

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 10, 1927

I.—GENERAL; PLANT; MACHINERY.

Kinetics of catalysed gas reactions in flow systems. A. F. BENTON (Ind. Eng. Chem., 1927, 19, 494—497).—It is assumed that the phenomenon of catalysis is a result of adsorption or of the formation of a superficial layer of an intermediate compound. The variation of adsorption with pressure is given by $s/S = ap/(1+ap)$, where s/S is the proportion of the area covered. When adsorption is small it is therefore proportional to the pressure. In the case of two reacting gases it will be proportional to the product of their partial pressures. If of two gases one is strongly adsorbed $dp/dt = k_1 S p_2 \cdot ap_1/(1+ap_1)$ and the partial pressure of the product is inversely proportional to the rate of passage of the mixture, or the yield is independent of this rate. The divergence from this result in the synthesis of ammonia is explained by adsorption of the product. From these considerations equations are deduced to cover special cases, the effective pressure of each gas being taken to be the mean of the pressures in the entering and exit gases. The catalysis of hydrogen and oxygen by silver is a case where oxygen and water are strongly adsorbed, but not hydrogen. Here it is shown that the yield should vary with the square root of the rate of flow. In the synthesis of ammonia, where neither of the reacting gases is strongly adsorbed, the equation is $xv = 3k_1 S p_1 6ax - k_2 S$, where x is the volume fraction of ammonia and v the rate of gas flow. In the case of the contact sulphuric acid reaction, all three gases are strongly adsorbed. In the conditions met with in practice the yield is $k_1 S/aF - k_2 S/2a$ where F is the fractional conversion of sulphur dioxide. Experimental results are in each case compared with these equations. It is shown that with uniform conditions over the cross-section, the shape of the catalyst mass is immaterial.

C. IRWIN.

Condensation of a gas mixture to form an ideal solution. J. H. SIMONS (Ind. Eng. Chem., 1927, 19, 482—485).—The quantity relations in the condensation of a gas mixture of three or more components can only be calculated on the assumption of an ideal solution. This assumption is nearly true for the liquefaction of air and for the condensation of members of a homologous series, e.g., petrol vapour. A series of equations is developed by which, the vapour pressures of the pure components being known, the dew point and the point of total condensation may be calculated and the composition of vapour and liquid at these or intermediate temperatures ascertained. Composition curves based on calculation are given for the condensation of oxygen, nitrogen, and argon between 85.5° Abs. and 82.3° Abs., the dew

point and point of total condensation at 1.395 atm., respectively. These show good general agreement with the results of experiment.

C. IRWIN.

Automatic devices for the extraction of powdered materials. S. PALKIN and H. R. WATKINS (Ind. Eng. Chem., 1927, 19, 535—537; cf. B., 1925, 566).—Two types of extractor are described in which the solvent flows upwards through the material to be extracted, thus maintaining it in a state of loose suspension and ensuring complete extraction. The first type consists of a boiling tube connected at the top to a reflux condenser, and having near its mid-point a side tube bent downwards to connect with a flask containing the solvent. The filtering medium, which may be absorbent cotton, is tightly sandwiched between the wall of the boiling tube and a cylinder of wire gauze, which latter has a folded groove running from the top to the point of entry of the side tube, to permit free passage of the solvent vapour to the reflux condenser. Down the centre of the boiling tube passes a narrow tube, closed at the lower end by a pad of absorbent cotton and funnel shaped at the top to receive the condensed solvent. The material to be extracted is situated in the annular space between the gauze and the inner tube. When in operation, the condensed solvent flows down the central tube, passes upwards through the solid material, and after filtering through the gauze and cotton, overflows into the heating vessel. In the second type the heating flask is provided with a long vertical tube sealed through the bottom of a wide boiling tube. Over the top of the inner tube is inverted a long test-tube, which rests loosely on the bottom of the boiling tube, in which is contained the powdered material suspended in solvent. Solvent vapour from the flask passes upwards through the inner tube, and, escaping from the bottom of the test-tube, maintains the solid in constant agitation. The filtering and overflow devices are substantially the same as in the first type, and the whole extraction vessel is water-jacketed. A liquid seal inside the heating flask prevents vapour entering the return pipe.

H. J. DOWDEN.

Continuous extraction apparatus. A. H. CLARK (Ind. Eng. Chem., 1927, 19, 534—535).—The apparatus described is of the Soxhlet type, capable of dealing with large quantities of material. The container consists of a pharmaceutical percolator, the bottom of which is closed by a cork and connected to the flask containing the solvent through a straight tube provided with a stop-cock, and having a vertically-directed insertion below the cock. (Alternatively, this side tube may be replaced by a suitably bent tube passing into the solvent flask).

The top of the percolator is closed by a cork or clamped wooden disc and washer, and is connected to the usual reflux condenser through a straight tube having a side tube branching downwards. The two side tubes are connected by rubber tubing or a Liebig condenser, which for less volatile solvents, *e.g.*, alcohol, is used as a steam jacket to prevent undue condensation. When in operation the outflow from the percolator is regulated by the stop-cock to equal the rate of condensation in the reflux. The extraction then becomes continuous, there is no siphoning, with its inherent irregularities, involved, and the operation can be stopped at any moment by closing the stop-cock. H. J. DOWDEN.

Lubricating oils. Laboratory tests in relation to practical results. A. G. MARSHALL and C. H. BARTON (J.S.C.I., 1927, 46, 130—138 t).—The validity of a number of laboratory tests usually applied to lubricating oils was examined by means of engine trials. It was found that oiliness had no practical significance in the lubrication of internal-combustion engines. Oxidation and coking tests of the types at present in use were found to give results which enormously exaggerated the difference between different lubricating oils in those cases in which they were in line with practical results, and in many cases they were found to be blended with residual filtered cylinder stock, with the result of "improving" their tests from the popular specification point of view. Such blending, however, increases the danger due to deposits in service. Evaluation of an oil by means of the rate of change of viscosity with temperature was also found to be misleading.

Glass wool as insulator. BATES.—See VIII.

PATENTS.

Method of furnacing in thermo-chemical treatment. C. B. JACOBS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,625,610, 19.4.27. Appl., 24.2.21).—Charges which shrink during thermo-chemical synthetic reactions are treated until a substantial shrinkage occurs, a further charge is then added before the completion of the original batch, and the augmented batch is treated to completion. W. G. CAREY.

Apparatus for producing refrigeration. Storing device for gas and liquids. F. G. KEYES, Assr. to NATIONAL REFRIGERATING Co. (U.S.P. 1,622,519—1,622,523, 29.3.27. Appl. [A—E], 19.2.20, 11.5.20, 8.6.21, 6.11.23, and 4.12.23).—In a refrigerating system comprising a still, a condenser, and refrigerator proper, with a cooling means used alternately for the still and the condenser, electrical means are used to effect the change over and cut off the heat from the still, which may be controlled by the level of liquid in the condenser or in the refrigerator. Check valves may be used in the pipe connexions between the still and the condenser, and between the refrigerator and still, whilst a capillary tube may be used between the condenser and refrigerator so that liquid may continually pass to the refrigerator while the still is working. In (E) a form of electrically heated still is described. B. M. VENABLES.

Heating granular materials. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (E.P. 268,599, 21.6.26).—

Granular materials such as contact masses, or sulphur and granulated coal for the production of carbon disulphide, are heated uniformly in an electric furnace by forcing gases through them to maintain constant agitation. Steam superheated at 250° thus agitates coke granulated between 1—10 mm. in a furnace heated electrically by steel electrodes, and water-gas is produced continuously.

W. G. CAREY.

Apparatus for detecting and determining impurities and dissolved matter in water and other fluids. EVERSLED & VIGNOLES, LTD., and C. E. PERRY (E.P. 268,597, 18.6.26).—In apparatus, such as the "Dionic Water Tester" described in E.P. 23,706 of 1907 and 12,735 of 1914 (cf. B., 1908, 1036; 1915, 454), in which the impurities and dissolved matter contained in water are ascertained from a measurement of the electrical conductivity of the water, the temperature correction is effected by varying the effective electrical length of the column of liquid being tested, a temperature scale being provided to enable this length to be readily adjusted.

J. S. G. THOMAS.

Separation of constituents of ternary gaseous mixtures. W. WILKINSON, Assr. to AIR REDUCTION Co., INC. (U.S.P. 1,619,909, 8.3.27. Appl., 15.10.25).—A primary liquefaction and rectification separates a liquid rich in the least volatile constituent and a gas rich in the most volatile constituent. Intermediate vapours are withdrawn to another rectifier not in heat communication with the primary one, but the gaseous product of the auxiliary is liquefied by heat exchange with a cold product of the primary rectifier and returned to the auxiliary rectifier. The liquid product from the auxiliary is returned to the primary rectifier.

B. M. VENABLES.

Gas analysis apparatus. R. W. JAMES. From BROWN INSTRUMENT Co. (E.P. 268,637, 3.9.26).—In gas analysis apparatus of the catharometer type in which the relative thermal conductivities of two gases or gas mixtures are compared electrically, the cells in which the gases are respectively received are formed in a single metallic block, *e.g.*, of aluminium, which may be coated with wax or varnish, the block being contained in a metallic housing provided with gas passages, so that temperature differences in the two gases are minimised. The gas passages are provided with filters for cleaning and drying the gases during passage to the respective cells. J. S. G. THOMAS.

Centrifugal separator. T. C. THOMSEN, Assr. to KOEFOED, HAUBERG, MARSTRAND, & HELWEG AKTIESELSKABET TITAN (U.S.P. 1,625,919, 26.4.27. Appl., 9.7.26. Conv., 8.3.26).—See E.P. 259,514; B., 1926, 999.

Separating gaseous mixtures. J. LE ROUGE, Assr. to SOC. L'AIR LIQUIDE (SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (U.S.P. 1,626,345, 26.4.27. Appl., 1.2.23. Conv., 16.3.22).—See E.P. 195,046; B., 1923, 638 A.

[Heating chamber] for gas- or oil-fired furnaces. R. F. HISLOP (E.P. 268,421, 29.12.25).

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

Composition of coal. Resolution of coal by solvents. C. COCKRAM and R. V. WHEELER (J.C.S., 1927, 700—718).—The γ -compounds of bituminous coals (compounds soluble in pyridine and chloroform—6.99% of coal), on extraction with (a) light petroleum, (b) ether, and (c) acetone, are separated into the following percentage fractions: (a) 33, (b) 37, (c) 12, and (d) residue, 18. From a study of the destructive distillation, oxidation with hydrogen peroxide and nitric acid, and ultimate analysis the compositions of the fractions are (a) saturated hydrocarbons, 40%; unsaturated hydrocarbons, 40%; resins, 20%; (b) resinols, resins, and resenes; (c) and (d) resin-like compounds. The treatment of bituminous coal with benzene under pressure at 200° gave an extract amounting to 5.96% of the coal. Fractionation of this extract with the above solvents gave products similar to those obtained by the pyridine-chloroform extraction. At 285° the extract amounted to 7.3% of the coal, but the general characters of the resulting fractions were the same. Tables showing the results obtained from different coals are given.

H. BURTON.

Differentiation of peat and lignite. U. SPRINGER and G. ABELE (Brennstoff-Chem., 1927, 8, 117—120).—Differentiation along geological, botanical, or petrographical lines is unreliable, as is also the physical method of determining the adsorptive power towards a dilute solution of ferric chloride, which varies considerably with the acidity and the fineness of the specimen. The degree of decomposition of the cellulose in the material may be measured by determining the degree of hydrolysis produced by 72% sulphuric acid, but the values found for different types of coal often lie very close together. A simpler and more reliable differentiation is afforded by the pentosan content of the material, which is very rarely less than 2% for peat or greater than 1% for lignite, though even so, intermediate types are inevitably met with.

W. T. K. BRAUNHOLTZ.

Path of travel of the gases in the coke oven. II. and III. W. E. DAVIES (Gas World, 1927, 86, Coking Sect., 2—5, 12—16; cf. B., 1927, 4).—A further discussion of the influence of the plastic layer resistance and the variation of the gas pressure in the hot and cool zones on the flow of gases in the coke oven. The rate of heating of successive layers of material across the oven and the amount of fissuring occurring in the coke layer are important factors in determining the pressure gradients. The work of Foxwell and others is criticised in the light of the author's theory of the mechanism of the coking process. A number of applications of the theory to coke-oven practice are summarised.

A. B. MANNING.

Reactivity of coke. G. AGDE and H. SCHMITT (Brennstoff-Chem., 1927, 8, 121—123).—The differences in reactivity exhibited by different cokes are attributed to somewhat other factors than those described by Nettlenbusch (cf. B., 1927, 208). The reactivity of a coke depends essentially on the accessibility of its carbon, particularly its amorphous carbon, to carbon dioxide, this being determined by the specific number, form, and

diameter of those pores of which the surface is accessible to the gas without unduly prolonged diffusion. The two principal factors in deciding this are the nature of the parent coal and the degree of decomposition of the tar coke (the solid decomposition product of non-expelled tar) corresponding to the carbonising temperature. Whilst graphite may be deposited on coke through decomposition of methane, it is also derived from decomposition of the tar coke at high temperatures. The reactivity of coke, under given experimental conditions, may vary as the experiment proceeds, owing either to partial gasification of the accessible carbon, or to an increase in the quantity of accessible carbon through changes in shape (e.g., due to fracturing).

W. T. K. BRAUNHOLTZ.

Determination of relative ignitabilities and combustibilities of domestic cokes. Tests on the possibilities of a "brazier and weighing method." T. F. E. RHEAD and R. E. JEFFERSON (J.S.C.I., 1927, 46, 166—172 r).—A method for determining the relative ignitability and combustibility of cokes for domestic use, by the continuous weighing of a coke fire in a brazier, has been partially investigated and some of the difficulties have been overcome. The apparatus used is briefly described, and results are given showing the effects of grade of coke, size of fire, and grate area on the rate of combustion.

Determination of the phenol content of gas liquor [crude ammonia liquor] and effluents from coke-oven by-product plants [and gasworks etc.]. H. BACH and H. UTHE (Brennstoff-Chem., 1927, 8, 120—121).—Existing volumetric and gravimetric methods of determining phenol in crude ammonia liquor are briefly discussed. An improved method, in which all sulphur compounds which are oxidised by bromide are completely removed, is to acidify the liquor (100 c.c.) and distil twice to very small bulk, filter the phenolic distillate, and boil gently under a reflux condenser until all hydrogen sulphide has been expelled (about $\frac{1}{4}$ hr.). About 1 c.c. of aqueous barium chloride and two drops of phenolphthalein are added, and then dilute sodium hydroxide in slight excess. The solution is again boiled to aggregate the precipitated barium sulphate and carbonate, and filtered, the phenol in the filtrate being determined with bromine by Koppeschaar's method.

W. T. K. BRAUNHOLTZ.

Vaporisation of petroleum. E. H. LESLIE and A. J. GOOD (Ind. Eng. Chem., 1927, 19, 453—460).—The vaporisation of a paraffin-base petroleum has been studied in an apparatus consisting of a heating coil and an equilibrium chamber in series, immersed in a constant-temperature bath. The liquid was fed at a constant rate of 10 c.c./min. into the heating coil and, on attaining equilibrium, the liquid and vapour phases were collected separately and analysed by a method giving their true b.p. curves. Equilibrium "single-flash" vaporisation of the petroleum was carried out by this method at 250°, 300°, 350°, etc., up to 600° F. The vapours all contained high-boiling compounds and the residues low-boiling compounds. The proportion of the distillate, i.e., the condensed vapour, which boiled below the flash temperature was approximately constant

and averaged 82.3%. In a second series of experiments the crude oil was first vaporised at 250°, the residue then vaporised at 300°, and so on up to 600° F.; this constitutes "successive-flash" vaporisation. It is less efficient than "single-flash" vaporisation in yield and in composition of the distillate, and also in heat requirement. The proportion of distillate boiling below the flash temperature was 80.61%. The results are discussed with regard to their application to petroleum distillation practice.

A. B. MANNING.

Petroleum lubricants. C. F. MABERY (Ind. Eng. Chem., 1927, 19, 526—529; cf. B., 1926, 810).—Determinations have been made of the sp. gr., viscosity, proportion distilling below 300° (30 mm.), and the sp. gr. and viscosity of the distillate and residue, of a number of lubricants from American petroleum. The behaviour of the original lubricant, of the distillate below 300°, and of the residue, in a Carpenter friction-testing machine, has also been studied. In some instances the distillate supports a heavier load than the original, and in others it breaks under a lighter load; the residues show similar variations in stability. The residual hydrocarbons obtained after extraction of the residue above 300° with an alcohol-ether solvent approximate to the series C_nH_{2n-8} for the Pennsylvania oils, and to C_nH_{2n-10} and C_nH_{2n-12} for the others. Two oils subjected to heavy use have been examined and found to have undergone little deterioration. There appears to be no direct relation between the viscosity and the stability under load, oils of the same viscosity showing wide variations in frictional tests. A series of oils of the same viscosity at 38° (320 sec.) showed a maximum difference in viscosity of 12 sec. at 98° and of 27 sec. at 54.4°.

A. B. MANNING.

Action of "antiknocks." A. EGERTON and S. F. GATES (Nature, 1927, 119, 427).—Antiknocks do not influence detonation in rapid combustion mixtures; they function in the initial stages of the combustion as negative catalysts. Those metals which give effective organo-metallic antiknocks are capable of forming higher oxides. Only those types of purely organic substances are effective as antiknocks which retard the oxidation, at the ordinary temperature, of certain liquid aldehydes.

A. A. ELDRIDGE.

Determination of [hard] asphaltum content of mineral oils. J. MARCUSSEN (Chem.-Ztg., 1927, 51, 190).—The methods used hitherto for the determination of asphaltum are neither concordant nor accurate. It is now proposed to use an ethereal solution of ferric chloride as the precipitant. 5 g. of oil are dissolved in 50 c.c. of ethyl ether and 5 c.c. of a 5% ethereal solution of ferric chloride added. The precipitate is filtered off and washed with ether, extracted with boiling ether, and dissolved in warm chloroform. This solution is agitated first with 5 c.c. of dilute hydrochloric acid, then with 5 c.c. of water, and finally, after evaporation to dryness at 105°, the residue is weighed.

H. MOORE.

Valuation of commercial motor spirits by Ostwald's index number method. W. OSTWALD (Petroleum, 1927, 23, 445—448).—The author traverses the criticisms raised by Kroch (B., 1927, 272), and

maintains that the method gives reliable commercial results, even with benzene-benzol mixtures. It is of use as indicating the amount of b.p. depression in alcohol mixtures.

H. MOORE.

Powdered coal for kiln firing. HARTFORD.—See VIII.

Linings for water-gas manufacture. BRADY.—See VIII.

PATENTS.

Coking retort oven. J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,613,068, 4.1.27. Appl., 14.2.21).—Each of the oven walls contains a series of up-flow and a series of down-flow flues which can be interconnected. Two channels communicate with each series of combustion flues, the channels being alternately used to supply fuel gas and air to the up-burning flues, and to receive the waste gases from the down-burning flues.

S. PEXTON.

Process for briquetting coal. S. R. WAGEL, Assr. to LEHIGH COAL & NAVIGATION Co. (U.S.P. 1,618,029, 15.2.27. Appl., 30.9.24).—Coal is bonded with clay, bitumen, and a binding agent which is effective between the temperature at which bitumen loses its binding qualities and that at which the clay becomes sufficiently baked to consolidate the whole.

S. PEXTON.

Binder for briquetting or moulding fuels, minerals, earths, and other finely-divided substances. A. TAPPING (E.P. 263,942, 6.10.25).—An aqueous pulp of starch containing alkali or sodium silicate and a preservative, e.g., cresol or formaldehyde, is mixed with pitch or bitumen of fairly high m.p. in a heated chamber under pressure. The temperature is maintained above the m.p. of the bitumen and the mixture is stirred. The moisture content of the product is regulated, after mixing, by allowing the escape of steam therefrom. The product may be used either in the liquid form, or cooled and the solid binder mixed and ground with the materials to be bonded.

S. PEXTON.

Manufacture of useful products by oxidising coal. W. A. BONE and R. QUARENDON, Assrs. to GAS LIGHT & COKE Co. (E.P. 268,006, 14.12.25).—Coal, or the residue obtained after heating coal under pressure with benzene, is oxidised, preferably at raised temperature, with a solution of caustic alkali, and an alkali manganate or permanganate, at ordinary pressure or in an autoclave. After filtration, the alkali salts of benzenoid carboxylic acids, e.g., mellitic acid, are separated.

W. G. CAREY.

Manufacture of liquid fuels. I. G. FARBENIND. A.-G. (E.P. 252,018, 10.5.26. Conv., 8.5.25. Addn. to E.P. 226,731; B., 1925, 163).—In liquid fuels containing iron carbonyl, the quantity of the latter is reduced by adding organic compounds of metals or metalloids soluble in benzene, e.g., methyl or ethyl borate or silicate, silicon or lead tetraethyl, zinc oleate, etc. The fuel may also contain organic halogen compounds such as ethylene chloride, chloroform, carbon tetrachloride, and/or organic compounds containing oxygen or nitrogen (cf. E.P. 252,019; B., 1927, 357).

W. G. CAREY.

Manufacture of combustible gas. W. D. WILCOX (U.S.P. 1,624,644, 12.4.27. Appl., 1.4.21).—A mixture

of coal- and water-gas is generated by passing steam through coal (contained in two generators) previously raised to incandescence by means of an air blast. The water-gas produced is superheated by passage through regenerators (previously heated by the combustion of the blast gases) and then passes upwards through a charge of fuel, having a layer of uncarbonised coal, to a gas outlet.
C. O. HARVEY.

Treatment of gas containing carbon monoxide. W. H. KNISKERN, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,614,072, 11.1.27. Appl., 21.9.23).—Gas to be purified from carbon monoxide is brought into contact with a catalyst in the presence of steam. The heat of treated gases is passed on to the incoming untreated gases, which are then brought into contact with circulating water, producing steam for the reaction.
S. PEXTON.

Desulphurising coal, water, or mixed gases for illuminating or heating. E. RAFFLOER, Assr. to W. E. LEUCHTENBERG (U.S.P. 1,624,625, 12.4.27. Appl., 3.1.25).—The gas is blown into a desulphurising chamber along with finely-divided material capable of absorbing sulphur.
C. O. HARVEY.

Apparatus for the destructive distillation of oil shale. L. R. ABERNATHY (U.S.P. 1,618,038, 15.2.27. Appl., 1.12.24).—Oil shale is distilled in superheated steam in a retort, dome shaped at the top. Within the dome is a conical hood from the underside of which the products of distillation are withdrawn. The raw shale entering the retort falls on to the outside of the hood, where it is preheated and distributed in the retort.
S. PEXTON.

Decolorising and clarifying agent [for petroleum]. H. S. CHRISTOPHER, Assr. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,617,476, 15.2.27. Appl., 30.9.22).—Pure hydrated aluminium silicate having the approximate composition $\text{Al}_2\text{O}_3 \cdot 12\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ is claimed.
S. PEXTON.

Apparatus for distilling hydrocarbons under pressure and a catalysing agent. F. M. HESS (U.S.P. 1,625,467, 19.4.27. Appl., 29.9.22).—The hydrocarbons to be cracked are vaporised in a still and the vapours passed through a primary dephlegmator and thence to a secondary dephlegmator into which a catalysing agent is introduced.
C. O. HARVEY.

