

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 2, 1929.

I.—GENERAL; PLANT; MACHINERY.

Rapid graphical method for calculation of steam-distillation problems. E. M. BAKER and E. E. PETTIBONE (Ind. Eng. Chem., 1929, 21, 562—564).—The method described enables the temperatures of steam distillation of organic compounds at various pressures to be read graphically. If the temperatures required for steam distillations are plotted against the temperatures at which water exerts vapour pressures equal to the sum of the partial pressures of steam and the substance being distilled, a straight line is obtained. Data are given for the construction of steam-distillation lines for carbon disulphide, acetone, chloroform, carbon tetrachloride, benzene, isopropyl iodide, chlorobenzene, aniline, and for several mixtures of higher paraffins.

H. INGLESON.

A.F.N. system of lixiviation. A. FONTAINE (Caliche, 1929, 11, 1—4).—A method of lixiviation is described whereby the solvent ascends in counter-current to the material to be extracted. Advantages claimed for the method are the intimate contact achieved between the two phases, resulting in rapid lixiviation and high yield, and the economy effected in heat and in manual labour.

H. F. GILBEE.

Jellies and gels. F. KIRCHHOF (Kautschuk, 1929, 5, 100—103, 140—142).—A review of characteristics of gels, with some reference to industrial products of this type.

D. F. TWISS.

Electrical heating. CARLETON.—See XI.

PATENTS.

Heat-exchange coil. J. STRINDLUND, Assr. to G. D. JENSSEN Co. (U.S.P. 1,713,456, 14.5.29. Appl., 5.5.27).—The pipe coil for the inner fluid is in the form of a vertical cylindrical vessel, the helical pipe being formed in the thickness of the wall, and between the turns of the pipe holes are formed through the walls to permit free circulation of the outer fluid which is contained in an outer casing.

B. M. VENABLES.

Apparatus for heat-treatment of fluids. W. WINSHIP (U.S.P. 1,712,372, 7.5.29. Appl., 30.3.26).—A number of vertical tubes are completely immersed in the fluid, which is in contact with both the inside and outside of the tubes. The tubes are of high-resistance material, are connected electrically in series (all or in groups), and are heated electrically.

B. M. VENABLES.

Apparatus for effecting the exchange of heat between fluids. F. BAILEY and F. H. JACKSON (B.P. 311,889, 2.4.28).—A heat exchanger of the type wherein heat-absorbing material is exposed alternately to the heating and cooling fluid is constructed of metallic ribbon

alternately flat and corrugated (or accordion-pleated), the pair being wound round a bobbin spirally, like a clock spring. There are several stories of spirals, the gases passing through edgeways, and the corrugations are preferably oblique to the ribbon, the obliquity being opposite in adjacent spirals.

B. M. VENABLES.

Cold interchanger for gas separation plant. M. FRÄNKEL (B.P. 294,944, 1.8.28. Ger., 1.8.27. Addn. to B.P. 246,172).—In a cold interchanger of the intermittent-regenerator type, in which the cold-absorbing mass comprises strips of metal, the filling is arranged in a number of zones, and at the cold end there is a greater mass of metal and smaller space for the gas than at the warmer end, progressively according to the temperature and volume of the gas. The strips are preferably corrugated, with the corrugations inclined to the general direction of flow. One method of assembling the strips comprises taking a pair of strips with corrugations transverse to the strip, but oppositely inclined, and winding the pair in a flat spiral up to the diameter of the casing, the whole forming one zone.

B. M. VENABLES.

Refrigerant lubricant and method of lubricating refrigerating machinery. R. F. MASSA, Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,696,642, 25.12.28. Appl., 9.10.24).—Glycols, preferably trimethylene glycol, containing less than 2% of water are used as lubricants.

R. BRIGHTMAN.

Artificial ice. M. EULE, Assr. to A. HARTMANN (U.S.P. 1,713,596, 21.5.29. Appl., 1.9.27. Ger., 28.9.26).—Clear ice is manufactured by removing the less soluble constituents from ordinary undistilled water, adding 5—6.5 g. of anhydrous sodium carbonate per m.³ of water, and freezing the water thus treated.

W. G. CAREY.

Dryer. W. T. MORIN, Assr. to PACIFIC ABRASIVE SUPPLY Co. (U.S.P. 1,713,237, 14.5.29. Appl., 11.1.28).—A chamber is provided with fixed flues in its walls, and the material to be dried is injected into the chamber downwardly, in such a manner as to avoid contact with the walls, but to be heated therefrom by radiation only. The heating gases, after passing through the flues, have a controllable choice of two routes: (a) directly upwards through the material, or (b) through a heat exchanger in which air is heated and passed through the material.

