, etc. C. ROSCOE (E.P. 271,799, 29.3.27).—A jaw-crusher in which the eccentric shaft and toggle motion is outside the fixed jaw, and the swing jaw at the opposite end of the frame, the motion being transmitted by rods outside the frame. B. M. VENABLES.

Still and the like. W. E. ENGLISH and J. R. HANNAN (E.P. 271,567, 26.2.26).—In heaters for stills containing a number of elements each comprising a steam supply tube with open end within an outlet tube with closed end, each element projects through and is detachably secured to a tube plate, and is provided with a valve on both the supply and drain tubes, and with a test cock outside the tube plate. The renewal of faulty elements while working is facilitated. B. M. VENABLES.

Centrifugal machine. SHARPLES SPECIALTY CO. (E.P. 268,321, 10.3.27. Conv., 26.3.26).—The bowl of a super-centrifugal machine is provided with a ported head and detachable cap; for cleaning, it is only necessary to remove the latter, the former remaining undisturbed so that the very accurate balance of the rotor may be preserved. B. M. VENABLES.

Obtaining and storing gases under pressure. C. W. P. HEYLANDT (E.P. 271,692, 28.8.26).—The liquefied gases having low b.p. are evaporated in a high-pressure tank by a source of heat, and are led as water-free compressed gases into another pressure-resisting tank. The tank in which the liquid is stored and evaporated is coated on the interior and/or exterior with a heat-insulating material to facilitate the introduction and slow evaporation of the liquid. W. G. CAREY.

Fire-extinguishing liquid. K. KUBIERSCHKY and W. SCHULTZE (U.S.P. 1,633,383, 21.6.27. Appl., 16.3.26. Conv., 3.3.25).—The liquid consists of liquid sulphur dioxide mixed with carbon tetrachloride. H. ROYAL-DAWSON.

Treatment of liquids. C. LOURENS, Assr. to N. V. ALGEM. NORIT MAATSCHAPPIJ (U.S.P. 1,634,154, 28.6.27. Appl., 11.11.22. Conv., 14.11.21).—See E.P. 163,505; B., 1921, 456 A.

Centrifugal machines and processes. L. D. JONES and A. U. AYRES, Assrs. to SHARPLES SPECIALTY CO. (U.S.P. 1,634,245—6, 28.6.27. Appl., [A], 13.4.27, [B], 1.5.24).—See E.P. 233,327; B., 1926, 343.

[Ash-cooling screens for] furnaces. BABCOCK & WILCOX, LTD. From DEUT. BABCOCK & WILCOX DAMPFKESSELWERKE A.-G. (E.P. 264,097—8, 24.9.26).

Arrangement for the accurate control of cooling, especially in furnaces. E. ROUCKA (E.P. 265,546, 27.10.26. Conv., 3.2.26).

[Precast concrete blocks for] tunnel linings. S. JOHANNESSEN (E.P. 272,397, 20.12.26. Conv., 12.11.26).

Method and apparatus for spraying. E. G. GRESHAM (E.P. 247,170, 23.1.26).

[Connexions for spouts of] centrifugal separator installations. AKTIEBOLAGET SEPARATOR (E.P. 269,883, 5.4.27. Conv., 24.4.26).

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

Improvement in Eschka's determination of sulphur [in coal]. H. TER MEULEN (Chem. Weekblad, 1927, 24, 205—206).—The sulphur contained in the volatile products, which escapes in the ordinary determination, may be arrested by passing the gases mixed with air over incandescent asbestos and then through potassium hydroxide. The results with this modification are from 3 to 5% higher than with the ordinary method. S. I. LEVY.

Velocity of reaction between carbon dioxide and different species of carbonised fuels. J. A. LEFFLER (Jernkontorets Annaler, 1926, 110, 165—208).—A study of the reaction $\text{CO}_2 + \text{C} = 2\text{CO}$ at 650—1150° with birch, red fir, spruce, and beech charcoals, and at 800—1400° with metallurgical coke.

CHEMICAL ABSTRACTS.

Steaming in continuous vertical retorts: theory and practice. N. H. HUMPHRYS (Gas J., 1927, 178, 370—371, 506—507, 635—637).—Advantages of steaming coal in gas retorts include the resulting diluting and conserving actions, as well as a greater regularity in the quantity and quality of gas produced, and the possibility of working to a closer margin above the declared calorific value. The quality of the coke and tar is improved, as are the working conditions. Comparative figures are given for the actual and calculated yields of therms and total gas (including its individual components) obtained by introducing varying quantities of steam. W. T. K. BRAUNHOLTZ.

Experiments on complete gasification. K. BUNTE (Gas- u. Wasserfach, 1927, 70, 617—621).—A weight balance, a carbon balance, a water balance, and a heat balance have been drawn up for a producer in operation at the Weisenau gas works. In a three days' run, the producer being in operation for two periods of 5 and 6 hrs., respectively, each day, 4.7 tons of coal (moisture 2.4%, ash 5.7%, volatile matter 33.0%) were gasified, producing 5335 cub. m. of gas (0°, 760 mm.), of 3410 kg.-cal./cub. m. mean gross calorific value. The total thermal value of the gas amounted to 49.4% of that of the coal used. Carbonisation of the coal and subsequent conversion of the coke into water-gas, after allowing a proportion for heating the retorts, would give 108.2 cub. m. of "mixed gas" of 3510

kg.-cal./cub. m. The total thermal value of the gas would amount to 48.5% of that of the coal, but by this process there would be obtained in addition 6.6% of tar.

A. B. MANNING.

Record of the gases from coke ovens at Routchenkovo. FEODOROFF (Chim. et Ind., 1927, 17, 729—736).—The installation consists of 4 batteries of 35 ovens each, the normal charge for each being 8 tons of coal and the duration of the coking 36 hrs. The products of distillation of the coal are worked up for the recovery of ammonia, benzol, and tar. About 8800 cub. m. of gas are produced per hour, the yield being 347 cub. m. per ton of coal carbonised in the ovens. The distribution of the gas at various stages of the process is tabulated.

H. M. LANGTON.

Permissible sulphur content of gas oils for the production of [carburetted] water-gas. G. A. BRENDER & BRANDIS (Het Gas, 1926, 46, 430—434; Chem. Zentr., 1927, I, 1096—1097).—Large-scale experiments have shown that, using gas oils containing 0.27—2.82% S, the sulphur content of the purified carburetted water-gas ranged from 16.6 to 27.2 g./100 cub. m., or, after allowing for 12.3 g. from the coke, from 4.3 to 14.9 g./100 cub. m., whilst purified coal gas contains at least 40 g./100 cub. m. The crude gas contained 0.22—0.51% H_2S , or 0.04—0.33% after allowing for 0.18% from the coke itself, as compared with 0.8% in coal gas. The required maximum sulphur content for gas oils of 0.5% is, therefore, without justification, for even with oils containing several times that amount the increase in the hydrogen sulphide of the crude gas, and in the sulphur compounds, particularly carbon disulphide, of the purified gas, are inconsiderable compared with the amounts present in coal gas.

A. B. MANNING.

Hydrocarbons in gas from the low-temperature carbonisation of coal. F. E. FREY and W. P. YANT (Ind. Eng. Chem., 1927, 19, 488—492).—The gas from the carbonisation of a non-coking Utah coal in a current of superheated steam at 700° has been analysed by fractional distillation (cf. B., 1927, 177), and a further study made of the hydrocarbon fractions. Acetylene and its homologues were absent. From the bromine addition products of the unsaturated constituents in the four-carbon-atom fraction *trans*- $\alpha\beta\gamma\delta$ -tetrabromobutane was isolated in amount equivalent to 0.002% of butadiene in the gas. Analysis of the dibromides showed that the butylene fraction (0.52% of the gas) consisted of 41% of *as*-dimethylethylene, 24% of ethylethylene, and 35% of *s*-dimethylethylene. The butane fraction (0.37% of the gas) consisted of 20% of isobutane and 80% of *n*-butane. Methylcyclopropane was absent.

A. B. MANNING.

Separation of individual saturated and unsaturated hydrocarbons in coal gas by fractional distillation. F. E. FREY and W. P. YANT (Ind. Eng. Chem., 1927, 19, 492—493; cf. preceding abstract).—A modification of the Shepherd and Porter method of gas analysis by fractional distillation at low temperatures is described, which permits the separation of the constituents into fractions containing (1) oxygen, nitrogen, hydrogen, carbon monoxide, and methane, (2) ethane

and ethylene, (3) propane and propylene, and (4) butane and butylene, respectively. Each fraction can then be analysed in a simple Orsat apparatus. The method can be applied to 50—2000 c.c. or more of gas, according to its content of these hydrocarbons. An analysis requires 5—6 hrs.

A. B. MANNING.

Formation of carbides in the system metal-carbon-hydrogen and metal-carbon-oxygen. G. MEYER and F. E. C. SCHEFFER (Rec. trav. chim., 1927, 46, 359—368).—Metal carbides rich in carbon, stable at low temperatures, and decomposed by heat, are probably formed during the production of synthetic petrol from water-gas in the presence of a metallic catalyst (Fischer and Tropsch, B., 1926, 475), and a nickel carbide of this character has, in fact, been isolated (Scheffer, Dokkum, and Al, A., 1927, 29; Meyer and Scheffer, *ibid.*, 220). The general principles involved in the study of the systems metal-carbon-hydrogen and metal-carbon-oxygen from the point of view of the phase rule are discussed.

M. S. BURR.

Combustion limits under high pressure of mixtures of air with inflammable gases and vapours. E. BERL and G. WERNER (Z. angew. Chem., 1927, 40, 245—250).—The curve showing the lower limit of complete combustion of hydrogen-air mixtures starts at 7.0% of hydrogen at atmospheric pressure, rises steeply to 10.8% at 21 atm., then falls slowly to 8.4% at 210 atm., whereas the curve of the upper limit rises smoothly, concave to the pressure axis, from 60% of hydrogen at 1 atm. to 73.5% at 210 atm. with a weak ignition, but, with strong ignition, falls from 71.5% at 1 atm. to 69% at 10 atm., then slowly rises to 74% at 210 atm. The upper limit of complete combustion of hydrogen-air mixtures under reduced pressure falls fairly smoothly with decrease of pressure to 20% of hydrogen at 240 mm., whereas the lower limit remains constant down to 400 mm., then increases sharply to 20% at 240 mm., consequently air-hydrogen mixtures of any composition do not react with a weak ignition at pressures lower than 240 mm. The lower limit curves for ethylene-air and methane-air mixtures resemble in shape the corresponding hydrogen-air curve, both showing a high lower limit at 20 atm. The upper limit curve of ethylene-air mixtures starts at 16% of ethylene at 1 atm., and rises extraordinarily steeply to 68% at 90 atm., then very slowly to 71% at 380 atm. The combustion range of methane-air mixtures decreases from 6.6—12.7% of methane at 1 atm. to a minimum of 7.5—12% at 21 atm., then rapidly increases to 5.2—46% at 400 atm. Ignition of mixtures of ethylene or methane with insufficient air for complete combustion under just sufficient pressure for reaction to take place results in the preferential oxidation of the hydrogen, but with further increase in the pressure the proportion of carbon dioxide and especially of carbon monoxide formed increases rapidly; thus, of the oxygen in a 40% ethylene-air mixture, 83% appears as water, 3% as carbon dioxide, and 14% as carbon monoxide after ignition under 40 atm. pressure, whereas under 92 atm. these figures are 50, 10, and 40% respectively. Air saturated with ether vapour at 20° is outside the combustion zone at 1 atm., but

at 4 atm. the mixture commences to burn with a weak ignition, the rate of reaction reaching explosive violence at 19 atm., and finally falling to zero again at 30 atm. Similar results are obtained with benzene-air mixtures, and also by raising the temperature while keeping the pressure constant, there being an upper and lower limit of temperature for all pressures within which range only does combustion take place. A. R. POWELL.

Conradson coke-test for motor oils. F. SCHULZ and M. KOHOUT (Petroleum, 23, 554—557).—The Conradson coke-test as carried out by A.S.T.M. methods shows considerable experimental errors; these can be diminished if 2 g. of the oil are weighed into a porcelain crucible (Rosenthal 103, No. 4) and put into the cold Conradson apparatus, which is then heated for 20 min. with the Tirrill burner (height of flame 20 cm.), the bottom of the iron crucible being 5 cm. above the mouth of the burner. Results of such determinations are given.

W. N. HOYTE.

Effects of the corona discharge on petroleum. J. J. JAKOSKY (U.S. Bur. Mines, Tech. Paper, 1926, No. 375, 21 pp).—Treatment of cracked gasoline (approximately 36% unsaturated) with hydrogen activated by the corona discharge gave no perceptible effect. Natural gas on being submitted to the discharge at 20° and 100° slowly dissociated into carbon black and hydrogen; no polymerisation was noted; at 500° a very small amount of tarry matter, possibly due solely to the heat, was obtained. On submitting benzene vapour to the discharge a light, slightly viscous, brown liquid was slowly produced; the odour was similar to that of pinol. In experiments on vapour-phase cracking below 600° the effects of a high-frequency corona discharge and a low-frequency 60-cycle discharge were inappreciable. Above 600° the greatest cracking was obtained with the high-frequency discharge, and the least with no discharge. The unsaturation of the oil was least with no discharge, and greatest with the high frequency; thus at 800°, 54% unsaturation was observed with the high-frequency, 34% with the low-frequency; and 31% with no discharge. In general, more uncondensable gas was obtained when the high-frequency discharge was used. Tests on the precipitation of hot vapours by a corona discharge showed that below a certain critical temperature most of the vapour was precipitated on the walls of the tube; above this temperature no precipitation took place. The critical temperature coincided with the change in the vapour from a visible cloudiness to an invisible gas. The corona discharge itself produced no cracking of oil vapour. The increased cracking obtained by the use of the corona during the thermal cracking of oils appears to be due to precipitation of the hot oil vapours against the walls of the cracking tube, and to the decreased amount of channelling by the electric wind set up by the discharge.

W. N. HOYTE.

Regeneration of used lubricating and transformer oils. R. A. WISCHIN (Petroleum, 1927, 23, 546—551).—The degradation of the oil is due to the atmospheric oxidation of the less stable parts of the oil, lactones or acids with carbonyl groups in particular being formed. Soaps of these acids are formed with

alkali from the air or from entrained boiler-feed water. These soaps act as catalysts in furthering the oxidation, as also do the asphalts formed as oxidation products; the action thus takes place with increasing velocity. For oil regeneration the Benmann floridin process is recommended. The best working temperature is 60—90°, and the floridin must be granular. If necessary, the oil is subjected to a preliminary treatment (filtration or precipitation with sulphuric acid etc.) to remove metallic particles and carbon sludge. About 1 hr.'s contact is the optimum for all oils, and the size of granules should be chosen accordingly. The regenerated oil has a slight acidity due to lactones which are adsorbed with difficulty by floridin, but are not harmful in lubricating oil. W. N. HOYTE.

Helium in Canada. ELWORTHY.—See VII.

Use of alcohol in arson. MAULHARDT.—See XX.

PATENTS.

Apparatus for separating coal from dirt and like foreign substances. W. H. BERRISFORD (E.P. 271,633, 10.5.26).—The jigger carries a step on to which material to be separated is pushed from a stationary step by virtue of the reciprocating motion of the jigger. As the jigger recedes below the stationary step, the material is pushed over the front edge of the movable step. Between these two steps may be situated an inclined plate, which divides the coal into two streams—clean coal and dirt—dust being removed by an air-current.

R. A. A. TAYLOR.

Treatment of coal. INTERNAT. COMBUSTION ENGINEERING CORP., Assees. of W. RUNGE and E. A. PACKARD (E.P. 264,169, 4.2.26. Conv., 12.3.25).—The coal is first pulverised in a mill in the presence of hot air or flue gases at about 100°, and is further heated by admixture with preheated air or flue gases in its passage to a separator. The degree of heating can be controlled and the coal either oxidised or carbonised as required. The separated material then falls into a retort which has at the bottom a coke-cooling chamber. The gases from the retort are used in the preheaters and may be admixed with air from the separator.

A. C. MONKHOUSE.

Apparatus for drying and distilling lignite, peat, non-coking coals, and other similar carbonaceous matter. H. DEBAUCHE (E.P. 270,921, 20.7.26).—The fuel is carbonised in a cylindrical firebrick retort which has in its centre a multi-ring cylinder consisting of a number of annular tapered collars. A variation in the thickness of the charge is obtained by varying the diameter of the collars, the carbonising space being 4 in. at the top of the retort and 2 in. wide at the bottom, according to the nature of the fuel. The distillation gases pass between the collars and are withdrawn by adjustable vertical pipes, arranged at different levels, to a common collecting main at the base of the retort. To prevent ingress of air, a suitable feeding hopper is used, and the semi-coke produced is withdrawn through a slide valve into a cooling box cooled by circulating water. After screening, the coke above 5 mm. is ready for sale and that below 5 mm. is further treated and briquetted.

A. C. MONKHOUSE.

Transforming lignite and similar carbonaceous matter into fuel of high calorific value for industrial and domestic purposes. H. DEBAUCHE (E.P. 270,976, 29.11.26).—Semi-coke produced by methods described in, *e.g.*, E.P. 270,921 (cf. preceding abstract), is screened as it issues from the retort, the coke below 5 mm. size being treated in an enclosed system filled with an inert gas to prevent oxidation of the coke and to conserve the sensible heat for the subsequent manufacture of briquettes. The coke at 180° is elevated by an enclosed conveyor and fed on to a screen of mesh dependent on the grading required for the briquettes; the larger material passes to a crusher, and is then fed back to the screen. The coke is then fed by conveyor to a tower from which it is discharged to a worm conveyor, the opposite end of which is being fed with pitch from an adjacent tower. The proportions are controllable and the mixture is conveyed to the briquetting plant. In experiments with lignite, the semi-coke above 8 mm. was used for domestic grates, 5½–8 mm. for boilers. The coke, 0–5 mm., gave a cohesion of 25% less than that of 0–1 mm., was 6% less dense, absorbed 4½ times more water, and required 7.5% less pitch.

A. C. MONKHOUSE.

Manufacture of motor fuel. R. CROSS, Assr. to GASOLINE PRODUCTS CO., INC. (U.S.P. 1,631,401, 7.6.27. Appl., 12.9.25).—An anti-knock motor fuel is produced by extracting petroleum oil with liquid sulphur dioxide, separating the undissolved hydrocarbons, and cracking them.

C. O. HARVEY.

Manufacture of gas suitable for heating and lighting purposes. S. J. B. TULLY and O. E. YEO (E.P. 271,765, 31.12.26).—The plant comprises a gas producer, a superposed retort, and a carburettor. Surrounding the retort is a chequer brickwork heating-jacket in communication with the producer and the carburettor, or the latter may constitute an independent portion of the heating jacket. The water-gas made is passed up through the retort, where it is enriched by coal gas, the mixture being led off independently of the carburettor; or the water-gas is passed through the heating jacket and then enriched by cracked oil vapours in the carburettor, whence it may pass to a gas-fixing or gas-treatment chamber.

R. A. A. TAYLOR.

Water-gas [from powdered fuel]. I. G. FARBENIND. A.-G., Assees. of F. WINKLER (G.P. 437,970, 28.9.22).—The air and the steam are blown into the producer in such a way that the whole of the charge of powdered fuel is given an up-and-down rotary motion.

A. B. MANNING.

Recovering ammonia from gases. R. O. E. DAVIS and L. B. OLMSTEAD (U.S.P. 1,631,720, 7.6.27. Appl., 13.6.21).—The ammonia is absorbed in a liquid cooled to about 0°, and is subsequently recovered by raising the temperature of the solution to 40–50°.

C. O. HARVEY.

Distillation of solid carbonaceous material. M. J. TRUMBLE (E.P. 271,740, 12.11.26).—The material is charged into a series of retorts, into the first of which superheated steam is introduced. The steam, now mixed with distillation products, is passed through the second retort, a part of the condensable distillation products

being removed, and so on throughout the whole series. The steam and remaining distillation products are then passed through a superheater which restores the mixture to the initial temperature of the steam. The resulting hot mixture is then passed through a second series of retorts as through the first. The portion of the distillation products removed may be the heavier oils by condensation or other fractions by dephlegmation.

R. A. A. TAYLOR.

Distillation of oil shale. C. T. LOUGHREY (U.S.P. 1,631,637, 7.6.27. Appl., 5.5.21).—The shale is distilled by passage down a column up which a heating medium is passed, the vapours being withdrawn by the partial vacuum produced by injecting a liquid under pressure into the withdrawn vapours. The liquid undergoes re-circulation and injection.

C. O. HARVEY.

Preparation of light hydrocarbons from heavy hydrocarbons or cyclic derivatives. A. J. KLING and J. M. F. D. FLORENTIN (F.P. 607,155, 30.11.25. Conv., 12.6.25).—Heavy hydrocarbons are heated with hydrogen under pressure in the presence of a catalyst, *e.g.*, halogen compounds of magnesium, zinc, iron, aluminium, and chromium.

A. B. MANNING.

Conversion of methane gas into hydrocarbons of higher carbon content. PÉTROLE SYNTHÉTIQUE SOC. ANON., and A. FOLLIET (E.P. 271,767, 1.1.27. Addn. to E.P. 261,267; B., 1927, 100).—The prior process is modified by imposing upon the heated gases a vacuum of from 20 to 50 cm. of mercury. The walls of the reaction chamber are of steel lined with refractory material.

C. O. HARVEY.

Treatment of mineral oils by heat to obtain lower-boiling oils. W. J. PERELIS (E.P. 271,601, 24.3.26).—In a cracking process, wherein the production of gas and coke is reduced and economy of heat is effected by means of interchangers, and which is suitable for the treatment of all types of oil (crudes, gas oils, kerosenes, etc.), the oil passes through a series of heating coils and is alternately heated at a cracking temperature, cooled during continued flow, and then reheated. The heaters are arranged for repeated reversals of the direction of flow.

C. O. HARVEY.

Sweetening hydrocarbon oils. M. G. PAULUS, Assr. to STANDARD OIL CO. (U.S.P. 1,628,423, 10.5.27. Appl., 28.5.25).—Hydrogen sulphide, contained in still gases, is dissolved in sour light hydrocarbon oils which are then treated with doctor (sodium plumbite) solution.

C. O. HARVEY.

Preparation of emulsifying agents etc. I. G. FARBENIND. A.-G., Assees. of W. PUNGS and M. LUTHER (G.P. 438,180, 9.9.22).—The oxidation products of solid paraffins are treated with an alkali hydroxide in amount not more than necessary to saponify the saponifiable constituents. Hydrocarbons, or other liquids insoluble in water, and containing, if necessary, sulphur or other materials in solution, are added before and after saponification. In addition to its use as an emulsifying agent, *e.g.*, for drilling oils etc., the product may be used directly as a lubricating oil.