Extraction of bituminous materials. I. G. FARBER, ENIND. A.-G., Assees. of E. REISSMANN and A. RICHTER (G.P. 437,010, 8.11.25).—The fraction of mineral oils, tars, etc. which is soluble in liquid sulphur dioxide is used as an extracting agent; higher yields are obtained than when benzene is used.
A. B. MANNING.

Manufacture of an emulsion. D. FINLEY, Assr. to PARAFFINE COS., INC. (U.S.P. 1,625,304, 19.4.27. Appl., 17.1.23).—Hot bitumen is added slowly with agitation to lime slaked with water, with which is mixed a hot solution of alum, the temperature being maintained.
H. ROYAL-DAWSON.

Treatment of emulsions of hydrocarbon oils and water. STANDARD DEVELOPMENT CO. From C. F. PESTER (Can. P. 247,810, 25.6.24).—The emulsions are treated with $\frac{1}{4}$ –5% of the sludge obtained in refining

mineral lubricating oils with fuming or concentrated sulphuric acid, and the mixture is heated at about 77°.

A. B. MANNING.

Iron carbonyl preparation. BADISCHE ANILIN- u. SODA-FABR. From A. MITTASCH and M. MÜLLER-CUNRADI (Can. P. 262,601, 11.7.25).—Iron carbonyl is dissolved in a hydrocarbon, the solution containing at least 20% of the former. Hydrocarbon substitution products which are inert to iron carbonyl can also be used as solvents.
A. B. MANNING.

Conversion of petroleum hydrocarbons. R. CROSS, Assr. to GASOLINE PRODUCTS Co. (U.S.P. 1,624,778, 12.4.27. Appl., 29.10.23).—The oil accumulates in a pool in an initial stage, and passes thence through a heating stage to a converter which has open vapour communication with the initial stage, whereby the evolved vapours pass through the relatively cooler oil and thence to the dephlegmator and condenser. The unvaporised oil provides energy for producing a turbulent condition in the converter and maintaining the carbon in suspension.
C. O. HARVEY.

Preparation and treatment of olefines. PETROLEUM CHEMICAL CORP., Assees. of H. S. DAVIS and W. J. MURRAY (E.P. 248,375, 24.2.26. Conv., 24.2.25).—The olefine mixtures resulting from the cracking of petroleum vapours at a nearly constant temperature between 600° and 650° are subjected to a physical fractionation in suitable plant of which a diagram and detailed description are given. Three main fractions are collected containing: (1) ethylene and propylene, (2) Δ^a - and Δ^b -butylenes and isobutylene, and (3) Δ^a - and Δ^b -amylenes, isopropylethylene, *as*-methyl ethylethylene, and trimethylethylene, together with hexylenes and higher olefines. The fractions are then selectively absorbed in diluted sulphuric acids of various strengths or in hydrochloric acid, which effects a further separation of their constituents owing to their varying reactivity with acid. The resulting alkylsulphuric acids or alkyl chlorides, on hydrolysis, yield the corresponding alcohols. *E.g.*, from the cracking of "gas oil" ($d\ 0.856$) at 601° there are obtained per barrel (42 gals.) 2.6 gals. of the amylenes fraction. This is agitated with 12.2 lb. of 65% sulphuric acid at below 20° for 6 hrs., allowed to settle, and the lower acid layer separated, neutralised, and distilled, when 0.28 gal. of crude tertiary alcohols (chiefly amyl) is obtained, resulting from the interaction of trimethylethylene, methylethylethylene, and some higher olefines with the acid, and subsequent hydrolysis. The upper layer of residual olefines, containing the amylenes and isopropylethylene, is distilled up to 60°, yielding 1.1 gals. of distillate. This is treated with 15.3 lb. of 77% sulphuric acid at 35–40° for 6 hrs., when the above olefines are absorbed. After settling, the lower acid layer is diluted with water so as to reduce the acid strength below 20%, and distilled to obtain 0.56 gal. of crude secondary amyl alcohols. Similarly, the butylene fraction (102 cub. ft.) obtained from the gas oil is treated with 65% sulphuric acid, which extracts isobutylene etc., and yields, on hydrolysis, 0.28 gal. of tertiary butyl alcohols. The residue, on treatment with 77% sulphuric acid, produces on hydrolysis 0.46 gal. of secondary alcohols formed from the butylenes. A. DAVIDSON.

Pulverised fuel burners. H. A. PROCTER (E.P. 268,417, 30.1.26).

Apparatus for the generation of gas from liquid hydrocarbons for combustion, lighting, and other purposes. R. E. GOLDSBROUGH, and GOLDSBROUGH PATENTS CO., LTD. (E.P. 266,765, 1.9.25).

Heating granular materials (E.P. 268,599).—See I.

Gas analysis apparatus (E.P. 268,637).—See I.

Material similar to compressed asphalt (E.P. 260,621).—See IX.

Bituminous emulsions (E.P. 268,411).—See IX.

Organic substances from gas mixtures (E.P. 262,404).—See XX.

III.—TAR AND TAR PRODUCTS.

Determination of phenol. BACH and UTHE.—See II.

PATENTS.

Separation of the components of low-temperature tar without distillation. ZEICHE M. STINNES, and A. WEINDEL (G.P. 433,455, 5.9.23).—The crude tar is treated with benzene at the ordinary temperature, and, after separation of the precipitated pitch and removal of the benzene by distillation, the residual oil is resolved into neutral oil and phenols by treatment with alcohol of less than 60% strength. The use of caustic soda being avoided and the solvents used being recoverable, the process is economical. A. B. MANNING.

Separation of the phenols and neutral oils of low-temperature tar. ZEICHE M. STINNES, and A. WEINDEL (G.P. 436,444, 22.7.22. Addn. to G.P. 433,268).—The procedure of the principal patent is modified by the use of a mixture of alcohol and a concentrated solution of ammonia, the mixture containing not more than 60% of alcohol for the extraction of the phenols. The solubility of the phenols in alcohol is increased several times by the addition of ammonia, so that extraction is facilitated, and the phenols are recovered directly in a highly pure form. Thus 32% of phenols, purity 98–99%, were recovered by one extraction from a low-temperature tar of which the total phenol content was 40%. The ammonia and alcohol are recovered by distillation without appreciable loss. A. B. MANNING.

Asphalt substitute. J. DRESCHER (Austr. P. 104,402, 4.12.23).—Lignite tar, heated if necessary, is mixed with sulphur, resin, and a mineral filler such as sand or basalt, the mixture brought to 80°, and treated with powdered lime. A. B. MANNING.

Distillation of tar and the like. W. B. DAVIDSON, A. C. MICHIE, and E. W. MUDDIMAN (U.S.P. 1,626,588, 26.4.27. Appl., 23.4.26. Conv., 1.4.25).—See E.P. 255,919; B., 1926, 815.

Bituminous emulsions (E.P. 268,411).—See IX.

IV.—DYESTUFFS AND INTERMEDIATES.

Food dye. H. JOHNSON and P. STAUB (Ind. Eng. Chem., 1927, 19, 497–498).—The green dye known as Fast Green F.C.F., now proposed for admission to the

list of permitted colours, is formed by the condensation of 2 mols. of ethylbenzylanilinesulphonic acid with 1 mol. of *p*-hydroxybenzaldehyde-*o*-sulphonic acid followed by oxidation with lead peroxide. The *p*-hydroxybenzaldehydesulphonic acid is prepared from *p*-nitrotoluene-*o*-sulphonic acid by oxidation in alkaline hypochlorite solution to the corresponding stilbene compound, which is converted into *p*-nitrobenzaldehyde-*o*-sulphonic acid by oxidation with alkaline permanganate. The *p*-nitro-compound is reduced with ferrous carbonate, diazotised, and warmed at 80–90° with dilute sulphuric acid, whereby *p*-hydroxybenzaldehyde-*o*-sulphonic acid results. The dye is superior, both as regards fastness and intensity, to the dyes at present in use. H. J. DOWDEN.

PATENTS.

Preparation of azo dyes. I. G. FARBENIND. A.-G. (F.P. 611,004, 12.2.26. Conv., 13.2.25).—Azo dyes for acetate silk and wool are obtained by coupling diazotised toluidine- ω -sulphonic acids with aromatic amines which contain no sulphonic or carboxylic groups. Examples are: *p*-toluidine- ω -sulphonic acid \rightarrow diphenylamine (yellow), 5-nitro-*o*-toluidine- ω -sulphonic acid \rightarrow benzyl-ethylaniline (red), 3:5-dinitro-*o*-toluidine- ω -sulphonic acid \rightarrow ethyl- β -naphthylamine (blue-violet). The dinitro-compound is obtained by amination of 2-chloro-3:5-dinitrotoluene- ω -sulphonic acid. C. HOLLINS.

Preparation of [azo] dyes containing chromium. GES. FÜR CHEM. IND. IN BASLE (F.P. 609,518, 19.1.26. Conv., 16.2.25).—Azo dyes containing chelate groups are heated with a solution of chromic chromate. Examples are: 4-chloroaniline-3-sulphonic acid \rightarrow salicylic acid (greenish-yellow on wool); 4-chloro-2-aminophenol-5-sulphonic acid \rightarrow 1-phenyl-3-methyl-5-pyrazolone (red); 1-amino-2-naphthol-4-sulphonic acid \rightarrow 1-*m*-nitrophenyl-3-methyl-5-pyrazolone (bluish-red). C. HOLLINS.

Preparation of vat dyes of the anthracene series containing nitrogen. I. G. FARBENIND. A.-G., Assees. of M. KUNZ (G.P. 436,537, 17.2.25).—Amino- and diamino-anthraquinones or their arylidene derivatives, and substitution products of these types, are condensed with aldehydoanthraquinones or their derivatives to give vat dyes. Vat dyes are thus obtained from 2-aminoanthraquinone and 2-aldehydoanthraquinone (orange-yellow) or 1-chloro-2-aldehydoanthraquinone (yellow) or 1-amino-2-aldehydoanthraquinone (claret); from 1-chloro-2-aminoanthraquinone and 1-amino-2-aldehydoanthraquinone (claret-red) or 1-azido-2-aldehydoanthraquinone (salmon-pink product, m.p. 331–333°); from 1-chloro-2-benzylideneaminoanthraquinone and 2-aldehydoanthraquinone (orange-yellow); from 2:6-dibenzylidenediaminoanthraquinone and 2-aldehydoanthraquinone (yellow), or 1-amino-2-aldehydoanthraquinone (claret product, m.p. over 350°), or 1-chloro-2-aldehydoanthraquinone (yellow product, m.p. over 360°); and from 2-benzylideneamino-3-methylanthraquinone and 1-amino-2-aldehydoanthraquinone (pink product, m.p. above 340°). C. HOLLINS.

Preparation of condensation products of the anthraquinone series [isodibenzanthrones]. I. G. FARBENIND. A.-G., Assees. of A. WOLFRAM (G.P. 436,077, 12.8.24. Addn. to G.P. 426,710. Cf. E.P. 255,277; B.,

1926, 780).—3 : 9-Diaroylperylene, heated with aluminium chloride at high temperatures, are converted into isodibenzanthrones. C. HOLLINS.

Preparation of acid wool dyes of the anthraquinone series. I. G. FARBERIND. A.-G., Assees. of W. MIEG (G.P. 436,539, 15.11.24).—The sulphonated (acridinated) products from dianthraquinonylamines are reduced, e.g., with sodium hydrogen sulphide, probably to dihydroacridines with elimination of one sulphonic group. 1 : 1'-Dianthraquinonylamine, after disulphonation with 15% oleum at 95–100°, is reduced by sodium hydrogen sulphide to a compound, which in the presence of bisulphite dyes wool an olive-green, becoming brown on after-chroming. Di- α -anthraquinonyl-1 : 5-diaminoanthraquinone yields a similar dye. From 1 : 2-dianthraquinonylamine by treatment with 15% oleum and subsequent reduction with alkaline sodium sulphide there is obtained a red acid dye; reduction with copper dust and sulphuric acid in place of sodium sulphide leads to a yellow-brown acid dye, giving brown shades on after-chroming. Di- α -anthraquinonyl-2 : 6-diaminoanthraquinone, from 2 : 6-dichloroanthraquinone and 1-aminoanthraquinone, gives, by treatment with 10% oleum at 95–100° followed by reduction with alkaline hyposulphite, a brownish-red acid dye. C. HOLLINS.

Manufacture of vat dyestuffs. M. P. SCHMIDT, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,625,826, 26.4.27. Appl., 20.11.23. Conv., 25.11.22).—See E.P. 207,553; B., 1924, 1008.

Manufacture of dyestuffs containing chromium. F. STRAUB, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,626,167—9, 26.4.27. Appl., 29.1.26. Conv., [A], 14.2.25, [B, C], 16.2.25).—See E.P. 247,556; B., 1926, 910, and F.P. 609,518, preceding.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Constituents of cell-wall of flax fibre. A. E. CASHMORE (J.C.S., 1927, 718–731).—The "cellulose-complex" obtained from flax fibre by freeing the fibre from extraneous matter, from pectin by ammonium oxalate treatment, and from hemicellulose by washing with 4% aqueous sodium hydroxide, had an α -cellulose content of 82–83%. Hydrolysis of the cellulose-complex with 2% barium hydroxide solution at 100° gave galactose together with small amounts of pentoses. With 5% sulphuric acid at 100° practically no hydrolysis took place, but at 130–140° dextrose, galactose, fucose, xylose, and traces of "uronic acid" were produced. Part of the dextrose produced is obtained by the hydrolysis of the non-cellulose portion of the complex. Water at 140–150° hydrolyses the complex yielding a hemicellulose (3.5% of weight of fibre) containing about 21% of "uronic anhydride," probably mainly galacturonic anhydride, 60% of galactose, and 10–14% of rhamnose. In addition, there is obtained a syrup soluble in 70% alcohol (1% of weight of fibre) containing xylose and fucose. At temperatures above 175° water disintegrates the fibre. H. BURTON.

Determination of cellulose by saccharification. A. KIESEL and N. SEMIGANOVSKY (Ber., 1927, 60, [B], 333–338).—Dry cellulose is preserved with 7–10

times its amount of 80% sulphuric acid for 2½ hrs. at the atmospheric temperature. Water is added in the proportion of 15 c.c. for each c.c. of acid used, and the resulting solution is heated for 5 hrs. on the steam bath. Cellulose is thereby converted quantitatively into dextrose. The presence of proteins (gelatin and casein) is without influence on the change. Dextrose, mannose, galactose, laevulose, invert sugar, xylose, and arabinose retained 99.7, 97.7, 99.9, 26.0, 66.06, 72.2, and 84.5% of their reducing power after treatment with acid as above. For the determination of cellulose in botanical material, the substance is subjected to a preliminary treatment with 2% hydrochloric acid for 3–5 hrs. on the water bath, and saccharification of the residue is accomplished as described above, or, alternatively, the process may be applied to the "crude fibre" obtained by one of the customary methods. Direct saccharification leads to correct results only in the absence of ketoses and pentoses. H. WREN.

Sulphite boiling process. M. HÖNIG and W. FUCHS (Ber., 1927, 60, 782–786).—A very marked increase is observed between the consumption of iodine by original sulphite liquors and those which have been preserved in about 1% alkaline solution. The amount of loosely combined sulphur dioxide is greater than that which can be accounted for by the aldehydes and sugars present. Further, the amount of organically combined sulphurous acid in an original sulphite liquor is identical with that of a "fermented" liquor. The sulphur dioxide must therefore be considered to be combined with the ligninsulphonic acid. The presence of an aldehydic group in the latter is improbable since the copper number of carefully dialysed ligninsulphonic acid is negligible. It is probable that the sulphurous acid is united to tautomerically active phenols. H. WREN.

Unsaturated sugar complexes in wood. W. FUCHS (Ber., 1927, 60, 776–782).—The yield of sugars obtainable by the hydrolysis of wood with acids can be considerably increased at the expense of the lignin by suitable pre-treatment of the material with perbenzoic acid, particularly if this has been preceded by the action of 0.5% sulphuric acid at the atmospheric temperature. The increase is due to the more copious presence of fermentable sugars, presumably dextrose, in the solutions from the oxidised wood. The pentosans in the original wood are decomposed to a considerable extent under the conditions of the hydrolysis, and the pentosan content of all the solutions from the wood subjected to the varied treatments is small and almost equal. The observations lend support to the hypothesis of the presence of unsaturated sugar complexes in wood. H. WREN.

Action of sodium hydroxide on cellulose under high pressure. S. ODÉN and S. LINDBERG (Ind. Eng. Chem., 1927, 19, 132–133).—Cotton cellulose was treated with about 7 times its weight of approximately 3.5N-caustic soda solution and heated in an autoclave slowly to 372° and maintained at this temperature for some hours. After removal of gaseous reaction products the residual transparent yellow-brown solution was fractionated to recover methyl alcohol, acetone, and light oil, evaporated to dryness, and extracted with ether to

remove pitch. The residue, consisting of alkali salts of organic acids, was then steam-distilled at 110–585°, and the distillate fractionated under reduced pressure into light and heavy oils and pitchy matter. The reaction products resulting from these operations are tabulated and their quantities recalculated on the basis of 100 g. of pure ash-free cellulose. Ultimate analysis of these products reveals losses equivalent to about 3.25% of the original cellulose. The densities and refractive indexes of the oil fractions are recorded; these oils are highly unsaturated, and are possibly ketonic in character.

D. J. NORMAN.

Determination of soda. TEXTOR and HOFFMAN.—See VII.

Use for hydrocellulose. GARDNER.—See XIII.

Cellulose ester varnishes. DABISCH.—See XIII.

PATENTS.

Production of stiff fabrics which stand washing.

A.-G. CILANDER (E.P. 264,783, 1.3.26. Conv., 21.1.26).—Artificial silk is woven with vegetable or animal fibres and the resulting fabric exposed for a short time, *e.g.*, 15 secs., at about 15° to the action of an inorganic acid, *e.g.*, sulphuric acid (*d* 1.53). The strength of the acid, the duration of the treatment, and the proportion of artificial silk in the fabric determine the degree of stiffness.

D. J. NORMAN.

Manufacture of fibre board. R. W. HILTON, Assr. to KEMPER-THOMAS Co. (U.S.P. 1,624,599, 12.4.27. Appl., 1.11.23).—Weather-resistant fibre board is made by thoroughly mixing fibre pulp in an aqueous solution of soap and adding lead acetate to precipitate insoluble lead soap, the amount of soap being sufficient to provide not over 3% of insoluble soap in the fibre.

W. G. CAREY.

Manufacture of strawboard. M. E. PENNINGTON and A. B. DAVIS (U.S.P. 1,625,090, 19.4.27. Appl., 18.4.25).—The strawboard comprises disintegrated new straw which has been disinfected and steam distilled, rendering it inodorous and sterile.

H. ROYAL-DAWSON.

Waterproof and like materials. C. HÄFELE (E.P. 266,813, 3.12.25).—Two or more webs of fibrous material are compounded by means of a sulphur-free rubber solution containing approximately 10% and 5%, respectively, of finely-powdered zinc oxide and barium sulphate. The composite web is then satinised and dried by passing it between rollers at 140–160°.

D. J. NORMAN.

Protection of animal fibres against attack by alkaline liquids. M. BERGMANN (G.P. 437,836, 14.10.23).—Sulphite-cellulose waste liquors in which the active groups have been oxidised, reduced, or halogenated, or the active constituents of the liquor after they have been subjected to one of these processes, are used for protecting the fibres during treatment with alkaline liquids.

L. A. COLES.

Manufacture and use of artificial silk and the like. BRITISH ENKA ARTIFICIAL SILK Co., LTD., Assees. of N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (E.P. 244,496, 15.12.25. Conv., 15.12.24).—The affinity for dyestuffs of artificial silk consisting of or containing cellulose silk, *e.g.*, viscose, may be increased by treatment with an oxidising agent, to increase the content of oxycellulose,

or decreased by treatment with liquids which, whilst having a solvent action on some or all of the non-cellulose constituents, have no appreciable action on cellulose. For example, the affinity for dyestuffs of viscose may be increased by exposure for about 2 hrs. at the ordinary temperature to a solution of sodium hypochlorite containing 5% of available chlorine, or decreased by heating for about 24 hrs. at 150° with glycerin. D. J. NORMAN.

Manufacture of artificial threads or filaments.

SOC. POUR LA FABR. DE LA SOIE "RHODIASETA" (E.P. 259,190, 31.8.26. Conv., 1.10.25. Addn. to E.P. 233,384; B., 1925, 587).—In a modified process for the dry spinning of artificial threads involving the use of temperature-controlling elements, as described in the prior patent, the controlling elements are connected in parallel, and in order to obtain different temperatures in different parts of a cell more than one element may be used, the temperature-controlling fluid to one of them being cooled if necessary.

B. P. RIDGE.

Treatment of cellulose acetate or products made therewith. BRITISH CELANESE, LTD., and G. H. ELLIS (E.P. 266,777, 29.10.25).—Pattern or other effects

are produced on textile fabrics made from or containing cellulose acetate threads or yarns by the local application (by printing, stencilling, etc.) to the fabric of one or more substances which modify the delustering action of hot or boiling aqueous media or moist steam, and afterwards subjecting the material to such delustering treatment, preferably with moist steam. The applied substances may be mechanical resists, solvents for cellulose acetates, or substances soluble in cellulose acetate, those of the last two groups which are not sufficiently soluble in water being either dissolved in liquids which are non-solvents for cellulose acetate, or dispersed colloiddally in aqueous media. The delustering may be prevented, retarded, or accelerated by the agents used, and the effects may be further enhanced by dyeing the fabric with suitable dyestuffs.

B. P. RIDGE.

Non-inflammable plastic masses. PATHÉ CINÉMA, ANC. ETABL. PATHÉ FRÈRES (F.P. 612,414, 30.6.25).—Cellulose esters or ethers are mixed with the phosphoric esters of halogen-substituted aliphatic alcohols and the usual plasticisers and highly chlorinated compounds. *E.g.*, 1 kg. of nitrocellulose is mixed with 400 g. of trichloroethyl phosphate and 400 g. of a plasticiser (tricresyl phosphate etc.), and the whole dissolved in a volatile liquid. The mixtures serve for the production of artificial fibres, plastic masses, films, etc.

B. FULLMAN.

Manufacture of coated laid paper. G. W. JOHNSON. From A. M. COLLINS MANUFACTURING Co. (E.P. 267,013, 13.9.26).—Ordinary paper is coated preferably on both sides with, *e.g.*, a solution of casein or glue, optionally in admixture with fillers and colouring agents, and is then passed between embossing rollers which impress a laid pattern on the coating. The lines thus formed are blended with the main coating while this is still moist in order to give the effect of a watermark in the body paper. The sheet may, if desired, receive a second plain coating, and is finally rolled to produce an even smooth surface suitable for high-grade printing.

D. J. NORMAN.

Manufacture of a waterproof [paper] composition. L. KIRSCHBRAUN (U.S.P. 1,621,791, 22.3.27. Appl., 20.10.20. Renewed 8.10.26).—Molten asphalt mixed with oleic acid is emulsified by agitation with water containing colloidal clay, and the emulsion is incorporated with a suitable paper or other fibrous stock, and treated with a substance, *e.g.*, lime, which precipitates oleic acid as an insoluble oleate. The treated stock is then passed through a papering machine and dried. T. S. WHEELER.