B. M. VENABLES.

Dryer. L. J. ROBB, Assr. to HEYL & PATTERSON, Inc. (U.S.P. 1,706,708, 26.3.29. Appl., 3.7.26).—A drying chamber is provided with an inclined chute having perforated top and bottom plates through which the drying gases pass, and with an outlet above the top plate for discharging the material.

L. A. COLES.

Dryer. D. S. BAKER (U.S.P. 1,706,993, 26.3.29. Appl., 29.6.26).—The passage for the drying medium through the apparatus is provided with reheaters situated between each pair of a series of supports for the material being dried. The extent of the reheating is controlled by regulating the proportion of the stream of drying medium brought into contact with each reheater, and without varying the quantity of heat supplied to the reheaters.

L. A. COLES.

Crushing machine. L. P. WALKER (U.S.P. 1,712,369, 7.5.29. Appl., 23.7.27).—In a gyratory crusher the usual pulley and bevel gear drive a central pinion which drives a number of surrounding pinions each provided with an eccentric driving member or wide cam; the cams act in unison to gyrate the end of the crusher shaft.

B. M. VENABLES.

Machine for disintegrating, grinding, or threshing. B. BIALY (B.P. 289,889, 3.5.28. Poland, 6.5.27).—The machine comprises a closed casing (which may have a smooth interior) and a high-speed rotor comprising one or more discs with projections; all or some of the discs are formed with central apertures, the drive being transmitted from the shaft across the apertures by spider arms that act as fan blades.

B. M. VENABLES.

Laboratory mill. S. W. WILEY (U.S.P. 1,706,643, 26.3.29. Appl., 8.11.26).—An outer casing fitted internally with adjustable stationary knives contains an inner rotating member also fitted with knives. A removable screen slides into lateral grooves in the lower part of the casing, and is held in place by a hinged door.

L. A. COLES.

Mixing machine. J. T. SIMPSON (U.S.P. 1,706,417, 26.3.29. Appl., 30.8.26).—The machine is provided with means for conveying material through it, mixing and mulling the material, and adjusting the conveyor so as to vary the length of time during which the material is subjected to the mixing and mulling operations.

L. A. COLES.

Apparatus for treating solutions. H. FRISCHER (U.S.P. 1,711,638, 7.5.29. Appl., 30.11.27. Ger., 6.12.26).—An apparatus suitable for dissolving, filtering, and washing comprises a sieve- or filter-drum provided with stirring blades on its outside, rotating within a vessel.

B. M. VENABLES.

Concentrating apparatus [for solutions]. G. H. PASSELECQ (U.S.P. 1,711,614, 7.5.29. Appl., 12.1.25. Fr., 14.1.24).—A number of evaporating chambers have a common vapour outlet, but the solution passes through the chambers in series, being reheated in the pipes between chambers in such a way as to produce transfer of the fluid by thermosiphon action.

B. M. VENABLES.

Device for cooling liquids. L. W. HASSENSALL (U.S.P. 1,712,701, 14.5.29. Appl., 20.4.28).—Frozen gas is placed in a receptacle which is submerged in the tank of liquor to be cooled, the outlet for gas being through a \cap -shaped pipe from the open or perforated end of which the gas bubbles out.

B. M. VENABLES.

Plant for the production of crystals. J. R. ELLISON (B.P. 311,935, 10.5.28).—A continuous crystal-

liser in the form of a long rocking trough from one end of which the crystals are intended to be discharged, suspended in the mother-liquor without the aid of a mechanical conveying device, is provided with travelling blade members, which travel to and fro adjacent to the inner surface of the trough and serve to loosen, but not to convey, crystals adhering to the surface of the trough.

B. M. VENABLES.

Operation of centrifugal separators. AKTIEBO-LAGET SEPARATOR (B.P. 296,670, 27.8.28. Swed., 3.9.27).—Solid matter which is collected on the wall of a bowl of a separator (used, say, for cleaning oil) is discharged by means of a flush of water, or other liquid such as brine, intermittently applied without stopping the bowl. The flush liquid is preferably led through special channels to the zone where the solids have collected so that it will have acquired a good tangential speed. Just before admitting the flush it is preferable to slow up the bowl, which is accelerated immediately after, thus producing eddies. By using an intermittent instead of continuous flush the amount of oil wasted in the emulsion of solids, oil, and water is much reduced.