A. B. MANNING.

Extraction of paraffin wax from brown coal. A. MAILHE (Addn. No. 30,634, 5.2.25, to F.P. 598,502;

B., 1926, 574).—The finely-powdered brown coal is treated with the vapours of organic solvents, and the extract so obtained with liquids in which paraffin wax is soluble, *e.g.*, the higher homologues of acetone.

A. B. MANNING.

Treatment [purification] of sludge acid. F. M. ROGERS, F. V. GRIMM, and G. L. WENDT, ASSRS. to STANDARD OIL Co. (U.S.P. 1,630,074, 24.5.27. Appl., 14.8.24).—Waste sulphuric acid from oil-refining is diluted to *d* 1.38, and passed through a tank at 65°, in which it is subjected to electrolysis by a current of 3–3.5 amp./dm.² to oxidise organic matter present.

T. S. WHEELER.

Rustproofing oil. R. E. WILKIN, ASSR. to STANDARD OIL Co. (U.S.P. 1,630,101, 24.5.27. Appl., 13.3.25).—An oil for protecting ferrous metals against rust comprises a hydrocarbon oil heavier than gas oil, containing from 3–35% of sodium salts of sulphonic acids obtained in refining petroleum.

T. S. WHEELER.

Coating gasometers. P. LECHLER Co., ASSEES. of R. MEZGER (U.S.P. 1,630,202, 24.5.27. Appl., 10.7.25. Conv., 11.9.22).—A solution of petroleum pitch in mineral oil is used to cover the surface of the water sealing a gasometer, so that with the movement of the bell, it coats the latter and protects it from corrosion.

T. S. WHEELER.

Carbonisation of fuel briquettes. E. B. A. ZWOYER, ASSR. to GENERAL FUEL BRIQUETTE CORP. (U.S.P. 1,632,052, 14.6.27. Appl., 9.2.23).—See E.P. 211,101; B., 1924, 858.

Method and apparatus for centrifugally separating substances [*e.g.*, wax from oils]. L. D. JONES, ASSR. to SHARPLES SPECIALTY Co. (U.S.P. 1,634,242–4, 28.6.27. Appl. [A], 9.4.27, [B, C], 7.10.21).—See E.P. 191,750; B., 1923, 703 A.

Emulsified solid grease. M. J. HEITMANN (U.S.P. 1,632,027, 14.6.27. Appl., 8.4.25. Conv., 10.4.24).—See E.P. 232,259; B., 1926, 478.

Production of petrol. F. TINKER (U.S.P. 1,632,011, 14.6.27. Appl., 25.9.22. Conv., 7.1.22).—See E.P. 195,465; B., 1923, 541 A.

Burner for use with liquid fuel, gas, etc. J. DAVIES (E.P. 271,575, 27.2.26).

Device [grate] for burning solid fuels. J. MARTIN (E.P. 265,191, 24.1.27. Conv., 29.1.26).

Coke-cooling plant. C. SCHWARTZ (E.P. 256,919, 5.1.26. Conv., 12.8.25. Addn. to E.P. 252,118; B., 1926, 780).

[Valves for] condensers for use in the recovery of by-products arising in the carbonisation of coal and the like. D. M. HENSHAW (E.P. 272,293, 11.3.26).

Cooling of coke and utilisation of the heat arising therefrom. COLLIN & Co., and J. SCHÄFER (E.P. 270,780, 14.1.26).

Apparatus for generating oil gas. A. E. HODGSON and N. C. JONES (E.P. 271,649, 31.5.26).

Handling coke discharged from coke ovens. N. V. SILICA EN OVENBOUW MIJ, and O. FRÖHLICH (E.P. 272,410, 19.1.27).

Carbonising plant (G.P. 436,515).—See III.

Activated charcoal (E.P. 257,917).—See VII.

III.—TAR AND TAR PRODUCTS.

Cresol-soap systems. JENČIČ.—See XII.

PATENTS.

Carbonising plant and generator for the production of low-temperature tar. C. ERHARD (G.P. 436,515, 13.12.24).—The gases and vapours evolved from a retort or producer pass through conduits, which are provided with blowers, into a dryer arranged vertically above the producer. In the dryer they pass through a heating arrangement which is supplied with a mixture of gas and steam from an external heater. The gases, steam, and other volatile products from the carbonisation of the fuel are withdrawn through a tar separator by means of a high-pressure blower, and compressed into a pressure chamber in which the steam condenses, whilst the gases pass to a holder. The gas and steam mixture supplied to both producer and dryer are taken from the circuit at a point between the tar separator and the high-pressure blower, and are heated in a special apparatus to such a temperature that the mixture of gases and vapours leave the dryer at 110°, the most favourable temperature for tar removal.

A. B. MANNING.

Separation of liquids (E.P. 271,130).—See I.

IV.—DYESTUFFS AND INTERMEDIATES.

Behaviour of oxides of lead towards dyestuffs.

II. A. N. ADAMSON and J. K. WOOD (J. Soc. Dyers and Col., 1927, 43, 192–194; cf. B., 1927, 324).—Hydrated lead monoxide shows no tendency to absorb basic dyes, neither dyeing of the oxide nor formation of precipitate taking place with Methylene Blue, Safranine T, Methyl Violet 10B, or Toluidine Blue. With acid dyes, such as Disulphine Green T and Patent Blue, the oxide is uniformly dyed without formation of precipitate, the absorption curves obtained in these cases by plotting the amount of dye remaining in solution against the amount taken up by the solid having more or less resemblance to ordinary adsorption isotherms. With Patent Blue it was found that a sample of oxide (moisture content 6.12%) prepared by precipitation with the minimum of alkali always adsorbed more dyestuff than a sample (moisture content 4.62%) obtained by precipitation with large excess of sodium hydroxide. Whilst the difference in adsorption powers may be partly due to the difference in moisture content, it may also be due to the presence of adsorbed alkali in the second sample, which would consequently be more electro-negative than the first sample. With other acid dyes, *e.g.*, Methyl Orange, Orange II, Bordeaux B, and Orange IV, less regular results were obtained, dyeing of the oxide being accompanied by formation of a considerable amount of flocculent precipitate above the layer of hydrated oxide. Bordeaux B was removed in approximately constant amount independent of the concentration, and Orange IV was completely removed from dilute solutions by the sample of oxide having moisture content 6.12%. It is probable that absorption is accompanied by more or less chemical combination, and the compound obtained (probably the lead salt of the dye acid) itself absorbs a further amount of the dyestuff. The absorption curves in these four cases represent a resultant of the various processes.

R. BRIGHTMAN.

PATENTS.

Dyes and dyeing. B. WYLLAM, J. E. G. HARRIS, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 271,533, 21.1.26).—Sulphuric esters of leuco-compounds are obtained by adding the esterifying agent directly to the hyposulphite vat together with additional sodium hydroxide and a tertiary amine (pyridine). The esterifying agents used are alkyl chlorosulphonates, chlorosulphonic acid, oleum, sulphur trioxide, or the reaction product of any of these with pyridine. C. HOLLINS.

Dyes and dyeing. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 271,537, 26.1.26).—Flavanthrone and its derivatives (e.g., dichloroflavanthrone) are reduced to leuco-compounds in cold concentrated sulphuric acid by treatment with a metal (copper). The leuco-compounds as prepared are stable, and may be used for conversion into leuco-esters by the processes of prior patents, and for dyeing and printing. C. HOLLINS.

Process of reducing vat dyes to their leuco-derivatives. C. H. MARSHALK (E.P. 271,569, 26.2.26).—The dry vat dye is dissolved or suspended in an anhydrous organic solvent (particularly dimethylaniline) and is reduced with hydrogen in the presence of a suitable catalyst (e.g., nickel, cobalt, iron, or copper on a pumice or kieselguhr carrier). The leuco-compound produced dissolves in the dimethylaniline, and is ready for conversion into a leuco-ester ("Indigo-sol"). C. HOLLINS.

Preparation of condensation products of the benzanthrone series. I. G. FARBENIND. A.-G., Assees. of H. NERESHEIMER (G.P. 438,467, 26.10.24. Addn. to G.P. 407,838).—Purer products and better yields are obtained by using for the condensation of benzanthrone a diluent such as benzene, *o*-dichlorobenzene, etc. in the presence of a mixture of alkali hydroxide and alkali alkoxide as condensing agent. The reaction proceeds at low temperature, e.g., 20–25° or 60°. All oxygen should be displaced by means of an inert gas. C. HOLLINS.

Manufacture of α -aroyl- β -naphthols and of condensation products of the benzanthrone series. I. G. FARBENIND. A.-G., Asses. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 248,791, 9.3.26. Conv., 9.3.25).— β -Naphthyl esters of aromatic carboxylic acids are converted by acid condensing agents (aluminium chloride) in the presence of a diluent into α -aroyl- β -naphthols, which may be further condensed to give hydroxybenzanthrone or dihydroxydibenzpyrenequinones. In the absence of a diluent, sodium aluminium chloride brings about both reactions in one operation. 2 : 6-Dihydroxynaphthalene dibenzoate, m.p. 217°, heated with aluminium chloride in tetrachloroethane, yields 2 : 6-dihydroxy-1 : 5-dibenzoylnaphthalene, m.p. 275°, from which, by heating with sodium aluminium chloride at 140–150°, 1 : 6-dihydroxy-3 : 4 : 8 : 9-dibenzopyrene-5 : 10-quinone, together with 2 : 6-dihydroxybenzanthrone, m.p. 250–252°, are obtained. β -Naphthyl benzoates similarly yields 1-benzoyl-2-naphthol (phenyl 2-hydroxy-1-naphthyl ketone), m.p. 137–139°, and 4-hydroxybenzanthrone, m.p. 176°. C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series] containing nitrogen. I. G. FARBENIND. A.-G.,

Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 254,742, 2.7.26. Conv., 2.7.25).—3-Bromo- or -chloro-benzantrones are nitrated (probably in the 8-position), the products are reduced, and then fused with alkali, preferably with the addition of alcohol, to give vat dyes which give reddish-grey to black shades, becoming green when chlorinated. Dyes are thus obtained from 3-bromo-8(?)-aminoanthraquinone, m.p. 274–278°, prepared from 3-bromonitroanthraquinone, m.p. 290°, and 3-chloro-8(?)-aminoanthraquinone, m.p. 290–291°, from 3-chloronitroanthraquinone, m.p. 284–285°. C. HOLLINS.

Manufacture of halogenated indantrones. BRIT. DYESTUFFS CORP., LTD., W. D. ROGERS, W. V. STUBBINGS, and F. W. EMERSON (E.P. 271,181, 1.3.26).—The halogenation of indanthrone purified by the sulphuric acid method of E.P. 9269 of 1914 (B., 1915, 900) yields products fast to chlorine. The degree of halogenation necessary is much lower than when a purified indanthrone is used. C. HOLLINS.

Manufacture of carbocyclic or heterocyclic compounds. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 251,997, 5.5.26. Conv., 5.5.25).—Fluorosulphonic acid is used for ring-closure in the manufacture of carbocyclic and heterocyclic compounds. Improved yields are claimed, due to smaller tendency to sulphonation. Examples of the cyclisation of thioglycolic acids to thioindoxyls and thioindigos (fluorosulphonic acid being also an oxidising agent), and of 4-amino-1-*o*-carboxyanilinoanthraquinone to the acridone described in E.P. 25,184 of 1910 (B., 1911, 1111) are given. C. HOLLINS.

Triarylmethane dyes from tetra-alkyldiaminobenzophenone and arylated ethylenediamines. **Triarylmethane dyes from tetra-alkyldiaminobenzophenones and di- α -naphthylethylenediamine.** I. G. FARBENIND. A.-G., Assees. of H. POLIKIER and H. HÄHLE (U.S.P. 1,629,884–5, 24.5.27. Appl., [A, B], 6.5.26. Conv., [A], 14.3.25; [B], 9.4.25).—(A) *s*-Diphenylethylenediamines non-substituted in the 4 : 4'-positions, and (B) *s*-di- α -naphthylethylenediamine, are condensed with (A) 1 or (B) 2 mols. of a 4 : 4'-tetra-alkyldiaminobenzophenone, e.g., 4 : 4'-tetramethyldiaminobenzophenone, by heating with phosphoryl chloride in presence of toluene at 100°, to yield dyes, which give strong, clear, fast, violet shades on tannin-mordanted cotton and wool. T. S. WHEELER.

Manufacture of anthraquinone derivatives. BRITISH DYESTUFFS CORP., LTD., and A. SHEPHERDSON (E.P. 271,602, 27.3.26).—In the condensation of unsulphonated arylamines with unsulphonated halogenoanthraquinones the large excess of arylamine usually employed as a diluent is replaced by sodium acetate crystals or sodium acetate and water. The quantity of arylamine may be reduced to about 1.5 mols. instead of 6–10 mols. C. HOLLINS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G., Assees. of A.-G. FÜR ANILIN-FABR. (E.P. 253,488, 16.4.26. Conv., 9.6.25).—Diazo compounds are coupled with a variety of azines obtained by alkaline fusion of naphthazenes and dinaphthazenes containing a sulphonic group in position 8 in the naphthalene ring and also

containing other hydroxyl or sulphonic groups. The azines have affinity for cotton and may be coupled on the fibre. The alkaline fusion product of the azine prepared by condensing 1:2-naphthaquinone-4:8-disulphonic acid or 4-hydroxy-1:2-naphthaquinone-8-sulphonic acid with *o*-phenylenediamine, is coupled with diazotised sulphanilic acid for a yellow wool dye; with diazotised naphthionic acid for a red wool dye; with diazotised 4-aminoazobenzene-4'-sulphonic acid for a red wool or direct dye; with diazotised *p*-toluidine-*m*-sulphonic acid for lake browns; with diazotised 4-chloro-2-amino-phenol-5-sulphonic acid or 6-nitro-2-aminophenol-4-sulphonic acid for a chrome violet-brown; on the fibre with diazotised *p*-nitroaniline (yellow) or tetrazotised dianisidine (violet-brown). Other azines used are obtained by alkaline fusion of the condensation products of 4-hydroxy-1:2-naphthaquinone-8-sulphonic acid with 1:2-naphthylenediamine, or 4-nitro-*o*-phenylenediamine; or of 1:2-naphthaquinone-3:8-disulphonic acid with *o*-phenylenediamine; or of 1:2-naphthaquinone-8-sulphonic acid with 1:2-naphthylenediamine-6-sulphonic acid.

C. HOLLINS.

Water-insoluble colours or dyes. E. C. R. MARKS. From E. I. DU PONT DE NEMOURS & Co. (E.P. 271,580, 2.3.26).—Dyes for nitrocellulose products and for spirit varnishes are obtained by converting acid dyes into their aryl-, diaryl-, or triaryl-guanidine salts, which are insoluble in water but soluble in organic solvents. Thus, di-*m*-xylylguanidine, dissolved in acetic acid, is added to an aqueous solution of Chrysophenine, giving a yellow precipitate soluble in acetone, methyl alcohol, or ethyl acetate. Di-*o*-tolylguanidine, diphenylguanidine, and phenyldi-*o*-tolylguanidine may also be used, and the process may be applied to all dyes containing sulphonic or carboxylic acid groups.

C. HOLLINS.

Manufacture of intermediate products for azo dyes. BRIT. SYNTHETICS LTD., and E. B. HIGGINS (E.P. 271,146, 17.2.26. Addn. to E.P. 262,958; B., 1927, 102).—The processes of the earlier patent are extended to the preparation of 2:3-hydroxynaphthoic arylamides from arylamines containing negative substituents, *e.g.*, *m*- and *p*-nitroanilines, *m*-nitro-*p*-toluidine, 2:5-dichloroaniline, sulphanilic acid.

C. HOLLINS.

Manufacture of diacetylacetylarlylenediamine. A. ZITSCHER and R. SCHMITT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,634,090, 28.6.27. Appl., 15.5.23).—See E.P. 211,772; B., 1924, 413.

Production of N-dihydro-1:2:1':2'-anthraquinoneazine. E. C. R. MARKS. From DU PONT DE NEMOURS & Co. (E.P. 271,942, 2.3.25).—See U.S.P. 1,580,700; B., 1926, 577.

Manufacture of chromable azo dyestuffs. F. STRAUB and H. SCHNEIDER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,633,874, 28.6.27. Appl., 21.7.26. Conv., 24.9.25).—See E.P. 258,854; B., 1927, 518.

Manufacture of azo dyestuffs and chromium derivatives thereof. SOC. CHEM. IND. IN BASLE (GES. FÜR CHEM. IND. IN BASEL) (E.P. 254,708, 25.6.26. Conv., 3.7.25).—See U.S.P. 1,623,005; B., 1927, 469.

Coloured leather (E.P. 248,767).—See XV.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Pancreatic digestion of wool. L. MEUNIER, P. CHAMBARD, and H. COMTE (Compt. rend., 1927, 184, 1208—1210).—Wool which has been allowed to swell in alkaline solutions is attacked by pancreatic enzymes. With a concentration of enzyme of 2 g./litre, the binding cement between the cells is broken down. When the alkaline solution has p_H 10, the cells enclosed in the external sheath are detached, whereas when the concentration of the enzyme is increased to 5 g./litre, the sheath itself is broken down. There is no pancreatic attack when wool is steeped in a solution having p_H 4 (the isoelectric point of the wool).

M. CLARK.

Structural picture of the cotton hair as an aid to the interpretation of some phenomena. F. P. SLATER (J. Soc. Dyers and Col., 1927, 43, 181—185).—From consideration of the growth of the cotton hair and from examination of the mature hair in elevation and in cross-section, a structural picture of the cotton hair is developed which shows that there is an enormous surface on which dyes can be adsorbed. The variation in shade of dyed yarns with the type of cotton used, and the fact that the depth of shade corresponds with the order of wall thickness are in accordance with the structural picture thus developed. The large variation in wall thickness with any pure strain sample of cotton accounts for variations in the characteristics of raw cotton. Immature hairs in which no secondary wall cellulose has been deposited are different, both chemically and physically, from normal hairs, and often emerge undyed from the dyebath.

R. BRIGHTMAN.

Determination of copper number [of cellulose]. D. CLIBBENS and A. GEAKE (Papier-Fabr., 1927, 25, 401).—The superiority of the Schwalbe-Braidy over the Schwalbe-Häggglund method in the range of low copper numbers is well established, but it may be that for higher copper numbers and for the investigation of wood cellulose the former method is unsuitable, without modification, and that the latter is better. This suggests that the copper number is dependent on the physical or mechanical structure of the cellulose, and it is doubtful, therefore, whether a single method is suitable for both cotton and wood cellulose. The degree of swelling of cotton cellulose exerts no influence on its Methylene Blue absorption, *e.g.*, mercerised cotton has no higher absorption than unmercerised. If, however, cotton is dried after treatment with dilute sulphuric or phosphoric acid, an increased absorption is given which is dependent, not upon the swelling of the cellulose, but upon the fixation of acid. Cottons of different origins show markedly different absorptions, possibly due to purely chemical differences, and not to different degrees of swelling. The absorption of water, which, in a physico-chemical sense, is taken as the most exact measure of the degree of swelling, shows no significant differences for cottons of different origin. For textile purposes both copper number and Methylene Blue absorption measurements are necessary if the conditions of oxidation are unknown, since the production of oxycellulose may be characterised either by low Methylene Blue absorption and high copper number or by high absorption and low copper number, according to the p_H value of the bleaching liquor.

B. P. RIDGE.

PATENTS.

Method of chemically varying artificial fibres. HEBERLEIN & Co., A.-G. (E.P. 261,794, 22.11.26. Conv., 21.11.25).—Artificial silk fibres are treated with a halogen compound of phosphorus in a suitable solvent (*e.g.*, phosphorus trichloride in toluene) in the absence of alkali. In order to neutralise acid liberated, an alkaline salt (sodium carbonate) or an agent which neutralises acid without having an alkaline reaction (sodium acetate) is added, if required. Viscose silk so treated is rendered insensitive to substantive dyes, whilst acetate silk may afterwards be dyed with basic dyes. B. P. RIDGE.

Treating crape waste. G. BONNARD (E.P. 270,727, 4.5.27. Conv., 10.5.26).—The material is passed through an opening machine, carded, the sliver cut up into lengths of about 4 cm. (which are re-carded if necessary), treated with boiling water containing soap in order to separate the fibres, washed, dried, and again carded before spinning. B. P. RIDGE.

Manufacture of artificial silk. SYNTHETA, A.-G. (E.P. 265,577, 27.1.27. Conv., 4.2.26).—The threads (of nitrocellulose or acetate silk) are spun directly into an atmosphere of the hot solvent vapours with the exclusion of any other atmosphere, the desired concentration of this atmosphere being maintained by the removal and condensation of surplus vapour. The difficulty of recovering solvent from dilute mixtures with air is thus avoided. B. P. RIDGE.

Manufacture of artificial silk. D. A. MCKENZIE, Assr. to VISCOSE Co. (U.S.P. 1,630,197, 24.5.27. Appl., 28.12.23).—The annular fibre cake of fragile artificial silk formed in the spinning box is placed on a carrier which is then rotated. Washing water is delivered to the interior of the cake, and passes through it under the action of centrifugal force. The washed material is transferred on the carrier to the drying chambers and thence to the skeining machines. T. S. WHEELER.

Production of carbohydrate compounds. W. HARRISON (E.P. 264,261, 24.10.25).—Cellulose or starch xanthate, prepared in the usual manner, is oxidised with nitrous acid in the presence of acetic acid and in the absence of other mineral acid. The bulky, spongy mass of sulphide $(\text{RO} \cdot \text{CS})_2\text{S}$, or disulphide, $(\text{RO} \cdot \text{CS} \cdot \text{S})_2$, so formed is broken up and washed with water. In place of nitrous acid, nitrogen trioxide or tetroxide may be used, and the process is applicable to other carbohydrates or carbohydrate mixtures. C. HOLLINS.

Extraction of pure cellulose from the bagasse of sugar cane. E. C. H. VALET (U.S.P. 1,630,147, 24.5.27. Appl., 9.8.26. Conv., 14.6.26).—Bagasse, cut into small portions, is extracted with 3–5% calcium oxide solution, and is then heated under 1.5 atm. pressure with a mixture of 6% sodium hydroxide solution and 2–3% sodium sulphite solution for 4–6 hrs. The cellulose obtained is cleaned by treatment with steam and bleached. T. S. WHEELER.