Manufacture of artificial silk and the like from viscose solutions. W. P. DREAPER (U.S.P. 1,626,454, 26.4.27. Appl., 18.4.25. Conv., 2.5.24).—See E.P. 239,254; B., 1925, 915.

Solvents for cellulose ester (E.P. 255,406).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Mordanting and dyeing. V. [Mechanism of the weighting of silk.] E. ELÖD, L. TEICHMANN, and E. PIEPER (Z. angew. Chem., 1927, 40, 262–264).—The isoelectric point of a Japanese silk in the raw state was p_H 5.1, and after weighting with tin silicophosphate, 4.1. A similar displacement of this point is obtained by prolonged treatment first in hydrochloric acid then in sodium hydroxide solutions equipotential with the solutions used in the commercial weighting process. These results appear to confirm Elöd's theory of the mechanism of this process (B., 1924, 939; 1926, 316). A. R. POWELL.

Action of iron blacks on leather. WOODROFFE and DEW.—See XV.

PATENTS.

Dyeing wool and silk. I. G. FARBENIND. A.-G. (F.P. 610,308, 1.2.26. Conv., 7.2.25).—Even shades on wool and silk mixtures are obtained by dyeing with azo dyes derived from pyrazolones and containing in either component one or more carbalkoxy-groups.

C. HOLLINS.

Dyeing leather with acid azo dyes. I. G. FARBENIND. A.-G. (F.P. 609,904, 23.1.26. Conv., 3.2.25).—Brown shades on leather are produced by using dyes of the type: amine \rightarrow resorcinol \leftarrow aminonaphtholsulphonic acid, or amine \rightarrow resorcinol \leftarrow aminonaphtholsulphonic acid \leftarrow amine. In place of resorcinol an unsulphonated derivative may be used, and in place of the aminonaphtholsulphonic acid a naphthylaminesulphonic acid.

C. HOLLINS.

Dyeing and printing of cellulose esters. I. G. FARBENIND. A.-G. (F.P. 610,539, 3.2.26. Conv., 6.3.25).—Sulphamic acids derived from coloured amines, especially from aminoanthraquinones, are dyed on cellulose acetate silk from an acid bath containing sodium sulphate. The sulphamic acid from 1:4-diaminoanthraquinone gives a violet-red, that from 4:8-diaminoanthrarufin a reddish-blue. For printing, the sodium sulphamate is mixed with glycerol and a thickener.

C. HOLLINS.

Calico printing process. I. G. FARBENIND. A.-G., Assees. of E. MÜNCH and K. H. MEYER (G.P. 435,092, 10.2.25. Addn. to G.P. 433,153; B., 1927, 216).—The

hydroxymethylformamide of the earlier patent is replaced in part or entirely by formamide. Formamide is a good solvent for dyes, especially for basic dyes, and this property is enhanced by addition of resorcinol. The mixture enables basic dyes to be fixed by steaming without addition of tannin or other fixing medium.

C. HOLLINS.

Pattern printing on cloth with the help of wax. F. ADLER (Austr. P. 104,395, 9.10.25. Conv., 7.11.24 and 13.7.25).—The material to be printed is laid on a flat bed of wax and then printed with a heated metal templet carrying the pattern, so that the wax under the printed portions melts and penetrates the material. The wax may be moulded on a cylinder and the pattern carried on a cylindrical printing roller. A waxed cloth of suitable length may be used in place of solid wax. The material to be printed should be stretched at a short distance above the wax bed so that after printing it frees itself from the reserving layer and adhesion is prevented.

C. HOLLINS.

Preparation of padding-baths. I. G. FARBENIND. A.-G. (F.P. 610,519, 2.2.26. Conv., 3.2.25).—Aromatic or partly hydrogenated aromatic sulphonic acids or their alkyl, aralkyl, or alkylaralkyl derivatives, or salts of these, are added to padding baths containing aniline salt, β -naphthol, 2:3-hydroxynaphthoic arylamides, etc. in order to increase the penetrating power of the solution, the resulting dyeings being thus rendered faster to washing and rubbing. Suitable sulphonic acids are those derived from benzylated naphthalenes, or the condensation of naphthalenesulphonic acids with cyclohexanol, benzyl alcohol or chloride, or tetralin dichloride, or of tetralinsulphonic acid with cyclohexanol.

C. HOLLINS.

Dyeing [acetylcelluloses]. P. RABE, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,622,122, 22.3.27. Appl., 11.4.24. Conv., 9.5.23).—See E.P. 215,783; B., 1925, 801.

Bleached and dyed furs (E.P. 264,969).—See XV.

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

Solubility of calcium phosphates in citric acid. J. GRAFTIAU (Bull. Soc. chim. Belg., 1927, 36, 165–171). Experiments in which a number of naturally occurring calcium phosphates were treated with a 2% solution of citric acid, initially in strict accordance with the requirements of the Wagner method, the treatment being then repeated on the extracted material till dissolution was complete, show that under these conditions the whole of the phosphoric acid is finally brought into solution. The rate of dissolution is markedly influenced by the state of aggregation. In general, the argillaceous phosphates of Liège and limestone phosphates require 4–5 successive extractions. Florida phosphates, land pebble, and hard rock, are much more resistant, and 8–11 successive extractions are necessary for the complete dissolution of the phosphoric acid. In the case of limestone phosphates application of the Wagner method leads to results which are difficult to interpret, since a not inconsiderable portion of the citric acid is immediately

neutralised by the calcium carbonate present. A specimen of limestone phosphate which yielded only 13.5% of its total phosphate content on a first treatment with a 2% solution of citric acid, yielded 40.8% and 31.1%, respectively, during the second and third treatments.

J. S. CARTER.

Determination of soda by electrical conductivity.

C. K. TEXTOR and W. F. HOFFMAN (Paper Trade J., 1927, 84, 201—205).—The method may be applied to cooking liquor, black liquor, washed pulp, and leached black ash. Although it is empirical, there is a definite relation between the total soda content and the specific conductivity of the liquors and effluents under the uniform conditions prevailing in any given mill.

CHEMICAL ABSTRACTS.

Potash from greensand. II. Adsorption from the vapour phase by glaucosil.

C. W. WHITTAKER and E. J. FOX (Ind. Eng. Chem., 1927, 19, 467—469).—Glaucosil is the siliceous residue left after the extraction of greensand with acid. It is grey in colour, white after ignition, and is more active chemically than crystalline silica. Adsorptions of benzene, xylene, water vapour, and carbon tetrachloride were determined from the pure vapours, and the total percentages by weight taken up were found to be 40.5, 31.0, and 61.0, respectively, for the three organic vapours mentioned. Water is also strongly adsorbed, and can be driven off only by heating at 200—250°. Adsorption-time curves are given showing a gradual fall from 100% to zero. In some cases initial low efficiencies were found when starting the apparatus, ascribed to a phase of preliminary activation.

C. IRWIN.

Determination of thiocyanates in the presence of chlorides, sulphides, and cyanides. Z. CHARATZ (Chem.-Ztg., 1927, 51, 251).—The crude potash of beet vinasses often contains potassium thiocyanate (Italian samples up to 1% or more), which may be thus determined: 20 g. of the potash are dissolved in water, hydrochloric acid is added to neutrality or feeble acidity, the liquid boiled, and diluted to 200 c.c. In each of two identical 25 c.c. vessels are placed 0.5 c.c. of 0.4% ferric sulphate solution and 1 c.c. of 1:2 hydrochloric acid. The first is then filled with water, and the second treated with 5 c.c. of the test solution and filled with water. The blank is then titrated with 0.5% potassium thiocyanate solution until the colours are identical. 0.04% of thiocyanate in the crude substance may be thus determined. The presence of more than 0.1% of ferrocyanide is disturbing, but the difficulty is avoided by working at high dilution; chlorides and sulphides have no effect. Using ammonium thiocyanate, the thiocyanate in ammonium sulphate may be thus determined. Cyanides are determined after conversion into thiocyanate by treatment with ammonium sulphide.

B. FULLMAN.

Volumetric determination of carbon dioxide in carbonates. J. LINDNER and F. HERNLER (Z. angew. Chem., 1927, 40, 462—464).—The material is decomposed with 4—10% hydrochloric acid in a slow current of air free from carbon dioxide, and the issuing gases are passed through an absorption tube in which is placed a definite volume of 0.1*N*-barium hydroxide containing 1% of

barium chloride and 0.3 c.c. of a 0.1% solution of phenolphthalein as indicator. Without filtering off the precipitated barium carbonate the excess of barium hydroxide is titrated with 0.1*N*-hydrochloric acid containing 3% of barium chloride. A compact apparatus for carrying out the determination without exposure of the solutions to the atmosphere is illustrated diagrammatically.

A. R. POWELL.

Ratio of sulphur trioxide to phosphoric anhydride [in acid phosphate].

J. B. THOMAS and C. C. HOWES (Amer. Fertiliser, 1926, 65, [8], 28—30).—The most economical ratio is determined by consideration of the cost of the acid in relation to the curve obtained by plotting the ratio of sulphur trioxide to phosphoric anhydride against the percentage availability of the phosphate. The method is not applicable in the manufacture of acid phosphate from rock, which differs greatly in composition from that used in establishing the ratio.

CHEMICAL ABSTRACTS.

Kinetics of catalysed gas reactions. BENTON.—See I.

Detection of mercury vapour. NORDLANDER.—See X.

Cyanides in the blast-furnace. KINNEY and GUERNSEY.—See X.

Liquids for mineralogical analyses. SULLIVAN.—See X.

Determination of phosphoric acid in fertilisers. BORDEIANU.—See XVI.

PATENTS.

Production of concentrated solutions of alkali cyanides. DEUTSCHE GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER, and K. ANDRICH (E.P. 268,420, 23.12.25).—Hydrocyanic acid gas is absorbed, preferably below 15°, in an aqueous mixture of alkali sulphate and lime, starting with a quantity of alkali sulphate insufficient to cause precipitation of a double salt of alkali sulphate and calcium sulphate (in the case of sodium sulphate not more than 28%), and continuing the admission of hydrocyanic acid until some calcium cyanide is formed, which is transformed into alkali cyanide by the addition of more alkali sulphate. Excess of alkali sulphate is avoided by adding it in portions, at intervals, so that calcium cyanide is formed in the intervals.

W. G. CAREY.

Manufacture of potassium nitrate. A. LAMBERT (E.P. 267,996, 18.11.25).—Sylvite, carnallite, kainite, or other material containing potassium chloride is partially purified from sodium chloride by concentrating a solution, which is then mixed with a solution of caliche similarly purified; the mixture is further concentrated by evaporation to separate further sodium chloride, and the potassium nitrate formed by the double decomposition is crystallised.

W. G. CAREY.

Manufacture of ammonium nitrate in water solution and simultaneous concentration thereof. Rapid evaporation to dryness of ammonium nitrate solutions. AZOGENO S.-A. PER LA FABR. DELL'AMMONIACA SINTETICA E PROD. DERIVATI, and

C. TONIOLO (E.P. 247,227—8, 8.2.26. Conv., 7.2.25).—(A) Nitric acid diluted with ammonium nitrate solution is neutralised with gaseous ammonia mixed with air heated at 80—100°. The heat developed by the reaction is thus utilised to evaporate some of the water contained in the acid. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 6048 of 1915 and 11,731 of 1888.] (B) Molten ammonium nitrate, still containing some water (10—20%), is sprayed over a mass of pulverised, cold or hot ammonium nitrate, and a current of cold or hot air is introduced at the particular time at which the molten salt incorporates itself with the pulverised salt. W. G. CAREY.

Preparation of an alkaline, iron-treated carbon suitable for cyanide synthesis. J. MICHAEL & Co. (G.P. 435,975, 6.3.24).—Porous carbon is impregnated with a solution containing an alkali carbonate and a complex iron salt (sodium ferrocyanide, sodium iron tartrate, etc.) which gives no precipitate with alkali carbonate. Good yields of cyanide are claimed, using smaller proportions of iron than usual. C. HOLLINS.

Decreasing the rate of crystallisation of borax from brine. A. W. GAUGER and H. H. STORCH, Assrs. to BURNHAM CHEMICAL Co. (U.S.P. 1,621,927, 22.3.27. Appl., 29.8.25).—The crystallisation of borax during the evaporation of brine is inhibited by the addition of 0.05—0.1% of rosin, or of the extract obtained by treating coniferous sawdust or the bush *Larrea mexicana* with sodium carbonate solution. T. S. WHEELER.

Manufacture of rouge. P. J. HESS, Assr. to PITTSBURGH PLATE GLASS Co. (U.S.P. 1,618,086, 15.2.27. Appl., 1.2.21).—Ferrous sulphate is heated at 870—940° to drive off the water of crystallisation and to convert it into a compound consisting of approximately 2 pts. by wt. of ferric oxide to 1 pt. of ferric sulphate. This compound is mixed with water to form a polishing mixture. H. ROYAL-DAWSON.

Production of oxides. F. THARALDSEN (Nor. P. 40,618, 29.1.23).—Material to be oxidised is heated below its fusion point under oxidising conditions, mixed with a flux and reducing agents and fused, and then the oxidation is completed. L. A. COLES.

Manufacture of lead nitrate. W. H. BENTLEY, B. CATLOW, and W. BLYTHE & Co., LTD. (E.P. 268,104, 24.2.26).—Metallic lead is alternately lowered into and raised out of a lead nitrate solution, the moist lead being oxidised by a current of air and the film of oxide dissolved by the solution. Nitric acid is added to the liquor as required, keeping it neutral or slightly basic, and solid lead nitrate is obtained by crystallisation, after slightly acidifying a portion of the liquor. W. G. CAREY.

Regeneration of catalysts used in the production of phosphorus pentoxide or phosphoric acid by the interaction of phosphorus or phosphoretted hydrogen and water vapour. I. G. FARBERIND. A.-G. (E.P. 259,201, 20.9.26. Conv., 29.9.25).—The catalysts are regenerated in the reaction vessel by suspending the inflow of raw material and passing hydrogen containing a small amount of oxygen through the heated vessel. W. G. CAREY.

Production of hydrogen. E. EDWIN (F.P. 612,238, 8.10.25. Conv., 17.10.24).—Gases containing carbon dioxide, obtained in the reduction of iron oxide formed by treating iron with steam for the production of hydrogen, are reduced by passage through a high-tension electric arc and returned to the process. L. A. COLES.

Manufacture of moulded sulphur. RHENANIA VEREIN CHEM. FABR. A.-G. (E.P. 265,540, 30.6.26. Conv., 3.2.26).—Moulds of aluminium or its alloys are employed and are cooled with water or air. The length joints are provided with flanges so arranged that the plastic or elastic filling between them does not come into contact with the molten sulphur. W. G. CAREY.

Causticising units or apparatus. W. D. MOUNT (E.P. 265,669, 12.8.25).—See F.P. 603,190; B., 1927, 187.

Gas analysis apparatus (E.P. 268,637).—See I.

Heating granular materials (E.P. 268,599).—See I.

Hydrogen and oxygen (E.P. 268,426).—See XI.

VIII.—GLASS; CERAMICS.

Glass annealing. J. W. FRENCH (J. Soc. Glass Tech., 1927, 11, 10—19).—Conditions of importance in annealing are (1) heat emissivity of the surface at all temperatures, (2) thermal expansion, (3) thermal conductivity, (4) cohesion, (5) viscosity, (6) surface layer, (7) homogeneity, (8) form and dimensions. The problem, in general, does not lend itself to mathematical treatment. Much evidence supports the theory that glass is always coated with an amorphous surface layer with properties differing from those of the underlying material, and the layer yields with great readiness to small impact forces. The annealing process is discussed and two types of lehr designed for annealing optical glass are described. A. COUSEN.

Stress systems and photo-elastic phenomena [in glass]. F. W. PRESTON (J. Soc. Glass Tech., 1927, 11, 23—29).—Photo-elastic phenomena can only be used successfully in studying the stress system in glass when restricted to two-dimensional cases, namely, by using plane parallel laminæ with stresses confined to the plane of the laminæ. With equal stresses at right angles, the ordinary polariscope indicates no strain, and, for accuracy, an instrument showing change of absolute refractive index parallel to each principal stress is needed. A. COUSEN.

Economics of the [glass] annealing process. E. A. COAD-PRYOR (J. Soc. Glass Tech., 1927, 11, 30—36).—The temperature of containers straight from machines on entering the lehr ranged from 580° to 778°. An insulated lehr was designed such that the heat carried in by the ware was practically sufficient to maintain the annealing temperature. For safety, a small amount of additional heat was supplied by preheating the slats of the belt. A. COUSEN.

Investigation of the glassy state by the method of enforced crystallisation. J. F. PONOMAREV (J. Soc. Glass Tech., 1927, 11, 39—52; cf. B., 1926, 877).—The m.p. diagrams of the following systems were determined by the method of enforced crystallisation: (1) $\text{Na}_2\text{O}, 2\text{B}_2\text{O}_3\text{—B}_2\text{O}_3$, (2) $\text{Na}_2\text{O}, 2\text{B}_2\text{O}_3\text{—}3\text{CaO}, \text{P}_2\text{O}_5$, (3) $\text{Na}_2\text{O}, 2\text{B}_2\text{O}_3\text{—Al}_2\text{O}_3$, (4) $\text{Na}_2\text{O}, 2\text{B}_2\text{O}_3\text{—MgO}$, (5) Na_2O ,

$2\text{B}_2\text{O}_3\text{--CaO}$, (6) $\text{Na}_2\text{O}, 2\text{B}_2\text{O}_3\text{--ZnO}$, (7) $\text{Na}_2\text{O}, 2\text{B}_2\text{O}_3\text{--PbO}$. From its physico-chemical characteristics glass is defined as a strongly under-cooled fusion which, on crystallising from its molten mass, gives crystals of the same chemical composition as the mass itself.

A. COUSEN.

Transparent zirconia-containing glasses. V. DIMBLEBY, S. ENGLISH, E. M. FIRTH, F. W. HODKIN, and W. E. S. TURNER (J. Soc. Glass Tech., 1927, 11, 52—64).—Transparent glasses were obtained in which soda of the parent glass $6\text{SiO}_2, 1.7\text{Na}_2\text{O}, 0.3\text{CaO}$ was molecularly replaced by zirconia in amounts up to 8% of the glass. A similar percentage of zirconia was also introduced into soda-magnesia-silica glasses. Series of glasses in which soda of the glass $6\text{SiO}_2, 2\text{Na}_2\text{O}$ was successively replaced by 0.5 to 4 mols. of zirconia melted at temperatures from 1400° to 1450° and worked at $1150\text{--}1350^\circ$. High proportions of the oxide gave viscous glasses with a high rate of setting, and with increase of zirconia both annealing temperature and density increased. Similar results were obtained when silica on the parent glass was replaced by zirconia, and the glass $5\text{SiO}_2, \text{ZrO}_2, 2\text{Na}_2\text{O}$ melted at 1500° , worked at 1350° , was very viscous, and set rapidly. The cubical thermal expansion constant for zirconia is 6.9×10^{-8} , and the oxide is exceptionally valuable for producing resistance towards water, hydrochloric acid, and alkaline reagents.

A. COUSEN.

Function of arsenic in potash-lead oxide-silica glasses. S. ENGLISH, E. M. FIRTH, and W. E. S. TURNER (J. Soc. Glass Tech., 1927, 11, 65—76).—To a normal crystal glass batch containing potassium nitrate, arsenious oxide was added in amounts up to 50 pts. per 1000 of sand, three types of batch being used with moisture contents (1) below 1%, (2) 1—2%, (3) 4%. Generally, 80—90% of the arsenic was retained, of which about 80% was in quinquevalent form, and these values were independent of moisture content. Progressive addition increased the rate of melting, but a maximum fining rate was reached with 20—30 pts. per 1000 of sand. With 2—5 pts. of arsenious oxide per 1000 of sand the most favourable melting and fining results occurred with 1—2% of moisture, with 10 pts. or more per 1000 variation of moisture did not affect melting and fining rates. Glasses from batches with 4% of moisture were the most viscous. The results with regard to retention of arsenious oxide applied also with lead crystal-phosphate opal glasses.

A. COUSEN.

Insulation of ceramic kilns. S. E. JOSI and A. E. HUBBARD (Trans. Ceram. Soc., 1926, 25, 185—199).—The principles of heat insulation are outlined briefly, and the advantages of insulating kiln walls and crowns are pointed out. In tunnel kilns, insulating powder 9—12 in. thick is used over the crown, and a layer of insulating bricks 4—6 in. thick is built into the walls. The method of insulating periodic kilns is described and illustrated.

F. SALT.

Application of powdered coal as a tunnel-kiln fuel firing hard-fired common brick. F. M. HARTFORD (J. Amer. Ceram. Soc., 1926, 9, 684—689).—Slack coal, ground in a ball-mill to pass 30-mesh, is passed with air to a distributing fan, so constructed that a

mixture of powdered coal with about 30% of the air required for complete combustion is drawn over and distributed in equal parts to 10 burners. The stream of air and coal enters the rear of the burner, and is met by a horizontal stream of air for complete combustion.

F. SALT.

Specifications for lining and checker brick for water-gas manufacture. E. J. BRADY (J. Amer. Ceram. Soc., 1926, 9, 667—678).—Tentative specifications for the purchase of refractories for the water-gas industry are presented, together with details of the process and equipment used in the industry, and tests prescribed in the specifications.

F. SALT.

Refractory materials of South Wales. W. R. D. JONES (Proc. S. Wales Inst. Eng., 1927, 43, 115—140).—The occurrence, preparation, properties, and uses of silica, fireclay, and dolomite refractories of South Wales are discussed. The silica material is obtained from the basal grit of the millstone grit series; the average silica content is about 95%, with 0.2—1.5% of alumina. The fireclays are obtained from the Lower Coal Series and the Pennant Series; the silica content is 60—68%, and the alumina 20—25%.

F. SALT.

Spalling of bricks. F. W. PRESTON (J. Amer. Ceram. Soc., 1926, 9, 654—658).—Norton's theory of spalling (B., 1925, 17) is criticised. The initial fissure in a typical case of spalling enters the brick normally to the isothermals, at right-angles to the tension, and makes its exit tangentially at other edges. Further, the isothermal planes, which are parallel to the face of the solid, are lines of principal stress, and therefore of zero shear. Once formed (at right-angles to the isothermals), the fissure advances in a direction at right angles to the lines of principal tension; i.e., the direction is related simply to the stress distribution at a given moment, but not to the temperature distribution. Diffusivity has no effect on the spalling tendency, but, on the other hand, the time elapsing between quenching and spalling is a function of the diffusivity.

F. SALT.

Effect of grain-size of fluxes and of non-plastic materials on the cone melting point of clays.

H. SALMANG (Ber. Deut. Keram. Ges., 1926, 7, 100—109).—Refractory tests were carried out on mixtures of kaolin and quartz; kaolin and felspar; kaolin, quartz, and felspar; and kaolin and potash mica. The quartz, felspar, and mica were ground, and elutriated by decantation in glass cylinders into five fractions, varying in average grain-size from 2.3μ to 150μ . Two series of mixtures were prepared by adding these materials to kaolin to the extent of 25% and 50%. The tests were carried out at two different rates of heating, viz., 15° and 5° per min. The effect of reducing the grain-size of the fluxes and quartz on the m.p. was most marked at the rapid rate of heating; in every case the slower heating tended to minimise this effect. In mixtures containing 50% of kaolin, 25% of felspar, and 25% of quartz, the m.p. was reduced from cone 31 to cone 27 by reducing the grain-size from 400μ to 2.3μ .