B. M. VENABLES.

Centrifugal treatment of substances. L. D. JONES, Assr. to SHARPLES SPECIALTY CO. (U.S.P. 1,711,533, 7.5.29. Appl., 25.6.27).—A centrifugal machine from which the substance or a constituent is delivered in dispersed form has that substance maintained in contact with an atmosphere saturated with the vapour of the substance, to prevent undesirable changes in the composition of the dispersion.

B. M. VENABLES.

Brake for centrifugals. R. A. STEPS (U.S.P. 1,713,502, 14.5.29. Appl., 23.6.26).—A brake drum is fastened to the belt pulley with the interposition of heat-insulating material.

B. M. VENABLES.

Concentrator. A. H. STEBBINS (U.S.P. 1,712,589, 14.5.29. Appl., 28.4.27).—A vertical receptacle has side walls which converge downwardly; at the bottom are a number of concentric annular collecting zones for different products. The pulp is supplied in the form of a thin annular sheet in a downward direction at a part of the receptacle where the diameter is largest.

B. M. VENABLES.

Effecting reaction between liquids tending to form tight emulsions. F. H. MCBERTY, Assr. to DE LAVAL SEPARATOR CO. (U.S.P. 1,698,270, 8.1.29. Appl., 16.8.24).—The liquids are circulated continuously and separated after partial reaction, the separated liquids being mixed and returned to the stream, and the operation repeated until reaction is complete. *E.g.*, used motor crank-case oil and trisodium phosphate solution are circulated to and from a tank, the circuit including a heater to raise the temperature of the oil to, *e.g.*, 80°. Part or all of the stream from the heater is run through a centrifuge, and the two components are returned to the tank. Agglomerated carbon accumulates in the centrifuge, and when the remainder is taken up by the sodium phosphate solution the latter and the clear oil are separately discharged from the circuit.

R. BRIGHTMAN.

Fractionating apparatus. F. P. RISDON, Assrs. to TEXAS CO. (U.S.P. 1,711,656, 7.5.29. Appl., 21.8.24).—

Bubbling trays with loose bubble caps are assembled in nests, the top tray only of each nest having means to secure the caps. The nests are supported on flanges inside the shell of the tower. B. M. VENABLES.

Condensation of vapours. W. HILDEBRANDT, ASSR. TO GASOLINE CORP. (U.S.P. 1,712,825, 14.5.29. Appl., 23.3.21 Renewed 6.10.28).—The vapour passes from a distilling header, through a continuous cooling coil, to a collecting header at a lower level. From the collecting header pipes rise to a gas header, and another pipe falls to a liquor collector and settler from which lead separate draw-off pipes for lighter and heavier liquids (oil and water), the latter leaving at the bottom of the settler but being afterwards brought up to a level only slightly below the oil outlet. B. M. VENABLES.

Recovery of gases or vapours taken up by adsorbents. N. V. NORITVEREENIGING VERKOOP CENTRALE, ASSEES. OF ALGEM. NORIT MAATSCHAPPIJ (B.P. 308,313, 11.1.28. Ger., 11.1.27).—The saturated adsorbent is treated with a hot or boiling liquid, *e.g.*, water, having a higher b.p. than the adsorbed substance. An aqueous solution of an organic substance may be used as the displacement liquid, the solution being one which is soluble in the adsorbed substance and also easily adsorbed itself. Or the adsorbent may be impregnated with a lyophile colloid which permits adsorption of the vapour when dry and releases the adsorbed substance when treated with hot or boiling water or other aqueous liquid. A. B. MANNING.

Air cleaner. M. L. BLAIR, ASSR. TO AC SPARK PLUG CO. (U.S.P. 1,712,947, 14.9.29. Appl., 4.12.26).—The air enters an outer cylindrical chamber at one end in a whirling manner, and passes through a filter to an inner chamber constituting the outlet pipe for clean air. Tangential outlets for heavy dust are provided in the outer cylindrical wall. B. M. VENABLES.

[Oscillation damper for] manometers and the like. H. WADE. FROM L'AIR LIQUIDE, SOC. ANON. POUR L'ETUDE & L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 311,967, 21.6.28).—A damper is inserted in the pipe leading to the manometer comprising a number of elastic discs clamped together round their periphery and provided with holes which are in staggered relation to each other. The discs are arranged to bend in only one axial direction for each group, but there are preferably two groups, one on either side of an apertured rigid wall, the discs of the one group being thus constrained to bend in the opposite direction to those of the other group. B. M. VENABLES.