Production of plastic masses from cellulose derivatives. PATHÉ CINÉMA (ANC. ÉTABL. PATHÉ FRÈRES) (F.P. 606,969, 10.3.25).—Non-inflammable products are obtained by dissolving cellulose derivatives, such as cellulose acetate, nitrate, or nitroacetate, or ethyl- or benzyl-cellulose, in phosphoric acid esters

containing one or more aliphatic radicals and one or more aromatic radicals, such as diphenylmethyl, diphenylethyl, phenyldimethyl, and phenyldiethyl phosphate. L. A. COLES.

Production of celluloid-like masses. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 247,174 and 263,076, 27.1.26. Conv., [A], 9.2.25; [B], 21.12.25. Addn. to E.P. 247,174, preceding).—(A) Phenol esters of phosphoric acid, of the highest purity, are mixed with nitrocellulose containing not more than 11% N, with or without the addition of solvents and diluents, and the mixture is worked up in the usual manner. (B) Softening agents (*e.g.*, esters of phthalic acid etc.) or hardening agents (*e.g.*, chloral etc.) are added prior to or during working up. B. P. RIDGE.

Manufacture of paper pulp. Treatment of plant material. F. K. FISH, JUN. (U.S.P. 1,633,730—1, 28.6.27. Appl., 11.7.25. Conv., [A], 22.12.24, [B], 20.12.24).—See E.P. 244,788—9; B., 1926, 871.

Preparation of paper pulp. F. K. FISH, JUN. (U.S.P. 1,633,736, 28.6.27. Appl., 24.5.26. Conv., 7.7.25).—See E.P. 255,030; B., 1927, 474.

Means for extracting fibres from the stalks of flax, hemp, and like plants. B. P. VON EHRENTHAL and K. SCHOLZ (E.P. 271,938, 10.12.25).

Continuously operating machine for phosphating and washing fabrics. M. RATIGNIER (E.P. 272,073, 13.9.26).

Vulcanised products (E.P. 271,553).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Testing dyestuffs for fastness to washing. S. R. TROTMAN (J. Soc. Dyers and Col., 1927, 43, 192).—A dyeing is made under standard conditions, and, after washing and drying, a weighed portion of the dyed sample is tested for fastness to washing under the standard conditions. At the end of the test the soap liquor is squeezed back into the bath, the sample washed with hot water, and the washings are added to the bath. The soap liquor and washings are cooled and made up to a measured volume. The quantity of dyestuff in this solution is then determined by placing an aliquot part in a Nessler cylinder and running a standard solution of the dyestuff, *e.g.*, 0.01%, into a second cylinder containing the same volume and concentration of soap solution as the diluted dye liquor, until the colours of two solutions match. More accurate results can be obtained with a Lovibond tintometer. From the volume of dye solution used the quantity of dyestuff extracted during washing is calculated and expressed as a percentage on the weight of fabric used. The method affords consistent numerical results on the same dyestuff, and enables a number of dyestuffs to be arranged in order of fastness to washing. R. BRIGHTMAN.

PATENTS.

Grounding or padding of textile materials. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (E.P. 271,543, 16.2.26).—Grounding and padding baths are improved by the addition of alkylated and/or aralkylated aromatic, or partly hydrogenated aromatic, sulphonic acids or sulphonates, phenolic derivatives being excluded.

Examples are: sulphonated benzyl- or polybenzyl-naphthalenes; condensation products of naphthalene-sulphonic acids with alcohols or mixtures of alcohols or with benzyl chloride, or tetralin dichlorides, or chlorinated paraffin oil; tetralinsulphonic acid condensed with cyclohexanol; naphthalene condensed with benzyl chloride and butyl alcohol and sulphonated; anthracene benzylated and sulphonated. The effect of these substances is to assist penetration of the fibre and also to disperse the grounding or padding agent when this is not completely soluble. C. HOLLINS.

Production of azo dyes on silk. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 253,865, 18.3.26. Conv., 20.6.25).—Degummed silk is impregnated at 20–30°, preferably in presence of a protective colloid, with a non-sulphonated arylamide of 2:3-hydroxynaphthoic acid or with a coupling component containing a reactive methylene group, and then coupled with a diazo component. Raw silk may also be degummed and bleached after dyeing in this manner. 2:3-Hydroxynaphthoic β -naphthylamide coupled with diazotised 4-nitro-*o*-anisidine gives a scarlet; bisacetoacetyltolidine with diazotised *o*-chloroaniline a yellow which does not bleed during degumming; bis-2:3-hydroxynaphthoyldianisidine with diazotised 5-nitro-*o*-anisidine a bordeaux. C. HOLLINS.

Production of fast mixed dyeings on silk. I. G. FARBENIND. A.-G. (E.P. 263,088, 21.7.26. Conv., 21.12.25. Addn. to E.P. 253,865; preceding abstract).—The raw or degummed silk is impregnated with a coupling component as in the prior patent and with the leuco-compound of a vat dye. The leuco-compound is oxidised and the ice-colour developed with a diazo component. Thus silk, impregnated with leuco-dibenzamidoanthrarufin and 2:3-hydroxynaphthoic *p*-anisidine, oxidised and developed with diazotised 5-nitro-*o*-anisidine, is dyed bordeaux; bisacetoacetyltolidine and leuco-Indanthrene Blue-Green B, oxidised and developed with *o*-chloroaniline, give a green. C. HOLLINS.

Preparations for dyeing cellulose esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 271,550, 22.2.26).—Insoluble or sparingly soluble compounds, suitable for dyeing cellulose esters either directly or by coupling on the fibre, are mixed with molasses. Other substances (protective colloids) may be added. Vat dyes are excluded. A preparation of aminoazobenzene ground with about 3 pts. of molasses and a little water gives on mere dilution a dyebath suitable for dyeing cellulose acetate. An intimate mixture of 1:4:5:8-tetra-aminoanthraquinone, molasses, sulphite-cellulose waste liquor, and sodium di-*p*-tolyl phosphate forms a homogeneous paste from which no dye separates on dilution. C. HOLLINS.

Dyeing, printing, or stencilling of cellulose acetate materials. R. CLAVEL (E.P. 270,987, 10.11.25).—Cellulose acetate materials after being weighted with tin phosphate or other insoluble salts (particularly of tin, zinc, tungsten, or aluminium) have affinity for direct, acid, basic, and other dyes, e.g., Janus Black D, Rhodamine G, Capri Blue, etc. C. HOLLINS.

Cross-dyeing cellulose fabric. J. C. WATSON, Assr. to T. P. GATES (U.S.P. 1,629,769 and 1,629,770,

24.5.27. Appl., 20.7.26).—(A) A process for cross-dyeing woven cotton and artificial silk fabrics comprises treating the warp threads during slashing (sizing) with a naphthol or other azo-dye coupling component, treating the filling threads with a different naphthol, weaving the fabric, and then treating it with a diazotised aromatic amine. Alternatively, the warp threads alone are treated. (B) The warp and filling threads are pretreated with leuco-compounds of two different vat dyes, and the woven fabric is treated with an oxidising agent.

T. S. WHEELER.

Dyeing films, plates, and other non-fibrous articles constructed of cellulose. E. CZAPEK (F.P. 605,661, 3.11.25).—Solutions of dyestuffs soluble in alkalis, but insoluble, or nearly so, in acids, are added to alkaline cellulose solutions, especially viscose solutions, and the cellulose and dyestuff are precipitated simultaneously with sulphuric acid or with ammonium chloride and hydrochloric acid. L. A. COLES.

Washing or dyeing machine. A. K. CROAD. From COWLES ENGINEERING CORP. (E.P. 271,749, 27.11.26).

Dyes and dyeing (E.P. 271,533 and 271,537).—See IV

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

U.S. Government master specification for soda ash. (U.S. Bur. Standards, Circ. No. 314, Nov. 23, 1926. 5 pp.).—The loss in weight after drying a sample for 1 hr. at 150–155° must not exceed 1%, and the total alkalinity of the material thus dried must not be less than 58%, calculated as Na₂O, matter insoluble in water shall not exceed 0.25%, and neither hydroxide nor bicarbonate must be present. 30 g. shall have a volume of 55–65 c.c., and not more than 0.5% shall fail to pass a No. 4 sieve (mesh 0.187 in.). Hydroxide is tested for by adding 10% barium chloride solution to a solution of the soda ash until no more precipitate forms, filtering, and adding a drop of 10% silver nitrate solution to the filtrate. A dark coloration indicates hydroxide. For the total alkalinity the solution is titrated with 0.5*N*-sulphuric acid, using methyl orange; another similar amount is then taken and about 1 c.c. less acid is added and the free carbon dioxide expelled by boiling, after which methyl orange is added and the titration completed. For bicarbonate the original solution is titrated with 0.5*N*-caustic soda until a drop produces instantly a dark colour with silver nitrate on a spot plate.

W. G. CAREY.

U.S. Government master specification for caustic soda (lye, for cleaning purposes). (U.S. Bur. Standards, Circ. No. 315, Nov. 23, 1926. 3 pp.).—Not less than 90% of sodium hydroxide and not more than 4% of carbonate calculated as sodium carbonate must be present. For the hydroxide determination an aqueous solution of 20 g./litre is made, 50 c.c. of 10% barium chloride solution are added to 50 c.c. of this solution and titrated with 0.5*N*-sulphuric acid, using phenolphthalein as indicator; the carbonate is determined in the same solution by adding methyl orange and titrating the barium carbonate. W. G. CAREY.

U.S. Government master specification for laundry soda (washing soda). (U.S. Bur. Standards,

Circ. No. 316, Nov. 23, 1926. 4 pp.).—Laundry soda is a mixture of sodium carbonate and bicarbonate, the total alkalinity of which shall be 39–41%, calculated as Na_2O , and matter insoluble in water must not exceed 0.1%. If the sample contains hydroxide it is rejected. Determinations are as for soda ash (cf. *supra*).

W. G. CAREY.

U.S. Government master specification for granular sodium carbonate (monohydrate crystals). (U.S. Bur. Standards, Circ. No. 317, Nov. 23, 1926. 5 pp.).—The total alkalinity of this substance, calculated as Na_2O , must not be less than 48.5%, matter insoluble in water must not exceed 0.1%, and neither hydroxide nor bicarbonate must be present. The technique of the hydroxide test and determination of total alkalinity and bicarbonate are the same as for soda ash (cf. *supra*).

W. G. CAREY.

Decomposition of phosphate rock by sulphuric acid. W. STOLLENWERK (Z. angew. Chem., 1927, 40, 613–620).—Phosphate was ground to a paste with water, the paste added to sulphuric acid, the mixture stirred for a given time, then filtered, and the residue washed free from acidity. Curves and tables were obtained showing the relation between the percentage of phosphoric acid dissolved and time, temperature, and concentration of the sulphuric acid used. The conversion is decreased, though the commencement of the reaction is hastened, by rise in temperature; the use of stronger sulphuric acid retards the reaction, though at lower temperatures (18°) the conversion is not decreased. Rise of temperature also increases the proportion of iron and aluminium dissolved. This quantity also varies with the time, but is not much affected by the strength of the sulphuric acid used. The behaviour of the reaction mass suggests that the decomposition of calcium phosphate by sulphuric acid proceeds in two stages. To test this, the sulphate content of the filtrate was determined. After allowing for the solubility of calcium sulphate in phosphoric acid, which was re-determined, a large deficiency was found in the free sulphuric acid content as deduced from the phosphoric acid liberated. It was shown in an indirect manner that solid calcium hydrogen sulphate is not formed under these conditions. Analyses of the filtrates suggested the following reactions (1) in the cold, $3\text{Ca}_3(\text{PO}_4)_2 + 9\text{H}_2\text{SO}_4 = 2\text{CaHPO}_4 + 4\text{H}_3\text{PO}_4 + 7\text{CaSO}_4 + 2\text{H}_2\text{SO}_4$; (2) at 50°, $3\text{Ca}_3(\text{PO}_4)_2 + 9\text{H}_2\text{SO}_4 = \text{Ca}_3(\text{PO}_4)_2 + 4\text{H}_3\text{PO}_4 + 6\text{CaSO}_4 + 3\text{H}_2\text{SO}_4$. Probably the dilution of the sulphuric acid checks the reaction, but the gradual conversion of anhydrite into gypsum effects a reconcentration. A microscopical examination confirmed this view. The complete reaction $\text{Ca}_3(\text{PO}_4)_2 + 3\text{H}_2\text{SO}_4 = 3\text{CaSO}_4 + 2\text{H}_3\text{PO}_4$ may be taken to be unimolecular as tricalcium phosphate is practically insoluble. A calculation of the velocity coefficient on this assumption gave the values 0.0159 at 18° and 0.0263 at 30°. The constancy of these justifies the assumption, but at higher temperatures the reaction appears to be bimolecular.

C. IRWIN.

Volumetric assay of iodides. A. J. JONES (Pharm. J., 1927, 118, 723–724).—Iodides in presence of chlorides and bromides are determined by titration with

standard potassium iodate solution in presence of hydrochloric acid.

E. H. SHARPLES.

Helium in Canada. R. T. ELWORTHY (Canada Dept. Mines, 1926, No. 679. 64 pp.).—Technical research in the extraction of helium from natural gases in Canada and the United States has shown the existence of several rich helium-bearing supplies, and the sources of helium have been studied, with the result that more than 25 million cub. ft. have been extracted from natural gas in Texas. The methods used for extraction depend on the removal of gaseous helium (b.p. — 269°) from the other liquefied constituents of the gas. The procedure of analysis of gases containing helium is described. From the results of large-scale experimental plants operated by the Linde Air Products Corp., Air Reduction Co., and the Jeffreys–Norton process, it was decided to instal a Linde plant to treat 5 million cub. ft. of gas per day, and the cost of extraction has been reduced progressively to \$24 per 1000 cub. ft. The helium content of gases at two points in Alberta and four points in Ontario ranges from 2 to 8 pts. per 1000, compared with 0.01–2 recorded for samples of natural gas from other parts of the world, and from the six positions a supply of over 5 million cub. ft. of helium per year is estimated, whilst the U.S. authorities state that it would be possible to extract yearly 50 million cub. ft. from gas which is being regularly used. The advantages of helium as compared with hydrogen for use in airships, apart from non-inflammability, are the lower diffusion of helium and the possibility of heating the gas-bags to expand the gas and counteract its greater density. Helium contaminated by diffusion of air into the gas-bags is purified by liquefying the contained oxygen and nitrogen or by the absorption of these gases by means of activated charcoal at low temperatures. The cost of storage and transportation of helium has been materially reduced by the development of large high-pressure steel cylinders. Cylinders 40 ft. long, $4\frac{1}{2}$ ft. in diameter, with walls 2 in. thick, charged to 2000 lb./sq. in. have a capacity of 200,000 cub. ft. of gas.

C. A. KING.

Formation of carbides. MEYER and SCHEFFER.—See II.

Iron in silicates. SARVER.—See VIII.

PATENTS.

Production of sulphuric acid. METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 262,397, 22.10.26. Conv., 7.12.25).—A mechanical mixing apparatus producing intimate contact between gases containing sulphur dioxide and finely-divided nitrosylsulphuric acid is employed in addition to a Glover tower plant of the usual type, and a similar mixing apparatus is interposed between each pair of chambers or towers (cf. E.P. 184,966; B., 1922, 858 A).

W. G. CAREY.

Manufacture of sulphuric acid. E. L. LARISON (U.S.P. 1,631,139, 7.6.27. Appl., 3.3.26).—Acid denitrated in a denitrating tower is delivered to the inlet of a nitrogen oxide recovery tower, and a portion of the acid from this tower is returned to the inlet, the remainder being sent to the denitrating tower.

W. G. CAREY.

Stabilising hydrocyanic acid. DEUTSCHE GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER, and F. KERSCHBAUM (E.P. 271,236, 5.5.26).—Kieselguhr for

absorbing and stabilising hydrocyanic acid is roasted, preferably in presence of a large amount of air, and is then freed from basic substances by treatment with acids or acid salts, the excess of acid being removed by washing. Esters which act as irritants, *e.g.*, chloroacetates, or which have a stabilising effect, *e.g.*, methyl chloroformate, may be used instead of acids.

W. G. CAREY.

Production of phosphoric acid. I. G. FARBERNIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 246,128, 5.1.26. Conv., 17.1.25).—In the combustion of phosphorus (or of gases containing it) with air, corrosion of the wall of the combustion chamber is avoided by continuously wetting the wall with phosphoric acid solution, either by employing a horizontal rotating tube as combustion chamber or, if an empty vertical shaft is used, by irrigating the walls with the solution.

W. G. CAREY.

Manufacture of phosphorus or phosphoric acid and concurrently of activated charcoal. Soc. POUR L'EXPLOIT. DES PROC. E. URBAIN (E.P. 257,917, 24.8.26. Conv., 4.9.25).—Monocalcium phosphate or phosphoric acid is heated with considerable excess of charcoal or carbonisable organic matter for 8–12 hrs., the mass being in granular form. The phosphorus is collected and purified, or is burnt before condensation, the activated charcoal which remains being washed with acid to dissolve any basic phosphate.

W. G. CAREY.

Discharging ammonium sulphate from saturating tanks. F. J. COLLIN, A.-G. ZUR VERWERTUNG VON BRENNSTOFFEN U. METALLEN (E.P. 268,309, 21.2.27. Conv., 24.3.26).—Compressed air is employed, and is heated in the saturating tank by the gases and vapours, thus cooling the vapours so that condensable substances are separated therefrom.

W. G. CAREY.

Purification of gases for the synthesis of ammonia. K. MÜLLER (E.P. 261,388, 10.11.26. Conv., 12.11.25).—After preliminary cleaning with lime or caustic alkali the gases are freed from final traces of oxygen, carbon monoxide and dioxide, sulphurous gases, and steam by absorbing the impurities in sodium hydride under pressure at 200° or higher. The active surface of the hydride is increased by mixing therewith inorganic substances, especially metals.

W. G. CAREY.

Treatment of zinc chloride-bearing solutions. ORKLA GRUBE-AKTIEBOLAG (E.P. 252,388, 21.5.26. Conv., 22.5.25).—The zinc is precipitated with less than the equivalent amount of lime at 30–40°, and, after removal of the mother-liquor, the resulting basic zinc chloride is treated with not more than an equivalent quantity of lime at about 70° to form zinc oxide, which is dried and sintered at 1000–1200° without removal of the calcium chloride. The quantity of lime is adjusted so that the subsequent sinterings remove cadmium etc. by volatilisation of the chloride.

W. G. CAREY.

Utilising the liquors derived from chloridising roasting pyrites cinder and the like. REYMER-SHOLMS GAMLA IND. AKTIEBOLAG (E.P. 260,294 and 260,297, [A], 22.10.26; [B], 23.10.26. Conv. [A, B], 24.10.25).—(A) The liquor first obtained on lixiviating

the roasted material and containing salts mostly in the form of chlorides is kept separate from the later leachings, which consist mostly of sulphates. Copper is precipitated from the chloride liquor by means of zinc, and the solution free from iron is suitably treated for the recovery of silver and cobalt, after which it is cooled to crystallise sodium sulphate. The sulphate liquor is treated with cement copper to precipitate cuprous chloride, and, after treatment with an alkaline reagent to precipitate other metals, the liquor is evaporated to produce sodium sulphate. (B) Sufficient chlorine ions are allowed in the sulphate liquor to be equivalent to the cupric copper ions, and the cuprous chloride precipitated is dissolved in the solution obtained after treatment of the chloride liquor (or in any suitable solution), the copper being then obtained by cementation.

C. A. KING.

Recovery of vanadium from solutions. R. H. STEVENS, G. C. NORRIS, and W. N. WATSON (E.P. 269,779 and 269,780, 23.8.26).—(A) Vanadium in solution is converted into the quinquivalent form, and precipitated, in the presence of a ferric salt, wholly or partly as ferric vanadate by the addition of a base, *e.g.*, lime. The precipitate is washed free from calcium sulphate by means of water or brine. (B) Preliminary purification of vanadium solutions is effected by adding a base to a solution containing quadrivalent vanadium. Silica, iron, alumina, and any other impurities which may be precipitated are removed, and the vanadium in solution is then converted into either the quinque- or ter-valent condition to facilitate its subsequent precipitation.

C. A. KING.

Conversion of lead sulphate into lead carbonate. S. C. SMITH (E.P. 272,053, 29.6.26. Cf. E.P. 239,257; B., 1925, 881).—In the manufacture of lead carbonate by passing carbon dioxide into a suspension of lead sulphate in ammonia solution, the product contains the minimum amount of lead sulphate if the carbon dioxide is introduced at such a rate that free carbon dioxide in the solution is avoided.

W. G. CAREY.

Preparation of lithium carbonate. H. WEIDMANN, ASSR. to ALLIED PROCESS CORP. (U.S.P. 1,623,513, 5.4.27. Appl., 12.5.26. Conv., 29.5.25).—Potassium chloride is added to a concentrated aqueous solution of lithium and potassium sulphates, and the precipitated potassium sulphate is separated from the solution. To the resulting solution rich in lithium chloride, potassium carbonate is added. Lithium carbonate in good yield is precipitated and potassium chloride regenerated, which can be used again in the first part of the process.

W. G. CAREY.

Manufacture of aluminium oxide. A./S. NORSK ALUMINIUM Co. (E.P. 252,399, 21.5.26. Conv., 23.5.25).—Silica-bearing slags containing calcium aluminate are pulverised and then leached and precipitated with a hot solution of sodium carbonate containing not more than 10 g./litre of free alkali hydroxide, to decrease the amount of silica dissolved and to produce a granular aluminium hydroxide precipitate. After filtration, the solution is treated with carbon dioxide to reconvert the greater part of the free caustic alkali into carbonate, so that the solution is available for further leaching.

W. G. CAREY.

Production of phosphorus. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 247,219, 6.2.26. Conv., 6.2.25).—Hot crude gases from phosphorus furnaces are freed from dust by passing them through a filter chamber maintained at a temperature above the dew point of phosphorus by internal electrical heating or external heating by combustion gases, and, if desired, subsequently submitting them to an electrical dust-removing treatment. The filter chamber may be filled with the same materials as are used for charging the furnace, either in a moist or dry condition.

W. G. CAREY.

Purification of hydrogen obtained by the inter-action of phosphorus and steam. I. G. FARBENIND. A.-G. (E.P. 262,455, 3.12.26. Conv., 4.12.25).—After condensing the bulk of the phosphorus acids the hydrogen, without previous cooling, is passed over active charcoal or silica gel, the substances adsorbed, *e.g.*, hydrogen phosphide, being oxidised by air during or after adsorption and the resulting oxides and acids of phosphorus being extracted with water or acids.