F. SALT.

Effect of zinc oxide on the colouring properties of chromium, cobalt, and iron oxides. D. S. PILLAI (Trans. Ceram. Soc., 1926, 25, 209—219).—Experiments

were carried out with three sets of triangular mixtures: cobalt, chromium, zinc; cobalt, iron, zinc; and chromium, iron, zinc. In this way 46 colour mixtures were obtained. The effect of various glazes on these mixtures was studied. Satisfactory colours were obtained from nearly all the mixtures, and they were suitable as under-glaze, on-glaze, and in-glaze colours, and as stains. Zinc oxide improved the cobalt blues, and formed a good range of browns, chocolate, and brownish-yellows with iron oxide. Chrome green is gradually destroyed by zinc owing to formation of zinc chromate. The green tints are improved, and the browns maintained with a magnesia glaze. F. SALT.

Use of opacifying media containing antimony in the enamel industry. H. HAUPT and G. POPP (Z. angew. Chem., 1927, 40, 218–221).—Two “quinquevalent antimony compounds,” sold under the trade names of “Leukonin” and “Timonox,” were examined in order to determine whether, during the process of manufacturing cooking utensils, the antimony was reduced to the toxic trivalent form, and whether this was soluble in boiling liquids containing organic acids. Solubility in a boiling 3% solution of tartaric acid was determined on the two compounds separately in powder form, and on frits made from them and melted at 1400°. Enamelled ware was also prepared by adding the compounds (a) to the frit, and (b) to the mill mixture, and tested in 1% tartaric acid, 4% acetic acid, 1% potash solution, and water. Domestic conditions were also simulated by the use of boiling sauerkraut and apple sauce. Leukonin was practically free from trivalent antimony, whereas Timonox contained this form only. The above methods of preparing Leukonin enamels had little effect on the solubility; a small proportion of the quinquevalent antimony is always reduced to the trivalent form, traces of which go into solution with tartaric acid. The quantity, however, is so minute as to be hygienically harmless. On the other hand, Timonox is quite unsuitable for the manufacture of domestic ware. F. SALT.

Resistance of enamel to attack by mineral acids. A. KERSTAN (Sprechsaal, 1926, 59, 708–709; Chem. Zentr., 1927, I, 342).—Enamel is attacked most readily by hydrochloric acid and nitric acid when the acids are of 25% and 35% strength respectively. Hydrochloric acid attacks the borates and part of the alkalis; nitric acid dissolves principally sodium oxide, potash enamels being far more resistant to its action. L. A. COLES.

Glass wool as insulator for refrigeration purposes. H. C. BATES (J. Amer. Ceram. Soc., 1926, 9, 690–692).—The insulating properties of glass wool of various densities, powdered cork, magnesia-asbestos composition, slag wool, Sil-o-Cel powder, cotton waste, and sheep's wool were determined by observing the rate at which ice melted in a vessel insulated with these materials. The change in volume of a mixture of ice and water was observed by means of a graduated tube leading from the vessel. The most effective packing for glass wool was that corresponding to a density of 0.065 g./c.c. The various conductivity values are tabulated, showing the high value of dry glass wool as a heat insulator. F. SALT.

Determination of silica in refractories. STADELER.—See X.

PATENTS.

Production of refractory material. C. DEGUIDE (F.P. 612,094, 19.6.25).—Mixtures of barium silicate containing from 1 mol. or more of barium oxide per 1 mol. of silica, with water-glass, alumina, silica, tar, and water, are moulded and calcined, yielding bricks suitable for lining furnaces and cement kilns.

L. A. COLES.

Clay bodies or material for making tiles, slabs, sanitary and other ware. W. GOURDJIAN and G. L. JONES (E.P. 268,204, 30.7.26).—Gum arabic is incorporated with the clay to produce when fired a hard ceramic article. B. W. CLARKE.

Manufacture of blue glazed ceramic articles with the appearance of old Egyptian ware. M. PICK (G.P. 436,182, 7.6.24).—A glaze consisting of water-soluble alkali salts with the addition of copper oxide is burnt at a low temperature on a low-burnt body rich in silica. The subsequent glazing is carried out at a temperature sufficiently low to prevent the complete fusion of the silica in the glaze. B. W. CLARKE.

Metallising glass surfaces. SOC. NOUVELLE DE MÉTALLISATION (F.P. 612,401, 27.6.25).—The metal is sprayed by Schoop's process on to the glass surface coated with a layer of gelatin or similar material mixed with glycerin, at such a temperature that it is of a suitable consistency, and the layer is hardened by treatment with formaldehyde. L. A. COLES.

Glass furnace. UNITED GLASS BOTTLE MANUFACTURERS, LTD., and T. C. MOORSHEAD (E.P. 268,432, 31.12.25).

Manufacture of sheet glass. A. E. WHITE. From PITTSBURGH PLATE GLASS Co. (E.P. 269,300, 9.3.26).

Rouge (U.S.P. 1,618,086).—See VII.

IX.—BUILDING MATERIALS.

Report of the Stone Preservation Committee (Dept. Sci. Ind. Res., March, 1927. 33 pp.).—A detailed examination of the causes of decay in stone is being made before attempting to devise or recommend methods of preservation. The microstructure of building stones, as revealed by an examination of sections mounted in a specially developed synthetic resin, is apparently of primary importance, and the distribution of pores in the stone, i.e., of the surface open to attack, is probably a preponderating factor in determining the rate of attack. Lack of information on this point in previous researches renders correlation of results of other workers very uncertain, and probably accounts for the widely differing results obtained in practice with various methods of preservation, e.g., limewashing. Experiments are being made on test pillars treated with various preservatives which appear to offer the highest initial resistance to attack. A preliminary bacterial investigation has led to the isolation of a special strain apparently only found in stonework. B. W. CLARKE.

Action of potash end-liquors and mother-liquors on cements. CALAME (Kali, 1926, 20, 328—336; Chem. Zentr., 1927, I, 342).—Cement with a low lime content, such as blast-furnace cement, is most resistant to attack by the liquors, and its properties in this respect are improved if Portland cement having a high resistance to attack by the liquors is used in its manufacture. Blast-furnace cement containing not too much clinker, mixed with about 10% of trass, is recommended.

L. A. COLES.

Hardening of roads containing silicates. R. FERRET (Compt. rend., 1927, 184, 935—937).—Stony or calcareous materials are rendered hard by a solution of sodium silicate (SiO_2 24.9%; Na_2O 7.3%) owing to the separation of gelatinous silica which subsequently coagulates, and binds the particles of stone into a resistant and insoluble mass. This occurs by means of adsorption, and since it is favoured by drying, dialysis, and the action of carbon dioxide, the different effects obtained under various working conditions may be explained.

J. GRANT.

Use of p_H colorimetric reagents for the recognition of green or dry wood. R. LEGENDRE (Compt. rend., 1927, 184, 949).—As indicated by the use of methyl red, the p_H values for macerated wood vary between 4.4 and 6.0, according to the age of the wood.

B. W. ANDERSON.

PATENTS.

Composition for building walls, floors, and structures. I. F. SHELLARD (E.P. 268,633, 27.8.26).—Portland cement (1 pt.) is mixed with 5 pts. of an aggregate consisting of 1 pt. of clean stone chippings and 4 pts. of clayey stone dust, arranged so that the aggregate contains just over 5% of clay.

B. W. CLARKE.

Production of cast articles from volcanic stones or like materials or mixtures thereof. C. KRÜGER (E.P. 262,413, 22.11.26. Conv., 2.12.25).—Volcanic stone, blast-furnace slag, or the like is heated so that only the non-crystalline matrix and the easily fusible crystals are fused, the difficultly fusible crystals remaining to serve as crystallisation centres on cooling, with the addition of other minerals not readily fusible to act as further centres if required.

B. W. CLARKE.

Acid-resisting cement. SCHMELZBASALT-A.-G., and C. TRENZEN (G.P. 435,913, 7.7.25).—Quartz, quartzite, or other acid-resisting material is mixed with a colloidal solution of water-glass (d 1.308—1.332) and barium hydroxide, with or without the addition of assistant or protective colloids.

D. WOODROFFE.

Manufacture of material similar to compressed asphalt. H. H. SCHRODER (E.P. 260,621, 30.10.26. Conv., 2.11.25).—Limestone marl having a sp. gr. about half that of compact rock limestone is intimately mixed in a granulated form, at a temperature above normal, with sufficient bitumen to obtain an incompact mass, which is moulded in the cold state and under moderate pressure to form an asphalt-like substance suitable for use as a road surface and the like.

B. W. CLARKE.

Concrete block composition. M. E. VON MACH, JUN. (U.S.P. 1,623,985, 12.4.27. Appl., 13.4.22).—The

residue of burned fuel is treated with a mineral acid to remove sulphides, washed to remove soluble matter, and mixed with cement, finely-divided wood, lime, and plaster of Paris.

B. W. CLARKE.

Bituminous emulsions [for road-making]. F. LEVY (E.P. 268,411, 3.12.25).—Bitumen is heated at 85—107° and agitated with a hot dilute aqueous solution of sodium silicate, forming an emulsion without the aid of fatty or saponifiable matter.

B. W. CLARKE.

Apparatus for drying and seasoning timber. A. M. KOBOLKE (U.S.P. 1,625,548, 19.4.27. Appl., 25.7.23. Conv., 25.7.22).—Steam from a boiler is distributed throughout a hermetically sealed kiln which is connected to a vacuum pump, and is provided with a condenser to cause condensation inside the kiln.

B. W. CLARKE.

Facing or preserving walls and other surfaces. J. W. DOUGAL (E.P. 268,016, 21.12.25).

Cement and lime burning. G. E. HEYL (U.S.P. 1,625,853, 26.4.27. Appl., 4.3.26. Conv., 5.3.26).—See E.P. 252,780; B., 1926, 668.

[Corrugated asbestos-cement] roofing and covering materials for buildings. C. C. FIELD (E.P. 268,499, 10.2.26).

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

Gaseous reduction of iron ores. H. KAMURA (Trans. Amer. Electrochem. Soc., 1927, 51, 413—422).—Iron ores are rapidly reduced by carbon monoxide at about 900°, giving a very pure sponge iron relatively free from silicon, manganese, and phosphorus. The reduced mass may be pressed into briquettes for melting in the electric or open-hearth furnace, giving a pure malleable iron or steel of any desired composition, and a slag containing all the impurities. Theoretical calculations substantiated by experiments show that the fuel consumption in the process would be about 0.3 ton of coke and 0.5 ton of coal per ton of iron produced, compared with 1 ton of coke per ton of iron in the usual blast-furnace process.

A. R. POWELL.

Composition of materials from various elevations in an iron blast-furnace. S. P. KINNEY (U.S. Bur. Mines, Tech. Papers, 1926, No. 397. 22 pp.).—In a blast-furnace working southern red and brown ores and pyrite sinter, 80—85% of the iron was reduced by the time it had reached the base of the shaft, 19½ ft. above the tuyère level. The sponge iron at that point contained considerable impurities, i.e., more than one third of the carbon and manganese and one half of the phosphorus content. Most of the silicon entered the metal in the bosh of the furnace, and was increased rapidly in the tuyère zone by the oxidation of part of the manganese, the content of which was reduced from 1.1% to 0.53% in the cast. More (0.07%) than the tolerant amount of sulphur had been absorbed at the base of the shaft, increasing to twice the tolerant amount (0.05%) at a point 27 in. above the tuyère level. Desulphurisation

occurred between this point and the tuyère. Preferential absorption of sulphur by lime in the upper part of the furnace was demonstrated. C. A. KING.

Occurrence, distribution, and significance of alkali cyanides in the iron blast-furnace. S. P. KINNEY and E. W. GUERNSEY (U.S. Bur. Mines, Tech. Papers, 1926, No. 390. 37 pp.).—The cyanide content of measured quantities of blast-furnace gas drawn from different planes of a blast-furnace by means of a water-cooled sampling tube was determined. At the tuyère level and also on a plane 27 in. above, the concentration of cyanides per 1000 cub. ft. of gas ranges from nothing at the wall to 3–4 oz. at the centre, about 3 oz. at the base of the shaft, and 10–20 oz. at the slag notch, although wide variations due to condensation occurred. Considering the probable effect of withdrawing a portion of the gas for cyanide recovery there is good reason to believe that the rate of formation of cyanide is determined largely by the rate at which alkali can be displaced from silicates, and that the cyanide content of the gases where cyanide is being formed is much below the concentration for equilibrium with the liquid phase in the charge. It seems unlikely that the furnace operation would be affected adversely by the withdrawal of a small part of the gas, and it is estimated that the daily recovery of crude cyanide would be 1040 lb. if one tenth of the gas at the base of the shaft was withdrawn, a recovery which would be commercially significant. C. A. KING.

Influence of rust-film thickness upon the rate of corrosion of steels. E. L. CHAPPELL (Ind. Eng. Chem., 1927, 19, 464–467).—The rate of corrosion of a steel in the atmosphere depends on its chemical constitution, and the thin film of rust formed has no protective effect. A number of such films on steels of varying composition after 6 years' exposure were all found to be 0.0005 cm. thick. Tests with an artificial covering of filter paper of varying thickness showed that, with films over 0.02 cm. thick, the corrosion is inversely proportional to the thickness and independent of the chemical composition. Such films only occur naturally in steel permanently under water. For such conditions, unless the rust film is removed, which is inadvisable, the chemical constitution of the steel is of no importance. C. IRWIN.

Determination of sulphur in steel. W. HERWIG (Chem.-Ztg., 1927, 51, 275).—A battery of vertical glass tubes has delivery tubes leading into potassium hydroxide solution. Hydrogen sulphide evolved on dissolving the steel samples in a hydrochloric-sulphuric acid mixture in the battery is trapped in the potassium hydroxide, and is then determined by acidification and titration with iodine. The temperature of the dissolution tubes is kept at 110–120° by immersion in an oil bath. The time of operation is from 10–15 min. L. M. CLARK.

Utilisation of manganiferous iron ores. T. L. JOSEPH, P. H. ROYSTER, and S. P. KINNEY (U.S. Bur. Mines, Tech. Papers, 1926, No. 393. 28 pp.).—Manganiferous iron ores from the Cuyuna district of Minnesota, which have a high content of phosphorus and alumina, and are low in silica, have been reduced in an experimental blast-furnace 31 ft. high and 6½ ft. in diameter

at the bosh. No difficulty was experienced in operating with a burden of 100% of brown ore, though careful adjustment of the charge was necessary on account of the high ratio of alumina to silica. The metal contained 11.68–14.4% and the slag 4.8–12% Mn. There was considerable variation in gas composition across the furnace, and the experiment is not considered to be completed. The utilisation of pig iron such as was produced in the experimental blast-furnace depends on the elimination of phosphorus, which averages 0.6%, and it is suggested that this might be accomplished by either a converter process or a duplex process using a converter and an open-hearth. C. A. KING.

Influence of molybdenum on medium-carbon steels containing nickel and chromium. J. A. JONES (Res. Dept., Woolwich, R.D. Rept., 1926, No. 67. 59 pp.).—The examination of a number of nickel, chromium, and nickel-chromium steels containing molybdenum, showed that increase in molybdenum content slightly raised Acl and lowered Ar1, the quantitative effect of the latter influence varying considerably in different steels, though the initial temperature is without effect on the position of the Ar1 point. When the rate of cooling is slow molybdenum effects more perfect hardening than nickel or chromium, and mass effect is less marked, thereby giving greater uniformity of properties throughout the walls of large forgings after treatment. Although the changes in the properties of steel due to the presence of molybdenum are progressive with increase of molybdenum, the most marked effect is produced in nickel-chromium steels by the addition of not more than 0.5% Mo. Generally, the presence of molybdenum improves the values of mechanical tests and reduces the susceptibility of steel to temper brittleness. The best combination of properties is provided by nickel-chromium-molybdenum steels, closely approached and in certain particulars exceeded by nickel-molybdenum and chromium-molybdenum steels. An appendix contains the detailed results of a large number of experiments. C. A. KING.

Decomposition of austenite during quenching. R. L. DOWDELL and O. E. HARDER (Trans. Amer. Soc. Steel Treat., 1927, 11, 217–232).—Tests were made on 10 high-carbon or alloy steels which were quenched from high temperatures to retain the maximum amounts of austenite. The results show that quenching in oil produces more austenite than the more drastic quenching in water, which, owing to greater temperature differences between the centre and periphery, causes higher tensional stresses at the edges and assists the formation of martensite. A compression stress in the core or the absence of tension favours the retention of austenite. If the core is a region of tensional stresses the structure is martensitic throughout. On quenching from a higher temperature the austenite grains are larger and more stable, the stability being further increased by the larger amount of carbon taken into solution by the austenite. T. H. BURNHAM.

Rapid analysis of brass. S. VON BOGDANDY and M. POLANYI (Z. Metallk., 1927, 19, 164–165).—On heating brass at 1190° in a vacuum the zinc and lead are completely distilled out of the metal, leaving a residue of

copper with traces of iron. The operation is carried out in a silica tube in the lower end of which is placed a small silica test-tube containing 0.15–0.2 g. of the brass. The upper part of the outer tube is connected to the vacuum pump and water-cooled, whilst the lower part is placed in a platinum-wound, electric resistance furnace previously heated at 1190°. After 5–6 min. the tube is removed from the furnace, cooled in water, and the copper button weighed. By repeating the process on the sublimate, but using a temperature of 600°, the zinc alone distills, leaving a residue of lead. The results for copper in a plain brass are within 0.1% of the theoretical.

A. R. POWELL.

Controllable variables in the submerged corrosion of metals. O. B. J. FRASER, D. E. ACKERMAN, and J. W. SANDS (Ind. Eng. Chem., 1927, 19, 332–338).—The corrosion rate of monel metal in 5% sulphuric acid solution varies directly with oxygen concentration, reaching a maximum when the solution is saturated in oxygen. Increase in the rate of aeration causes a rapid increase in the corrosion up to a point, after which further increase has little effect. With increasing sulphuric acid concentration a maximum corrosion rate is reached in 5% of acid, the rate then decreasing until, with 80% of acid, an abrupt and rapid increase indicates a change in the corrosion reaction. The rate of corrosion increases with the velocity of relative movement between the test piece and the solution, but the rate of change gradually diminishes, and tends to become constant when the velocity is 200 ft./min. With increasing temperature, the rate of corrosion increases logarithmically to a maximum at 70°, after which the rate diminishes owing to the rapid decrease in the solubility of oxygen at the higher temperatures. The finish of the test-piece surface has little effect on the corrosion rate.

L. M. CLARK.

Heavy liquids for mineralogical analyses. J. D. SULLIVAN (U.S. Bur. Mines, Tech. Papers, 1926, No. 381. 26 pp.).—A review of the sp. gr. and uses of heavy liquids and mixtures for use in determining the density of, and separating, minerals. Bromoform–carbon tetrachloride or acetylene tetrabromide–carbon tetrachloride, d 1.58–2.89; stannic bromide–carbon tetrachloride, d 1.58–3.35; tin tetrabromide–antimony tribromide, d 3.11–3.64; thallous silver nitrate solution, d 1–4.6; thallous formate solution, d 2.86–4.94; and mercuric chloride–mercuric iodide–antimony trichloride, d up to 5.2, are recommended. For routine work the Mississippi Valley Station of the Bureau of Mines have simplified practice to two heavy liquids only. Acetylene tetrabromide is recommended for all low-gravity work, and thallous formate–malonate, which is miscible in water, for work on minerals of high density (d 4.3). As a less expensive reagent, thallous formate may be used up to d 3.5.

C. A. KING.

Recovery of molybdenite from the ore. H. A. DOERNER (U.S. Bur. Mines, Tech. Papers, 1926, No. 399. 13 pp.).—All the known deposits of molybdenum contain considerable quantities of molybdenite occurring as an oxidation product of molybdenite which is recovered readily by flotation. A number of reagents were tested under a great variety of conditions, and in no

test was there any indication that molybdenite could be floated. Gravity methods of separation are obviously useless because of the low sp. gr. and softness of molybdenite, nor did the dielectric method (cf. Hatfield; B., 1924, 261) prove promising. Two leaching methods showed the possibility of larger scale operations in which (A) the ore is extracted with a hot solution of sodium carbonate, acidified with sulphuric acid, a small quantity of sodium acetate being added, and digested with lead sulphate; molybdenum is completely precipitated as lead molybdate which can be converted to molybdic acid, or (B) the ore is digested with warm dilute sulphuric acid, and the solution neutralised with scrap iron, molybdenum being precipitated as a hydrate of the lower oxide, MoO_2 . The separation of molybdenum from all other constituents of the ore is practically complete by treating the roasted ore at 450° with chlorine.

C. A. KING.

Determination of phosphorus in phosphorus alloys. B. SALKIN (Ind. Eng. Chem., 1927, 19, 416–417).—The alloy is dissolved in dilute aqua regia solution, and any phosphine escaping is absorbed in a bromine-concentrated nitric acid solution. The two solutions containing phosphorus are combined, and the usual procedure for determination of phosphorus in such solutions is followed.

L. M. CLARK.

Sources of error in the determination of phosphoric acid by the molybdate–magnesia method. II. J. M. McCANDLESS and J. I. BURTON (Ind. Eng. Chem., 1927, 19, 406–409; cf. B., 1925, 80).—Magnesium ammonium phosphate precipitated by addition of magnesia mixture to a hydrochloric acid, ammoniacal, or neutral solution of ammonium phosphomolybdate always contains molybdenum, the purity being greatest in the precipitate from neutral solution. The more concentrated the solution, the greater will be the amount of molybdenum carried down. Ignition at 1000° volatilises most of the molybdenum. When the residue is grey or black after ignition it still contains molybdenum, which can only be volatilised with a loss of phosphoric acid. It is recommended that the precipitate should be filtered on an asbestos mat in a platinum Gooch crucible, and heated to constant weight in a blast-flame. Molybdenum in magnesium pyrophosphate is determined colorimetrically as sulphide by comparison with standards. Microcosmic salt is suggested as a phosphate standard.

L. M. CLARK.

Determination of silicon and silica in aluminium. PRETTNER (Chem.-Ztg., 1927, 51, 261).—Methods for the determination of silicon combined and free in aluminium alloys, which depend on removal of silica from a mixture of silicon and silica by treatment with hydrofluoric acid, are criticised, since silicon deposited from solution when an alloy is chilled readily oxidises on ignition. The mixture of silicon and silica is preferably dried at 200°.

L. M. CLARK.

Determination of silica in ores, slags, fluxes, and refractory materials containing fluorine. A. STADELER (Stahl u. Eisen, 1927, 47, 662–664).—Comparative tests on five methods of determining silica in materials containing fluorine showed that fusion with alkali carbonate followed by precipitation of the silica with ammonium carbonate and ammoniacal zinc oxide

solution gives the most accurate results. Good results are obtained by fusion with alkali carbonate followed by evaporation with hydrofluoric acid only when the material contains less than 1% of fluorine. For the determination of silica in fluorspar the powdered mineral is extracted with acetic acid, and the residue calcined at 800°, weighed, evaporated with hydrofluoric acid, again heated at 800°, and weighed again; the difference is silica.