Oxides of sulphur for fire prevention (B.P. 312,746).—See VII. **Drying of solid matter from liquids** (B.P. 312,433).—See XVIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Explosions in coal mines and permitted explosives: historical record. (SIR) F. L. NATHAN (Fuel, 1929, 8, 256—295).—A summary is given of the results of the various investigations into the causes and the prevention of explosions in coal mines, both in Great Britain and abroad, from 1828 to the present day. The factors which have been principally studied are

(a) the influence of the presence of coal dust on the ignitibility of gaseous mixtures, (b) the composition and testing of safety explosives, (c) the length and duration of flame from an explosive as affecting the ignition of firedamp-air or coal dust-air mixtures, and (d) the influence of the dimensions of the gallery, the density of loading, the size of the detonator, etc. on the charge limit. A. B. MANNING.

Recent research on production and utilisation of coke. W. T. K. BRAUNHOLTZ (Gas World, 1929, 90, Coking Sect., 65—68).—A number of different cokes have been examined by the "Micum" trommel test (the standard German test for the mechanical strength of coke) and the shatter test (standard English and American method). It is found that if 50 lb. instead of the specified 50 kg. of coke are used in the full-scale trommel test, more breakage occurs with the smaller quantity, owing presumably to the cushioning action with the larger amount. If the trommel length is halved, 50 lb. of coke used in such an apparatus give the same result as 100 lb. of coke in the full-size trommel test; this is an advantage when the quantities of coke available are small. There is an approximate concordance between the results given by the trommel and shatter tests, but it is concluded that the trommel index of a coke cannot be calculated with accuracy from its shatter index or *vice versa*; the two tests place the cokes in the same order of merit as regards their tendencies to "fines" production, but it is shown that the trommel test is far more drastic than the shatter test. Experiments on the determination and nature of the volatile matter in coke and a comparison of the weights of different cokes occupying 1 cub. ft. are described. The sieve analysis, distribution of the ash, and the proximate analysis of a number of coals, as charged to the ovens, are also given, and it is indicated that (a) the degree of fineness of the coal charged to coke ovens shows little variation from plant to plant, (b) there is a distinct tendency for the middle sizes to contain least ash, and (c) Cumberland coking coals have higher moisture and volatile matter contents than Durham coals.

C. B. MARSON.

Absorption characteristics of coals. B. PENTEGOV and R. NJANKOVSKAJA (Mem. Univ. d'Etat Extr. Orient, 1927, No. 6, 18 pp.; Chem. Zentr., 1928, ii, 2762).—Brown and bituminous coals are differentiated by their adsorptive power towards ferric chloride, oxalic acid, and methylene-blue in aqueous solution.

A. A. ELDRIDGE.

Measurement and value of the plasticity of coal. J. A. JACKSON (Gas World, 1929, 90, 715—717).—A coal briquette, 0.5 in. long and weighing 2 g., is heated in a silica tube, and rests on a thermocouple; the rate of heating is adjusted to give a rise of 10° per min. On the coal rests a glass or silica pointer the movement of which indicates expansion of the briquette, which occurs freely as the coal becomes plastic. Results are given for a number of coals, showing m.p., plastic range, degree of expansion, and proximate analysis, and the values obtained with coals suitable for use in continuous vertical retorts are indicated.

R. H. GRIFFITH.

Nitrogen compounds in coal. K. ISHIBASHI (J. Fuel Soc. Japan, 1929, 8, 64—65).—A sub-bituminous Fushun coal treated with phenol yielded 30% of extract. The original coal contained 2.02% N, of which 20.8% was contained in the extract and 77.7% in the residue. On carbonisation of these two fractions at 1000° the former evolved 14.4% and the latter 20% of its nitrogen in the form of ammonia. From an examination of the nitrogen compounds in the extract it is concluded that they are the source of the basic compounds in the tar, whilst those in the residue form the principal source of the ammonia. A. B. MANNING.

Liquefaction of coking coal. H. NOVÁK and J. HUBÁČEK (Paliva a Topeni, 1927, 9, 145—158; Chem. Zentr., 1928, i, 1120).—A coking coal from Schneidemühl was hydrogenated at 400—500° under 200 atm. pressure. Within 2 hrs. 50% of the dry coal had been converted into oil. Tin, antimony, bismuth, etc. had no catalytic effect on the reaction. The products of the hydrogenation, which in amount were about double those obtained by ordinary distillation, differed completely from the latter in chemical composition. They were quite mobile at the ordinary temperature, and contained more than 50% of constituents boiling below 250°. The low-temperature tar contained only 4% of benzine and solar oil. The benzine and petroleum of the low-temperature tar was equal in quality to that obtained from crude oils. The higher-boiling oils were formed of gas oils and paraffinic substances, thus resembling in composition the ordinary tar from this coal. A. B. MANNING.

Desulphuration of coal. E. GRÜNERT (J. pr. Chem., 1929, [ii], 122, 1—120).—A series of investigations has been carried out on the main reactions involved in the removal of sulphur from coal during distillation.