W. G. CAREY.

Calcination of limestone and the like in vertical kilns fired with lean gas. HAUTS FOURNEAUX & ACIERIES DE DIFFERDANGE-ST. INGBERT-RUMELANGE, SOC. ANON., and E. LAVANDIER (E.P. 246,485, 21.1.26. Conv., 21.1.25).—The gas is burnt under pressure in mixing burners with sufficient air for complete combustion, the products of combustion being injected into the kiln through nozzles which localise a temperature zone in the kiln of at least 900°.

W. G. CAREY.

Manufacture of cyanides of alkali-forming metals. CALIFORNIA CYANIDE CO., INC., Assees. of F. J. METZGER (E.P. 261,722, 14.10.26. Conv., 23.11.25).—See U.S.P. 1,573,732; B., 1926, 486.

Production of aluminium chloride. E. R. WOLCOTT, Assr. to TEXAS CO. (U.S.P. 1,633,835, 28.6.27. Appl., 2.2.21).—See E.P. 175,006; B., 1922, 252 A.

Filtration of solutions and the like. J. BLUMENFELD (U.S.P. 1,633,621, 28.6.27. Appl., 28.7.24. Conv., 3.8.23).—See E.P. 228,814; B., 1925, 315.

Adsorbents (E.P. 271,564).—See I.

Ammonia from gases (U.S.P. 1,631,720).—See II.

Electrolytic apparatus (E.P. 271,685 and 271,726).—See XI.

Aurothiosulphate compounds (E.P. 261,048).—See XX.

VIII.—GLASS; CERAMICS.

Structure and constitution of glass. W. ROSENHAIN (J. Soc. Glass Tech., 1927, 11, 77–97).—As a tentative working hypothesis, complex amorphous solids, such as glass, are considered as assemblages of atoms in which certain molecular groupings may occur with greater or less frequency, but in which the intra-molecular linkages need not be very different in kind or intensity from the other atomic linkages which exist, or come into being, when the substance is cooled down into the quasi-solid state. Formation of linkages will be limited by the inter-atomic distances and the angular relations between adjacent atoms. A portion only, probably

small, of the inter-atomic linkages are operative, and the latent heat of fusion, representing the energy stored in unsaturated bonds, is largely retained in cooling and setting, so explaining the small heat evolution on cooling. With rise of temperature a point is reached above which each increment entails the breakage of certain linkages and the storage of energy or latent heat, and the heat added is used partly for this purpose and partly for increasing the amplitude of atomic thermal oscillation. The apparent specific heat of a vitreous substance should thus be greater than that of a crystalline substance of the same composition, and available data appear to bear out the point. There is an intermediate stage between elastic solid and viscous liquid in which rupture of bonds may occur for a time after application of stress, but with storage of sufficient energy to stop, after a time, further rupture; when stress is removed there will be gradual and incomplete return to the original configuration. The hypothesis fits the observed facts with regard to the slight thermal effect when glass is heated, and the variation of thermal expansion with temperature. The apparent modulus of elasticity should be decidedly higher than that of the same substance in crystalline form. Since solid diffusion is probably caused by "slip" mechanism, such cannot occur in cold amorphous solids, and diffusion at higher temperatures is probably due to viscous flow. The presence of molecular aggregates may result in neutral zones.

A. COUSEN.

Shrinkage of diaspore clays. I. S. M. PHELPS (J. Amer. Ceram. Soc., 1926, 9, 659–666).—Three types of diaspore clays were studied in order to determine the nature and cause of the shrinkage which takes place in them after prolonged use. Under given conditions of time and temperature, the shrinkage varied inversely as the alumina content of the clays. The duration of the firing period had a marked effect upon shrinkage. The bonding (plastic) portion and the granular portion were separated by the aid of deflocculation and tested; considerable differences were observed in their porosity, shrinkage, and chemical composition. In the process of manufacture, diaspore refractories should receive sufficient heat treatment to produce constancy of volume within practicable limits.

F. SALT.

Hypothesis explaining some characteristics of clay. J. VERSLUYS (Proc. K. Akad. Wetensch. Amsterdam, 1927, 30, 104–112).—A theoretical and mathematical paper in which it is shown by comparison with actual experimental curves that the tensile strengths of soils in their dependence on grain size and water content are directly calculable from the surface tension of the pendular water between the individual grains. The contracting effect of pendular water alone is insufficient to explain completely the peculiar phenomena exhibited by clay, *e.g.*, expansion and contraction on change in the proportion of water, for which purpose it is necessary to assume the existence of a power of expansion resulting from the possibly multimolecular film of adsorbed moisture which envelops each clay particle. Considering an ideal case in which two spherical particles are caused to approach with progressive displacement of the adsorbed film near the points of contact, it is shown that as the removal of adsorbed moisture proceeds the power

of expansion of the clay progressively increases. When clay dries out two forces are involved, the contracting force which increases as the water content decreases, and the expanding force, the equilibrium being continually shifted. An interpretation of the phenomena of quicksands is suggested, based on the probable behaviour of a mass of wet sand towards a sequence of physical disturbances. J. S. CARTER.

Clay analysis for control purposes. W. P. ECKDAHL (Chemist-Analyst, 1925, No. 45, 5—7; Chem. Zentr., 1927, I, 170).—A 0.5 g. sample is fused with sodium carbonate, dissolved in 60 c.c. of 1:1 hydrochloric acid and 12 c.c. of acetic acid, the undissolved silica filtered off, and iron oxide, alumina, and silica are precipitated by concentrated ammonia, after which the liquid is boiled and filtered. Lime and magnesia are determined in the filtrate. The precipitate is dissolved in hydrochloric acid, reprecipitated, filtered, ignited, and weighed, treated with hydrofluoric acid and weighed again. B. W. CLARKE.

Determination of ferrous iron in silicates. L. A. SARVER (J. Amer. Chem. Soc., 1927, 49, 1472—1477).—The use of diphenylamine as an internal indicator in the titration of ferrous iron by potassium dichromate (cf. Knop, B., 1924, 404) is unaffected by the presence of hydrofluoric acid. The latter acid, however, decomposes potassium dichromate at higher temperatures so that the analysis of silicates by decomposition in presence of excess of dichromate, to eliminate oxidation by atmospheric oxygen, becomes impossible. A modified Pratt method of analysis is described (cf. Hillebrand, U.S. Geol. Survey Bull., 1919, No. 700, p. 203). 0.5 g. of silicate is boiled with 10 c.c. of either 12*N*-hydrochloric acid or 18*N*-sulphuric acid in a platinum crucible having a close-fitting transparent bakelite cover fitted with a bakelite funnel and an entrance tube. Air-free carbon dioxide is swept through the crucible for 10—15 min. during the boiling, and then 7 c.c. of 48% hydrofluoric acid are added through the funnel. After gently boiling for 10 min. the whole is cooled in a current of carbon dioxide, an excess of standard dichromate solution is added through the funnel, and the contents of the crucible are poured on to solid boric acid and titrated with ferrous iron solution using diphenylamine as indicator. The results are somewhat higher than those obtained in an ordinary crucible, but show good concordance. S. K. TWEEDY.

Effect of different forms of silica in porcelain bodies. H. HIRSCH (Ber. Deut. Keram. Ges., 1926, 7, 49—88).—Tests were made on a porcelain body consisting of 50 pts. of kaolin, 20 pts. of Norwegian feldspar, and 30 pts. of quartz. The raw quartz was added in a number of different forms: Scandinavian vein quartz, rock crystal, flint, geyserite, two kinds of quartz sand, and the washings from kaolin. These materials were examined mineralogically, and photomicrographs are given. The porcelain samples were tested for shrinkage, porosity, true and apparent sp. gr., cone m.p., coefficient of thermal expansion, tensile strength, resistance to the Brinell test, impact, electric breakdown, and translucency (photometric method); they were also examined in thin sections microscopically. Vein quartz produced by far the greatest translucency, especially in the hard

porcelain series. Of the three strength tests, the Brinell test gave the most uniform and reliable results; the highest figures in these tests were obtained with pre-fired flint and quartz sand. These two materials, and the kaolin residues, also gave better results than quartz in the electrical test. Raw flint gave the lowest results in all these tests. It is concluded that crystalline quartz is only used on account of its effect upon translucency. Flint, quartz sand, and especially kaolin residues produce bodies which are stronger both mechanically and electrically. A study of thin sections revealed no direct relationship between microstructure and physical properties. The formation of mullite crystals was identified both microscopically and by chemical analysis. F. SALT.

Structure of the crystalline phases of porcelain. L. B. STROUTINSKY (J. Russ. Phys. Chem. Soc., 1927, 59, 137—145).—Sillimanite subjected to X-ray analysis gives the following measurements: $a = 741 \text{ \AA.}$; $b = 7.67 \text{ \AA.}$; $c = 5.72 \text{ \AA.}$ It is found to consist of 4 mols. of $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$. Andalusite also consists of 4 mols., and has the corresponding measurements: 7.76, 7.86, and 5.51 \AA. Mullite gives figures pointing to $\frac{1}{2}\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$, and, as this is impossible, the substance is either a mixture, or has been assigned an erroneous formula.

E. ROTHSTEIN.

PATENTS.

Production of potash glass. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 245,785, 8.1.26. Conv., 8.1.25).—Potash glass is obtained by the use of potassium bicarbonate as a batch material; this substance is easily obtained pure, is non-hygroscopic, and promotes active mixing during melting. A. COUSEN.

Manufacture of silica glass articles. "QUARTZ & SILICE" (E.P. 266,711, 21.2.27. Conv., 24.2.26).—Addition of further material to vitreous silica articles is obtained by packing a mass of pulverulent silica in the desired region and fusing this to the original body by an apparatus containing, preferably, electrical resistance or high-frequency induction heating elements. Further parts may be so added to existing ware or articles of large dimensions built in successive stages. A. COUSEN.

Cement [ceramic] composition. C. E. KRAUS (U.S.P. 1,629,714, 24.5.27. Appl., 23.9.24. Cf. U.S.P. 1,509,406; B., 1925, 11).—Bentonite is added to kaolin or other clay to increase its plasticity when mixed with water. T. S. WHEELER.

Ceramic insulating material. H. H. SORTWELL, Assr. to STAR PORCELAIN Co. (U.S.P. 1,633,462, 21.6.27. Appl., 27.2.24).—A black porcelain insulating material consists of 45 pts. of black iron ore, 35 pts. of red-burning clay, 15 pts. of potter's flint, and 5 pts. of feldspar. H. ROYAL-DAWSON.

Manufacture of an article of sillimanite-bonded granular material. M. C. BOOZE, Assr. to NORTON Co. (U.S.P. 1,616,525, 8.2.27. Appl., 20.5.21).—A ceramic article is made of graded super-refractory grains united by a small quantity of a synthetic sillimanite bonding material of the general formula $x\text{Al}_2\text{O}_3 \cdot y\text{SiO}_2$. Thus, crystalline alumina is bonded with a mixture of 7 pts. of

alumina (99% Al_2O_3) and 15 pts. of kaolin. The mixture after being moulded is fired at cone 16. H. HOLMES.

Colourless Crookes glass. P. V. W. GELL, C. E. GOULD, W. M. HAMPTON, and H. S. MARTIN, Assrs. to CHANCE BROS. & Co., LTD. (U.S.P. 1,634,182, 28.6.27. Appl., 11.5.25).—See E.P. 256,737; B., 1926, 878.

Method and apparatus for producing a glass article [by severance]. E. C. R. MARKS. From LIBBEY GLASS MANUF. CO. (E.P. 272,308, 15.3.26).

Annealing ceramic wares (E.P. 248,394).—See I.

IX.—BUILDING MATERIALS.

PATENTS.

Conversion of slags into cement. S. MICHELSEN (E.P. 267,539, 10.3.27. Conv., 10.3.26).—Gypsum or other compounds containing sulphur are mixed with slag and heated so that the manganous oxide is partly or wholly converted into manganous sulphide. Limestone is then added if necessary in order to give the slag the required composition for use as cement.

B. W. CLARKE.

Calcining limestone (E.P. 246,485).—See VII.

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

Transformation of retained austenite into martensite by stress. K. HONDA and K. IWASÉ (Sci. Rep. Tôhoku Imp. Univ., 1927, 16, 1—8).—By measurements of changes in hardness and intensity of magnetisation, it has been shown that retained austenite in a quenched chromium steel is transformed into the stable martensite by cold hammering. On quenching plates of different thickness with oil and water, respectively, it has been found that, when the thickness of the plates is 3 mm. or more, water-quenched specimens are harder (*i.e.*, contain more martensite) than oil-quenched. This is in accordance with Mathews' observations (B., 1925, 807; 1926, 132) and also with Bain's view (Trans. Amer. Soc. Steel Treat., 1925, 8, 14) that the internal stress produced by quenching promotes the transformation of retained austenite to martensite, the stress being greater in water-quenching. The contrary effect, however, is observed when the thickness of the plate is below 3 mm. In both methods of quenching the hardness increases with the thickness of the plate. When the steel is quenched in liquid air the hardness diminishes as the thickness of the plate increases. It is suggested that, although the supercooling of the quenched austenite facilitates transformation into martensite, the stress set up due to expansion may, after a certain point, prevent a further transformation of the remaining austenite, this stress naturally increasing with thickness.

M. S. BURR.

Nitridation of steels. L. GUILLET (Compt. rend., 1927, 184, 1296—1299; cf. B., 1926, 410).—Tests on nitrided complex steels show increases in hardness particularly with chromium-tungsten-vanadium steels, pearlitic and martensitic nickel-vanadium steels, and nickel-manganese steels. The presence of vanadium, especially in nickel steels, has an important influence. Such steels are attacked by acid, alkali, and salt solutions

to variable extents for different samples, and more so than ordinary cemented steel. The nitrogen is present probably as a complex nitride, and its concentration is highest on the surface of the metal. J. GRANT.

Thermal changes in iron-manganese alloys, low in carbon. (SIR) R. HADFIELD (Proc. Roy. Soc., 1927, A, 115, 120—132).—The object of the investigation was the determination of the temperature of the critical magnetic changes in a series of iron-manganese alloys, practically free from carbon, containing 1.70—38.90% Mn. Magnetic transformations are confined to magnetic alloys with less than 16% Mn. The non-magnetic alloys above 16% show no magnetic transformations, no matter how heat-treated, or even when cooled to the lowest known temperatures. For all the magnetic alloys, a reversible magnetic transformation is found at practically the same temperature as the A2 magnetic change in iron (in this case, at 765—780° on heating, and at 767—776° on cooling). For alloys containing 6.75—14.30% Mn, a doubling of the magnetic transformation is observed on cooling, although not on heating. That is, the first recovery of magnetisation which takes place on cooling only holds over a limited range of temperature, the alloy becoming again practically non-magnetic, until finally, on further cooling, the full magnetic properties reappear and remain down to atmospheric temperature. The second critical point is progressively lowered with increase of manganese and decreases in intensity, and is not a fixed point for each alloy, but on repetition appears at a lower temperature. No such variation occurs in the position of the upper magnetic transformation. The magnetic strength of the alloys appears to become progressively weaker with increase of manganese. Heating and cooling curves of the inverse rate type are reproduced, and the relation between the magnetic changes and the thermal evolutions shown by the curves is discussed. It is suggested that the non-magnetic properties of the higher-percentage alloys may be due to chemical combination of the iron and manganese, the most probable formula for the resulting compound being Fe_3Mn .

L. L. BIRCUMSHAW.

Tensile tests on alloy crystals. I. Solid solution alloys of aluminium and zinc. II. Solid solution alloys of copper and zinc. III. Conclusions. C. F. ELAM (Proc. Roy. Soc., 1927, A, 115, 133—147, 148—166, 167—169).—I. The results of tensile tests on single crystals of six alloys containing from 4.45 to 18.50% Zn showed that, with increasing percentage of zinc, the amount of extension before fracture tended to decrease while the breaking load increased. There was considerable variation especially in the elongation between crystals of the same composition. All the alloys show a well-marked yield point, particularly those with a high zinc content. Alloys containing 5—7.5% Zn usually show a double wedge-shaped fracture, as in the case of pure aluminium, whilst those containing over 10% Zn show a straight cleavage fracture (cf. A., 1925, ii, 954). Certain crystals containing 5—7.5% Zn showed an intermediate structure in which the double wedge was replaced by a single wedge asymmetric to the axis of the specimen. This may be due to the fact that, as the zinc content increases, the possible amount of extension

before fracture decreases, so that fracture may occur before the position for slip on two planes is reached. The results from both distortion and X-ray measurements with 10 and 15% zinc alloys indicate that the crystals deform by slip on an octahedral (111) plane in a direction represented by the normal to a (110) plane. This was confirmed by observation of slip-bands. There is also evidence that the 10% alloy crystal slipped on a second plane for about the last 10% of its extension. In many specimens the plane of fracture agreed very closely with the slip-plane, but in some the two made a small angle of from 10° to 20° with each other. Calculation of the shear stress on the slip-plane and the amount of shear showed that the resistance to shear increased considerably with increasing zinc content. The maximum increase in hardness occurs in the early stages of deformation, but the shear stress increases right up to the breaking-point.

II. Tensile tests have been carried out on a number of brass crystals containing about 70–80% Cu, which had been prepared by a method previously described (A., 1926, 1085). Distortion and X-ray measurements indicate that deformation occurs by slip first on the (111) plane in the (110) direction, and then on the (111) plane in the (011) direction. The most noticeable features of the distortion of these crystals are, firstly, the enormous extension (160%) before fracture, compared with copper, which rarely exceeds 60%, and, secondly, the uniformity of the crystal in its final condition, considering the abrupt changes in shape when the slip changes from one plane to the other. Calculations were made of the shear stresses on both planes; the shear stress at the beginning of the test is very little different from that of copper. The different octahedral planes in the crystal appear to be unequally resistant to shear, and it is suggested that slip tends to proceed more easily along a plane upon which it has once begun.

III. The results obtained by Rosbaud and Schmid (A., 1925, ii, 488) in their investigation of the effects of alloying on distortion in the case of zinc-cadmium and zinc-tin alloys are in general agreement with those described in the preceding investigation on the aluminium-zinc alloys. The fact that, in many respects, the results obtained with brass crystals appear to be directly opposite to those with aluminium-zinc crystals may possibly be ascribed to the different methods of preparing the crystals. It is suggested that the alloying atoms take up different positions when the crystals grow from the liquid from those occupied when the metal recrystallises in the solid, and it is probable that alloy crystals grown in the solid by a process of straining and heat-treatment are more uniform in structure than those grown from the liquid.

L. L. BIRCUMSHAW.

Cementation of aluminium and duralumin after double electrolytic deposition. J. COURNOT and E. PEROT (Compt. rend., 1927, 184, 1250–1252; cf. B., 1927, 167).—The cementation of aluminium and duralumin coated electrolytically with copper and then with nickel, chromium, cobalt, or cadmium has been attempted by heating in a sand-bath or tubular electric furnace under varying conditions of deposition and cementation. Folds were produced at cementation temperatures above 575°, and sounder surfaces resulted from sand-bath treatment and double cementation. The total degree

of penetration depends principally upon the copper deposit. The cemented zone consisted of a solid solution surrounded by a eutectic phase analogous with Al-Cu, and a few hard constituents produced by combination of the copper with the metal forming the outside deposit.

J. GRANT.

Influence of small quantities of bismuth, tin, lead, etc. on the structure and working properties of gold and gold alloys. L. NOWACK (Z. Metallk., 1927, 19, 238–244; cf. A., 1926, 896).—The most objectionable impurities in gold and gold alloys are bismuth, tellurium, and lead, less than 0.1% of which renders the metal unworkable owing to the segregation of bismuth, AuTe₂, and Au₂Pb along the grain boundaries. Prolonged annealing of metal containing these impurities serves to enhance their deleterious effect. More than 0.1% Sb renders gold brittle owing to the separation of the eutectic, but 0.1% Al, up to 1% Sn, and 2–3% Fe have little effect other than a slight hardening action. Characteristic photomicrographs of gold with various impurities are reproduced showing the modifying action of the impurity on the grain structure.

A. R. POWELL.

Recrystallisation and annealing of precious metal [silver-copper-gold] alloys. L. STERNER-RAINER (Z. Metallk., 1927, 19, 149–153, 245–248).—The hardness of cold-rolled silver-copper alloys (80% Ag) increases slightly with rise of annealing temperature to 315°, then decreases rapidly. The tensile strength and elastic limit decrease slightly, and the elongation is increased by keeping the alloy in boiling water for 1–1.5 hr. The rate at which annealing takes place increases rapidly with rise of temperature above 315°, but even at 720° annealing is not completed in less than 80 min.; at 750°, however, the maximum ductility is obtained in 20 min., and further heating decreases it again. The best method of ascertaining when a sample of cold-rolled metal is completely annealed is to determine its elongation, as this property is more drastically affected by heat treatment than any other. Thus, after 60% reduction in thickness the metal has an elongation of 4.4%, which increases to a maximum of 36.2% at 720°. Similar behaviour is shown by 14-carat gold containing equal proportions of silver and copper. After a 10% rolling the tensile strength, elastic limit, and hardness increase on annealing at 350°, and, to a smaller extent, at 450°; with a 50% reduction by rolling, however, annealing above 300° causes a rapid softening. The hardening action of a low-temperature anneal is hardly noticeable with 18-carat gold, and does not occur with alloys of higher carat.

A. R. POWELL.

Determination of vanadium in iron minerals and rocks. V. A. SILBERMITZ and L. V. ROZHKOVA (J. Russ. Phys. Chem. Soc., 1927, 59, 121–124).—A new colorimetric method for determining vanadium present in minerals and rocks which are soluble in acids or water is described. The pulverised rock is dissolved in nitric acid, the chlorides are precipitated with silver nitrate, and the organic matter is oxidised with potassium permanganate, excess of the latter being removed by the addition of hydrogen peroxide solution. Phosphoric acid is now added, the excess of hydrogen peroxide being destroyed by boiling, and then ammonium molybdate

solution. The colour obtained is compared with a standard chart prepared from standard solutions of ammonium vanadate. The method is quite as accurate as the more usual methods. E. ROTHSTEIN.