A. R. POWELL.

Selenium sulphide. A detector for mercury vapour. B. W. NORDLANDER (Ind. Eng. Chem., 1927, 19, 518—521).—A solution of aluminium chloride containing 100 mg. of aluminium per litre is saturated with hydrogen sulphide. At the same time selenious acid is run in. The yellow precipitate is collected, washed, and dried, and is applied to smooth, dense, but not highly glazed paper with cotton wool. This paper is darkened by mercury vapour, the shade varying with the concentration, time of exposure, and temperature. At a velocity of 1 m./sec. and a temperature of 70°, 1 pt. of mercury in 4 million pts. of air give a colour change in 4 min. A machine devised to measure similar concentrations by the time required to match a shade is described. The test is specific for mercury liquid or vapour, and is not given by mercury ions in solution.

C. IRWIN.

Determination of mercury vapour. B. W. NORDLANDER (Ind. Eng. Chem., 1927, 19, 522—524).—The attempt to remove mercury vapour from air by agitation with a spray of dilute aqua regia leaves the content of mercury in the air (as shown by selenium sulphide; cf. preceding abstract) almost unchanged. A similar result was found when the air was passed through a weighed glass tube filled with gold leaf and glass wool and the amalgamated mercury weighed. In both cases the proportion of mercury removed was about 0.2% of that present, and the methods lead to totally erroneous results.

C. IRWIN.

PATENTS.

Heat-treatment of alloy steels. P. R. KUEHNRICH (E.P. 267,687, 19.2.26).—A process for hardening and imparting stainless qualities to steel containing 10—20% Cr and 1—1.7% C, with or without other metals in addition to iron, consists in heating it out of contact with air and preferably in a salt bath at 1150°, cooling in air, reheating at 650—700°, air-cooling, reheating in a salt bath at 1020°, and quenching in paraffin, oil, or water.

C. A. KING.

[Rustless cast iron] alloy. P. D. MERICA and J. S. VANICK, ASSRS. to INTERNAT. NICKEL CO. (U.S.P. 1,626,248, 26.4.27. Appl., 26.9.25).—Cast iron containing carbon within the usual ranges is rendered corrosion-resistant by the addition of 5—35% Ni and 2—15% Cr, the percentage of nickel exceeding that of the chromium.

F. G. CROSSE.

Steel of high-temperature stability. W. R. SHIMER, ASSR. to BETHLEHEM STEEL CO. (U.S.P. 1,624,075, 12.4.27. Appl., 29.11.24).—A low-carbon, tough steel contains 0.4—0.6% Cr, 0.5—0.8% Mn, and also 1—1.5% W to preserve the strength of the steel at high temperatures.

C. A. KING.

Copper alloys. M. G. CORSON (E.P. 268,200, 21.7.26).—Copper alloys which do not anneal at relatively high temperatures contain 0.3—2% Cr or 1—6% Co. The alloy is quenched at 900—975°, and is then subjected to mechanical working.

C. A. KING.

Copper alloys. METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 268,225, 21.9.26. Conv., 15.4.26).—An alloy suitable for bearings contains 78.5—93.8% Cu, 5—9.5% Al, 1—6% Mn, 0.1—3% Fe, and 0.1—3% Ni.

C. A. KING.

[Aluminium] alloy. A. PACZ, ASSR. to ALUMINUM CO. OF AMERICA (U.S.P. 1,595,058, 3.8.26. Appl., 19.12.24).—An aluminium base alloy contains 3—15% Si, 1.0—1.5% Cu, and 0.5% Mn.

F. SALT.

Treatment of zinc ores and other zinciferous materials. W. JOB (E.P. 268,201, 22.7.26).—Zinc ores, or zinciferous materials containing lead, copper, or noble metals are smelted in a blast furnace to volatilise lead and part of the zinc. Copper and the noble metals are recovered in the form of a matte, and the zinc-bearing slag is smelted again. In a shaft furnace suitable for this process the hearth is constricted so that the distance between opposite tuyères may be less than 1 m. The shaft above the tuyère zone is bulged out so that the gases pass up the centre of the furnace, and smelting of the charge near the side walls is largely prevented. Additional air is supplied to the upper part of the furnace (cf. U.S.P. 1,526,910; B., 1925, 323).

C. A. KING.

Extraction of lead and zinc from ores and metallurgical products. F. KRUPP GRUSONWERK A.-G. (E.P. 256,601, 24.7.26. Conv., 8.8.25).—Ore containing lead and zinc is heated under oxidising conditions in a rotary furnace to volatilise lead oxide, and the residue, mixed with reducing agents, is further heated to volatilise zinc which is collected separately (cf. E.P. 24,009 of 1910; B., 1911, 1320).

C. A. KING.

Tin-plate and sheet-mill furnace. R. THOMAS & CO., LTD., and T. F. DAVIES (E.P. 267,592 16.12.25).—The heating chamber of a sheet-mill furnace is protected from the direct action of flames from the combustion chamber by what is essentially a false crown extending from the bridge at least two thirds of the length of the furnace. Inspection apertures are provided through the arch and bridge extension, both of which incline downwardly toward the exit port. Subsidiary ports may be provided at each corner of the furnace bottom connecting with the main flue which underlies the bed of the furnace.

C. A. KING.

Electrolytic deposition of cadmium. E. C. R. MARKS. From GRASSELLI CHEMICAL CO. (E.P. 265,292, 4.11.25).—Cadmium may be deposited from a solution containing a cadmium compound (e.g., the oxide, hydroxide, cyanide, or sulphate), a cyanide of cadmium, ammonium, or other metal, an ammonium compound such as ammonia solution, the cyanide, or sulphate, and an alkaline reagent such as ammonia solution or the hydroxide of an alkali metal. The ammonium salt tends to prevent the deposition of other metals, such as zinc, along with the cadmium. The bath may also contain a colloidal substance such as starch or gulac, which helps to improve the quality of the cadmium

plate. The electrolysis is conducted under carefully regulated conditions of concentration of electrolyte, temperature, voltage, and current density. The surface area of the cadmium anodes should be 2–3 times that of the cathodes. The process may be adapted to the determination of cadmium in the presence of zinc.

M. E. NOTTAGE.

Cleaning metal. S. R. MASON, ASST. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,625,484, 19.4.27. Appl., 7.6.23).—Metal is subjected first to an alternating current in an electrolyte of ferric chloride and hydrochloric acid, and then, after removing the scale and rust, as an anode to direct current in a solution of sulphuric acid.

H. ROYAL-DAWSON.

Apparatus for determining the percentage of carbon in iron or steel. C. J. G. MALMBERG and J. G. HOLMSTRÖM (U.S.P. 1,626,005, 26.4.27. Appl., 26.6.24).—See E.P. 184,215; B., 1922, 763A.

Treatment of steel ingots. J. V. ELLIS, and UNITED STEEL COMPANIES, LTD. (E.P. 268,123, 18.3.26).

Hearths of blast and like furnaces. E. BARTEN (E.P. 267,468, 30.11.26. Conv., 9.3.26).

XI.—ELECTROTECHNICS.

PATENTS.

Electric furnace. F. T. COPE, ASST. to ELECTRIC FURNACE CO. (U.S.P. 1,624,217, 12.4.27. Appl., 9.11.25).—A hearth comprising a number of plates having lugs at opposite edges for engagement under the edges of adjoining plates is spaced above a refractory base, and a resistor is mounted between the hearth and base.

J. S. G. THOMAS.

Affixing [metallic oxide] coatings on incandescence lamp filaments. H. K. RICHARDSON, ASST. to WESTINGHOUSE LAMP CO. (U.S.P. 1,624,071, 12.4.27. Appl., 28.7.21).—A high potential is established between the filament and another electrode connected thereto, in a rarified atmosphere in the presence of the powdered metallic oxide, so as to effect bodily transfer of the powdered particles to the filament without dissociation.

J. S. G. THOMAS.

Thermionic electrode. BRITISH THOMSON-HOUSTON CO., LTD., ASSEES. of G. M. J. MACKAY (E.P. 267,142, 4.3.27. Conv., 6.3.26).—In a thermionic electrode of the type described in E.P. 208,728—9 (B., 1925, 391), a metal container, e.g., of nickel, encloses a core of film-forming material, e.g., caesium, rubidium, barium, or calcium, of materially lower electron affinity, which is able to diffuse through the wall of the container at high temperatures. Alternatively, the core may consist of a mixture of a compound of one of the said metals, e.g., caesium chloride, and a reducing agent, e.g., powdered magnesium or calcium, so that at the operating temperature of the electrode the desired metal is produced without the formation of deleterious products.

J. S. G. THOMAS.

High-pressure system for electrolytic processes, more especially for the production of hydrogen and oxygen. J. E. NOEGGERATH (E.P. 268,426, 30.12.25).—An electrolyser adapted for high-pressure electrolytic systems has a cross-section so reduced in

area as to be only sufficient to accommodate the gas bubbles passing through it when operating at high pressure. The vessel consists of a number of individual cells connected in series, the distance between electrodes of different polarity being greater at the bottom than at the top, whilst, in order to obtain smaller electrolyte-filled spaces, parts of the vessel are insulated against the electrolyte, so that the walls of the vessel can be used for conducting current. Partitions separating electrodes are made of asbestos cord, and yield to the variations of volume due to the different pressures of the gases.

W. G. CAREY.

Galvanic battery. C. DE MIERES (E.P. 268,047, 28.12.25).—A secondary battery comprises a positive electrode of graphite, artificial carbon, or other electrical conductor not attacked by chlorine or bromine, covered with spongy silver, a narrower and shorter negative electrode of zinc, and an electrolyte consisting of an aqueous solution of zinc chloride mixed with one or more other halogen salts of zinc, e.g., zinc bromide. The voltage of the cell is 2.2.

J. S. G. THOMAS.

Impregnating porous substances or fillers with artificial resins. BAKELITE GES.M.B.H. (F.P. 611,264, 15.2.26. Conv., 17.2.25).—The material is impregnated with a colloidal solution of artificial resin, e.g., phenol-formaldehyde condensation products, and the resin precipitated by means of water. Thus, sawdust is mixed with a 50% solution of artificial resin, then treated with water, centrifuged, and dried. Similarly, paper pulp, fibrous material, asbestos, or mica may be treated with a colloidal suspension of artificial resins and soap solution. The product is suitable for making electric insulators etc. and for lining chemical plant.

Electric arc welding or coating with material. INTERNAT. GENERAL ELECTRIC CO., INC., ASSEES. of ALLGEM. ELEKTRICITÄTS-GES. (E.P. 262,453, 3.12.26. Conv., 3.12.25).

Production of electrodes for electric accumulators. P. HAGSPIHL (E.P. 265,918, 22.10.26. Conv., 13.2.26).

Manufacture of [plates for] accumulators or electrical storage batteries. S. J. WILLIAMS (E.P. 268,179, 28.5.26).

Gas analysis apparatus (E.P. 268,637).—See I.

Apparatus for determining impurities in water (E.P. 268,597).—See I.

XII.—FATS; OILS; WAXES.

Marine animal oils. I. Squalene and spinacene. E. ANDRÉ and H. CANAL (Ann. Chim., 1927, [x], 7, 69–112).—A detailed account of an investigation, a preliminary note of which has already appeared (A., 1925, i, 1373). The liver oil from *Cetorhinus maximus*, *Scymnus lichia*, and *Centrophorus granulosus* was separated by hydrolysis into fatty acids, alcohols, and non-hydrolysable matter, physical and other constants of each fraction from the various specimens being given. Conversion of the unsaturated hydrocarbons from the three species, after purification by fractional distillation under reduced

pressure, into hydrochlorides and hydrobromides, and fractional crystallisation of these from acetone yields, respectively, hydrochlorides, $C_{28}H_{46} \cdot 6HCl$, m.p. 107—108°, and $C_{30}H_{50} \cdot 6HCl$, m.p. 143—145°; $C_{30}H_{50} \cdot 6HCl$, m.p. 107—108°, and $C_{31}H_{52} \cdot 6HCl$, m.p. 143—145°; $C_{30}H_{50} \cdot 6HCl$, m.p. 106—107°, and $C_{31}H_{52} \cdot 6HCl$, m.p. 141—143°; hydrobromides, $C_{28}H_{46} \cdot 6HBr$, m.p. 106°, and $C_{30}H_{50} \cdot 6HBr$, m.p. 130—132°; $C_{28}H_{46} \cdot 6HBr$, m.p. 105°, and $C_{30}H_{50} \cdot 6HBr$, m.p. 130—132°; $C_{29}H_{48} \cdot 6HBr$, m.p. 105°, and $C_{30}H_{50} \cdot 6HBr$, m.p. 132—134°. As a result of a large number of analyses by different methods, it is found that the hydrohalide with the lower m.p. always contains a larger proportion of halogen, but traces of hydrogen halide are lost during the crystallisations, and that, moreover, the addition of hydrogen halide to the six ethylenic linkages is not complete, a carefully purified sample of squalene hydrochloride, the analysis of which corresponded with a C_{30} formula, losing sufficient halogen after several months to agree with a C_{31} formula. Re-treatment of this sample with hydrogen chloride in ether gave a specimen corresponding with C_{29} , and in acetone, with one corresponding with the formula $C_{28}H_{46} \cdot 6HCl$. These facts show that the constitution of the hydrocarbon cannot be determined by analysis of the hydrohalides alone (cf. Heilbron, Kamm, and Owens; A., 1926, 816, whose work is criticised on this ground), the authors holding the view that the formula $C_{28}H_{46}$ (or even $C_{27}H_{44}$ from its relation to cholesterol) for squalene is more probable than that of higher homologues. J. W. BAKER.

Determination of the iodine value according to the German Pharmacopœia. VI. K. H. BAUER and P. MANICKE (Pharm. Zentr., 1927, 68, 241—250).—Comparative determinations of the iodine values of drying, semi-drying, and non-drying oils and fats by various methods are described. The Hanus method gave results lying within the limits given in the G.P. VI., and with 33—100% excess of halogen no notable differences were observed, except with cod-liver oil, which, with 33% excess, gave an iodine value of 160.9, and with 100% excess, 164.7. Reaction periods varying from 15 min. to 2 hrs. produced no marked variation in the results. The Kaufmann method gave values agreeing generally with those given by the Hanus method. Varying excesses of halogen had no influence on the iodine value except with linseed oil, which gave higher values with excess. The pyridine sulphate dibromide method of Rosenmund-Kühnemann gave rather lower values than the above two methods, even with large excess of halogen and longer reaction period. Winkler's method—the official G.P. VI. method—gave results with hard fats agreeing with those given by the Hanus and Kaufmann methods, but the oils gave generally higher values. The excess of halogen used varied from 15% to 200%, and a more satisfactory end-point was obtained by determining the excess of halogen by the addition of potassium iodide and titration with thiosulphate. The method gave results falling within the official limits, but it is doubtful if it is superior to either the Hanus or the Kaufmann method. E. H. SHARPLES.

PATENTS.

Preservation of oils. F. W. NITARDY, Assr. to E. R. SQUIBB & SONS (U.S.P. 1,624,358, 12.4.27. Appl.,

21.1.24).—Castor oil, from which odour and taste have been removed, and containing a small proportion of ethyl alcohol, is claimed. S. S. WOOLF.

Manufacture of soap [filaments]. A. H. CHARLTON, and T. B. ROWE & Co., LTD. (E.P. 267,999, 24.11.25).—Liquid soap, preferably containing about 63% of fatty acids, with or without the addition of sodium carbonate, sodium silicate, etc., is forced through suitable orifices under a pressure of 15—60 lb./sq. in., and the solidified filaments so formed are subsequently dried. The liquid soap may be aerated before extrusion. S. S. WOOLF.

Pattern printing using wax (Austr. P. 104,395).—See VI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

“Oxyns.” IV. The autoxidation process.

A. EIBNER and H. MUNZERT (Chem. Umschau, 1927, 34, 89—96, 101—108; cf. B., 1926, 888).—Previous work and existing theories on the processes involved in the oxidation of drying oils are comprehensively and critically recapitulated. In developing the concept of “ideal drying of an oil,” the various factors influencing a drying oil film are discussed, and an expression is devised analysing the total increase in weight of a film into its components due to these separate influences. The gradual increase of acidity is shown to be due to the hydrophilic nature of the drying film. Evidence is adduced for the absence of true hydroxy-acids in “oxyn acids,” but reduction, acetylation, benzoylation, etc. of these fail to yield crystallisable products that would lend themselves to further investigation. The oxyn acids are probably non-hydroxylic intermediates between peroxy-acids and true hydroxy-acids (contrary to the views of Auer), and various possibilities of tautomerism are discussed. Dried oil films and artificially oxidised oils, blown oils, Uviol oils, etc. (“liquid oxyns”) yield dark-coloured soaps and acids on saponification and acidifying, whereas the soaps and acids obtained from fresh oils and stand oils are of lighter colour. True hydroxy-acids of the C_{18} group and their salts are colourless, crystalline, and water-soluble. Tung oil is the most suitable oil for experimental use owing to its freedom in the main from mixed glycerides, and its capabilities of yielding purifiable products, and its so-called anomalous behaviour is shown not to militate against its value in this connexion. The influence of the glycerin residue in the dried oil film is definite, since freed tung oxyn acids, their soaps, and methyl and ethyl esters are dark coloured, whereas the glycerides are colourless. Analysis of the methyl ester of tung oxyn acids reveals no true hydroxy-acids, and favours the Böeseken and Ravenswaay formulation of elæostearic acid as a conjugated triply-unsaturated substance (B., 1925, 813). A synthetic glyceride of a true hydroxy-acid of the C_{18} group was in no way comparable with an oil film and saponified to a colourless soap. Lines of investigation on many points arising in the present paper are indicated. S. S. WOOLF.

Laboratory examination of paints. A. A. KRAEFF (Chem. Weekblad, 1927, 24, 210—219).—Paint films for comparative examination of physical properties are prepared by spreading known quantities over paper

surfaces of definite area previously coated with gum arabic; after drying, the film is removed by soaking in water and washed free from the gum. Chemical examination shows whether the pigment is basic (rust-preventing) or acidic; all acidic pigments accelerate rusting, except chromates, which induce passivity. The permeability of the films by moisture is determined by Gardner's method of closing tubes containing sulphuric acid by films of the paint, and noting the increase of weight after keeping in a damp atmosphere. The permeability is determined chiefly by the oil used and the size of the pigment particles, and by chemical reaction between the oil and the pigment. Tables and graphs of extensions and breaking strains are quoted from work of Gardner and of Nelson, and discussed. The effects of sulphur dioxide and of moisture are examined by exposing films to these reagents in closed vessels, and observing the change in the extensibility. The determination of covering power by means of Pfund's cryptometer is described and some data are recorded, and the use of the Gardner-Holdt flowmeter for determination of brushing consistency is described. Methods of carrying out practical observations on resistance to wind and weather are suggested.

S. I. LEVY.

Physical properties of some grades of whiting. Keeping properties and mobility of paints. H. A. GARDNER (U.S. Paint Manufs. Assoc., Sept., 1926, Circ. No. 283, 116—123).—The following properties of 10 samples of whiting (natural and precipitated) are tabulated:—fineness, oil absorption, sp. gr., bulk density, alkali number (number of milligrams of potassium hydroxide required by the boiling water extract of 1 g. of sample), colour (whiteness in oil), and times of settling when shaken in water. The settling, alone and when thinned with an equal volume of light petroleum, mobility on keeping, etc. of a series of experimental paints of both exterior and interior types were observed, and it is shown that ready-mixed paints containing 20—30% of precipitated whiting of high oil absorption may show greater mobility than paints of similar composition containing natural whiting of lower oil absorption.

S. S. WOOLF.

Toxic compounds for anti-fouling compositions. H. A. GARDNER (U.S. Paint Manufs. Assoc., Sept., 1926, Circ. No. 287, 147—149).—A series of submersion tests on anti-fouling paints on metal panels show that red cuprous oxide is the best form of copper for use in such paints, and that copper cyanide is also fairly satisfactory. Mercuric oxide (red or yellow) has excellent toxic qualities, alone or mixed with cuprous oxide, and to a lesser extent with the cyanide.

S. S. WOOLF.

Volatility of acetone-benzene paint and varnish removers. J. M. WEISS (Ind. Eng. Chem., 1927, 19, 485—487).—The addition of 3—5% of paraffin wax to mixtures of benzene and acetone results in an extraordinary reduction in the rate of evaporation of the solution when exposed to air, due to the formation of a surface film of wax which is almost impervious to benzene vapour. The rates of evaporation of solutions containing wax have been compared with that of the solvent itself at 15.5°, 29.5°, and 37.5°. The reduction in evap-

oration is greater the higher the m.p. of the wax used. The results indicate that the industrial use of such mixtures as paint and varnish removers is not, under reasonable conditions of ventilation, attended with hazards due to the toxicity of the vapours.

A. B. MANNING.

Physical properties of films prepared from nitrocellulose of low viscosity, and the effect of concentration upon mobility. H. A. GARDNER (U.S. Paint Manufs. Assoc., Sept., 1926, Circ. No. 284, 124—132).—Lacquers made by dissolving samples of nitrocellulose of extra low viscosity (designated " $\frac{1}{3}$ sec." as against " $\frac{1}{2}$ sec." for the usual low-viscosity type) in a mixture of equal volumes of butyl acetate and toluol were tested, with the addition of plasticisers and pigments, for mobility at various concentrations and for tensile strength and elongation of their films. Promising results were obtained with concentrations of nitrocellulose more than double that possible when " $\frac{1}{2}$ sec." material is used. In the case of a further sample of nitrocellulose of exceedingly low viscosity, giving working solutions containing 45% of nitrocellulose, a danger point in viscosity reduction was indicated, since cracking, checking, and scaling occurred immediately the films dried. The possibility of modifying such products to serve as undercoats is suggested. Details of the falling-sphere method of determining the viscosity of clear lacquers are given.

S. S. WOOLF.

Analysis of cellulose ester varnishes. H. DABISCH (Farben-Ztg., 1927, 32, 1609—1611).—A general scheme for the analysis of nitrocellulose varnish is given, the initial step being the precipitation of the nitrocellulose by pouring the varnish into a suitable amount (determined by a preliminary experiment) of light petroleum, b.p. 35—50°. Addition of a little acetic acid is advantageous if the precipitate remains colloidal. After filtration and washing, solvents are separated from resins and plasticisers by fractional distillation of the filtrate, the fractions being subsequently further analysed. The b.p. and sp. gr. of all commonly occurring solvents are tabulated. In the distillation residue, resins and plasticisers are recognised by saponification, identification of alcohol residues, and re-esterification of the liberated acids according to the Wolff-Schulze method for separating resin and fatty acids. Conclusions as to the nature of the resins present will have been drawn from the presence or absence of cloudiness etc. in the original precipitation of the varnish. Similar details apply for cellulose acetate varnishes, whilst in the case of a blend containing both esters, methyl alcohol is used as precipitating agent, completely bringing down the acetate. The procedure is then as for nitrocellulose varnish.

S. S. WOOLF.

Possible use for hydrocellulose in paints and in lacquers. H. A. GARDNER (U.S. Paint Manufs. Assoc., Sept., 1926, Circ. No. 282, 113—115).—Hydrocellulose mixed with a high-grade lithopone flat wall paint yields a very thick granular paint that brushes out well and on stippling gives the "pulled" rough effect that has also previously been obtained by the use of plaster of Paris, asbestos fibre, etc. The product obtained by the nitration of dry hydrocellulose is, when washed and

dried, a grey granular material, fairly soluble in lacquer solvents to solutions yielding dark coloured films of apparently good strength. S. S. WOOLF.