I. *Hydrolysis of sulphur by the reaction* $3S + 2H_2O = SO_2 + 2H_2S$.—Dry sulphur dioxide and hydrogen sulphide do not react at room temperature, but in the presence of condensed water reaction takes place rapidly (Noack, Diss., Dresden, 1925). At 100° no appreciable reaction occurs between the moist gases, but sulphur is deposited on cooling. The conclusions of Lewis and Randall (A., 1918, ii, 159) are therefore erroneous. The reaction at higher temperatures (450—800°) has been investigated by passing the gases in equivalent amounts into a reaction vessel and analysing the rapidly cooled emergent gases. The reaction takes place on the walls of the vessel. At 300° equilibrium is reached very slowly. At 450° it may be attained by the use of quartz powder as a surface catalyst. At 600° a catalyst is no longer necessary. Confirmatory experiments on the action of steam on sulphur have also been made. The values for the equilibrium constant, $K = [H_2S]^4 \times [SO_2]^3 / [H_2O]^4 \times [S_2]^3$, between 450° and 600° (1.18 to 0.0062) are in good agreement, when extrapolated, with the results of Randall and von Bichowsky (A., 1918, ii, 159), but do not agree with those of Lewis and Randall (*loc. cit.*) at 450°, the difference being attributed to the neglect of the latter to take into account the dependence of the equilibria in sulphur vapour on the total pressure. The application of Nernst's approximation formula shows that the heat

of the reaction $3S_2$ (gas) + $4H_2O$ (gas) = $4H_2S$ + $2SO_2$ is not 12.7 kg.-cal., as calculated from data for other reactions, but is of the order of 32.0 kg.-cal., whilst the value calculated from the reaction isochore is 28.0 kg.-cal.

II. *Hydrolysis of sulphur in presence of amorphous carbon.*—Preliminary investigations of this reaction have been made. At 150—450° the carbon does not itself react, but catalyses the gaseous reaction. The behaviour varies somewhat with the type of carbon employed, but at 600° and above the sulphur dioxide is removed by reduction, and the formation of carbon disulphide begins at 800°. At room temperature active charcoal catalyses the reaction between dry sulphur dioxide and hydrogen sulphide.

III. *Interaction of iron pyrites, amorphous carbon, and steam.*—When steam and nitrogen are passed over pyrites, the latter is decomposed slowly at 500° and rapidly, but not completely, at 600°, the sulphur being hydrolysed to sulphur dioxide and hydrogen sulphide. The hydrolysis of the ferrous sulphide is less complete at the higher temperature owing to the coherence of the protective layer of iron oxide formed. The results are in agreement with those of Foerster and Geissler (B., 1923, 401 A). The amorphous carbon used includes sugar- and wood-charcoals, active charcoal, and acid-treated semi-cokes. When such carbons are heated in a stream of nitrogen and in a stream of nitrogen and steam at 500—600°, a comparison of the resulting gas mixtures shows that the ordinary water-gas reaction occurs. When a mixture of steam and nitrogen is passed over a mixture of amorphous carbon and pyrites containing 5—10% S at 500—600°, about half the sulphur is found as hydrogen sulphide in the resulting gas. No sulphur dioxide is obtained. The sulphur not remaining as ferrous sulphide is adsorbed by the carbon. The gas contains more carbon dioxide and less carbon monoxide and hydrogen than it does when no pyrites is present, owing either to the reduction of the sulphur dioxide or to an alteration of the catalytic effect of the solid on the water-gas reaction. When a mixture of amorphous carbon and pyrites is heated in a stream of nitrogen, although sufficient hydrogen is present to combine with the sulphur, only a small proportion of the latter is removed as hydrogen sulphide. It is adsorbed by the carbon, and is only removed by steam. It is shown that the increased formation of hydrogen sulphide in presence of steam probably results from the hydrolysis of the sulphur, since the equilibrium in this reaction is attained more rapidly than the equilibrium in the reaction between hydrogen and sulphur, which also comes into consideration. R. K. CALLOW.

Determination of sulphur in coal by means of the nephelometer. B. TYKAČ and J. STREIT (Paliva a Topeni, 1927, 9, 133—134; Chem. Zentr., 1928, i, 1123).—Muer's nephelometer has been used to determine the sulphur content of coal. The sulphur is converted into a soluble sulphate and to the solution, acidified with hydrochloric acid, excess of barium chloride is added. The flame of a candle, or preferably the filament of a carbon-filament lamp, is viewed through a cylinder, which is then gradually filled with

the turbid solution until the flame or the filament just disappears. The height of the liquid column in cm. (c) is related to the number of mg. of sulphur present by the formula $S = 0.6 + 15.3c$, the constants of which, however, must be redetermined each time the apparatus is used against solutions of known sulphate content. The apparatus gives very accurate results.