Synthetic testing for flotation. C. G. McLACHLAN (Trans. Canad. Inst. Min. Met., 1926, 29, 251–280).—Laboratory experiments on the flotation of certain minerals by selective reagents, *e.g.*, oleic acid, aniline, showed that selective adsorption of oils is not the primary controlling factor in flotation. The forces involved are surface forces, and consequently the relative magnitude of these forces increases as the size of the mineral particle decreases. The electrostatic charge normally possessed by a mineral particle plays a very minor part. In certain cases of added electrolytes, it was evident that definite chemical interaction was necessary for successful working; *e.g.*, copper sulphate promotes the flotation of sphalerite by coating it with a film of copper sulphide. Similarly, the beneficial action of sulphuric acid in the treatment of certain zinc ores is due to copper associated with these ores going into solution as sulphate. The atomic arrangements of a number of sulphides, oxides, and carbonates were examined. In chalcopyrite and pyrite the faces of the unit crystal contain metal atoms only; in molybdenite, sulphur and metal atoms; whilst in sphalerite the zinc and sulphur atoms are interchangeable. In non-floatable minerals, *e.g.*, oxides of copper, zinc, arsenic, and members of the calcite group of carbonates, oxygen and not sulphur atoms are present in the crystal faces, and it is suggested that only surfaces which contain elements having an affinity for oxygen are capable of flotation. C. A. KING.

PATENTS.

Heat-treatment of steel. N. D. CHOPRA and F. J. BULLEN (E.P. 271,606, 1.4.26).—Steel is heat-treated in contact with furnace gases which have been treated with a mixture of an alkaline-earth oxide (10–50%) and carbonaceous material (4–20% of free carbon). An exhausted carburising mixture forms a suitable material. Steel turnings or other form of steel which exposes a large surface to the furnace gases may surround the article to be treated. C. A. KING.

Treatment [to prevent corrosion] of iron and steel articles. METALS PROTECTION CORP., Assees. of C. H. HUMPHRIES (E.P. 264,788, 23.8.26. Conv., 25.1.26).—Articles of iron or steel are subjected, with or without the aid of electrolysis, to a solution containing chromic acid so as to form a surface film containing iron and chromium oxides, and chromates. The coating formed is adherent and resistant to acid fumes, and forms a good surface for the after-application of paint or enamel. C. A. KING.

Making grey-iron castings of any desired structure. MASCHINENFABR. ESSLINGEN (E.P. 260,990, 2.11.26. Conv., 6.11.25).—The total content of carbon and silicon in iron for a casting of definite thickness is selected from a graph showing the relation of structure with the wall thickness and the content of carbon and silicon. C. A. KING.

Magnetisable material. E. GÜMLICH (E.P. 262,153, 30.11.26. Conv. 30.11.25).—An iron alloy of high

magnetic permeability contains more than 40% Ni and 5–20% Mn. C. A. KING.

Bearing metal. L. SEMPELL (E.P. 272,065, 4.8.26).—A bearing metal is made by fusing together 8–25% Sn, 1–4% of ammonium or calcium molybdate (1–2.5% of a 70% ferromolybdenum alloy), 0.5–2% Ni, 0.5–2% Cu, 13–20% Sb, the remainder being lead. C. A. KING.

Bearing metal. P. KEMP and T. KITTL (U.S.P. 1,632,604, 14.6.27. Appl., 10.8.25. Conv., 23.8.24).—The alloy consists of 65–77% Pb, 3–14% Sn, 10–27% Sb, and 0.7–2.5% As. F. G. CROSSE.

Treatment of zinciferous flue dust. F. KRUPP GRUSONWERK A.-G. (E.P. 265,558, 29.12.26. Conv., 4.2.26).—Zinciferous flue dust is heated in a rotary furnace at a temperature sufficiently high to sinter the material and at the same time volatilise the impurities, *e.g.*, arsenic, antimony, tin, cadmium, lead, and various acid radicals. The temperature in the furnace may approach the volatilisation point of zinc, and fluxes may be added to assist in the purification. C. A. KING.

Acid extraction of metals such as zinc and vanadium. R. H. STEVENS, G. C. NORRIS, and W. N. WATSON (E.P. 269,777, 23.8.26).—An ore containing both a relatively easily and difficultly soluble metal is extracted with a dilute acid to dissolve the easily soluble metal. The residue, if necessary after being reground, is then leached with hot acid of suitable concentration, *e.g.*, for vanadium, preferably about 50 g. of sulphuric acid per litre, and the strongly acid solution is returned as leaching acid in the first leaching step. C. A. KING.

[Desulphurising] treatment of nickel-containing mattes. H. WADE. From INTERNAT. NICKEL CO. (E.P. 271,282, 16.7.26).—Nickel or nickel-copper mattes are converted by blowing gases containing oxygen through the molten matte at 1380–1750°, and reducing the oxygen content of the blast during the later stage of blowing. Additional heat may be supplied by combustion above the bath in the converter, which is lined with fused alumina bonded with kaolin. C. A. KING.

Copper-tin-nickel alloy. R. OZLBERGER (E.P. 268,798, 31.3.27. Conv., 3.4.26).—A copper alloy containing about 7.3% Sn and 0.25% Ni. C. A. KING.

Solders for aluminium and its alloys. E. C. R. MARKS. From VERGO ALUMINIUM U. METALLVERWERTUNGS-GES.M.B.H. (E.P. 271,662, 29.6.26).—The solder consists of an alloy containing up to 70% Al together with tin, zinc, copper, iron, silicon, and lithium; *e.g.*, the constituents may consist of 450 pts. of aluminium, 370 pts. of tin, 180 pts. of zinc, 20 pts. of copper, 15 pts. of silicon, 10 pts. of lithium, and 5 pts. of iron. C. A. KING.

Production of corrosion-resistant articles. ALUMINIUM CO. OF AMERICA, Assess. of E. H. DIX, JUN. (E.P. 271,798, 28.3.27. Conv., 22.1.27).—An impure aluminium or aluminium alloy is protected from corrosion by casting it in contact with a plate or casing of corrosion-resistant aluminium or alloy. The latter may be in contact with a mould by which heat is removed at such a rate as to permit only surface fusion of the aluminium. The compound article may be pressed or otherwise hotworked to improve cohesion. C. A. KING.

Manufacture of a light aluminium alloy. V. E. HYBINETTE (E.P. 271,597, 19.3.26. Cf. U.S.P. 1,579,481; B., 1926, 496).—A primary alloy containing 60–75% Al together with nickel and metals of the chromium group is prepared and introduced into molten aluminium to give a final alloy containing not less than 94% Al, 0.5–3% Ni, and 0.1–0.75% of metals of the chromium group. As hardening elements may be introduced 0.1–0.75% Cu (Mn) and 0.1–1% Fe. C. A. KING.

Bright-annealing [muffle] furnace. SIEMENS-SCHUCKERTWERKE G.M.B.H. (E.P. 262,433, 1.12.26. Conv., 3.12.25).—The time taken by the material annealed to cool down is considerably reduced by arranging axially in the muffle a tube, closed at both ends in a heat-insulating manner, which can be opened at both ends while the material is cooling so that air from outside the muffle enters the tube. If desired the tube may be coated internally with enamel. J. S. G. THOMAS.

Process and apparatus for splitting up metal alloys into their various component kinds of crystals by centrifuging. K. SCHMIDT (E.P. 271,293, 10.8.26).—Metal alloys, particularly easily fusible alloys, are introduced into a centrifuge of the basket type, the temperature being so adjusted that one type of crystal present in the alloy is in the solid state while all the remaining components are liquefied. This condition is maintained until the solid and liquid constituents have been separated by centrifuging. C. A. KING.

Furnace for smelting and refining iron, steel, and other metals. P. KÜHN (E.P. 248,012, 23.2.26. Conv., 23.2.25).—In a hearth furnace having regenerators for preheating air for combustion directly connected to each end of the hearth, a rich fuel is injected through openings in the crown directly on to the charge and into the current of air entering from the regenerator. C. A. KING.

Treatment of zinc-bearing ores and metallurgical products. H. W. GEPP, H. HEY, G. RIGG, R. H. STEVENS, and R. T. D. WILLIAMS, ASSTS. to ELECTROLYTIC ZINC CO. OF AUSTRALASIA, LTD. (U.S.P. 1,634,100, 28.6.27. Appl., 30.6.23. Conv., 1.8.22)—See E.P. 201,918; B., 1924, 679.

Heat-resisting alloy. G. R. BROPHY, ASSR. to GENERAL ELECTRIC CO. (U.S.P. 1,633,805, 28.6.27. Appl., 17.6.21).—See E.P. 179,306; B., 1922, 505 A.

Alloy. W. E. RUDER, ASSR. to GENERAL ELECTRIC CO. (U.S.P. 1,633,826, 28.6.27. Appl., 7.6.23. Renewed 29.4.27).—See E.P. 216,389; B., 1924, 637.

Annealing, carburising, bright-annealing, and other heat-treatment furnaces. T. R. SPECK (E.P. 271,918, 22.1. and 14.10.26)

[Sealing means for] electric smelting furnaces. A.-G. BROWN, BOVERI & CIE. (E.P. 263,872, 31.12.26. Conv., 2.1.26).

Annealing metals (E.P. 248,394).—See I.

Rustproofing oils (U.S.P. 1,630,101).—See II.

XI.—ELECTROTECHNICS.

PATENTS.

Electrolytic apparatus and electrodes therefor. W. G. ALLAN (E.P. 271,685, 4.8.26).—In an electrolytic

cell for the decomposition of water, electrodes and their separating diaphragms are made of a sinuous or zig-zag shape, the electrodes being secured mechanically and electrically to opposite cell walls, whilst the diaphragm is supported by a holder carried by the cell ends, so that the total electrode area is large relative to the projected area and the current paths are of large total cross-sectional area. J. S. G. THOMAS.

Electrolytic apparatus and electrodes therefor. J. P. SCOTT (E.P. 271,726, 27.10.26).—In an electrolytic cell for the decomposition of water, using relatively high current densities, sinuous electrodes are arranged longitudinally within the cell, and are supported in co-operating pairs, whilst a pervious diaphragm is arranged between the members of each pair, so that evolved gases are formed in separate compartments and pass out at the top of the cell. J. S. G. THOMAS.

Gaseous electric conduction apparatus [rectifier]. RAYTHEON MANUF. Co., ASSEES. of C. G. SMITH (E.P. 263,108, 15.11.26. Conv., 21.12.25).—An electric discharge device operating by conduction between electrodes within an enclosure contains two gases, one of which, *e.g.*, caesium or other alkali metal or alkaline-earth metal, ionises more readily than the other, *e.g.*, helium, which is preferably inert with respect to the alkali vapour. The more easily ionisable gas is ionised by light radiation from a heated filament for starting purposes. J. S. G. THOMAS.

Incandescence lamp. O. V. MAURER (U.S.P. 1,630,128, 24.5.27. Appl., 17.7.25).—An incandescence lamp containing a tantalum filament is filled with pure argon at atmospheric pressure. T. S. WHEELER.

Electric furnace for heating gases at high temperatures. A.-G. BROWN, BOVERI & CIE. (E.P. 264,849, 20.1.27. Conv., 22.1.26).—Long, narrow, refractory insulating tubes through which the gas to be heated passes, and which contain nickel-chromium heating spirals, are embedded in passages in a refractory stone filling of a steel tube provided with end covers serving as supports, and which carry connexions for the gas inlet and outlet pipes. J. S. G. THOMAS.

Electric arc welding. BRITISH THOMSON-HOUSTON CO., LTD. FROM GENERAL ELECTRIC CO. (E.P. 272,067, 7.8.26).—Methyl alcohol or acetone etc. is dissociated in the arc so as to produce an atmosphere composed substantially of carbon monoxide and hydrogen surrounding the arc and the molten portion of the work. J. S. G. THOMAS.

Electrical resistance thermometer. A. BURSILL, and ELECTROFLO METERS Co., LTD. (E.P. 271,921, 27.1.26).—A resistance element is supplied with alternating current, and is so connected that variations of current due to variations of its resistance cause variations of current in a moving coil disposed in the field of an alternating current electromagnet energised from the same source as the resistance element. J. S. G. THOMAS.

Electrical insulating compound. A. E. L. SCANES, and METROPOLITAN-VICKERS ELECTRICAL Co., LTD. (E.P. 271,979, 22.3.26).—A plastic or fluid electrically insulating material, *e.g.*, material of the nature of pitch,

is intimately mixed with a substance or mixture of substances, *e.g.*, ammonium sulphate, ammonium phosphate, and carbon tetrachloride, which when heated evolve a gas (or gases) which precludes combustion. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 186,861, 3344 of 1906; also in pursuance of Sect. 8, Sub-sect. 2, to E.P. 252,856.] J. S. G. THOMAS.

Treatment of natural fibrous material for use in or as electric insulating material. STANDARD TELEPHONES AND CABLES, LTD. From WESTERN ELECTRIC CO., INC. (E.P. 271,948, 4.3.26).—Fibrous material is freed from extraneous ionisable matter by washing for 1–3 hrs. with water substantially free from such matter at 80–100° and subsequently treating the washed material with a solution of an electrolyte, *e.g.*, calcium hydroxide, which is irreversibly adsorbed by the surfaces of the fibres. J. S. G. THOMAS.

Incandescence lamp. G. R. FONDA, Assr. to GENERAL ELECTRIC CO. (U.S.P. 1,632,647, 14.6.27. Appl., 24.1.25).—See E.P. 246,516; B., 1926, 413.

[Electrode economiser for] electric furnaces. SIR W. G. ARMSTRONG, WHITWORTH & CO., LTD., and E. WINDLE (E.P. 271,739, 12.11.26).

[Contacts for self-baking] electrodes for electric furnaces. NORSKE AKTIESELSKAB FOR ELEKTROKEM. IND. (E.P. 262,722, 3.8.26. Conv., 9.12.25).

Electrolytic [pressure] system [for the generation of gases]. J. E. NOEGGERATH (E.P. 245,119, 18.12.25. Conv., 19.12.24).

[Electrodes for] gas-filled discharge tube. H. WADE. From N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 269,657, 20.1.26).

Selenium and like cells. FOXGROVE MACHINERY CO., LTD., and F. GROVER (E.P. 272,126, 31.1.27).

Treatment of sludge acid (U.S.P. 1,630,074).—See II.

Magnetisable material (E.P. 262,153).—See X.

Sterilising water (E.P. 271,721).—See XXIII.

XII.—FATS; OILS; WAXES.

Separation of liquid and solid fatty acids by Twitchell's method. A. STEGER and H. W. SCHEFFERS (Rec. trav. chim., 1927, 46, 402–408).—The various methods of separating liquid and solid fatty acids by means of the differences in solubility of their metallic salts in organic solvents are criticised owing to the disturbing influence of their mutual solubilities, and, in the case of the salts of bivalent metals, to the formation of mixed saturated–unsaturated salts. The method recommended is Twitchell's modification (A., 1921, ii, 662) of Varrentrapp's lead salt–ether method, using alcohol as the solvent. It is concluded from a large mass of experimental data that the separation is strongly influenced by the nature of the solid fatty acids, stearic acid separating better than palmitic acid, and the latter better than a mixture of equal parts of the two. The nature of the unsaturated acids appears to have much less effect. Unsaturated solid

acids such as elaidic behave mainly as saturated acids with a low m.p., but the separation is less complete.

E. HOLMES.

Addition of iodine to unsaturated oils, fats, and fatty acids in an organic solvent. II. Determination of a mixture of oleic and elaidic acids. III. J. P. K. VAN DER STEUR (Rec. trav. chim., 1927, 46, 409–413, 414–416).—II. In extension of earlier work (B., 1927, 494) a method is developed whereby it is possible to determine the amounts of oleic and elaidic acids in a mixture of the two, higher unsaturated acids being absent. A method of calculating equilibrium constants for various mixtures is given, starting from the values 94.7 and 5.0, at 0°, for oleic and elaidic acids, respectively. These exhibit good agreement with the observed values, and are best shown graphically for the purposes of the determination. Further values are given for the equilibrium constants at 19.5°, oleic acid 26.3, elaidic 2.0, linoleic 17.1, erucic 28.8, and brassidic 1.8, the first two showing the enormous influence of temperature on this constant. III. Equilibrium between solutions of iodine and unsaturated fatty materials in benzene is dependent on temperature. The equilibrium constants of the various compounds examined in benzene solution bear the same relation to one another, but are approximately one third of the values obtained in carbon tetrachloride solution, both at 19.5°. Comparison of the values for peanut oil and its fatty acids shows that the glycerol linkings have no influence on the equilibrium point.

E. HOLMES.

Grape seed oil. J. F. CARRIÈRE and R. CAMPREDON (Chim. et Ind., 1927, 17, 723–728).—The quality and yield of oil depend on mode of collection, degree of maturity of grape, and on climate and soil where cultivated, being 9–12% for fresh pips or 15–17% in the dry state. As 100 kg. of residue (45% of water) arising from the grapes give 20% of pips (40% of water) and 1 hectolitre of wine corresponds to only 12 kg. of residue, it follows that scarcely 200 g. of oil per hectolitre of wine are produced. It is economical to extract the oil on the spot either by crushing or solvent extraction. The residue, after fermentation of the grape juice, is treated in continuous presses to separate the seeds or pips from the pulp, the seeds being then dried until a humidity of only 20–25% is attained. Works operating day and night treat 5600–6000 kg. of seeds per 24 hrs., *i.e.*, extract 7–8 hectolitres of oil. When freshly prepared from seed of good quality the proportion of free acidity in the oil is low (0.44–4.7% for one factory). The residue of the industry, *i.e.*, the spent seed, is an excellent fertiliser containing 2% N, 0.5% P₂O₅, and 0.7% K₂O.

H. M. LANGTON.

Prevention of autoxidation of olein. A. BAG and W. NOWIKOV (Chem. Umschau, 1927, 34, 175–176).—The tendency for olein and various unsaturated fatty oils to undergo autoxidation has been examined by determining, in the Mackey apparatus, the rise in temperature of wadding impregnated with the test liquid. The effect of the addition of small amounts of amines, aldehydes, organic acids, phenols, and alcohols has been determined, and it is shown that, whereas olein and cottonseed oil attain a temperature of 140° in from

1 to 2 hrs., on the addition of 1% of β -naphthol they reach only 100° after 4 hrs. These results have been substantiated on the technical scale. It is also shown that the addition of 1% of β -naphthol to linseed oil prevents polymerisation and oxidation for at least two years, the viscosity and iodine value of the sample being unaltered. The action is regarded as negative catalysis. E. HOLMES.

Cresol-soap systems. II. Gelatinisation tendency of cresol-soap systems. S. JENČIČ (Kolloid-Z., 1927, 42, 168—174. Cf. B., 1927, 494).—Sodium salts of the fatty acids show a pronounced lyophilic character towards cresol in absence of water. At the ordinary temperature the gelatinisation tendency of cresol-soap systems depends on the nature of the fatty acid, rising with increase of mol. wt. (for acids of even number of carbon atoms). The sodium salts of the lower members of the acetic acid series, although crystalloidal in water, give gels in cresol. The gelatinisation tendency of sodium salts of unsaturated fatty acids with 18 or more carbon atoms and 1 or 2 double linkings is lower than that of saturated fatty acids containing the same number of carbon atoms. Sodium ricinoleate possesses the lowest tendency to gelatinise. E. S. HEDGES.

PATENTS.

Bleaching wool fat. I. LIFSCHÜTZ (G.P. 438,292, 14.10.22. Addn. to G.P. 324,667; B., 1920, 790 A).—Products obtained by the partial or complete saponification of wool fat by the process described in the prior patent are bleached by exposure in a finely-divided form to the action of atmospheric oxygen. Bleaching may be accelerated by the addition of other oxidising agents or of oxygen carriers. L. A. COLES.

Deodorising, clarifying, and dehydrating vegetable and animal oils and fats. E. FORAY (Addn. No. 31,090, 2.10.25, to F.P. 601,919; B., 1926, 987).—Sulphuric acid is added to a mixture of the oil or fat with water, zinc dust, and wood charcoal or decolorising charcoal, and when reaction has ceased the aqueous liquor is removed and the fat is washed with water until neutral. L. A. COLES.

Removal of suspended matters from oils and organic solvents. H. HEY (U.S.P. 1,633,941—2, 28.6.27. Appl., [A], 26.11.21, [B], 11.3.24. Conv., 16.12.20).—See E.P. 176,540; B., 1922, 334 A.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Testing the mechanical properties of paint and varnish films. I. H. WOLFF and G. ZEIDLER (Farben-Ztg., 1927, 32, 2135—2136).—An apparatus is described by the use of which the elasticity and tensile strength of detached paint and varnish films may be determined under constantly increasing load, attained by flow of water from an upper to a lower chamber, the film being suspended between them. The apparatus records and shuts off automatically. S. S. WOOLF.

Accelerated paint testing. M. SCHULZ (Farben-Ztg., 1927, 32, 2128—2133).—The functions of the components of accelerated weathering cycles are discussed, and descriptions and photographs of actual cycles in use in various German laboratories are given. In tests

in which the concordance between the results of accelerated and ordinary exposure tests is well illustrated, it is established that, in the protection of iron against rust, red lead-oil primers are superior to nitrocellulose primers, whilst sand-blasting the iron surface decreases the protective action of paint applied thereto.

S. S. WOOLF.

Influence of plasticising on the mechanical-elastic properties of artificial and natural plastic substances. II. Artificial resins. O. MANFRED and J. OBRIST (Kolloid-Z., 1927, 42, 174—180).—The "streaming effect" described in a previous paper (A., 1927, 514) has been further examined. The character of artificial resins is less dependent on their chemical constitution than on their state of dispersity and re-aggregation. Stirring during the process of aggregation has a favourable influence on the orientation of the rod-like particles. The modulus of elasticity of a number of artificial resins has been determined. E. S. HEDGES.

Grape seed oil. CARRIÈRE and CAMPREDON.—See XII.

PATENTS.

Water paint or distemper. C. J. MILLAR (E.P. 271,149, 18.2.26).—On applying one coat of a distemper consisting of a loosely-bound emulsion of water, varnish or oil, an organic agglutinant, and pigments, to absorbent or non-absorbent surfaces, partial separation ensues, an even coating being furnished by one portion, whilst the remainder segregates to form irregular designs of decorative value, the appearance of wall paper being obtained. A preferred composition is:—140 pts. of a casein solution, 140 pts. of varnish, 260 pts. of pigment, and 100 pts. of water. S. S. WOOLF.

Production of low-viscosity lacquer and film. E. M. FLAHERTY, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,629,999, 24.5.27. Appl., 23.5.21).—The viscosity of a solution of pyroxylin in the usual solvents is greatly reduced by allowing it to remain for several weeks with a small quantity of sodium acetate dissolved in methyl alcohol. The resulting solution, though containing 25—30% of pyroxylin, can be used without thinning in the preparation of lacquers, which give films of considerable thickness. T. S. WHEELER.

Manufacture of water-soluble condensation products. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 248,782, 8.3.26. Conv., 9.3.25).—A halogenated simple or mixed aliphatic ketone or ether or a halogenated aliphatic aldehyde is condensed with a phenol in the presence or absence of a condensing agent, and the product, after washing, is rendered water-soluble by treatment with concentrated sulphuric acid. S. S. WOOLF.