Examination of turpentine oil used in large-scale chemical industry. C. AUSTERWEIL (Chem.-Ztg., 1927, 51, 249—251).—In the manufacture of synthetic camphor and perfumes (terpin hydrate and terpineol), true turpentine oil (from coniferous balsamic resin) should be used, only α -pinene and nopinene being of use. The oil should be free from water, *e.g.*, there should be no turbidity on treatment with 2 to 3 vols. of benzine; the acid value should not exceed 1, and foreign hydrocarbons, the distillation products of resins, should be absent. The α -pinene and nopinene content is examined thus: the optical rotation should not exceed $\pm 39^\circ 50'$ (pure α -pinene). Distillation after drying over anhydrous sodium sulphate should give a 90% yield of a product having b.p. 153.5—165°/760 mm. The fractions should have $d_{15}^{20} 0.862$ —0.874, $n_{15}^{20} 1.4625$ —1.4755, and the ratio of the optical rotations for the mercury green ($\lambda=5460$) and yellow ($\lambda=5780$) lines should be between 1.061 (nopinene) and 1.134 (α -pinene). If the residue of distillation is 10% or more, it is steam-distilled, and the product should have an optical rotation within 10% of that of the original oil. B. FULLMAN.

Bleaching of shellac. E. R. WATSON and H. M. MULANY (J. Indian Chem. Soc., 1926, 3, 255—257).—Shellac when bleached with the minimum quantity of hypochlorite solution (1 pt. of bleaching powder to 6.5 pts. of shellac), precipitated in a granular form by acidification of the alkaline solution in the cold, treated in aqueous suspension with 1 pt. of 10% sodium thio-sulphate solution to remove absorbed chlorine, thoroughly washed, and air-dried, can be stored for long periods without any appreciable diminution of its solubility in alcohol. J. W. BAKER.

PATENTS.

Manufacture of carbon black pigments or compositions containing the same. W. H. WHATMOUGH (E.P. 267,625, 22.12.25).—A composite black pigment is produced by precipitating one or more pigments, *e.g.*, Prussian blue, in an aqueous suspension of finely-divided carbon pigment particles. If an oleaginous substance be also present in suspension, the precipitated mass is suitable for use in paints, inks, etc. S. S. WOOLF.

Production of titanium pigments. E. C. R. MARKS. From J. IRWIN and R. H. MONK (E.P. 267,788, 6.8.26).—A titanium pigment substantially free from the higher oxides of titanium is prepared by calcining a finely-ground mixture of precipitated titanium hydroxide with 2—20% of carbonaceous material at 750—900°. S. S. WOOLF.

Application of coloured pigments in suspension in gelatin or glue solutions by means of sprays. R. HEINRICH (G.P. 435,835, 15.9.25).—The precipitation of the pigment is retarded by using a medium which tends to jellyify on cooling. D. WOODROFFE.

Potentially reactive liquid coating compositions. E. C. R. MARKS. From BAKELITE CORP. (E.P. 267,736, 27.4.26).—A phenol is heated under reflux with a fatty oil (preferably tung oil) and a converting agent, *e.g.*,

phosphoric acid, anhydrous aluminium chloride, sulphur, oxalic acid, etc., until suitable viscosity is attained when a hardening agent containing methylene groups, *e.g.*, hexamethylenetetramine, is added in amount not exceeding 3% by wt. of the phenol, the necessity for elimination of excess by washing being avoided by this limitation. Reaction proceeds to the formation of a non-reactive, permanently fusible resin which may be used as a varnish base or may be rendered potentially active by dissolving it while hot in a suitable solvent and adding a further quantity of the hardening agent.

S. S. WOOLF.

Manufacture of aldehyde condensation products. SOC. CHEM. IND. IN BASLE (E.P. 260,288, 22.10.26. Conv., 22.10.25).—Araldehyde, alone or in polymerised form, or admixed with other aldehydes, is condensed in one or more stages with carbamide or a derivative thereof in a closed vessel, in the presence of a catalyst, *e.g.*, acetic acid, sulphuric acid, etc. Water, methyl or ethyl alcohol, glycerol, etc. may be added as solvents or diluents, to be separated at the end of the reaction.

S. S. WOOLF.

Manufacture of linoxyn-like substances. W. O. HERMANN and H. DEUTSCH, ASSTS. to CONSORTIUM FÜR ELEKTROCHEM. IND. (U.S.P. 1,625,852, 26.4.27. Appl., 13.6.25. Conv., 18.6.24).—See G.P. 434,318; B., 1927, 305.

Impregnating fillers with artificial resins (F.P. 611,264).—See XI.

Tanning condensation products (G.P. 436,446).—See XV.

Photographic process (E.P. 265,769).—See XXI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Influence of butaldehyde-ammonia in vulcanisation. C. A. HALLAS and T. J. DRAKELEY (J.S.C.I., 1927, 46, 178—179 T).—The effect of the addition of 1% and 2% of butaldehyde-ammonia on the vulcanisation of various rubber mixes was determined. Butaldehyde-ammonia was a powerful accelerator of vulcanisation which was not activated by zinc oxide. In pure rubber-sulphur mixes it acted also as a softening agent, and in mixes containing zinc oxide the softness increased with the time of vulcanisation. In conjunction with diphenylguanidine the softening effect was very marked at the longer times of vulcanisation. The softening effect was not appreciable in a more heavily compounded vulcanisate.

Oxidation of caoutchouc. J. MCG. ROBERTSON and J. A. MAIR (J.S.C.I., 1927, 46, 41—49 T).—Purified rubber on oxidation with varying proportions of potassium permanganate in neutral or alkaline solution gives a series of complex, pale yellow or colourless, oxygenated resinous products which are insoluble in acetone, alcohol, or alkalis, but easily soluble in ether, benzene, ethyl acetate, etc. In spite of their inertness these products are probably hydroxy-compounds, analysis indicating mere addition of oxygen without hydration. Formic, acetic, oxalic, and laevulinic acids, also colloidal resin acids are likewise obtained. Oxidation of rubber in a mixture of chloroform and acetic acid by hydrogen

peroxide yields as primary product an alcoholic substance of composition $C_{59}H_{102}O_{16}$, some carbon dioxide also being liberated; the alcoholic substance on oxidation with chromyl chloride gives an aldehyde (capable of yielding a disemicarbazone), whilst on atmospheric oxidation it forms a resin acid apparently with fission of its molecule into two approximately equal parts. Treatment of rubber in carbon tetrachloride with chromyl chloride and decomposition of the primary product with water gives as main products a brown aldehydic resin (oxidisable to a resin acid) and a complex insoluble material containing chromium and chlorine; the latter, which probably arises from polymerisation of the chromyl chloride compound during decomposition, loses its chromium and chlorine when heated with acetic acid or aqueous sodium carbonate, forming an aldehydic resin or a resin acid respectively. D. F. TWISS.

PATENTS.

Method of preserving [rubber] latex and product obtained thereby. GENERAL RUBBER CO., ASSEES. OF A. E. JURY and O. H. SMITH (E.P. 268,219, 10.9.26. Conv., 8.6.26).—Rubber latex is preserved by the addition of sufficient alkaline phosphate, *e.g.*, trisodium phosphate, to reduce the hydrogen-ion content to below 1×10^{-7} ; a bactericide, *e.g.*, formaldehyde, is also introduced. For long storage suitable proportions are 0.5% of formaldehyde and 1.5% of trisodium phosphate.

D. F. TWISS.

Production of elastic, waterproof, adherent coatings. H. QUITNER (Austr. P. 104,398, 30.12.25).—Paper, fibrous materials, etc. are treated with latex of rubber, gutta-percha, or balata and solutions or emulsions of natural or artificial resins, especially shellac, the latex and the resin dispersion being applied either jointly or successively. The rubber latex may be dilute, concentrated, or vulcanised, or may be compounded and the product subsequently vulcanised.

D. F. TWISS.

Waterproof materials (E.P. 266,813).—See V.

XV.—LEATHER; GLUE.

Destructive and preservative effects of neutral salts upon hide substance. II. A. W. THOMAS and M. W. KELLY (Ind. Eng. Chem., 1927, 19, 477–480; *cf.* B., 1926, 23).—Experiments were made on hide powder to determine the action of saturated solutions of sodium, potassium, calcium, and magnesium chlorides, and sodium sulphate, respectively, at 7°, 20–25°, and 37.5°, also of saturated solutions of sodium chloride–sodium sulphate mixtures at 20–25° and 37.5° respectively, and of saturated sodium chloride solutions containing calcium chloride. There was increased hydrolysis with increased temperature in all cases except sodium sulphate. Sodium sulphate solutions showed no hydrolysis at 37.5°, and an increasing amount, though small, down to 7°; this is explained by the greater content of sodium sulphate of a saturated solution at the higher temperature. The different hydrolytic effects of the different salts were not attributable to differences in p_H value. Sodium sulphate is a hide preservative. There was less hydrolysis with

various mixtures of sodium chloride and sodium sulphate than with sodium chloride alone. A solution saturated with a salt mixture of 5 mols. of sodium chloride and 1 mol. of sodium sulphate preserved hide powder as well as a saturated sodium sulphate solution. Calcium chloride produced 18% hydrolysis in 7 days; sodium chloride gave 6% at 20–25°, but the admixture of calcium chloride diminished this hydrolysis. The addition of 8% of calcium chloride to sodium chloride renders the latter a better hide preservative. D. WOODROFFE.

Action of bates and methods of measuring the enzymatic activity of natural and artificial bates. G. HUGONIN (J. Soc. Leather Trades' Chem., 1927, 12, 26–42; *cf.* B., 1927, 342).—The enzymic activity can be measured by determining the effect on substrates of casein, gelatin, or fibrin respectively, by the Soerensen formaldehyde method or that of Wilson and Merrill (B., 1926, 205). Nine different samples of pancreatin were examined by these methods and by their action on elastin and fats respectively. The Fuld–Gross and Northrop casein methods and the keratose method gave figures of similar order of strength. The activities as determined by their action on collagen and elastin respectively were quite different from the others. The Willstätter and Wohlgemuth methods indicating the activities on fats gave inconcordant results, but they showed that pancreatin possesses lipolytic properties, and further, that the various enzymes in commercial pancreatins are present in very varying proportions. The determination of the activity of a pancreatin should be made on the same product, without using type products. The determination of the quantity of elastin dissolved by a sample of bate can be made under well-defined conditions on the same sample of elastin, but the digestive power of a bate on gelatin, casein, or fibrin is liable to show variations. All the different activities on the various skin components should be determined in order to obtain the practical value of the bate. The Wilson–Merrill method is the best for determining the activity on collagen, elastin, and keratose, and thus the action of a pancreatin or bate on all the constituents of the skin. Willstätter's method is preferable to that of Wohlgemuth for determining the lipolytic activity, since it provides for p_H control and yields easily reproducible results. D. WOODROFFE.

Action of iron blacks on leather. D. WOODROFFE and D. H. DEW (J. Soc. Leather Trades' Chem., 1927, 12, 42–46).—Pieces of sumac-tanned skiver were treated with solutions containing 5%, 2%, and 1% respectively of ferrous sulphate on the weight of the leather. The soaked leather was dried out, analysed, and kept for 28 months without showing any signs of deterioration. The finished skivers contained 0.97–1.27% of ferric and aluminium oxides. The mere presence of iron in a leather or treatment with iron salts does not appear to cause any deterioration of the leather. Leathers containing iron, which have rotted or weakened, have usually contained free sulphuric acid also.

D. WOODROFFE.

Commercial gelatins. Their jelly strength, gold number, and hydrogen-ion concentration. P. SEREX and M. W. GOODWIN (Ind. Eng. Chem., 1927,

19, 473—474).—The jelly strength, gold number, and p_H value were determined on 23 different commercial gelatins of varying grades. The gold numbers showed only slight differences, 0.0085—0.0150. The jelly strength varied from 33 to 339, and the p_H values from 3.99 to 6.70. There was no direct relation between the jelly strength, gold number, and p_H value. A considerable difference in jelly strength between the similar grades of different manufacturers was observed.

D. WOODROFFE.

PATENTS.

Bleaching leather and other products of animal origin. ÉTABL. REYNIER and C. SZMUKLER (F.P. 575,431, 26.12.23).—Wool, hair, pelts, tawed leather, etc. are bleached with dry ozone, and treated with a current of air until the odour of ozone has disappeared.

L. A. COLES.

Bleached and dyed furs. F. B. DEHN. From STEIN FUR DYEING Co., INC. (E.P. 264,969, 25.11.25).—Fur skins are protected against singeing or burning of the hair or leather and rendered suitable for bleaching by treatment with a ferrous salt, with or without addition of ammonium chloride to retard oxidation of the protective agent. The subsequent bleaching is preferably carried out by hydrogen peroxide or such substances as sodium peroxide or perborate, which generate hydrogen peroxide on treatment with acids.

A. DAVIDSON.

Treatment of leather goods. W. SCHMIDT (G.P. 435,685, 28.4.25).—Articles such as shoes are cleaned with soap solution and then treated with softening agents consisting of water-soluble and emulsifiable oils or fats, such as sulphonated animal, vegetable, or mineral oils.

L. A. COLES.

Treatment of animal tissue and products. F. W. WEBER, ASST. to MAYWOOD CHEMICAL WORKS (U.S.P. 1,621,965, 22.3.27. Appl., 4.4.24. Renewed 6.8.26).—The basic acetates of the rare-earth metals can be employed as tanning agents in place of alum.

T. S. WHEELER.

Manufacture of soluble tanning condensation products from aromatic hydroxy-compounds. A. KÄMPF (G.P. 436,446, 21.6.23).—The condensation products derived from phenols or naphthols and natural resins or their constituents in presence of acid catalysts are treated with sulphonating agents, or natural resins or their constituents are treated with aromatic hydroxy-sulphonic acids in presence of an acid catalyst. Colophony is heated with phenol and sulphuric acid to give a clear solution. After the gradual addition of more sulphuric acid the temperature rises to 70°. The product is soluble in water, and gives a heavy precipitate with gelatin. Similar products can be obtained from colophony and cresol or α - or β -naphthol. In using turpentine and phenol or pinene and α -naphthol, the phenol must be dissolved in the turpentine or its derivatives, condensed with a few drops of sulphuric acid, and sulphonated. The products when dry are brittle resinous substances giving dark yellowish-brown to brownish-green solutions. They are easily precipitated from aqueous solutions by dilute sulphuric acid. The solutions give heavy precipitates with gelatin unless they are neutralised.

D. WOODROFFE.

Dyeing leather (F.P. 609,904).—See VI.

XVI.—AGRICULTURE.

Fertility of a soil as related to the forms of its iron and manganese. P. H. BREWER and R. H. CARR (Soil Sci., 1927, 23, 165—173).—A type of toxicity present in an Indiana soil was unaffected by dressings of lime or commercial fertilisers. When, however, farmyard manure was added satisfactory yields were obtained. This is attributed to the reduction of some of the ferric iron present to the ferrous state, in which it is more available to plants. The determination of manganese dioxide in soil is discussed. The green colour produced when certain soils are allowed to remain for a long period in contact with alcoholic potassium thiocyanate is considered to be due to the presence of a colloidal manganese compound. It is only developed when the lime requirement of the soil has been sufficiently satisfied by dressings of lime or where insufficient available iron is present.

G. W. ROBINSON.

Outgo of calcium, magnesium, nitrates, and sulphates from high-calcic and high-magnesic limes incorporated in two soil zones. W. H. MACINTIRE (Soil Sci., 1927, 23, 175—197).—Results are given of lysimetric investigations in Tennessee of the loss in drainage of calcium, magnesium, nitrates, and sulphates, after surface and sub-surface incorporations of calcium hydroxide, burnt dolomite, and a corresponding lime-magnesia mixture, respectively, at a rate equivalent to 0.1% of quicklime reckoned on the soil. The losses of calcium and magnesia were greater with sub-surface than with surface incorporations. Losses of nitrate and sulphate were increased by the dressings.

G. W. ROBINSON.

Removal of nutrients from subsoil by lucerne. C. E. MILLAR (Soil Sci., 1927, 23, 261—269).—Exclusion of the top 8 in. of the roots of lucerne plants from the soil by means of glass cylinders did not decrease growth as measured by the weight of air-dry tops. Application of nutrient solution near the tap roots at various depths increased the growth of plants with the root system restricted, but had little effect on normally grown plants. The results indicate that the lower portions of the roots of lucerne are able to absorb nutrients in appreciable quantities.

C. T. GIMINGHAM.

Microflora and productivity of leached and non-leached alkali soils. J. E. GREAVES (Soil Sci., 1927, 23, 271—302).—The effect of leaching alkali soils on the numbers of micro-organisms and on the biological changes in the soil has been investigated. Both natural and artificial alkali soils were used, the latter being prepared by addition of various proportions of sodium chloride, sulphate, and carbonate to a normal productive soil. Leaching of the normal soil and of all the alkali soils increased the bacterial numbers, in some cases very greatly. Leaching, in many cases, increased ammonification to above that occurring in normal soil. Soil extract and stable manure were very effective in restoring ammonification in alkali soils. Nitrification was very low even after leaching, but was increased by inoculation with soil extract or stable manure. Sodium carbonate reduced nitrification more than the chloride or sulphate. Most of the soils showed nitrogen fixation, which was increased by leaching. Leaching also increased the

crop yields. The author considers that organic manures are essential in reclaiming alkali soils, even where leaching is employed. C. T. GIMINGHAM.

Normal moisture capacity of soils. C. F. SHAW (Soil Sci., 1927, 23, 303—317).—Experimental evidence indicates that when water is added at the surface to a mass of uniform soil and allowed to percolate downward, a condition of distribution is reached at which the water content at any depth will be approximately the same, except for a region of very rapid decrease in the water content in the lowest layers of the wetted soil. The term "normal moisture capacity" is proposed as an expression denoting the water content of a soil in this condition of uniform distribution; for medium-textured soils it is approximately the same as the moisture equivalent. The author holds that at this point the soil water is essentially in a static condition, and is not free to move under the normal film forces existing in the soil, but it is available for plants. Under these conditions, mulches are of little or no effect in conserving soil moisture. C. T. GIMINGHAM.

The hydrometer as a new and rapid method for determining the colloidal content of soils. G. J. BOUYOUCOS (Soil Sci., 1927, 23, 319—331).—The percentage of soil material which stays in suspension in water for 15 min. under standardised conditions is equal to the percentage of colloids in the soil, as determined by the heat of wetting method. The agreement is close for nearly all types of soils, the very few exceptions being soils abnormally rich in organic matter not thoroughly decomposed. A quick method for determining the colloidal content of soils is based on this relationship. An amount equivalent to 50 g. of oven-dry soil is dispersed in 1050 c.c. of water by very rapid mechanical stirring for 9 min. in a specially constructed vessel. 5 c.c. of *N*-potassium hydroxide are added, and, after settling for 15 min., the density of the soil suspension is determined by means of a sensitive hydrometer graduated to give grams of dry material per litre directly. The temperature must be taken into account; a change of 1° F. makes a difference of about 0.35% of soil colloids. C. T. GIMINGHAM.

Colorimetric determination of phosphoric acid, particularly in phosphatic fertilisers. C. V. BORDEIANU (Ann. sci. Univ. Jassy, 1927, 14, 353—362).—Citric acid interferes with the colorimetric determination of phosphoric acid by Bell and Doisy's method (A., 1920, ii, 769), but the following reaction may be used. A green ring, becoming blue, appears when a solution containing as little as 0.00005 mg. of phosphoric anhydride in 0.1 c.c. is poured on the surface of a mixture of 2 c.c. of an ammonium molybdate solution prepared as recommended by Fresenius with 1 c.c. of a solution made by adding to 1 g. of *p*-hydroxyphenylglycine 3—4 c.c. of water, then just sufficient nitric acid of *d* 1.12 to dissolve the solid, and making up to 10 c.c. with more water. Hypophosphites and arsenic compounds do not give this reaction, but ammonia and alkaline hydroxides interfere. For colorimetric determination, standard colours are made with a solution of potassium dihydrogen phosphate, which should be of approximately the same concentration with respect to phosphoric acid as the solution to

be analysed. The colour requires 15 min. to attain its maximum intensity. The presence of iron does not cause appreciable error if the ratio of phosphoric oxide to ferric oxide is not less than 0.2. Satisfactory results can be obtained with artificial manures, phosphorites, urine, and glycerophosphates. R. CUTHILL.

Measure of ionimetric acidity [hydrogen-ion concentration] by inversion of sucrose. Application to complex media: soils. V. VINCENT (Compt. rend., 1927, 184, 338—340).—Phosphate buffer mixtures of p_H 7.12 and 6.98 invert sucrose at 100° as if having p_H 5.20 and 4.80 respectively; this is due to increased dissociation at higher temperatures, or "reserve acidity." The application of this to the study of soils, including those containing calcium carbonate, is discussed. E. W. WIGNALL.

Ratio of sulphur trioxide to phosphoric anhydride [in acid phosphate]. THOMAS and HOWES.—See VII.

Solubility of calcium phosphates in citric acid. GRAFTIAU.—See VII.

Quality of bread from various wheats. GERICKE.—See XIX.

PATENTS.

Inducing germination and growth of seeds and plants. J. ZELLWEGER (U.S.P. 1,625,181, 19.4.27. Appl., 1.8.24).—The apparatus consists of a soil bed on a perforated or porous support with a passageway so arranged that air can pass upwards into the soil. The air is forced through by means of a fan, and moisture is supplied at a high temperature to the fan.

C. T. GIMINGHAM.

Destruction of weeds. A. RATHSACK (G.P. 436,405, 15.8.25).—The leaves of weeds are moistened by means of a special spraying device. Materials such as calcium cyanamide, kainit, or anhydrous ferrous sulphate are then applied as a fine powder. G. W. ROBINSON.

Plant sprays. FARBENFABR. VORM. F. BAYER & Co. (F.P. 596,552, 10.4.25. Conv., 19.4.24).—Plant sprays are prepared consisting of nucleus-substituted mercuric derivatives of organic bases with or without addition of other insecticidal, bactericidal, or fungicidal materials. *p*-Acetoxymercurianiline, its *N*-acetyl derivatives, or the products obtained by the reaction of mercuric acetate with toluidines or other nucleus-substituted anilines or *N*-alkylated aromatic amines, or heterocyclic bases such as pyridine, have a more powerful bactericidal and fungicidal effect than the mercury derivatives of phenols and hydroxy-carboxylic acids, and are more readily miscible with other bactericidal and fungicidal substances. G. W. ROBINSON.

Fertilisers. ASAHI GLASS Co., LTD. (Addn. No. 30,648, 18.2.25, to F.P. 556,211).—The colloidal magnesium silicate of the prior patent is changed into a product containing sodium, which is more stable in the colloidal state, by addition of sodium hydroxide or the use of excess of sodium silicate in its preparation. Potassium or ammonium may be introduced in place of sodium. The magnesium may also be replaced by other metals, such as manganese, and the silicic acid partly or completely replaced by boric acid or phosphoric acid.

G. W. ROBINSON.

Production of fertiliser from lignite ash. MASCHINENBAU-ANSTALT HUMBOLDT (G.P. 437,515, 20.4.24).—Harmful constituents are rendered innocuous and the active constituents are decomposed by sintering the ash under oxidising conditions; the product is fused in special kilns, and the clinker obtained is ground to a fine powder. L. A. COLES.