A. B. MANNING.

Oxidation of ammonia from [coke-oven] crude by-product liquors. G. A. PERLEY and W. P. WHITE (Ind. Eng. Chem., 1929, 21, 564—567).—The catalytic oxidation of the ammonia evolved by passing air through the heated liquor was completely prevented by the deposition of sulphur derived from hydrogen sulphide on the surface of the platinum gauze used. Various methods in use commercially for the removal of hydrogen sulphide from gases were employed without success. When the crude ammonia-air mixture was passed through a solution of nickel sulphate in ammonium sulphate the subsequent oxidation could be carried out satisfactorily. The following series of reactions is suggested to explain the removal of the hydrogen sulphide: $\text{NiSO}_4 + 2\text{NH}_4\text{HS} + \text{O} \rightarrow \text{S} + \text{NiS} + (\text{NH}_4)_2\text{SO}_4 + \text{H}_2\text{O}$; $\text{NiS} + 2\text{O}_2 \rightarrow \text{NiSO}_4$. The loss of ammonia in this liquid is very small.

H. INGLESON.

Problems in the determination of unsaturated hydrocarbons in gases. I. Separation by fractional distillation. H. S. DAVIS (Ind. Eng. Chem. [Anal.], 1929, 1, 61—64).—For fractional distillation of gas mixtures boiling between -162° and $+13^\circ$, a special column has been designed which consists of a low-pitch glass spiral mounted inside an evacuated and silvered glass jacket. The liquefied mixture of gases is distilled from a Dewar vessel provided with an electric heating system, and the distillate is collected in a graduated vessel which is also immersed in a Dewar flask. The apparatus has been satisfactorily tested with mixtures of benzene and toluene, alcohol and water, and olefine mixtures; for storage of the pure gases which can be prepared in this way small steel bottles are recommended.

R. H. GRIFFITH.

Corrosion and metal protection in gasworks' practice. MAAS (Gas- u. Wasserfach, 1929, 72, 573—578).—A general review of the fundamental causes of corrosion and of the particular problems presented by gasworks' plant is given. Simple precautions are suggested to avoid accumulation of water in contact with metallic surfaces, and different methods for protection of service pipes, by means of calorising, coating with bituminous substances, etc., are described.

R. H. GRIFFITH.

Effect of distillation conditions on the consistency of road tars. C. O. CONDRUP and H. M. SPIERS (Gas J., 1929, 186, 856).—The consistency of a tar is considerably altered by heating at or near its b.p.; a vertical-retort tar, for example, changed from a value of 2.8 to 10.5 sec. when boiled for 3 hrs., and to a smaller extent at lower temperatures. The quality of a distillate may consequently be altered not only by the fractions which are collected, but by the rate at which these are allowed to cool.

R. H. GRIFFITH.

Motor fuels and other products from the cracking of wood tars. J. C. MORRELL and G. EGLOFF (Ind. Eng. Chem., 1929, 21, 537—542).—A wood tar derived from the thermal decomposition of Douglas fir, cracked under pressures of 75, 100, and 120 lb./in.² and at temperatures ranging from 393° to 405° , yielded 26% of motor fuel free from tar acid based on the dry wood. By recycling the gas oil fraction a maximum yield of 33% of motor fuel was obtained. The anti-knock properties of the motor fuel were better than those of benzene, and analysis showed the content of aromatic and unsaturated compounds to be 97%. Pine tar oils from the destructive distillation of light-wood on cracking yielded about 22% and 41% of oil distilling within the motor-fuel range. Depending on the products desired, the pressure-distillate oil may be distilled to yield motor fuel and gas oil, or solvent oil, turpentine substitute, and pine oil equivalent. The gum-forming tendencies were in excess of those permissible in standard motor fuels, indicating the preferential use of such distillates in the manufacture of solvent oils, turpentine substitutes, flotation oils, paint thinners, etc. The light fraction (solvent oil) is a good solvent for crude rubber. Cracking should be carried out so that the residue is not run to coke, but remains suitable for the uses to which the original pine tar is applied. A mixture of tar derived from the thermal decomposition of hickory and oak cracked at 90 lb./in.² and 382° yielded approx. 24% of oil distilling in the motor-fuel range, but containing 52% of tar acids and 6% of tar bases. Refining of the cracked distillates follows that used for the cracked distillates of low-temperature tars and shale oils.