Manufacture of condensation products from carbamide or its derivatives and formaldehyde. SOC. CHEM. IND. IN BASLE (GES. FÜR CHEM. IND. IN BASEL) (E.P. 249,101, 1.3.26. Conv., 10.3.25).—Sufficient carbamide is added to the initial water-soluble condensation product of 1 mol. of carbamide with at least 2 mols. of formaldehyde, at any stage of the concentration, to bring the proportion to not less than 1.05 mols. of carbamide to 2 mols. of formaldehyde. The mixture is

converted into the solid hard final condensation product in the presence of an acid catalyst, which may be added at the same time as the additional carbamide or at a different stage of the concentration. S. S. WOOLF.

Manufacture of transparent and colourless condensation products of carbamide and solid polymerides of formaldehyde. F. E. K. STEPPES and H. O. TRAUN (H. TRAUN & SÖHNE) (E.P. 271,264, 22.6.26).—Solutions containing up to 75% of solid matter are obtained by dissolving carbamide in aqueous solutions of formaldehyde of strength exceeding 40% (prepared by the use of reagents which promote dissolution in water of solid polymerides of formaldehyde, *e.g.*, ammonia, alkalis, formates, acetates, sulphites, hydrochloric acid, aromatic sulphonic acids). On boiling these carbamide-paraldehyde solutions for a short while in an open vessel, viscous condensation products are obtained that may readily be hardened by further heating at 100°, the difficulties customarily experienced in the removal of water and the tendency to crack on hardening being obviated. S. S. WOOLF.

Production of a polymerised vinyl chloride modification [β -caouprene chloride]. L. A. VAN DYK (E.P. 255,837, 29.6.26).—If the polymerisation of vinyl chloride by means of ultra-violet rays or sunlight, with or without a catalyst (*e.g.*, a soluble lead salt), be continued beyond the acetone-soluble or α -stage, a new (β -) modification insoluble in acetone but soluble in monochlorobenzene is obtained, before conversion into the insoluble (γ and δ) forms. The β -polymerised vinyl chloride (β -caouprene chloride) may also be obtained from the γ and δ forms by heating them with aniline, quinoline, etc. Monochlorobenzene solutions of β -caouprene chloride to which is added a plasticiser, *e.g.*, a dichlorobenzene etc., yield flexible, transparent, and non-inflammable films on drying. S. S. WOOLF.

Production of a polymerised vinyl chloride [β -caouprene chloride]. L. A. VAN DYK (E.P. 260,550, 26.7.26).—The β -caouprene chloride described in E.P. 255,837 (cf. preceding abstract) is obtainable from the α -compound by prolonged action of ultra-violet rays and/or heat. Vinyl chloride is polymerised in a closed quartz vessel by means of sunlight or ultra-violet rays to α -caouprene chloride, and the unchanged vinyl chloride is distilled off. The α -compound is isolated by dissolution in acetone, and is submitted to further action of the rays or is heated at 50–135° until soluble in chlorobenzene but no longer soluble in acetone. C. HOLLINS.

Filter stones (E.P. 251,964–5).—See I.

Lead carbonate (E.P. 272,053).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Spiral structure of rubber. H. FEUCHTER (Kautschuk, 1927, 98–101, 122–124).—The structure of rubber is regarded as involving the polymerisation of isoprene or dimethyloctadiene into concentrically superposed spherical polyprene shells; the latex globule constitutes a single shell aggregate or co-molecule (cf. B., 1927, 148). It is suggested that each shell possesses residual affinity localised at a point, and that

the attachment of shell to shell occurs by means of these residual affinities, a succession of such bonds over a number of shells following a continuous spiral course. In ordinary rubber the globules are attached to one another by localised but movable surface valencies or co-valencies. The fibrous character of stretched rubber and the possibility of retraction after stretching are explained with the assumption of a reversible anisotropic phase-crystallisation. D. F. TWISS.

Fibre structure and Röntgen interference of stretched synthetic rubber. L. HOCK (Kautschuk, 1927, 125–126).—Contrary to a recently expressed view that so-called “synthetic rubber” is not really rubber because it fails to show the X-ray interferences exhibited by natural rubber, the former under suitable conditions actually does reveal the fibrous character and the interference phenomena characteristic of rubber. D. F. TWISS.

Sulphur and theory of accelerators of vulcanisation [of rubber]. P. SCHOLZ (Kautschuk, 1927, 101–103, 127–128).—Addition of small proportions of various organic accelerators of vulcanisation to molten sulphur expedites the attainment of equilibrium between the allotropic forms of the element. It is assumed therefore that such substances accelerate vulcanisation either by facilitating the conversion of S_λ into more chemically active S_π or S_μ , or that this change in the sulphur induces a parallel transformation of the rubber into a more active form (cf. J.S.C.I., 1921, 48 T). D. F. TWISS.

Ultramicroscopical studies on the theory of vulcanisation. H. DANNENBERG (Kautschuk, 1927, 104–105, 128–130).—The microscope and ultra-microscope are applied to the examination of the behaviour of mixtures of rubber (or of paraffin oil) with sulphur, zinc oxide, and an organic accelerator, when heated and subsequently cooled. When heated with rubber, well dispersed sulphur dissolves without melting. Further heating at 120° causes separation of a colloidal substance, probably S_μ ; this change appears to be coincident with incipient vulcanisation, but the deposition does not continue with longer heating. It is believed that vulcanisation is dependent on the change $S_\lambda \rightarrow S_\mu$, and that vulcanisation accelerators are substances capable of expediting this change. Although the mechanism of vulcanisation may be explained by S_μ being much more chemically active towards rubber, or by the formation of colloidal sulphur inducing “polymerisation” of the rubber, the view favoured is that vulcanised rubber is a dispersion of S_μ in raw rubber. D. F. TWISS.

Production of rubber articles from various preserved [rubber] latices and prevention of the development of tackiness. R. DITMAR (Gummi-Ztg., 1927, 41, 1688–1689).—For the manufacture of cold-vulcanised articles from preserved latex, ammonia is a better preservative than formaldehyde; caustic soda is very unsatisfactory. The undesirable development of tackiness in cold-vulcanised articles on storage can be prevented by soaking in a 35% solution of formaldehyde after vulcanisation, by giving this treatment before vulcanisation and either before or after wiping with benzene, or by freely applying colloidal clay before or after vulcanisation. D. F. TWISS.

PATENTS.

Vulcanised products for use in the manufacture of rubber goods, of paper or paper boards, or for coating fabrics. F. KAYE (E.P. 271,553, 23.2.26).—Emulsified oils or fats are treated with a vulcanising agent, *e.g.*, a sulphide or a polysulphide, or are mixed with rubber latex and then vulcanised. Substances may also be added to accelerate vulcanisation. The products may be allowed to gel, with the aid of a coagulant, *e.g.*, aluminium sulphate, if desired, and then sheeted, with or without the incorporation of fillers. Alternatively, the products may be beaten into paper pulp; a coagulant is then added and the mixture formed into paper or paper board.

D. F. TWISS.

Manufacture of moulded rubber articles from latex. J. MCGAVACK, ASSR. to NAUGATUCK CHEMICAL Co. (U.S.P. 1,629,924, 24.5.27. Appl., 24.3.26).—Rubber latex, with or without vulcanising agents and fillers, is mixed with an aqueous emulsion of pine tar and allowed to set in a mould. The product is dried first at ordinary, and then at higher, temperatures.

T. S. WHEELER.

Thickening and stabilising latex. M. C. TEAGUE, ASSR. to AMERICAN RUBBER Co. (U.S.P. 1,634,124, 28.6.27. Appl., 30.9.25).—See E.P. 235,232; B., 1925, 729.

XV.—LEATHER; GLUE.

Rendering gelatin insoluble. G. E. HOLDEN (J. Soc. Dyers and Col., 1927, 43, 194—196).—About 17.5% of soluble gelatin is converted into an insoluble form by heating at 120° for 8 hrs., the same proportion of insoluble gelatin being obtained when formaldehyde is used as fixing agent. The insoluble gelatin possesses approximately the same empirical composition as the soluble and the original gelatin. Both the insoluble and the untreated gelatin possess approximately the same absorptive powers for tannic acid, but the insoluble form showed increased affinity for basic dyestuffs when applied to cotton in association with tannic acid. It is probable that the altered dyeing properties are due to the conversion of a part of the original gelatin into an insoluble anhydride. The insoluble formo-gelatin compound formed with formaldehyde is decomposed by repeated washings with boiling water, and leaves fixed in an insoluble state approximately the same percentage of gelatin as is produced by the agency of heat. Samples of gelatin used in this work had the composition: C 50.21%, H 6.35%, N 17.72%, ash 1.85% (French); and C 50.42%, H 6.75%, N 17.61%, ash 1.95% (English).

R. BRIGHTMAN.

PATENTS.

Manufacture of tanning agents. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 260,543, 16.4.26. Conv., 28.10.25).—A salt of aluminium is mixed with a salt (other than a nitrite) of an alkaline-earth metal or of another bivalent metal, *e.g.*, zinc or magnesium. Thus, aqueous solutions of aluminium sulphate and magnesium sulphate are mixed, then concentrated to a paste, and the product is dissolved as required for tanning purposes. It produces a white, soft leather.

D. WOODROFFE.

Manufacture of coloured dressings for leather. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 248,767, 5.3.26. Conv., 6.3.25).—A pigment dye (*e.g.*, Hansa Yellow) is intimately ground with a substance of high viscosity capable of gelatinising cellulose esters (*e.g.*, tricresyl phosphate) and combined with a substance not capable of gelatinising cellulose esters (*e.g.*, butyl acetate), the mass being mixed with a celluloid or nitrocellulose varnish.

D. WOODROFFE.

Manufacture of artificial horn. BRITISH GLUES AND CHEMICALS, LTD., R. DUNCALFE, and H. J. COTES (E.P. 271,221, 13.4.26).—A nitrogenous substance (*e.g.*, chrome leather from which the glue-forming matter has been wholly or partially removed) is screened, pressed to reduce the water content, mixed with an absorbent binding material (*e.g.*, saponified resin, casein, calcium or magnesium chlorides) to which an organic acid may be added, and moulded. The product is then treated with a formaldehyde solution, washed, and finally dried.

D. WOODROFFE.

[Pressing devices for use in the] production of artificial horn and goods made therefrom. O. MANFRED (E.P. 269,761, 3.7.26).

XVI.—AGRICULTURE.

Effect of large applications of potassium and chlorine on the growth, leaf colour, and yield of potatoes. Chlorosis as a disturbance of ionic equilibrium in plants. K. MAIWALD (Z. Pflanz. Düng., 1927, A9, 57—98).—The pure salts, potassium chloride and sulphate, sodium sulphate, and calcium chloride, given separately in rather excessive amounts, with and without nitrogen as ammonium nitrate, produced characteristic effects on the growth of potatoes in vegetation experiments. It was clear that with potassium and sodium sulphates the effects were due solely or predominantly to the cations, with calcium chloride solely to the anions, and that with potassium chloride both anions and cations influenced the condition of the plants. Special attention was paid to leaf colour, and the plants were graded according to the depth of colour of the leaves. The classification adopted was shown to accord well with the actual content of chlorophyll, as determined by the method of Willstätter and Stoll. The influence of excess of chlorine ions caused a reduction in chlorophyll content sufficiently great to be classed as incipient chlorosis. Excess of potassium or sodium ions alone effected a reduction in leaf colour, as compared with normal plants, of about 25%, chlorine ions alone about 70%, and potassium and chlorine ions together, about 60%. When, however, ammonium nitrate was given in addition, this reduction in colour was converted into an increase over the normal of 25—35%. The author discusses his results in some detail in relation to the literature on the absorption of ions by plants and their effects, and considers that not only chlorosis, but a whole series of phenomena concerned with plant metabolism, can be attributed to the alteration of the equilibrium between physiologically important ions in the plant. The behaviour of the plants in regard to selective absorption of ions was closely similar to that

observed by Hoagland in experiments with barley in water culture (cf. B., 1924, 143). C. T. GIMMINGHAM.

Conception of the nutrient requirement of a soil and its determination. R. MEYER (Z. Pflanz. Düng., 1927, A9, 99—109).—The necessity for a clearer conception of the meaning to be attached to "the nutrient requirement of a soil" is pointed out. The idea must be considered from an economic point of view, and involves the factor of yield. It follows that Mitscherlich's method is the only one available for an exact determination, since it alone takes yield into account. It is particularly recommended that the yield factor be investigated simultaneously in the same experiment for more than one nutrient. C. T. GIMMINGHAM.

Comparative manuring trials on arable land and pastures. M. POPP, W. FELLING, and R. FLOESS (Fests. Versuchs-Stat. Oldenburg, 1926, 66; Bied. Zentr., 1927, 56, 205—207).—The potash content of hay in pastures is only of value as a measure of the available supplies of potash in the soil when nitrate and phosphate are present in optimum relative proportions. In cropping trials, basic slag gave better results than Algerian phosphate. The difference between the crop increases following the use of these two fertilisers was less with oats than with potatoes. Larger dressings of Algerian phosphate gave greater proportional crop increase with potatoes than with oats. Using similar amounts of phosphate as basic slag, the reverse proved to be the case. A. G. POLLARD.

Action of ammonium sulphate and of sodium nitrate on acid sandy soils. D. MEYER (Mitt. Deut. Landw.-Ges., 1926, 740—742; Bied. Zentr., 1927, 56, 208—209).—Increased soil acidity following the use of ammonium sulphate is not satisfactorily remedied by liming. The use of the nitrates of sodium and calcium as fertilisers on light sandy soils is to be recommended. A. G. POLLARD.

Manuring with phosphates. O. WOLTE and R. LEONHARDS (Mitt. Deut. Landw.-Ges., 1926, 701—706; Bied. Zentr., 1927, 56, 207—208).—Extensive experiments demonstrate the increased need of German soils for phosphate, as an outcome of the shortage of supplies of high-grade basic slag. Deficiencies of phosphate in soils are emphasised during dry periods. In most fertiliser trials adequate supplies of phosphate must be maintained in the soil if reliable results are to be obtained. A. G. POLLARD.

Action of various phosphatic manures. M. POPP and J. CONTZEN (Fests. Versuchs-Stat. Oldenburg, 1926, 48; Bied. Zentr., 1927, 56, 251—254).—Tests of the value of phosphates with and without lime on acid soils are recorded. On sandy soils lime increased the intake of phosphate by pasture grasses. A similar effect on peas was not observed. Crop increases following the use of basic slag on sandy soils were less when lime was used in addition. With Rhenania phosphate the reverse was the case. Colloidal phosphates were more effective on acid soils. Lime decreased the effectiveness of tri- and di-calcium phosphates, the effect being more marked in the case of the former. Lowered crop increases resulted from the use of lime with magnesium ammonium phosphate, the effect being due to changes in soil reaction

rather than to an adverse lime-magnesia ratio. The activity of superphosphate on sandy soils was increased by the use of lime. On moorland soils results were irregular, and frequently the reverse of those obtained on sandy soils. The intake of phosphate by plants appeared to be facilitated on moorland soils. A. G. POLLARD.

Accuracy of the various methods of measuring concentration of hydrogen ions in soil. C. OLSEN and K. LINDERSTRØM-LANG (Compt. rend. Trav. Lab. Carlsberg, 1927, 17, 1—27).—Comparison of the hydrogen electrode, quinhydrone electrode, and colorimetric methods for determination of the p_H of soils, based on the study of about 100 soil samples, leads to the conclusion that measurements made on soil suspensions with the quinhydrone electrode in some cases give a p_H value as much as 0.8 too high. The colorimetric method is also subject to errors, but, in this case, a correction can be made covering both the "indicator" error and the influence of carbon dioxide. A correction curve is given, by the use of which the p_H of soils can be measured with an accuracy of $\pm 0.15 p_H$ unit. This curve is applicable only to soil samples examined in their natural moist condition, the proportion of soil to water being 1:1 by vol., and the mixture being kept for 24 hrs., with frequent stirring before filtration. It is considered that the colorimetric method is to be preferred to the quinhydrone method, and that the accuracy of the determination, though not very great, is sufficient for the purposes of most soil investigations. C. T. GIMMINGHAM.

Total sulphur content of arable soil. G. BERTRAND and L. SILBERSTEIN (Compt. rend., 1927, 184, 1388—1390).—The sulphur content of samples of arable soil has been determined by oxidising a known weight of the soil with fuming nitric acid in an open flask, thereby fixing the volatile sulphur, and finally fusing in a nickel crucible with excess of potassium carbonate and sodium carbonate. The alkali sulphate is then determined with barium chloride in the usual way. Electrical heating must be used, as fusion over a gas burner gives high results. Determinations by this method gave values for the total sulphur varying from 0.202 to 5.175 g./kg. for samples of soil from various sources. Samples of soil from the same district also showed variations, but, in general, districts having the highest sulphur content in the soil were also the most fertile; the use of sulphates as fertilisers is consequently suggested. R. BRIGHTMAN.

Laws governing the porosity of soils and their application to agriculture. W. NITZSCH (Wiss. Veröff. Siemens-Konz., 1927, 5, 96—120).—Starting with the fact that the method of cultivating the ground influences the growth of plants, various physical factors are investigated. It is shown that there is an exact relation between the porosity, heat, and water content, and air permeability. The relation between the capillary and non-capillary components of the porosity are investigated. The water capacity of the soil as a whole is distinguished from that of its components, an increase in the proportion of fine particles increasing the capacity. Practical tests carried out from this point of view show that the results form a satisfactory basis for experiment. The ordinary methods of tilling the soil

leave much to be desired, and further work on these lines is necessary.

C. J. SMITHELLS.

PATENTS.

Composition for destroying weeds. H. E. HUGHES, ASSR. to WEED CONTROL CO. OF CALIFORNIA (U.S.P. 1,629,819, 24.5.27. Appl., 16.9.24).—The composition comprises a mixture of arsenic trioxide, dilute hydrochloric acid, potassium permanganate, and magnesium chloride.

T. S. WHEELER.

Manufacture of arsenate insecticides. H. W. WALKER (U.S.P. 1,629,557, 24.5.27. Appl., 14.5.26).—Arsenic trioxide is heated in presence or absence of water with an excess of precipitated calcium carbonate, and the product, consisting of calcium carbonate coated with calcium arsenite, is heated at 650° in air to oxidise the latter salt to calcium arsenate.

T. S. WHEELER.

XVII.—SUGARS; STARCHES; GUMS.

Sucrose crystallisation. I. A. KUKHARENKO and B. E. KRASIL'SHIKOV (Zapiski [Russia], 1926, 4, 59—67).—A viscosimeter, constructed on the principle of a spherical body falling in a liquid, is described. The readings may be made electrically. The relation between the speed of crystallisation and the viscosity, with varying supersaturation and temperature, is linear. At constant temperature an increase in viscosity is associated with an increased speed of crystallisation; at lower temperatures the viscosity is increased and the crystallisation retarded.

CHEMICAL ABSTRACTS.

Crystallisation of sugar solutions. J. DĚDEK and J. NOVÁČEK (Kolloid-Z., 1927, 42, 163—167).—A method is devised for determining the "crystallisability" of sugar solutions. A slightly undersaturated sugar solution is placed in a desiccator over calcium chloride. After evaporation has proceeded for some time, the solution is inoculated with finely-powdered sugar crystals; crystallisation takes place, and the weight of sugar crystals produced is taken as a measure of the "crystallisability" of the solution. Impure sugar solutions give smaller crystals, and the total weight of crystallised sugar is less than in the case of purified sugar solutions. Direct addition of colloids has the effect of diminishing the size of the crystals and reducing the "crystallisability." The mechanism of the process is discussed.

E. S. HEDGES.

Dry defecation. P. I. FILIP'EV (Zapiski [Russia], 1926, 4, 72—74).—A new type of defecator is described. Sugar does not decompose during dry defecation.

CHEMICAL ABSTRACTS.

Yeast-gum. Y. HASHITANI (J. Inst. Brew., 1927, 33, 347—351).—Yeast-gum is a mannan, and is present in large quantities in yeasts of weak fermenting power. It is a white, amorphous, and hygroscopic substance which does not reduce Fehling's solution, and an aqueous solution of which shows a strong foaming power. It is unacted upon by takadiastase, kashiwagidiastase, "digestine," pancreatin, and invertase, and acetic bacteria and yeast do not grow on it. During autolysis of yeast it can pass out of the cell wall, and is found present in the beer in small quantities. Since the amount of yeast-gum in yeast is generally in inverse proportion to that of

glycogen, it is considered that it does not function as a reserve substance. By depolymerisation of the gum by heating with glycerol, α -yeast-gum is obtained which is attacked by various micro-organisms, and also varies from yeast-gum in regard to its rotatory power, foaming power, and viscosity.

C. RANKEN.

Tragacanth and its mucilage. II. N. EVERS and T. McLACHLAN (Pharm. J., 1927, 118, 746—747).—The deterioration of tragacanth when kept for different periods under varying conditions has been examined. Judged from the strength of the mucilage prepared from it, tragacanth undergoes considerable deterioration on keeping, this being hastened by drying and prevented by keeping in a moist atmosphere. Mucilages made in the cold after keeping for a year have a better suspending power than those made by heating (cf. B., 1924, 802).

E. H. SHARPLES.

Action of bacilli on carbohydrates etc. WOKES and IRWIN.—See XIX.

PATENT.

Carbohydrate compounds (E.P. 264,261).—See V.

Cellulose from bagasse (U.S.P. 1,630,147).—See V.

XVIII.—FERMENTATION INDUSTRIES.

Original gravity [of beers]. R. H. HOPKINS (J. Inst. Brew., 1927, 33, 320—336).—The figures for "degrees of gravity lost" in the Mean Brewery Table from 0.8° Spirit Indication are shown to be below the true values when applied to the determination of original gravity of fully fermented beers. The errors arise as the result of averaging into the table readings taken on partly fermented worts. Other sources of discrepancy between declared and analytically determined original gravities include variations in yeast increase, in loss of alcohol, and in the rate of fermentation among breweries. All other factors being the same, slow fermentations yield beers with higher original gravities than rapid fermentations. By utilising figures published in the Original Gravity Report of Brown and Thorpe, a revised table is drawn up giving more accurate results.

C. RANKEN.

Yeast reproduction in worts of varying original gravities. R. H. HOPKINS (J. Inst. Brew., 1927, 33, 337—346).—Although yeast multiplication is considerably diminished in the latter stages of fermentation, yet it continues in slight degree as long as the fermentation function of the yeast is in operation, unless the oxygen supply gives out. After the cessation of multiplication, the continued increase of the yeast crops is due to the growth in size of the cells. This is especially marked in worts of high gravity. The yeast crops from worts of similar composition, up to an original gravity of 1060°, are approximately proportional to the original gravity. For gravities over 1060° the proportion does not hold.