Production of phosphate fertiliser. U. WEDGE, Assr. to I. P. THOMAS & SONS CO. (U.S.P. 1,624,195, 12.4.27. Appl., 7.2.23).—A dry mixture of phosphate rock, potassium chloride, and sodium carbonate is heated in a furnace at reaction temperature, when carbon dioxide is liberated and the phosphoric acid content of the rock rendered soluble. W. G. CAREY.

XVII.—SUGARS; STARCHES; GUMS.

Formation and nature of molasses. J. DĚDEK (Chem. Listy, 1927, 21, 96—101, 163—170).—The colloids separated by the dialysis of diffusion juice, of syrup, molasses, caramel, and beet pectins greatly diminish the velocity of crystallisation of sucrose, but in no case does their presence render a saturated sucrose solution non-crystallisable, *i.e.*, colloids are not the cause of the formation of molasses. Their retarding action is due to their adsorption on crystal surfaces, whereby they interfere with the further growth of the crystals. An examination of the saturation solubility of sucrose in solutions also saturated with respect to various salts shows that certain salts may exert a considerable influence on the crystallisation of sugar. Thus lithium chloride lowers the saturation solubility of sucrose to 43.4% of the normal, whilst potassium iodide raises it to 154.2% of the normal.

R. TRUSZKOWSKI.

Determination of sucrose in dried beetroot cossettes. L. EYNON and J. H. LANE (J.S.C.I., 1927, 46, 177—178 T).—In determining the sucrose content of dried beetroot cossettes by direct polarisation, not less than twice the normal weight, *i.e.*, 52 g. of the material, should be used to insure uniform results. The soluble matter in 52 g. of dried cossettes occupies, on the average, 22.65 c.c., so that the volume of extracting liquid required to make up a total volume of 400 c.c. (a half-normal solution) is $\{400 - (22.65 + 0.52W)\}$, W representing the % of water in the sample. Hence, if the average water content of dried cossettes is assumed to be 6% the amount of liquid required is 374.2 c.c. The procedure consists in heating 52 g. of the sample with 374.2 c.c. of dilute basic lead acetate solution (basic lead acetate solution of d 1.23—1.25 diluted 10- or 20-fold) in a water bath at about 80° for 1 hr., cooling to about 20°, filtering, and polarising the filtrate in a 400 mm. tube; the Ventzke reading represents the % of sucrose in the sample. If the filtrate is not quite brilliant it may be made so by the addition of 0.1 or 0.2 c.c. of acetic acid.

Sugar complexes in wood. FUCHS.—See V.

PATENTS.

Manufacture or recovery of substances from residual liquors of the beet sugar industry. L. MELIERSH-JACKSON. From LARROWE CONSTRUCTION CO. (E.P. 265,831, 17.5.26).—Beet sugar molasses contains

glutamic acid probably in the form of an amide. Desaccharified molasses (*e.g.*, Steffen's waste water, distillery slops, or vinasses, etc.) is treated with hydrogen chloride or hydrochloric acid below 70°. A mixture of betaine hydrochloride and inorganic chlorides separates, from which the former is removed by extraction with methyl or ethyl alcohol or concentrated hydrochloric acid. The original mother-liquor is heated at 80—85°, when the glutamic acid derivative is hydrolysed, and glutamic acid hydrochloride crystallises on cooling. The mother-liquor is further concentrated at 50° (or less) for the recovery of more betaine hydrochloride. In an example, Steffen's waste water (d 1.29—1.4) yielded 20% of betaine hydrochloride, 30% of mixed inorganic chlorides (mainly potassium), and 19% of glutamic acid hydrochloride, calculated on the dry matter of the waste water.

B. FULLMAN.

XVIII.—FERMENTATION INDUSTRIES.

Influence of hydrogen-ion concentration on development of colour during wort boiling. W. WINDISCH and P. KOLBACH (Woch. Brau., 1927, 44, 53—56, 64—67).—In experiments with worts prepared by decoction methods of mashing, as employed in continental brewing, it was found that adjustment of the hydrogen-ion concentration by addition of acid or alkali before boiling influenced profoundly the development of colour during the boiling process. At p_H 6.16 the increase of colour during 2 hrs.' boiling was about 100%, whilst at p_H 5.2 it was only about 30%. At any given p_H value, the increase of colour was proportional to the duration of boiling up to a period of 4 hrs. Hops contribute considerably to the development of colour, but the bitter acids (ether extract) have much less influence in this direction than the portions insoluble in ether. The development of colour during wort boiling is also influenced by the character of the malt and the hops used, and by aeration. It is suggested that in the technical analysis of malts the colour of the standard worts should be determined before and after boiling. J. H. LANE.

Significance of hydrogen-ion concentration in the brewery. H. LÜBERS (Woch. Brau., 1927, 44, 97—103).—The importance now rightly attached to hydrogen-ion concentration in brewing processes should not lead to neglect of titratable acidity. With the use of several indicators showing different p_H values, titration affords useful information regarding the buffer action of worts or extracts. For example, during the malting of barley the p_H value (of cold water extracts) remains almost constant at about 6.0—6.25, but in the course of 4—5 days the titratable acidity towards phenolphthalein (p_H 9) and the titratable alkalinity towards methyl orange (p_H 4.5) both increase to about double their initial values, indicating the formation of soluble buffer substances (phosphates) which stabilise the hydrogen-ion concentration. J. H. LANE.

Standardisation of malt analysis. F. DUCHÁČEK and V. L. ŽILA (Woch. Brau., 1926, 43, 455—460, 467—472).—Before 1903 there was fairly general uniformity in methods for malt analysis on the Continent. Owing

to the partial or regional adoption of modifications, recommended by a brewing convention at Bonn in 1914 and another at Hanover in 1920, there is now considerable diversity. In most countries the Balling table of densities of sugar solutions has been replaced by the more accurate one compiled by Plato in 1900 as the basis of extract determinations. The new table is calculated for $15^{\circ}/15^{\circ}$ in France and Sweden, for $20^{\circ}/4^{\circ}$ in North Germany, and for $17.5^{\circ}/17.5^{\circ}$ in Bavaria and Czechoslovakia. It indicates amounts of extract about 1% higher than those found by the Balling table. Several test samples of malts were sent by the authors for analysis to 11 laboratories in Germany, and the results are now compared and discussed, and some suggestions are made with a view of ensuring more concordant results. J. H. LANE.

Simplification of work in the analysis of malt and beer. DOEMENS (Woch. Brau., 1926, 43, 477—481).—Suggestions are made for saving time and work in routine analyses. The sp. gr. of worts can be rapidly and accurately determined by the float method, in which distilled water is added to 200 c.c. of the wort until a bulk, of accurately standardised density, neither floats nor sinks. By noting the temperature of the wort and the volume of water added, the sp. gr. of the wort can be at once ascertained from a table. The method is not so suitable for beers. In the pyknometric weighing of alcoholic distillates barometric variations may cause differences of as much as 5 mg. in the apparent weight of a filled 50 c.c. flask. J. H. LANE.

Head on beer from the physico-chemical point of view. C. GEYS (Woch. Brau., 1927, 44, 145—147).—The colloidal particles in beer which contribute most to head-formation and -retention are not the extremely small ones which are electrically charged and disinclined to coagulate, but those of larger size, which are almost or quite neutral, and therefore strongly inclined to coagulation. The latter type of particles tend to accumulate in the surface films, on which they confer stability. During the later stages of brewing, from the cooling of the wort onwards, there is, on the whole, a tendency for the colloidal constituents of the wort to increase in size. This is advantageous for head formation. Excessive coagulation may, however, be detrimental as it removes from the wort many of the particles which would otherwise assist the formation of foam. Long contact of beer with yeast may also be detrimental owing to the peptising action of yeast enzymes on colloids of low dispersion. J. H. LANE.

Aluminium in the brewery. M. VON SCHWARZ (Woch. Brau., 1926, 43, 523—525, 541—545).—Recent experience seems to indicate that aluminium of 99% purity gives better results as a material for brewing vessels than that of lower purity or even that of 99.5%. A defect that occasionally appears a few months after the metal has been rolled is the appearance of very small blisters on the surface, probably due to the presence of minute crystals of a compound FeAl_3 , formed when the metal is molten and accumulated at the surface in the subsequent rolling. On contact with an electrolyte this compound undergoes corrosion, yielding alumina and hydrogen, but the injury to the metal is usually slight.

Aluminium vessels encased in masonry with a layer of pitch between have sometimes corroded owing to the presence of chlorine in the pitch. Unencased vats and tanks are preferred. J. H. LANE.

Empiricism and science in brewing. W. WINDISCH (Woch. Brau., 1927, 44, 1—5, 13—17, 25—29, 37—41, 49—52, 61—64, 73—76, 85—92).—A personal appraisal of the contributions of science to some of the more important problems of the brewing industry. J. H. LANE.

Bottom- and top-fermentation yeasts. E. RATHKE and F. WINDISCH (Woch. Brau., 1926, 43, 537—541).—Mainly a description of the results obtained by Euler (cf. A., 1926, 1176). J. H. LANE.

Fermentation and ripening of beer. L. NATHAN (Woch. Brau., 1926, 43, 585—589, 599—602).—A defence of the Nathan process of brewing against certain criticisms. J. H. LANE.

Saccharification and fermentation of glycogen and starch by maltase-free yeasts. A. GOTTSCHALK (Woch. Brau., 1926, 43, 487—488).—See A., 1926, 544, 759.

PATENTS.

Denaturising of alcohol. M. TAGLIETTI and S. HIRSCH (Swiss P. 115,929, 18.3.25).—As denaturant, rectified oils from ichthyol-shale distillate are used.

S. S. WOOLF.

Dehydration of alcohol (F.P. 608,062).—See XX.

Treatment of waters for brewing (E.P. 261,708).—See XXIII.

XIX.—FOODS.

Quality of bread from wheats supplied with nitrogen at different stages of growth. W. F. GERICKE (Cereal Chem., 1927, 4, 73—86).—Six varieties of wheat received similar applications of sodium nitrate (equal to 100 lb. of nitrogen per acre) at the time of planting and at subsequent growth periods. In some cases addition of nitrogen caused no change in the time of ripening, in others the maturity was delayed. When nitrogen was applied 45 or 75 days after planting, the grain contained the greatest amount of protein. The variation of the quality of the flours, as expressed by the loaf volume and texture of the bread, with the protein content and time of ripening is discussed.

F. R. ENNOS.

Wheat and flour studies. X. Factors influencing the viscosity of flour-water suspensions. I. Effects of temperature, degree of hydration, and method of manipulation. A. H. JOHNSON (Cereal Chem., 1927, 4, 87—128).—The viscosity of flour-water suspensions depends on the quantity of electrolytes present and the degree of hydration of the flour constituents. The viscosity is greater the higher the temperature of the water used in the preliminary extraction, since the degree of hydration and the amount of electrolyte removed are increased. Lengthening the period of extraction at lower temperatures and shaking more vigorously have a similar effect. The increase in viscosity is accompanied by a diminution in the resistivity of the wash water, but there is no relationship between the two, owing to the effect of hydration of the

flour, especially at higher temperatures, on the viscosity of the suspensions. Although the electrolyte contained in flour probably consists mainly of dipotassium hydrogen phosphate, sufficient of other electrolytes is present to prevent the calculation of the ash content, by comparison of the resistivities of the decantates with those of this salt. At 50° a change begins to take place in the properties of the flour-water suspensions, as is shown by a sharp increase in the viscosity. By viscosity measurements made after removal of the proteins by pepsin, it is shown that the increase is due mainly to hydration of the proteins, and to a much less extent of the starch. The employment of the viscosimetric method for determining the rate of proteolytic activity in a flour suspension is only possible if the degree of hydration of the flour and the quantity of electrolytes present can be made constant. The method of extraction recommended is to wash with several litres of distilled water at 40° until a maximum viscosity is obtained.

F. R. ENNOS.

Determination of milk solids in food products. I. Direct method for milk proteins in cacao products. H. G. WATERMAN and H. A. LEPPER (Ind. Eng. Chem., 1927, 19, 501—506).—A method is described, directly applicable to milk chocolate, for the separation of casein, lactalbumin, and lactoglobulin as a group from the cacao protein group. Preliminary experiments showed the necessity of removing (a) fats by extraction with ether and (b) cacao proteins by first extracting with acidified alcohol (85% by vol. of anhydrous ethyl alcohol and 1% by vol. of glacial acetic acid), and then with a weakly acid aqueous medium (5 g. of anhydrous sodium sulphate in 1 litre of 0.015N-hydrochloric acid, equivalent to p_H 2.0). The residue is then dehydrated with alcohol followed by ether. Extraction of the milk proteins is performed at p_H 11.6—11.8 by means of a mixture of borate and caustic soda, the borate exerting a buffer action, so that the fall in p_H during the extraction is not more than 0.4—0.6 unit. After centrifuging and filtering, total nitrogen in the extract is determined by the Kjeldahl method. Milk proteins are then determined by selective precipitation with 0.1N-sulphuric acid at about p_H 4.5, which point is determined by titration with methyl red as indicator. After removal of the precipitate, non-milk protein nitrogen is determined in the filtrate by the Kjeldahl method. The difference between the two nitrogen figures represents that due to casein, lactalbumin, and lactoglobulin, the conversion factor 6.38 being used.

H. J. DOWDEN.

Examination of raw milk by the small plate method. A. M. VAN DEN BURGH (Pharm. Weekblad, 1927, 64, 417—423).—Comparative figures on the determination of bacteria in milk by the method devised by Frost in 1915 and modified by Clarenburg in 1925 are given, and the method is strongly recommended for the examination of raw milk. With pasteurised milk low results are obtained.

S. I. LEVY.

Bacteria causing spoilage of evaporated milk. C. D. KELLY (Trans. Roy. Soc. Canada, 1926, [iii], 20, V, 387—394).—Three types of spoilage are classified, the bacteria responsible for the changes described being

typical forms of *Bacillus cereus*, Frankland, *Bacillus simplex*, Gottheil, and *Bacillus megatherium*, De Bary.

A. WORMALL.

Critical solution temperatures and butter analysis. J. WAUTERS (Bull. Soc. chim. Belg., 1927, 36, 271—276).—Although a normal Crismer value (critical solution temperature in absolute or nearly absolute alcohol) alone is not a proof of absence of adulterants in butter, the sum of the Crismer value and the Reichert-Meissl value is less than the normal 83—84 when cacao butter, neutral lard, or oleomargarine is present. A further confirmation of adulteration is obtained by a determination of insoluble volatile acids (Wauters and Reychler method), which gives higher figures with adulterated than with pure butter. Priority as against Polenske is claimed.

C. HOLLINS.

Production of a "caramel" odour and flavour in dairy products by *Streptococcus lactis*. Lister. W. SADLER (Trans. Roy. Soc. Canada, 1926, [iii], 20, V, 395—409).—Organisms, classified as typical *Streptococcus lactis*, Lister, strains, proved to be responsible for the specific and undesirable flavour of a sample of butter. Remedial and preventive methods, based mainly on sterilisation of the apparatus, are outlined.

A. WORMALL.

Gas storage of fruit. F. KIDD, C. WEST, and M. N. KIDD (Dept. Sci. Ind. Res., Food Investigation Special Rep., 1927, No. 30. 87 pp.).—In an attempt to prolong the storage life of fruit by storing in an atmosphere rich in carbon dioxide, with or without temperature control, apples alone being studied, the fruit was stored in closed chambers, restricted ventilation being controlled by opening and closing air ports. The respiratory processes of the apples result in a rise of temperature and an increase in the carbon dioxide content of the atmosphere with a corresponding diminution in that of oxygen. The principal difficulties encountered in storing under restricted ventilation were an increase in humidity to 90—98% and an accumulation of various volatile organic products in the stagnant atmosphere. A high humidity as such has not been proved to be deleterious, but the actual deposition of water on the fruit encourages growth of fungi. The use of oiled-paper wrappers for the apples, or embedding in sawdust or oiled sawdust, removes the ill-effects of the stagnant atmosphere, which otherwise encourages superficial scald. An atmosphere of approximately 10% of carbon dioxide and 11% of oxygen was found to be the most suitable; higher concentrations of carbon dioxide led to a danger of wastage due to brown heart. With this atmosphere, increases in storage life of 50—100% were recorded. Rise in temperature due to self-heating was an important factor, and some form of temperature control was found to be desirable. A temperature of about 10° is believed to be the most suitable. At this temperature an increase in storage life of 100% on a semi-commercial scale has been recorded, but at higher or lower temperatures the efficiency of the method falls off. This is ascribed to variations in the concentration of oxygen and of carbon dioxide in solution in the tissue fluids. At too low temperatures the carbon dioxide concentration in the tissue fluids becomes too high and may lead to brown

heart; at too high temperatures the oxygen content is insufficient to support respiration. The preservation of the colour, firmness, and juiciness of the apples is associated with the prolongation of the storage life. The preserving action of the atmosphere used is found to be proportional to the reduced respiratory activity of the fruit. Discussing overseas transport of fruit, the authors ascribe the varying success of transport in unventilated holds to variations in the extent of accidental ventilation.

F. H. GEAKE.

Edible viscera. A. M. WRIGHT and J. C. FORSYTH (J.S.C.I., 1927, 46, 36—38 T).—Edible viscera and blood represent 24% of the dressed weight of the sheep, 20% of the lamb, and 14% of the steer. Results collected from the work of various other investigators show that vitamins-A and -B are widely distributed amongst the edible viscera, occurring chiefly in the kidney, liver, and brain, whilst vitamin-C also occurs in notable quantity in the kidney and the liver. The results of chemical analyses of the edible viscera from sheep and oxen are tabulated. A canned mixture of hearts and diaphragms marketed as "hearts and skirts" has a very high food value. Blood is a valuable source of albumin.

F. H. GEAKE.

Sterilisation of green fodder by a liquid medium. I. C. BRAHM, G. ANDRESEN, and R. PRILLWITZ (Biochem. Z., 1927, 181, 96—104).—If green fodder be kept in 0.1%—or, better, in 0.2%—hydrochloric acid, the butyric acid fermentation is largely suppressed. Acetic acid fermentation, however, is little affected.

C. R. HARRINGTON.

Food dye. JOHNSON and STAUB.—See IV.

PATENTS.

Treatment of condensed or evaporated milk. A. E. VIDAL. From ANDERSON-BARNGROVER MANUF. CO. (E.P. 268,138, 6.4.26).—The hermetically-sealed tins of condensed milk are passed through a steam-heated chamber with a number of divisions, each of which has a higher temperature than the preceding one, the temperature of the last being below that required for sterilisation. After automatic removal of the leaky tins, the remainder are heated in a second chamber at the sterilising temperature (115°), cooled in a third chamber at a pressure above atmospheric, and again passed through a leak detector.

F. R. ENNOS.

Manufacture of an invalid food from milk. K. HOEFELMAYR (E.P. 265,910, 23.4.26. Conv., 12.2.26).—Condensed milk, milk powder, or curdled milk is subjected to the action of ultra-violet light for some time. The product has antirachitic properties. [Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 236,197; B., 1925, 735.]

H. ROYAL-DAWSON.

Process for obtaining concentrated vitamin preparations. L. A. AGOPIAN (E.P. 268,655, 24.9.26; cf. E.P. 168,903; B., 1923, 470 A).—Fresh vegetables or fruit are crushed, and the mixture of juice and pulped residue is treated with a solution of lead acetate (80 pts.) and copper acetate (1 pt.). The precipitate, after separation by the filter press, is washed with water, dried, and powdered, the vitamin A being extracted with cold light

petroleum. The extract is concentrated *in vacuo*, and the vitamin either diluted with a suitable vegetable oil or further concentrated by extraction with 99% methyl alcohol.

F. R. ENNOS.

Obtaining extractives from coffee [grounds]. K. VON VIETINGHOFF (E.P. 268,241, 20.10.26).—Coffee grounds are heated with water under a pressure of 8—12 atm. at 160—200°, and the resulting solution, which contains a further 12—18% of aromatic extract, is evaporated if desired.

F. R. ENNOS.

Removing the bitter constituents from lupins. GES. FÜR LUPINEN-IND. M.B.H. (G.P. 437,355, 4.5.22. Addn. to G.P. 335,646; B., 1921, 599 A).—Loss of protein is diminished by using solutions containing nitrates, sulphates, or phosphates, instead of salt solution as described previously.

L. A. COLES.

Sterilising milk and other products in glass bottles or other containers. F. HUMBEECK (E.P. 268,603, 23.6.26).

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

Preparation of formaldehyde by catalytic dehydrogenation of methyl alcohol. I. J. C. GHOSH and J. B. BAKSI (J. Indian Chem. Soc., 1926, 3, 415—430).—The effects of physical aggregation, temperature, and the presence of various promoters on the copper catalyst in the preparation of formaldehyde by the catalytic dehydrogenation of methyl alcohol have been studied. A pure copper catalyst retains its activity for a longer period when the oxide (precipitated from copper acetate, not sulphate) is reduced in the form of small, loose pills or granules (between 10- and 20-mesh) than when it is used in the form of powder. Silver is useless as a promoter, whilst nickel increases the initial activity of the catalyst, which, however, rapidly loses its activity. The best promoters are thorium and cerium oxides. A catalyst prepared from copper acetate containing 0.1% of thorium nitrate at 205° had a space velocity of 900, and yielded approximately 15 g. of formaldehyde per hr. from 16.8 g. of methyl alcohol, and retained its activity unimpaired for an almost indefinite period. With increase in the percentage of thorium or rise of temperature the percentage decomposition of the formaldehyde into carbon monoxide and hydrogen increases. The most efficient catalyst is prepared from copper acetate containing 0.1% of cerous nitrate, which at a reaction temperature of 200° had a space velocity of 1774, and gave an almost quantitative yield of formaldehyde. This catalyst is about twice as efficient as the 0.1% thoria preparation.

J. W. BAKER.

Standards for rectified ethyl alcohol. A. V. RAKOVSKI (Trans. Inst. Pure Chem. Reagents, Moscow, 1927, 26—30).—Standards are suggested for the physical properties and for the permissible impurities in ordinary rectified, double rectified, and purest (prima prima) alcohol, and results of analysis are given for a number of samples received.

T. H. POPE.

Preparation of camphor from pinene. H. M. MULANY and E. R. WATSON (J. Indian Chem. Soc., 1926,

3, 253—254).—The preparation of camphor from Indian turpentine may be effected by heating the pinene with 2 pts. of salicylic acid at 110° for 17 hrs. and then gradually raising the temperature to 150° in 11 hrs., a 70.8% yield of bornyl salicylate being obtained. This, on hydrolysis with 5 pts. of 20% alcoholic potassium hydroxide solution for 8—10 hrs., gives borneol in 44% yield. The latter is oxidised to camphor by shaking with 5 pts. of 50% nitric acid for 3 hrs. (yield 50%).