H. S. GARLICK.

Road tar. H. MALLISON (Petroleum, 1929, 25, 399—403).—Three German road tars, viz., (a) road tar I, (b) anthracene oil tar 50/50, (c) anthracene oil tar 60/40, each of which has been found suitable for road making, have d^{15} not above 1.225; not more than 1% of water; not more than 1% of light oil (b.p. up to 170°); 12.0—24.0, 1.0—15.0, and 1.0—10.0% of middle oil (b.p. 170 — 270°); 4.0—12.0% of heavy oil (b.p. 270 — 300°); not more than 5.0, 3.0, and 3.0% (by vol.) of phenols; not more than 5.0, 3.0, and 3.0% of naphthalene; 5.0—18.0% of free carbon; viscosity (Hutchinson) 3—15, 1—15, and 20—80 sec.; and 55—65, 45—55, and 55—65% of pitch (softening point 60 — 75°). Sample (a), owing to its high content of middle oil, has a steep viscosity-temperature curve and more readily becomes mobile on heating; sample (b) dries more rapidly on the road; and sample (c) is more viscous, resembling rather the English road tar (II), the corresponding values for which are: 1.240, 1%, 1%, 10—18%, 1%, 10—18%, 6—12%, 4% (by vol.), 5%, 24%, and 20—100 sec. Naphthalene tends to render a tar more readily fluid on warming, hence the ease of penetration into the road material is greater, but owing to its volatility the naphthalene content must be limited to cut down loss by evaporation. The "gum test" or "resinification test" of Herrmann, in which the behaviour of a thin film of tar on a glass plate at 20° is noted, does not really indicate ease of resinification, but rather the tendency to thicken by reason of the

evaporation of volatile constituents. "Free carbon," usually regarded as a filler, is the portion insoluble in benzene; this is in reality a mixture of carbon and valuable asphaltic substances. The quantity of material insoluble in tar oil or, preferably, aniline-pyridine is a better indication of the free carbon content. The quantity of bitumen that can be taken into actual solution in a road tar is not greater than 10–20%. By replacing part of the pitch content of the tar by an equal quantity of bitumen, the softening point remains at 41–43°, whilst the ductility (Dow) at 10° falls from more than 100 cm. for tar or for tar with 10% of bitumen, to 70 cm. and 16 cm. for tar with 15% and 20% of bitumen, respectively.

W. S. NORRIS.

New Austrian specification for [testing] natural asphalts and petroleum asphalts. ANON. (Petroleum, 1929, 25, 404–410).—If it is necessary to melt the sample (methods for taking which are described) the container should be heated in an oil-bath at 75–100° above the softening point for the shortest possible time. The density of fluid or semi-fluid asphalt is determined by Lunge's method using a sp. gr. bottle; for solid asphalt a bath is prepared in which a sample, free from air bubbles, will just float, the density of the bath being then determined. Breaking point is determined by Church's method; softening point by the ring-and-ball method; ductility by the Dow-Smith ductilometer; flash point by means of an open-crucible, horizontal-flame, Marcusson apparatus, and the penetration test is carried out by Richardson's method. The bituminous content is ascertained by extracting 5–10 g. with 300 c.c. of chloroform, allowing it to settle, and filtering the insoluble portion, which is weighed after drying at 105°. For the determination of water the method D 95–23 T of the A.S.T.M. is employed, using xylene saturated with water. Ash is determined by ignition in a porcelain crucible, and sulphur by burning a sample in a current of air, using a transparent silica tube, and leading the products of combustion into neutralised hydrogen peroxide solution; the sulphuric acid in the latter is then titrated using 0.025*N*-sodium carbonate. Oleaginous constituents are determined by the method of Marcusson and Eickmann. The sample (20 g.) is dissolved in 30 c.c. of benzene and diluted with 440 c.c. of "normal benzene." After keeping, the filtered solution is washed three times with 30 c.c. of concentrated sulphuric acid, then with (50%) aqueous-alcoholic *N*-sodium hydroxide and with water, is evaporated, and the residue is weighed. A weighed portion of the residue is distilled to a coke residue and the paraffin content of the distillate determined by Holde's method. The acid value is determined by dissolving 5 g. in 25 c.c. of benzene, cooling, adding 100 c.c. of neutral 96% alcohol, and, after settling, titrating the filtered solution by means of alcoholic 0.1*N*-alkali hydroxide. Saponification value is determined by hydrolysing for 30 min. with 25 c.c. of benzene and 25 c.c. of alcoholic *N*-potassium hydroxide, diluting with 100 c.c. of neutral alcohol, and titrating. The stability of the sample is measured by determining the loss in weight when a portion is heated for 5 hrs. at 163° in an electrically-heated Heraeus air-bath.