C. RANKEN.

Influence of brewery water on the composition of wort and beer. V. KOUDELKA (Arhiv Hemiju, 1927, 1, 24—28).—The best water for the manufacture of beer is one the p_H of which is most favourable to the fermentation of wort. Waters rich in carbonates, particularly of magnesium, are unsuitable for breweries,

and cannot be improved by the addition of calcium sulphate.

R. TRUSZKOWSKI.

Microscopical investigation of the dregs of wine as a means of detecting fruit wine. A. WIDMER and O. E. KALBERER (*Z. Unters. Lebensm.*, 1927, 53, 193—208).—An attempt to detect the adulteration of grape wine with that made from apples or pears, by reason of the starch content of the wine dregs, is described. Investigation of fresh grape-must, however, showed that starch is present in this material also, being derived chiefly from the stalks. The number of starch grains visible in the field of the microscope varies within wide limits both for fresh wine and for wine suspected of being adulterated. The shapes and modes of fission of starch grains from both grapes and fruit are very similar, so that distinction between individual grains is difficult. The presence of fruit wine can be suspected when the number of starch grains is exceptionally high and when round single grains of approximately equal size predominate. The suspicion is strengthened if, in addition to the starch, cellular tissue of pears or apples can be detected.

H. J. DOWDEN.

Yeast-gum. HASHITANI—See XVII.

PATENTS.

Fermentation of sugar for the production of yeast and of alcohol. J. P. H. JANSEN (E.P. 270,770, and 271,336, 10.11.25).—Very small quantities of suitable salts, *e.g.*, phosphates, are added with pure culture yeasts to dilute solutions of sugar. (A) If the production of yeast is desired, the fermenting liquid is retained at 25–30°, and aerated by air or oxygen, the yeast being subsequently separated by centrifuging. (B) For the production of alcohol, hydrocyanic acid is added in place of the aeration.

C. RANKEN.

Oxidation process, especially for use in removing iron from wine and other organic liquids. L. E. GRANDCHAMP and J. L. WOLFF (F.P. 613,922, 4.8.25).—Wine is treated with oxydases to convert ferrous compounds into ferric compounds, which are precipitated by the tannic acid in the wine and removed.

L. A. COLES.

XIX.—FOODS.

Chemical composition of wheat. A. SCHHUKIN (*Naukh-Agron. Zhurnal* [Russia], 1926, 3, 379—396).—The hard wheats contain more gliadin and starch than the soft, and less total nitrogen and total protein. The quantity of starch per unit of gliadin is lower in the hard wheat. The size of the kernel within the same variety has little influence on the composition. During ripening the total nitrogen decreases and the amount of gliadin, glutenin, and starch increases. In hot, dry seasons the nitrogenous substances increase and the starch decreases; in wet seasons the reverse is the case. The increase in the concentration of the soil solution influences the composition in general. The gliadin increases as the starch decreases; increase in the starch content causes a decrease of the protein.

CHEMICAL ABSTRACTS.

Detection of neutralised cream. R. STROHECKER (*Z. Unters. Lebensm.*, 1927, 53, 221—227).—The

method of Tillmans and Luckenbach (*cf. B.*, 1925, 967) for the detection of neutralised milk has been applied to cream. Owing to the higher fat content correction is made for the greater volume of the fat phase by conversion of the hydrochloric acid titer of the iron serum (p_H 8.4—3.2) to its corresponding value for the aqueous phase (skim-milk). The acidity of the skim-milk is then read off from the curve given in the text, and from this value the acidity of the cream is calculated by multiplying by the factor $(100-f)/100$, where f is the volume of fat in 100 c.c. of cream. The difference between this value of acidity and the apparent acidity gives the extent to which the cream has been neutralised.

H. J. DOWDEN.

Gelatinisation of fruit pectins. H. LÜERS and K. LOCHMÜLLER (*Kolloid-Z.*, 1927, 42, 154—163).—A method is described for determining the gelatinising power of fruit pectins, and the results obtained with the apparatus are given. The gelatinising power of preparations of equal pectin content rises with increase in the sugar content; conversely, keeping the sugar content constant, the gelatinising power is proportional to the concentration of the pectin. The higher the pectin content, the lower is the concentration of sugar required for gelatinisation. The gelatinising power depends also on the hydrogen-ion concentration, the optimum value being p_H 2.95—3.05. The tendency to gelatinisation falls off slowly on the acid side and rapidly on the alkaline side. Pectins containing less than 7.3% of methoxyl do not gelatinise; gelatinisation occurs most readily when the methoxyl content is 11—12%.

E. S. HEDGES.

Volatile materials of food. J. KÖNIG and W. SCHREIBER (*Biochem. Z.*, 1927, 184, 105—124).—The volatile material obtained by heating various types of food (*e.g.*, flesh, potato, fruit, milk, egg, cauliflower, etc.) at 98—100° and removed in a stream of nitrogen is separated into carbon dioxide, hydrogen sulphide, mercaptan, phosphorus compounds, aldehydes, etc. It is found that the amount of phosphorus compounds runs parallel with the content of vitamins-A and -D.

P. W. CLUTTERBUCK.

Mucous cell content of cacao husks. C. GRIEBEL and A. MIERMEISTER (*Z. Unters. Lebensm.*, 1927, 53, 227—233).—The determination and identification of husk in cacao products has been attempted by measuring the area covered by the mucous cells in a weighed quantity of the material (*cf. B.*, 1926, 605). 1 pt. of husk and 9 pts. of sugar are intimately ground and 5 mg. of the powdered material are spread on a microscope slide with the aid of a few drops of benzene. The slide coating is fixed with collodion and stained with a 0.01% solution of Safranin T, whereby the mucous cells are coloured a bright orange, whilst the remaining tissue becomes deep red. The area of the cells is measured by means of a special counting microscope (*loc. cit.*). The values obtained for husks of the same native origin varied within wide limits. The same procedure was followed with fat-free cocoa powders treated with potassium bromide, and it was found that the variation in the mucous cell value followed very closely the variation in the number of scleridæ. It is possible to detect in a known cocoa powder the addition of a known

variety of husk, but such cases are rare and, in general, measurement of the mucous cell content of a cocoa powder is not so suitable as finding the scleridæ number for determining the husk content. H. J. DOWDEN.

Determination of [finely-ground] cacao husk. J. GROSSFELD (Z. Unters. Lebensm., 1927, 53, 233—236).—It is shown that the determination of husk when very finely ground is extremely difficult by the microscopical method of Griebel and Sonntag (cf. B., 1926, 605), and that the chemical analysis is very little different from that of pure cocoa. Determination of crude fibre and nitrogenous substances by microscopical and analytical methods, in three finely-ground cocoa powders containing husk, gave ratios of nitrogenous substance to crude fibre of 1.0—1.2, which would correspond to pure cocoa husk. Determination of husk from the crude fibre content by the Grossfeld method (cf. B., 1926, 688) gave more satisfactory results. 10 g. of the material were de-fatted by ether extraction and elutriated in the manner previously described. Sedimentation was allowed to proceed for a longer period than for ordinary cocoa powder, and filtration of the residue without suction occupied several days, owing to the colloidal nature of the finely-divided husk. The deposit was dried and the crude fibre determined. The husk content, as determined from the amounts of fibre in the sediment and the original material, ranged from 12.0—15.8%. It is concluded that, although, when very finely ground, part of the husk is hydrolysed and dissolved; this loss is compensated by the tendency of the more resistant portion, which is richer in husk, to collect in the sediment.

H. J. DOWDEN.

Detection and determination of vitamin-A and of vitamin-D in cod-liver oil and various food products. F. WOKES and S. G. WILLIMOTT (Pharm. J., 1927, 118, 752—757).—Zucker's method for the detection of vitamin-D (by its administration producing acid fæces in rats) has been examined and found suitable for quantitative use in assay methods. Attempts to apply the method to children were unsuccessful owing to difficulty in controlling the diet. Colour tests for vitamin-A indicate that the characteristics of true vitamin-blue are typical absorption bands of 590 and 617 $\mu\mu$, and the fading of the blue colour with parallel formation of red or yellow. The specificity of the colour tests has also been examined quantitatively, applying certain laws of physical chemistry, and satisfactory results have been obtained during detailed studies of the destruction of the vitamin by heat and by oxidation. When cod-liver oils are examined with purified antimony trichloride, differences in the stability of the vitamin can be detected by observing difference in the colour changes.

E. H. SHARPLES.

Antimony trichloride and some factors affecting its sensitivity as a reagent for vitamin-A. F. WOKES and J. R. BARR (Pharm. J., 1927, 118, 758—760).—A preliminary account of the properties and factors affecting the sensitivity of antimony trichloride for use in chloroform solutions according to the method of Carr and Price (A., 1926, 870). The following results were obtained. The chloroform should be dried with anhydrous calcium chloride and be free from phosgene,

chlorine, and hydrochloric acid. The antimony trichloride should be recrystallised from chloroform or other anhydrous solvent and stored in a desiccator. Solutions are preferably made in the cold, as heat may cause them to contain free chlorine which apparently accelerates the reaction between antimony trichloride and vitamin-A, thus diminishing the sensitivity of the reagent. This is also affected by the gradual separation of small amounts of a heavy oily liquid which is apparently a solution of chloroform in either antimony trichloride or some similar antimony compound analogous to the solution of water in phenol found in official carbolic acid solutions. The formation of this oily liquid is influenced by the presence of moisture and temperature and by exposure to air.

E. H. SHARPLES.

Use of certain carbohydrates and glucosides in the differentiation of members of the *Salmonella* group of food-poisoning bacilli. F. WOKES and J. H. IRWIN (Pharm. J., 1927, 118, 747—751).—Serological tests of twenty-one members of the *Salmonella* group of food-poisoning bacilli, together with their action on a series of twenty-four alcohols, carbohydrates, and glucosides, are described. The biochemical tests, generally, confirmed the serological classification, and for differentiation the best results were obtained with arabinose, xylose, sorbitol, mannitol, dulcitol, mannose, sucrose, and maltose. Of the glucosides, arbutin showed the greatest differentiation. Æsculin and salicin gave the same results and amygdalin and phloridzin were not acted upon.

E. H. SHARPLES.

Xylene numbers [in butter analysis]. A. VAN RAALTE (Z. Unters. Lebensm., 1927, 53, 236—244).—See B., 1926, 563.

PATENTS.

Flour improver. E. J. SULLIVAN (U.S.P. 1,630,143, 24.5.27. Appl., 26.8.26).—A mixture of monocalcium phosphate (50 pts.), ammonium chloride (25 pts.), calcium sulphate (25 pts.), and ferrous, manganese, or zinc sulphate (0.2 pt.) is used.

T. S. WHEELER.

Preservation of fruit. R. DE O. McDILL (U.S.P. 1,630,129, 24.5.27. Appl., 21.10.21).—Citrus fruit is preserved by coating it with a solution of paraffin wax in a volatile solvent, e.g., petrol, and allowing the solvent to evaporate.

T. S. WHEELER.

Preservation of fresh fruit, vegetables, and like food materials. A. J. H. HADDAN. From F. D. COLLINS (E.P. 271,626, 1.5.26).—The material, dehydrated to 25% of moisture, is chopped and ground, and after a short while is ground more finely and pressed into shapes.

W. G. CAREY.

Method of preserving fruits. W. B. McLAUGHLIN (U.S.P. 1,631,974, 14.6.27. Appl., 25.9.24).—The fruit is mixed with sugar and immediately milled to a fine condition before fermentation occurs, the mixture being maintained at a sufficiently low temperature to prevent fermentation until it is self-preserving.

H. ROYAL-DAWSON.

Preparation of fruit for the market. W. R. BARGER, L. A. HAWKINS, and C. P. BLATZ, Assrs. to PEOPLE OF THE UNITED STATES (U.S.P. 1,632,579, 14.6.27. Appl., 25.1.26).—The fruit is washed in hot

water and subjected to a bath of dilute antiseptic for 10 min. at a temperature of $1\cdot7^{\circ}$.

H. ROYAL-DAWSON.

Transference of vitamins. S. GRÖNNINGSAETER, Assr. to FISCHER HOLLINSHED Co., INC. (U.S.P. 1,629,618, 24.5.27. Appl., 2.11.26).—Cod-liver oil is heated with sodium hydroxide solution and alcohol at 80° for $\frac{1}{2}$ hr., a vegetable oil and water are added, and the mixture is agitated. The greater portion of the vitamins present pass into the added oil.

T. S. WHEELER.

Manufacture of powdered or dried milk products. N. C. CHRISTENSEN (E.P. 244,728, 16.11.25. Conv., 18.12.24).—See U.S.P. 1,574,233; B., 1926, 383.

Means for calculating or determining the required temperature of a liquid or semi-liquid [used in dough-making]. C. DAVIES (E.P. 272,017, 3.5.26).

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

Determination of small quantities of water in alcohol. E. L. SMITH (J.C.S., 1927, 1284—1288).—Sodium or potassium is dissolved in a known volume of the alcohol, dry ethyl acetate is added, and a portion of the mixture is immediately titrated with acid; after keeping at 70° for 30 min., the free alkali liberated by the action of the water in the alcohol on the ethoxide hydrolyses the ester, and the alkali concentration for successive time intervals is determined by titration, the titration at infinite time being determined, when necessary, by extrapolation of the titration-1/time curve. The difference between the initial and limiting titrations multiplied by a factor depending on the concentration of the acid gives the percentage of water in the alcohol. The determination may be hastened and the graphical extrapolation obviated by using considerable excess of alkali metal and of ester. The accuracy is 0.01%.

S. K. TWEEDY.

Dehydration of alcohol. E. L. SMITH (J.C.S., 1927, 1288—1290).—25 g. of ethyl succinate and 7 g. of sodium are added to each litre of alcohol (containing not more than 0.5% of water); after refluxing for 2 hrs. (cf. preceding abstract) the dehydrated alcohol is distilled off through a fractionating column. The formation of ethyl ether does not take place to any measurable extent during the dehydration. The product probably contains less than 0.01% of water.

S. K. TWEEDY.

Attempt to prove the use of alcohol in a case of arson. J. MAULHARDT (Arch. Pharm., 1927, 265, 255—256).—500 g. of the uncharred wood from the fire were submitted to steam-distillation, the distillate being fractionated; 20 c.c. of first runnings, d 0.9972, were obtained which gave no positive test for pyridine. Acetone was identified by the iodoform and other reactions. The solution reduced ammoniacal silver nitrate solution, but ethyl alcohol could not be identified either by Berthelot's ethyl benzoate method or by the iodoform reaction after the acetone had been "fixed." The same results were obtained in control tests on a piece of partially burnt pine wood.

S. COFFEY.

Modified Calvert test for ethyl phthalate. R. D. SCOTT and E. G. WILL (J. Amer. Pharm. Assoc., 1927, 16, 417—419).—The sensitivity of this test depends on the p_H at which the red colour denoting the presence of the ethyl phthalate develops, and a technique is described to adjust it to p_H 11—12, its optimum value. The sensitivity of the test is also increased by diminishing the amount of sulphuric acid, by increasing the amount of phenol, and by using a boiling water-bath instead of a small Bunsen flame.

E. A. LUNT.

Nicotine in tobacco. A. HEIDUSCHKA and F. MUTH (Pharm. Zentr., 1927, 68, 337—345, 353—361, 369—374).—The limit of detection of nicotine is raised by the resins present in the steam-distillate from tobacco, but not sufficiently to affect the quantitative methods. Nicotine was found in the smoke from various German "nicotine-free" and "nicotine-harmless" cigarettes, and in the urine of a non-smoker after indulgence in ordinary and "nicotine-harmless" cigarettes, the chemical tests being confirmed biologically. The method of Rasmussen (A., 1916, ii, 359) is the most exact for the determination of nicotine in tobacco, whilst for comparative analyses the method of Toth as modified by Rundshagen (cf. B., 1926, 214) is recommended. The known methods for the removal of nicotine from tobacco (by evaporation, distillation, extraction, and oxidation) have been examined, and it is concluded that they do not yield a nicotine-free, aromatic tobacco which can be used in manufacture. Proposals for standards for nicotine-free tobaccos are made.

B. FULLMAN.

Colour test for ergot alkaloids. N. EVERS (Pharm. J., 1927, 118, 721—723).—The following preliminary tests based on the colour reaction with a mixture of sulphuric acid and acetic anhydride are described. For *Ext. Ergotæ Liq.*, 2 c.c. of the sample are mixed with 1 c.c. of 10% ammonia solution and shaken with 15, 10, and 5 c.c. of ether. The ether is poured off through a filter and the filtrate evaporated to dryness. The residue is dissolved in 15 c.c. of glacial acetic acid, filtered, and 4 c.c. of the filtrate are mixed with 4 c.c. of 50% sulphuric acid (by vol.). On slowly mixing the two liquids a violet-blue colour is formed which develops to a full strength in about 12 hrs., and can be compared in a tintometer against standard ergotoxine solution. To test a sample of ergot, 2 g. of the powder are shaken for 2 hrs. with 1 c.c. of 10% ammonia solution, 2 c.c. of water, and 40 c.c. of ether. The ether extract is treated as above. Results with a number of extracts are given, and all preparations showing amounts of alkaloid above 0.065% by biological test gave a well-marked colour, but the test was not always reliable in indicating inactive specimens. A negative result with the colour test always indicated an inactive preparation. Experiments with commercial samples of ergotoxine phosphate and ergotinine citrate, the latter giving a similar colour to the former, are described.

E. H. SHARPLES.

Identification and determination of morphine in compound tincture of camphor. C. M. CAINES (Pharm. J., 1927, 118, 751—752).—Arising out of the examination of a sample of compound tincture of camphor from which the whole of the meconic acid had

been precipitated as insoluble calcium and iron meconates, a method for the determination of morphine based on its colour reaction with potassium iodate in acid solution is proposed. Confirmation of the presence of morphine may be obtained by precipitation and microchemical identification, and the method is applicable to determinations of small amounts of morphine in official solutions, pills, ampullæ, and tablets.

E. H. SHARPLES.

Japanese peppermint oil. H. SHINOSAKI, T. NAGASAWA, and H. MAKINO (Rep. Imp. Ind. Res. Inst., Osaka, 1927, 7, No. 15, 1–26).—About 10% of Hokkaido peppermint oil reacts with alkali sulphite; the reacting substance in the fraction of high b.p. is chiefly Δ^1 -menthen-3-one. Menthol obtained from menthenone by reduction with nickel and hydrogen or sodium and ethyl alcohol differs in physical properties from natural menthol. Hokkaido peppermint oil usually contains twice as much menthone and sulphite-reacting substances as Okayama peppermint oil, but the latter is richer (by 10%) in menthol.

CHEMICAL ABSTRACTS.

New constituent of lavender oil. Composition of castoreum. A. S. PFAU (Perf. & Essent. Oil Rec., 1927, 18, 205–206).—(A) Crystals deposited from a sample of lavender oil from Grasse were identified as the methyl ether of umbelliferone (7-methoxycoumarin), m.p. 117–117.5°, which has a noteworthy influence on the odour of lavender oil obtained by extraction. (B) The following compounds have been identified in castoreum: benzoic acid, benzyl alcohol, acetophenone, and *p*-ethylphenol. Other acids and phenols and a lactone having a characteristic odour are present (cf. Schimmels' Bericht, 1927).

E. H. SHARPLES.

Genus *Mentha*. S. B. GORDON (Perf. & Essent. Oil Rec., 1927, 18, 219–222).—A more detailed discussion of the biochemical significance of the presence of γ -methyl-*n*-butyl alcohol in *Mentha piperita* (cf. B., 1927, 346).

E. H. SHARPLES.

Determination of cineole. T. T. COCKING (Pharm. J., 1927, 118, 725–727).—The author's method (B., 1921, 869 A) has been applied to the determination of cineole in commercial camphor oils, and the f.p. of mixtures of *o*-cresol with cineole, camphor, and terpene in varying proportions have been determined. Curves and tables of the results are given, and by their use the cresineol method may be used for the determination of cineole in, e.g., mixtures of cineole and camphor, mixtures of terpene and cineole saturated with camphor, commercial light oil of camphor boiling below 200° or mixtures of similar composition, and crude camphor oil containing high-boiling constituents.

E. H. SHARPLES.

PATENTS.

Production of substantially pure methyl alcohol. E. AUDIBERT (E.P. 271,538, 30.1.26).—A boiling solution of pure copper nitrate or an organic copper salt (free from elements of the iron group) is treated with alkali, the precipitate washed, dried at 50°, mixed with 10–20% of the violet copper obtained by reducing black tetracupric hydroxide at 200° by Sabatier's method, and reduced with hydrogen or carbon monoxide at ordinary pressure, the temperature being kept below 200°. Alternatively, the copper nitrate or other copper salt

is calcined, the oxide obtained being mixed with violet copper and reduced as above. In the presence of the catalyst thus obtained the reaction $\text{CO} + 2\text{H}_2 = \text{CH}_3\cdot\text{OH}$ takes place exclusively at pressures of 100 atm. upwards, the mixed gases entering the catalyst space at 160–180°, and the temperature of the space being kept at not above 400–450°. At 150 atm. upwards several hundred grams of methyl alcohol per litre of catalytic space are produced per hour.

B. FULLMAN.

Manufacture of acetic acid and acetates. SYNTHETIC AMMONIA & NITRATES, LTD., P. A. SMITH, and H. G. SMITH (E.P. 271,589, 13.3.26).—Methyl alcohol vapour is caused to react under hydrogen pressure with sodium formate at 200–300°, potassium formate or water being added to keep the formate in a molten condition. Alternatively, gases tapped from a methyl alcohol synthesis plant containing methyl alcohol vapour, hydrogen, and carbon monoxide react under pressure with caustic alkali or an alkali carbonate at a temperature not exceeding 240°, some liquid phase being continually present. Acetic acid is obtained by distilling the sodium acetate with sulphuric acid.

W. G. CAREY.

Manufacture of glycol ethers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 271,169, 22.2.26).—The condensation of alkylene oxides with monohydric alcohols in presence of sulphuric acid, alkali metal alkoxides, or alkali metal salts of lower aliphatic acids as catalysts is claimed. Examples are glycol monomethyl, monoethyl, and monobutyl ethers from ethylene oxide, and an α -chloro- β - γ -dihydroxypropane monomethyl ether, b.p. 168–170°, from epichlorohydrin.