J. W. BAKER.

Manufacture of strychnine and brucine from *Nux vomica*. E. R. WATSON and H. D. SEN (J. Indian Chem. Soc., 1926, 3, 397—400).—Strychnine may be economically extracted from *Nux vomica* by heating the nuts (41 lb.) with 1.25 times their weight of water for 3 hrs. in an autoclave at 30 lb. pressure, mixing with freshly slaked lime (13 lb.), passing the product through a disintegrator, and extracting with 20 gals. of kerosene oil at 90° for 1 hr. with vigorous stirring in a closed pan. The alkaloid is extracted from the kerosene solution (separated by centrifuging) with 40 lb. of dilute sulphuric acid (containing 240 g. of acid, d 1.84), and on concentration of the acid liquor the strychnine sulphate (6.8 oz.) crystallises out. Alternatively, the alkaloid may be precipitated from the acid liquor with soda ash, and the brucine removed by extraction with acetone after drying. The insoluble residue is dissolved in hot, dilute sulphuric acid, from which the strychnine sulphate crystallises out on cooling.

J. W. BAKER.

Biological assay of the specific alkaloid of ergot. J. H. BURN and J. M. ELLIS (Pharm. J., 1927, 118, 384—387).—The method of Broom and Clark (J. Pharm. Exp. Ther., 1923, 22, 59) for the biological assay of the specific alkaloid of ergot gives results varying in accuracy from ± 10 to $\pm 25\%$. Examination by this method of extracts of ergots of different origin indicates the following content of alkaloid in the ergot: Portuguese 0.133%, Spanish 0.12—0.067%, Russian 0.005%, and Polish 0.03%. The identity in pharmacological activity of ergotamine and ergotoxin is confirmed.

B. FULLMAN.

Albumose component of *Argentum proteinicum*. C. MANNICH and T. GOLLASCH (Arch. Pharm., 1927, 265, 96—104).—Nine samples of Protargol of various dates and five other competitive brands all gave the biuret reaction, but only Protargol gave the tryptophan reactions with ferric chloride and formaldehyde and with glyoxylic acid, indicative of the presence of animal albumoses. Protargol also behaved differently towards precipitating agents such as absolute alcohol, 30% acetic acid, and 50% magnesium sulphate solution. Dialysis experiments and precipitation with dialysed ferric chloride solution furnished similar evidence; other brands differed from Protargol in that they contained a less uniform albumose, and more material remained unprecipitated by the iron sol.

S. COFFEY.

Detection of vegetable poisons and drugs in decomposed cadavers. A. BRÜNING and B. KRAFT (Arch. Pharm., 1927, 265, 106—109).—The remains of the viscera from a case of scopolamine poisoning, which ended fatally in 1923, were kept for three years, during which time decomposition took place. Scopolamine was

identified, however, in the decomposed material by the Stas-Otto method.

S. COFFEY.

Determination of total geraniol content in citronella oil and examination of this oil in general.

A. RECLAIRE and D. B. SPOELSTRA (Perf. Ess. Oil Rec., 1927, 18, 130—132).—The formation of citronellal diacetate during the acetylation of citronella oil has been confirmed (cf. de Jong, B., 1919, 232 A), and to the formation of this substance, which is unchanged by heating in a boiling water bath after the addition of water, are ascribed the discrepancies observed in the determination of the "total geraniol" content by different analysts. A method to eliminate the difficulties caused by the presence of citronellal is described. The oil is oximated according to the method of Dupont and Labaune (B., 1924, 926), and in a part of the oximated oil, in which the citronellal is present as citronellal-oxime, the nitrogen content is determined by Kjeldahl's method. From this figure the citronellal content is calculated and the geraniol is determined in the oximated oil by acetylation in the ordinary way. The sum of both values is called the "total geraniol" content. Analyses of five citronella oils by different methods and by independent analysts are described. The differences found in the determination of the citronellal, geraniol, and "total geraniol" by the author's methods are much smaller than those given by the recognised methods.

E. H. SHARPLES.

Determination of iodine values. J. BÖESEKEN and E. T. GELBER (Rec. trav. chim., 1927, 46, 158—171).—In contrast to the ideas of earlier workers the authors take the view that the low or absent iodine values in the case of certain compounds depend not only on the rate of addition of the halogens, but also on subsequent side-reactions of the saturated compound. The authors agree with Ingle (A., 1902, i, 528; 1904, ii, 456) that when the solution of Wijs is employed iodine and chlorine, but not iodine and hydroxyl, are added to the double linking, and they have used this solution to obtain rapidly true iodine values for many substances which contain only one double linking. The so-called negative groups, e.g., carboxyl, phenyl, acetyl, etc., have a retarding influence on the speed of the reaction. In the case of compounds having two (or three) double linkings, it is found that unconjugated systems react rapidly and completely in the normal way, whereas in conjugated systems addition takes place in accordance with the Thiele theory, and the first (or second) addition is rapid, but the final double linking is only slowly saturated. This affords a simple method of distinguishing between conjugated and unconjugated systems, and the authors obtain by Wijs' method iodine values for elæostearic acid which confirm the view that it contains three conjugated double linkings (A., 1925, i, 507). The influence of side-reactions is discussed, and in those compounds in which there is a double linking in the $\alpha\beta$ -position to a so-called negative group, the incorrect values are due either to the influence of iodine ions, or to the occurrence of equilibrium reactions, or to the solvent used (dissociating or non-dissociating). Marshall's solution (B., 1900, 213 A) is suitable for the determination of $\alpha\beta$ -unsaturated acids. Ingle's theory

of the hydrolysis of the iodochlorides is considered to be incorrect. J. M. GULLAND.

Chemical aspects of organic evolution. J. C. DRUMMOND (J.S.C.I., 1927, 46, 161—166 t).

Extraction devices. CLARK, also PALKIN and WALKINS.—See I.

Liquids for mineralogical analyses. SULLIVAN.—See X.

Turpentine oil in industry. AUSTERWEIL.—See XIII.

PATENTS.

Manufacture of concentrated solutions of formaldehyde or of paraformaldehyde. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (E.P. 267,768, 18.6.26).—Methyl alcohol, substantially free from water, is catalytically oxidised, and the reaction gases are rapidly cooled so as to condense a mixture of formaldehyde and water vapour, which is then fractionated by means of the uncondensed gases. The resulting concentrated solution of formaldehyde (about 60%) is allowed to solidify to a tallow-like mass, and may be converted into a dry powder by heating *in vacuo*. S. S. WOOLF.

Dehydration of alcohol. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (F.P. 608,062, 17.12.25. Conv., 18.12.24).—The condensed azeotropic mixture is treated with a concentrated solution of potassium carbonate, magnesium chloride, sodium sulphate, or sodium or potassium acetate. S. S. WOOLF.

Preparation of chloro-derivatives of ethane. I. G. FARBERIND. A.-G., Assees. of M. MÜLLER-CUNRADI (G.P. 436,999, 30.3.21).—Ethane, or a mixture of ethane and ethylene previously reduced, is treated with chlorine in the presence of a catalyst, particularly of active carbon. Equal volumes of ethane and chlorine, led over active carbon at 100—300°, give equal quantities of ethyl chloride and ethylidene dichloride, with small amounts of *ααα*-trichloroethane. If the mixture of ethylene with a little ethane and other gases adsorbed from coal-gas by active carbon is reduced with the hydrogen corresponding with the ethylene content, and is then passed with an equal volume of chlorine over heated active carbon, there are obtained ethyl chloride and some ethylidene dichloride. Other catalysts, *e.g.*, active silicic acid, may be used. C. HOLLINS.

Production of alkyl [tetraethyl] compound of lead. G. CALINGAERT, ASST. to GENERAL MOTORS CORP. (U.S.P. 1,622,233, 22.3.27. Appl., 17.1.24. Renewed 7.11.25. Cf. U.S.P. 1,539,297; B., 1925, 652).—An alloy of lead with 18% of sodium (50 pts.), ethyl iodide (64 pts.), and pyridine (2 pts.) are heated together at 72° under reflux, and water (4 pts.) is added during 2 hrs., at a rate less than that at which the reaction proceeds. The lead tetraethyl, which is formed in satisfactory yield, is removed by distillation with steam.

T. S. WHEELER.

Glycol ethers and cellulose ester solvents. CARBIDE AND CARBON CHEMICALS CORP., Assees. of J. G. DAVIDSON (E.P. 255,406, 21.10.25. Conv., 20.7.25).—As solvents for cellulose esters, mono- and poly-propylene glycol ethers, particularly the methyl, ethyl, and aryl

ethers, are used. The monoethers of monopropylene glycol are prepared by heating propylene oxide with the alcohol or phenol in an autoclave at 100—200°, whilst the diethers are obtained from propylene glycol or propylene chlorohydrin by boiling with an alkyl sulphate in presence of alkali. The monoethers of polypropylene glycols, *e.g.*, the ethyl ether of dipropylene glycol, are formed as by-products in the former reaction. The propylene glycol ethers show high solvent power for cellulose esters, the monoethers surpassing the diethers in this respect. They are miscible with most of the common solvents, and are free from disagreeable odour.

A. DAVIDSON.

Separation and purification of vanillin. J. TCHERNIAC (E.P. 268,158, 27.4.26).—Crude vanillin is converted into a somewhat concentrated solution of its sodium bisulphite compound (*e.g.*, 100 pts. of vanillin are dissolved in 328 pts. of a sodium bisulphite solution containing 12.5% of sulphur dioxide), which is then salted out, preferably by the addition of sodium chloride (other salts of sodium, or salts of other metals, *e.g.*, aluminium chloride, may be used); or the solution of the sodium bisulphite compound is treated with the equivalent of a salt (*e.g.*, potassium chloride) which will give an insoluble vanillin-bisulphite compound by double decomposition. The bisulphite compound yields substantially pure vanillin on acidification. B. FULLMAN.

Preparation of camphor from oil of turpentine. C. DANIER (F.P. 579,277, 7.6.23).—Turpentine oil of suitable origin is mixed with 1.5% of caoutchouc as polymerisation agent, and saturated with hydrogen chloride. The almost solid mass is melted with 10—20% of metallic chromium, and after slow cooling is decanted from impurities. The solidified oil is pressed, and yields a synthetic camphor of m.p. equal to that of natural camphor. The process, it is claimed, enables turpentines to be used which are otherwise valueless on account of their mercaptan content. C. HOLLINS.

Recovery of volatile organic substances from gas mixtures. I. G. FARBERIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 262,404, 3.11.26. Conv., 3.12.25).—Such solvents as liquid triaryl phosphates or solutions of solid in liquid triaryl phosphates are claimed, *e.g.*, mixtures of isomeric tricresyl phosphates, or the mixed esters obtained according to E.P. 181,835 (B., 1922, 647 A), or solutions of triphenyl phosphate in mixed tricresyl phosphates. The triaryl phosphates have the advantage of low inflammability and volatility, and high solvent power and b.p. B. FULLMAN.

Preparation of calcium acetylsalicylate. C. R. JACOB (F.P. 609,720, 21.1.26. Conv., 20.10.25).—Carbon dioxide is passed into a mixture of acetylsalicylic acid, calcium carbonate, and just sufficient water to allow the calcium acetylsalicylate to be precipitated as formed. The calcium carbonate is added gradually at the same time as the carbon dioxide. No hydrolysis of the acetyl group occurs. C. HOLLINS.

Preparation of γ -dialkylamino- α -hydroxy-acids and their derivatives (esters and amides). I. G. FARBERIND. A.-G., Assees. of O. EISLEB (G.P. 436,521, 7.12.23).—Dialkylaminoketones are converted into

cyanohydrins which give acids, esters, and amides by appropriate hydrolysis. The *cyanohydrin* of methyl β -dimethylaminoethyl ketone, treated with benzoyl chloride, gives an *O*-benzoate, b.p. 180°/9 mm. (*hydrochloride*, m.p. 206°). When the moist cyanohydrin is dissolved in 37% hydrochloric acid below 35°, heat is developed and a precipitate is formed, which dissolves on further heating, and by evaporation a mixture of ammonium chloride and the *hydrochloride* of γ -dimethylamino- α -hydroxy- α -methylbutyric acid is obtained. The crude hydrochloride is converted directly into the *methyl ester*, b.p. 85°/12 mm., *ethyl ester*, b.p. 85–90°/9 mm. or 219–222°/756 mm. (*O*-benzoate, b.p. 180°/9 mm.; *O*-benzoate *hydrochloride*, m.p. 162°), and *butyl ester*, b.p. 117–118°/9 mm. The *cyanohydrin* of methyl β -diethylaminoethyl ketone yields with alcoholic hydrogen chloride equal amounts of *ethyl γ -diethylamino- α -hydroxy- α -methylbutyrate*, b.p. 112–115°/9 mm. or 245–247°/756 mm. (*O*-benzoate, b.p. 195/10 mm.), and *γ -diethylamino- α -hydroxy- α -methylbutyramide*, m.p. 61–62°, b.p. 150–175°/8 mm. (*O*-benzoate *hydrochloride*, m.p. 167°). The *methyl ester*, b.p. 110°/12 mm. (*O*-benzoate *hydrochloride*, m.p. 110°), *propyl ester*, b.p. 125°/10 mm. or 259–261°/756 mm. (*O*-benzoate, b.p. 203°/10 mm.; *O*-benzoate *hydrochloride*, m.p. 114–116°), and *butyl ester*, b.p. 140°/12 mm. (*O*-benzoate, b.p. 215°/12 mm.; *O*-benzoate *hydrochloride*, m.p. 112°), are described. *Methyl β -dimethylaminoisopropyl ketone*, b.p. 60–64°/20 mm., gives a *cyanohydrin*, from which are obtained *ethyl γ -dimethylamino- α -hydroxy- α - β -dimethylbutyrate*, b.p. 95–100°/9 mm. or 223–227°/760 mm. (*O*-benzoate, b.p. 182°/8 mm.). The products have therapeutic interest.

C. HOLLINS.

Preparation of organic arsenic compounds.

DEUTSCHE GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESLER (Aust. P. 100,211, 4.3.22).—Halogenated aromatic aldehydes and ketones react with sodium arsenite in aqueous alcoholic solution at 150–200° in the presence or absence of copper or a copper compound. The arsenic acids so formed have trypanocidal action. Examples are *acetophenone-p*-arsenic acid, prepared from *p*-bromoacetophenone and sodium arsenite in aqueous alcohol at 160–170°, and *3-aminoacetophenone-4*-arsinic acid, decomp. 230° (*sodium salt* described), prepared from 4-bromo-3-aminoacetophenone, sodium arsenite, and copper powder in aqueous alcohol at 180°.

C. HOLLINS.

Purification of bisalkylxanthenes. A. ROTHMANN and H. STEIN, Assrs. to C. F. BOEHRINGER & SÖHNE, G.M.B.H. (U.S.P. 1,626,558, 26.4.27. Appl., 12.12.24. Conv., 28.1.24).—See G.P. 431,752; B., 1926, 902.

Products from coal (E.P. 268,006).—See II.

Preparation of olefines (E.P. 248,375).—See II.

Phenols from tar (G.P. 436,444).—See III.

Substances from residue liquors (E.P. 265,831).—See XVII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photochemical studies. IX. Action of certain oxidants on the latent image. A. REYCHLER (Bull. Soc.

chim. Belg., 1927, 36, 76–78; cf. B., 1926, 515).—An exposed plate is treated for a few minutes with an oxidising solution—chromic acid, acid ferricyanide, mercuric chloride, or acid permanganate. Immediate development gives a very attenuated negative since the insolated grains have been largely destroyed. Exposure to light, followed by development, gives a positive due to insolation of grains previously left unattacked. Removal of the unattacked grains with thiosulphate, followed by development (several hours in full daylight) with a developer made up from sodium sulphite, mercuric bromide, and metol, yields a very vigorous negative, due to development and intensification of the remains of the latent image.

C. HOLLINS.

PATENTS.

Mercurial printing surfaces. A. R. TRIST (E.P. 265,294, 5.11.25; cf. E.P. 225,928; B., 1925, 152).—A planographic printing plate having ink-retaining and mercurial ink-refusing areas, consists of an iron base on which is electro-deposited first a layer of metal with which mercury can amalgamate, *e.g.*, copper, and then a layer of metal unaffected by mercury to which printing ink will adhere, *e.g.*, nickel. Local resists are applied to the nickel layer to protect the printing areas, and the unprotected parts are then etched away to expose the copper layer and form recesses, which are then filled up with a series of alternately electro-deposited films of two metals, one of which forms a hard amalgam with mercury, such as silver or gold, whilst the other takes a high polish when treated with mercury, such as copper, a film of the latter being outermost; by this means diffusion is reduced to a minimum, and it is possible to arrange that each recess is exactly filled up. The non-printing areas are then treated with mercury.

M. E. NOTTAGE.

Photographic processes [involving synthetic resins from furfuraldehyde, pyrrole, and thiophen]. H. WADE, From WADSWORTH WATCH CASE Co. (E.P. 265,769, 4.2.26).—Resins produced by polymerisation of five-membered monoheterocyclic compounds such as furfuran, pyrrole, thiophen, and their derivatives, or by condensation of furfuraldehyde with ketones and amines, particularly β -naphthylamine, are applied as photosensitive films on suitable surfaces. A sensitiser consisting of a source of halogen, such as iodoform, may be incorporated with the resin. Photographic images can be formed in such media, and are developed by the use of solvents or by selective dyeing. *E.g.*, a mixture of 100 g. of furfuraldehyde and 100 g. of concentrated ammonia solution forms, at room temperature, or under gentle heating if necessary, a resinous condensation product. After drying at 105° it is dissolved in benzene or acetone and applied as a thin film to the surface on which the photographic print is to be made. After exposure to light, the image is developed in a 25% solution of benzene in turpentine.

A. DAVIDSON.

XXII.—EXPLOSIVES; MATCHES.

Detonation wave from solid explosives. W. C. HOLMES (J. Franklin Inst., 1927, 203, 549–559).—The detonation waves of explosives with a detonation velocity of 2000–2500 m./sec. are found to travel with an increase in velocity of 100–200 m. when the wave

leaves the explosives and travels in the surrounding air. In the case of explosives with a high detonation velocity of 5800—6400 m./sec. a decrease in velocity of 1200—1400 m./sec. is observed. It is found that beyond the surface of the explosive the wave travels for a certain distance at a constant velocity and then decreases steadily, and this zone of constant velocity seems to vary with the diameter of the explosive cartridge. With a cartridge of 4 in. diameter the distance is 25 in. compared with 10 in. for a diameter of 1.25 in. as determined by tests on spheres cut out from cartridges of various sizes. Values are calculated for the velocity of the detonation wave in air on the assumption that the wave travels with the velocity of propagation of the gaseous molecules, which is given by the Clausius formula, $v = 29.354 \sqrt{T/D}$ where T is the temperature ($^{\circ}\text{Abs.}$) and D is the density of the gas (air = 1). T is obtained by assuming that the gasification of the explosive takes place adiabatically, and that the Rankine formula is valid. Good agreement between calculated and observed velocities is obtained for blasting gelatin, high-velocity gelatin dynamite 60%, gelatin dynamite 40%, and dynamite 40%. S. BINNING.

PATENT.

Manufacture of detonating compositions for explosive and percussion caps. E. VON HERZ (U.S.P. 1,625,966, 26.4.27. Appl., 21.10.25. Conv., 23.10.24).—See E.P. 241,892; B., 1926, 966.

XXIII.—SANITATION; WATER PURIFICATION.

[Water] chlorination for algæ control. C. COHEN (J. Amer. Water Works Assoc., 1927, 17, 444—455).—In Texas conditions are extremely favourable for algæ multiplication, and the methods of control in general use, which involve treatment with copper sulphate and frequent tank and reservoir cleansings etc., are expensive. Laboratory experiments indicated that algal growth could be inhibited with the application of 1.0 p.p.m. of chlorine. The application of this amount to water entering clean reservoirs and the like was successful in preventing a recurrence of heavy algal growth, but complete freedom from algæ forms was not attained. In another case, where, after nozzle aeration and passage through a filter and prior to distribution, 24 hrs. settlement of the water was customary, the application of 1.1 p.p.m. of chlorine at the inlet of the settlement tanks, instead of 0.5 p.p.m. (which was usually applied to the settled water for bacterial control), considerably reduced the cost of algæ control, and the treated effluent was as satisfactory from a bacteriological point of view as previously. Further laboratory experiments indicated that dosages of 1.0 and 1.5 p.p.m. of chlorine are effective within periods of 6 and 3 hrs., respectively, and even 0.05 p.p.m. of residual chlorine exercises some effect after 12 hrs.; re-growth of algæ due to seeding and spores does not recur until 8 days from stoppage of chlorine treatment, and that protococcus forms, somewhat resistant to copper sulphate, were susceptible to the action of chlorine. W. T. LOCKETT.

Dissolved oxygen changes during filtration [of water]. W. U. GALLAHER (J. Amer. Water Works Assoc., 1927, 17, 476—480).—There is a gradual increase in the amount of oxygen removed by a filter as the

temperature of the applied water increases. Probably most of the oxygen is used up in the biological reactions in the filter bed; whether or not all is used in this manner has not been settled. Temperature may have some relation to air-binding in filters, but is not the sole cause of it. W. T. LOCKETT.

Zeolite softening [of water]. A. S. BEHRMAN (Ind. Eng. Chem., 1927, 19, 445—447).—Artificial zeolites made by fusion and natural products such as bentonite or glauconite are relatively slow in action, and have other disadvantages. A gel zeolite such as "crystalite" is prepared by mixing solutions of sodium silicate and aluminium sulphate under such conditions that no precipitate is formed. The whole mass sets to a gel which may be dried and granulated. Such a zeolite has an economical softening capacity of 14—23 kg. of calcium carbonate per cub. m., or double that of the best of the older materials. This can be considerably increased by a larger consumption of sodium chloride in case of peak loads. The softening plant for a given duty can, therefore, be made much smaller, and water consumption for regeneration and loss of head during treatment are both reduced. With fairly soft waters an upward flow can be used with advantage. In this case loss of head is nil, and a rate as high as 10—12 gals./sq. ft./min. can be employed. C. IRWIN.

PATENTS.

Treatment of sewage. T. W. LOVETT (E.P. 266,893, 16.2.26).—Aeration of sewage, prior to and after passing into activated-sludge aeration tanks, is effected by causing a part of the sewage under a head to flow first through a number of vertical tubes each having a part adapted to function as a Venturi tube and below the Venturi throat an air inlet, an enlargement, and a further vertical length, and, secondly, through a vessel which serves as a separator, wherein air ascends to the closed top of the vessel and passes by a conduit to the aeration tanks, while the liquid flows through a lower and open end of the vessel and passes direct to the aeration tanks, or by way of one or more similar Venturi tubes or like conduits. W. T. LOCKETT.

Purifying boiler feed water. A.-G. FÜR STICKSTOFFDÜNGER (E.P. 250,574, 1.4.26. Conv., 11.4.25).—Liberation of oxygen and/or other gases from feed water is effected by forcing the water at a suitable temperature through the nozzle of a water-jet pump into a pipe which, at its upper end, has an enlarged space adjacent to the nozzle. The pipe, at its lower end, is enclosed in a separating chamber, wherein a reduced pressure is maintained, and from which water vapour charged with the oxygen and/or other gases is removed by suction pump, and purified water escapes by a pipe at the base. W. T. LOCKETT.

Treatment of waters for brewing. E. JALOWETZ (E.P. 261,708, 13.8.26).—Either by the addition of gypsum or the removal of the carbonates of calcium and magnesium, the quantity of calcium sulphate in the water is so regulated as to be in exact stoichiometric proportion to the amount of calcium and magnesium carbonates present. C. RANKEN.

Apparatus for detecting and determining impurities in water (E.P. 268,597).—See I.