C. HOLLINS.

Manufacture of esters. I. G. FARBENIND. A.-G. (E.P. 259,204, 27.9.26. Conv., 5.10.25).—The removal of water formed in the interaction of an alcohol and an acid is accomplished by adding a liquid such as benzene, toluene, carbon tetrachloride, hexane, etc. insoluble in water, but capable of forming with water a mixture of lower b.p. than that of either component, and also lower than that of the ternary mixture of alcohol, water, and the added liquid. By carrying out the reaction under a pressure of, e.g., 12–15 atm., it is possible to distil off the water formed in the reaction, together with the added liquid, but with practically no alcohol. The added liquid is separated from the distillate and returned to the mixture. Catalysts such as sulphuric or phosphoric acid may also be added.

A. DAVIDSON.

Production of acid amides and salts. SYNTHETIC AMMONIA & NITRATES, LTD., P. A. SMITH, and H. G. SMITH (E.P. 271,969, 13.3.26).—Hydrogen cyanide is passed with an alcohol vapour (methyl alcohol) over a dehydrating catalyst (alumina, thoria) at a high temperature (for methyl alcohol, 250–300°), preferably under pressure. The reaction is: $\text{R}\cdot\text{OH} + \text{HCN} = \text{R}\cdot\text{CO}\cdot\text{NH}_2$. The acid amide may be hydrolysed with alkali, giving a salt and ammonia. Since the latter may be used with carbon monoxide for the production of hydrogen cyanide, the process may be made cyclic.

C. HOLLINS.

Manufacture of unsaturated aldehydes. H. RUPE (E.P. 267,954, 17.3.27. Conv., 22.3.26; cf. A., 1926, 821).

—Acetylenic alcohols of the type, $\text{CRR}'(\text{OH})\cdot\text{C}\equiv\text{CH}$, are converted by treatment with an acid or an acid derivative (best with formic acid) into ethylenic aldehydes, $\text{CRR}'\text{:CH}\cdot\text{CHO}$. The acetylenic alcohols, prepared usually from acetylene and a sodio-ketone, are warmed with 85% formic acid until reaction begins. cyclo *Hexylideneacetaldehyde* ($\text{R} + \text{R}' = [\text{CH}_2]_5$), b.p. 78–80°/10–11 mm. (semicarbazone, m.p. 200–205°), its 3-methyl derivative and its 2-methyl-5-isopropyl derivative, b.p. 115°/9.5 mm. (semicarbazone, m.p. 140.5°), $\beta\beta$ -dimethylacetaldehyde (semicarbazone, m.p. 244°), and β -methyl- β -ethylacetaldehyde, b.p. 133–134° (semicarbazone, m.p. 170°), are described. C. HOLLINS.

Manufacture of aralkylamines and their derivatives and intermediate products. I. G. FARBERIND. A.-G., Assees. of A.-G. FÜR ANILIN-FABR. (E.P. 249,883, 26.3.26. Conv., 30.3.25).— ω -Halogenated methylphthalimides are condensed with an aromatic compound, and the phthalyl group is removed from the product by hydrolysis. *N*-Chloromethylphthalimide is condensed in presence of zinc chloride with benzene to give benzylphthalimide, which is hydrolysed first with cold aqueous alcoholic alkali to form benzylphthalamic acid and finally with dilute acid to give benzylamine. *m*-Xylene gives 2:4-dimethylbenzylamine, m.p. 218–219° (hydrochloride, m.p. 210°); naphthalene gives *N*- α -naphthylmethylphthalimide, m.p. 175°, and (α -naphthylmethyl)amine; phenol and anisole give, respectively, *p*-hydroxybenzylamine, m.p. 107° (with a little *o*-hydroxybenzylamine, m.p. 129°), and *p*-methoxybenzylamine (*N*-*p*-methoxybenzylphthalimide, m.p. 104°); *o*-nitrotoluene gives 3-nitro-4-methylbenzylamine. The zinc chloride catalyst may be omitted in some cases; e.g., guaiacol and chloromethylphthalimide condense at 120° to *N*-4-hydroxy-3-methoxybenzylphthalimide, m.p. 154°, which is hydrolysed to 4-hydroxy-3-methoxybenzylamine. C. HOLLINS.

Manufacture of aromatic aldehydes. I. G. FARBERIND. A.-G., Assees. of A.-G. FÜR ANILIN-FABR. (E.P. 250,955, 15.4.26. Conv., 15.4.25).—Aralkylamines are condensed with compounds containing reactive halogen (especially 4-chloronitrobenzene-3-sulphonic acid), the secondary amine is oxidised to an azomethine derivative, and the latter is hydrolysed by mineral acid to give an aldehyde. In conjunction with the process of E.P. 249,883 (cf. preceding abstract) this forms a convenient method for introducing an aldehyde group into an aromatic hydrocarbon or derivative, e.g., vanillin from guaiacol. The preparation of 2:4-dimethylbenzaldehyde, anisaldehyde, vanillin, 3-chloro-4-methylbenzaldehyde, m.p. 232°, phenylacetaldehyde, α -naphthaldehyde, b.p. 134°/3 mm., and of the intermediate 4-nitro-1-aralkylamino-benzene-2-sulphonic acids and azomethines, is described. C. HOLLINS.

Preparation of di-iodotrimethylamine. L. GROUCHKINE (E.P. 272,113, 24.12.26).—*s*-Di-iodotrimethylamine, $\text{NMe}(\text{CH}_2\text{I})_2$, is stated to be produced when $\alpha\gamma$ -di-iodohydrin ($\alpha\gamma$ -di-iodo- β -hydroxypropane), obtained by the action of potassium iodide on $\alpha\gamma$ -dichlorohydrin, is treated with excess of 20% ammonia solution and the product heated at 150° for 1 hr. The substance contains 80% of iodine, and, being free from hydriodic and iodic acids, is suitable for subcutaneous or intravenous injection. C. HOLLINS.

Manufacture of chloro-iodo-compounds of 2-aminopyridine and its derivatives. CHEM. FABR. AUF ACTIEN VORM. E. SCHERING (E.P. 264,508, 12.1.27. Conv., 15.1.26).—On addition of iodine monochloride to a solution of 2-aminopyridine in dilute hydrochloric acid

$$\begin{array}{c} \text{CH}\cdot\text{CH}\cdot\text{C}\cdot\text{NH}_2\cdot\text{HCl} \\ \text{an additive compound, probably } \begin{array}{c} \parallel \\ \text{CH}\cdot\text{CH}\cdot\text{NCl} \end{array} \end{array}$$

m.p. 141°, is precipitated. The corresponding 2-acetamidopyridine chloroiodide (m.p. 155°), and the hydrochlorides of 2-ethylamino- (m.p. 90–91°), 2-isopropylamino- (m.p. 84–86°), 2-isoamylamino- (m.p. 50–51°), 2-diethylamino- (m.p. 54–55°), and 2-amino-6-methyl-3-ethyl- (m.p. 105–106°) pyridine chloroiodides are similarly prepared.

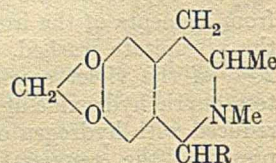
C. HOLLINS.

Manufacture of new compounds of bile acids. SOC. CHEM. IND. IN BASLE (GES. FÜR CHEM. IND. IN BASEL) (E.P. 257,900, 30.7.26. Conv., 2.9.25).—The salts of bile acids with asymmetric acylated aliphatic diamines are superior to the lecithin salts in emulsifying power (e.g., in keeping in colloidal solution the cholesterol and calcium bilirubinate of the bile), produce an increased flow of bile, and have marked antiseptic powers. The diamine derivatives, prepared according to E.P. 203,608 and 219,304 (B., 1923, 1152 A; 1925, 899), are mixed in molecular proportions with the bile acid in the presence or absence of a solvent, or the mixture may be triturated or fused. *N*-Oleyl-*N*'-diethylethylenediamine dehydrocholate, mp. 200°, *N*-isovaleryl-*N*'-diethylethylenediamine cholate, m.p. 90°, *N*-benzoyl-*N*'-diethylethylenediamine cholate, m.p. 99°, *N*-stearyl-*N*'-diethylethylenediamine cholate, m.p. 67°, deoxycholate, m.p. 58°, and apocholate, m.p. 132°, *N*-carbomethoxy-*N*'-diethylethylenediamine dehydrocholate, m.p. 199°, and *N*-stearyl-*N*'-diethylethylenediamine dehydrocholate, m.p. 133°, are described. C. HOLLINS.

Manufacture of mercaptobenzthiazole. W. J. KELLY, ASST. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,631,871, 7.6.27. Appl., 7.8.22).—Aniline is heated with sulphur and carbon disulphide at 150–300° under a pressure of 100–700 lb./sq. in. A. DAVIDSON.

Production of 3-pyridylhydrazine and its derivatives. DEUTSCHE GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (E.P. 259,961, 11.10.26. Conv., 17.10.25).—A 3-aminopyridine is diazotised by adding sodium nitrite solution to a solution of the base in cold concentrated hydrochloric acid, and the diazo compound is reduced with stannous chloride. The double tin salt which separates is decomposed with alkali and the hydrazine extracted with ether. 3-Hydrazinopyridine, m.p. 53–55°, and 2-chloro-5-hydrazinopyridine, m.p. 129–130°, are described. C. HOLLINS.

Preparation of derivatives of methylhydrastinine. CHEM. FABR. E. MERCK, ASSEES. of M. OBERLIN and H. MAEDER (G.P. 438,326, 2.8.25).—Intermediate products for pharmaceutical compounds are obtained by condensing methylhydrastinine in suitable media, usually in presence of alkali (sodium carbonate or sodium ethoxide) with organic compounds containing reactive hydrogen. The products described have the formula:



where R is *o*-nitrobenzyl (m.p. 121–122°; by conden-

sation with *o*-nitrotoluene); 2-nitro-5-methoxybenzyl (m.p. 154—155°); 6-nitro-3:4-methylenedioxybenzyl (m.p. 178—179°); 6-nitro-3:4-dimethoxybenzyl (m.p. 158—160°); 1-phenyl-3-methyl-5-hydroxy-4-pyrazolyl (decomp. 130—135°; from 1-phenyl-3-methyl-5-pyrazolone); 5-keto-1-phenyl-2:3-dimethyl-4-pyrazolonyl (m.p. 200—201°; from antipyrin); acetonyl (m.p. 119—120°; from acetone); α -carbethoxybenzyl (m.p. 111—112°; from ethyl phenylacetate); *o*-nitrobenzoyl (decomp. 155°; from *o*-nitrobenzaldehyde); 6-nitro-3:4-dimethoxybenzoyl (decomp. 139°); 2-nitro-3:4-dimethoxybenzoyl (m.p. 133°); 6-nitro-3:4-methylenedioxybenzoyl (decomp. 143—144°); α -(6-nitro-3:4-dimethoxyphenyl)allyl (m.p. 157—158°); and α -(6-nitro-3:4-methylenedioxyphenyl)allyl (m.p. 132—133°).
C. HOLLINS.

Stabilisation of molecular compounds of pyramidone and butylchloral hydrate. CHEM.-PHARM. A.-G. BAD HOMBURG (G.P. 438,984, 25.7.25).—The addition of 5—10% of hexamethylenetetramine to the molecular compound of pyramidone (4-dimethylamino-1-phenyl-2:3-dimethyl-5-pyrazolone) with butylchloral hydrate prevents its decomposition into a brown oil without affecting its therapeutic value. The mixture is a stable, white powder.
C. HOLLINS.

Preparation of a lecithin derivative. P. BERGELL (G.P. 438,328, 17.2.25).—Fresh, non-resinified lecithin is added to well-stirred anhydrous glycerol (*d* 1.26) at 90°. Solution occurs, followed by swelling to a glassy mass. This is kept for 24 hrs., finely ground in a paste mill, and worked for 1 hr. at 70—90° to form a product which is more stable and less easily hydrolysed than a simple solution of lecithin in glycerol. Mixed with concentrated sugar solutions it remains stable, and may be sterilised by heat. The product is very soluble in chloroform, from which it is incompletely precipitated by acetone, and dissolves in aqueous acetone or in ether. The lecithin is rapidly and completely racemised during the heating in glycerol, and the product is optically inactive.
C. HOLLINS.

Preparation of salts of lipid acids. BEHRING-WERKE A.-G. (G.P. 438,327, 2.11.24).—Yeasts or other micro-organisms, freed from nucleic acids by the process of G.P. 424,657 (B., 1926, 851), or the lipid acids obtained from cells thus treated, are converted into salts by treatment with basic organic dyes, organic bases (including alkaloids), or heavy-metal salts. The products are less irritant and have less tendency to induce fever than metal or dye salts of nucleic acids. Thus, yeast free from nucleic acids is shaken in aqueous suspension with 1% fuchsine solution and kept for 12 hrs. at 15°. Excess dye is washed out and the residue of yeast cells containing the fuchsine salt of lipid acid is dried. The yeast may be similarly treated with a quinine salt, with 3:3'-diamino-4:4'-dihydroxyarsenobenzene hydrochloride, or with mercuric chloride, to form corresponding lipid salts. Cells freed from nucleic acid, but still containing the proteins of the bacteria nuclei, give lipid salts mixed with salts of caryonic acid; the pharmacological properties are the same. The lipid acids isolated by the process of G.P. 424,657 may also be used.
C. HOLLINS.

Production of complex aurothiosulphate com-

pounds. F. J. E. ANDERSEN, I. SIESBYE, and N. J. H. WEITZMANN (DANSK CHEMO-THERAPEUTISK SELSKAB VED ANDERSEN, SIESBYE, & WEITZMANN) (E.P. 261,048, 8.11.26. Conv., 7.11.25).—Aurous bromide or iodide is dissolved in a partly alcoholic solution of the bromide or iodide of an alkali metal, alkaline-earth metal, or of ammonium. The thiosulphate of the same metal is added and, after remaining for some time at 45°, sufficient alcohol is added to precipitate the aurothiosulphate. The products are of use in the treatment of tubercular diseases.
A. DAVIDSON.

Manufacture of mercurimononitro-*o*-cresol and salts. G. W. RAIZISS, ASST. to ABBOTT LABORATORIES (U.S.P. 1,630,072, 24.5.27. Appl., 25.5.23. Cf. E.P. 236,538; B., 1925, 783).—3-Nitro-*o*-cresol is heated at 100° with a dilute acetic acid solution of mercuric acetate for $\frac{1}{2}$ hr., treated with alkali until faintly acid, boiled, and cooled to separate crystalline 4-acetoxymercuri-3-nitro-*o*-cresol, which, on treatment with dilute sodium hydroxide solution, yields crystals of sodium 4-hydroxymercuri-3-nitro-*o*-cresoxide, which is of therapeutic value.
T. S. WHEELER.

Preparation of a concentrated, stable solution of 3-acetamido-4-hydroxyphenylarsinic acid by the acid of its ammonium salt. ÉTABL. POULENC FRÈRES (E.P. 264,797, 3.11.26. Conv., 19.1.26).—3-Acetamido-4-hydroxyphenylarsinic acid is neutralised with 5*N*-ammonia solution; the ammonium salt is precipitated by addition of 2 vol. of alcohol and crystallised from alcohol at 60°. An aqueous solution (1 g. in 2—4 c.c.) containing a little sodium sulphite (0.1 g.) may be sterilised without deterioration.
C. HOLLINS.

Manufacture of organic arsenic compounds. F. LEHNHOFF-WYLD (E.P. 249,515, 15.3.26. Conv., 17.3.25. Addn. to E.P. 232,612; B., 1925, 970).—An alcoholic solution of 1 mol. of 3:3'-diamino-4:4'-dihydroxyarsenobenzene hydrochloride is mixed with a solution, in the same alcohol, of 2 mols. of a metallic salt such as zinc chloride. The solution so obtained is added slowly to a concentrated aqueous solution of 1 mol. of the sodium salt of 3:3'-diamino-4:4'-dihydroxyarsenobenzene-*NN'*-dimethylenesulphonate. After a few hours the mixture is shaken, neutralised with alcoholic sodium hydroxide, and the product precipitated.
A. DAVIDSON.

Manufacture of complex antimony compounds. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 271,940, 1.3.26).—Antimonyl derivatives of *o*-dihydroxybenzenes (e.g., pyrocatechol, gallic acid) react with 1 mol. of a neutral salt of an aliphatic acid containing "easily migratory hydrogen atoms" (e.g., malonic, lævulic, diacetylsuccinic acids) or of an aliphatic hydroxy-acid (tartaric, citric) to form new complex compounds of therapeutic value. Compounds from antimonyl pyrocatechol with sodium malonate, sodium lævulate, and sodium tartrate, and from antimonyl gallic acid with sodium malonate and sodium lævulate are described.
C. HOLLINS.

Manufacture of ureides of dialkyl- or arylalkylacetic acids. F. HOFFMANN-LA ROCHE & Co. A.-G. (E.P. 264,804, 11.12.26. Conv., 20.1.26).—Substituted acetureides, which have hypnotic properties, are obtained

by prolonged action of dilute alkali upon 5:5-disubstituted barbituric acids. 5-Allyl-5-isopropylbarbituric acid, boiled for 100 hrs. with 5% ammonia solution or 16—17% ammonium carbonate solution or for 80 hrs. with 1% sodium hydroxide solution gives α -isopropyl- $\Delta\gamma$ -pentenoylcarbamide, m.p. 194—194.5°. A mixture of sodium hydroxide, disodium hydrogen phosphate, and potassium dihydrogen phosphate may also be used for the hydrolysis, or the neutral calcium salt which may be boiled with water. 5:5-Dialkylbarbituric acid gives in 10 hrs. α -allyl- $\Delta\gamma$ -pentenoylcarbamide, m.p. 156—157°. α -Phenylbutyruide [α -phenylbutyrylcarbamide], m.p. 146—147°, is obtained from 5-phenyl-5-ethylbarbituric acid. C. HOLLINS.

Manufacture of pine extract for medical use. I. DANISCHEWSKY (E.P. 271,555, 23.2.26).—A pine needle extract completely soluble in water and containing approximately 12% of terpenes, pinenes, camphenes, and turpentine, 70% of alcohol etc., and 18% of water (slightly alkaline with sodium hydroxide) is obtained by mixing pine needle oil and alcohol or industrial methylated spirits in approximately equal quantities, and distilling at 80°. To the distillate is added 20—30% of distilled water slightly alkaline with sodium hydroxide, any oil which rises being separated. The liquid may be coloured with fluorescein etc. B. FULLMAN.

Manufacture of stabilised metaldehyde. E. LÜSCHER, Assr. to ELEKTRIZITÄTWERK LONZA (U.S.P. 1,631,875, 7.6.27. Appl., 26.6.22. Conv., 3.12.21).—See E.P. 189,074; B., 1923, 44 A.

Preparation of solutions of compounds [medicaments] insoluble or sparingly soluble in water. W. MINNICH, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,632,306, 14.6.27. Appl., 10.11.24. Conv., 19.11.23).—See G.P. 420,649; B., 1926, 341.

Manufacture of acridine derivatives. I. G. FARBERENIN, A.-G., Assees. of H. JENSCH (U.S.P. 1,629,873, 24.5.27. Appl., 3.9.21. Conv., 8.7.19).—See E.P. 176,038; B., 1922, 347 A, also A., 1922, i, 468.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic ripening nuclei. A. STEIGMANN (Kolloid-Z., 1927, 42, 183—186).—A discussion of the formation and growth of ripening nuclei. In the primary process, the sensitising sulphur compound is adsorbed and then reacts, especially in the warm, to form silver sulphide. In the secondary process, silver halide is reduced by activated hydrogen obtained from the gelatin under the catalytic influence of the silver sulphide nuclei. E. S. HEDGES.

PATENTS.

Preparation of photographic emulsion and material. S. E. SHEPPARD and R. F. PUNNETT, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,623,499, 5.4.27. Appl., 16.6.25).—An emulsion consists of a photographic silver salt suspended in a colloid, to which are added sensitising and reducing compounds upon which part at least of the sensitivity of the emulsion depends. The sensitising compound contains a bivalent atom of the sulphur group directly joined by a double linking to a

single metalloid atom, to which at least another group of atoms is attached. Such compounds include thiocarbamides, selenocarbamides, etc., or the corresponding carbimides. W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Removal of tetranitromethane from [technical] trinitrotoluene. R. H. GÄRTNER (U.S.P. 1,632,959, 21.6.27. Appl., 18.12.24. Conv., 31.12.23).—See G.P. 416,905; B., 1926, 110.

XXIII.—SANITATION; WATER PURIFICATION.

Efficiency of chlorinating sewage tank effluents. W. V. D. TIEDEMAN (Eng. News-Rec., 1927, 98, 944—948).—The method of operating sewage chlorinating plants by setting a fixed minimum dosage to be used throughout the year is inefficient or uneconomical or both. Experiments, with respect to the chlorination of tank effluents from the domestic sewage of Huntington, N.Y., have shown that chlorination on a residual chlorine basis, making use of the *o*-tolidine test to control the chlorine dosage, is to be preferred. Bacteriological examinations extending over a period of 12 months show excellent results when a trace or more of free chlorine is indicated by the test. With residuals of 0.2—0.6 p.p.m., following a 10 min. contact between chlorine and sewage, reductions of 99.9% of bacteria (as evidenced by the 37° agar count) and of *B. coli* were effected. The fine solids in the tank effluents were penetrated by chlorine when a residual of 0.2 p.p.m. or more was maintained and efficient disinfection of the solids resulted. It was also observed that chlorination results in a permanent reduction in the biochemical oxygen demand of the effluent. W. T. LOCKETT.

Removal of iron from a ground water. H. LÜHRIG (Gas- u. Wasserfach, 1927, 70, 621—623).—A town water supply was found to contain iron to the extent of 2.9 mg./litre after passing through the usual purification processes. This was at first attributed to the presence of iron sulphate, but as methods for the removal of this still failed it was concluded that the iron was present in some organic form not readily oxidised by air. This was confirmed by treatment of the water with potassium permanganate (2.5—3 mg./litre), after which the iron was readily removed. A. B. MANNING.

Cresol-soap systems. JENČIČ.—See XII.

PATENT.

Process and apparatus for electrolytically sterilising water. P. M. R. SALLES (E.P. 271,721, 25.10.26).—Water is submitted to electrolytic treatment under high anodic tension in the cold in such manner that ozone is formed at an anode of small superficial area, the cathode being of much larger area. Thus the cathode may be tubular in form, constituting a duct through which water flows, whilst the anode is composed of a platinum or platinum-coated wire arranged along the axis of the duct, the distance between cathode and anode being just sufficient to permit the flow of water and disengagement of the gas evolved. J. S. G. THOMAS.