W. S. NORRIS.

Petroleum asphalts and resins. N. I. TSCHERNOZHUKOV (Nef. Choz., 1928, 15, 670–673).—When heated with oxygen under pressure, paraffinum liquidum yielded oxygenated compounds (e.g., C 71.64, H 8.81, O 19.55%) free from sulphur. Neither asphaltenes nor carbenes were formed. Crude Balakhani, Grozni, and Emba oils gave asphaltenic sediments, e.g., C₁₇₂H₁₇₆O₁₄S; C₁₃₆H₁₄₂O₁₂S. Baku and Emba white oils were heated with sulphur and then oxidised; asphalt-like compounds were obtained. With sulphuric acid the sulphur-treated oils liberate sulphur dioxide. Hence asphaltenes are formed from crude oil by unknown sulphur compounds in various stages of oxidation.

CHEMICAL ABSTRACTS.

Determination of softening points of pitches and asphalts by the Kraemer-Sarnow method. D. HOLDE (Petroleum, 1929, 25, 411–412).—In such determinations attempts to obviate the danger of mercury poisoning (cf. Mallison, B., 1928, 661) by replacing the mercury by a small brass rod lead to somewhat too high and irregular values.

W. S. NORRIS.

Extraction of pyridine and phenol from crude benzene. H. A. J. PIETERS and M. J. MANNENS (Chem. Weekblad, 1929, 26, 286–290).—The procedure for removal of pyridine and phenol from the crude benzene obtained by the oil-washing of gas, by preliminary washing with dilute sulphuric acid and sodium hydroxide respectively, before the ordinary sulphuric acid and alkali purification, has been examined in detail for quantities of about 15 tons of crude benzene. The preliminary working gave recoveries of over 34 kg. of pyridine and 50 kg. of phenol in each case; a considerable economy is effected in the total sulphuric acid used, but this is offset by the greater quantity of alkali required. The methods of analysis are discussed; in the determination of pyridine, titration after distillation, using dimethyl-yellow and methylene-blue together, gives accurate results.

S. I. LEVY.

Sulphur compounds in pressure-cracked naphtha and cracked naphtha sludge. D. S. MCKITTRICK (Ind. Eng. Chem., 1929, 21, 585–592).—212 litres of naphtha (*d* 0.786) from the cracking of the fuel-oil fraction (*d* 0.953) of California Midway crude, b.p. range 30–250°, were extracted with liquid sulphur dioxide at –20° to –30°. The extract oil was submitted to a series of fractional extractions, first with aniline and then with ethylene glycol diacetate at –19° to –20°. The fractions were combined into groups depending on their sulphur content and submitted to further extraction. The final yield was 13.6 litres of oil (*d* 0.900) containing 5.5% S, which was 40% of the total sulphur in the original naphtha. The sulphur compounds in the extract oil were further concentrated by systematic fractional distillation into 2.5° cuts or less. Thiophen isomerides partially separated in the fractional distillation were purified through their mercuric chloride derivatives, and their composition was confirmed by the m.p. of this and of a second derivative. The following compounds were identified:—thiophen, 2- and 3-methyl- and -ethyl-thiophens, and 2:3- and 3:4-dimethylthiophens. An acid sludge formed in the low-temperature refining of naphtha was diluted and the separated tar

steam-distilled. The distilled oil was treated with concentrated sulphuric acid and the oil from the sludge thus obtained steam-distilled and then fractionally distilled into 2.5° cuts. The sulphur compounds in the sludge oil readily reacted with aqueous mercuric chloride solution to give thick oily precipitates which congealed to brown solids. With methyl iodide, water-soluble crystalline sulphonium compounds which sublimed without melting were obtained. Oxidation with potassium permanganate yielded sulphones, and additive compounds were formed with bromine. The evidence points to the sulphur compounds being present as cyclic sulphides, although no definite substances were identified.

H. S. GARLICK.

Determination of free sulphuric acid in light-oil sludge. F. S. BACON (Ind. Eng. Chem. [Anal.], 1929, 1, 89—92).—Five different methods have been found which are satisfactory for determining the free acid content of light-oil sludges. (A) A water extract of the sludge is treated with acid 10% barium chloride solution. (B) The sludge is weighed on to a thick mat of asbestos in a Gooch crucible, washed with chloroform until the washings are colourless, and then treated with water to dissolve sulphuric acid which still adheres to the asbestos. (C) The sludge is mixed with chloroform and poured into ice-water, which is then titrated with alkali before and after boiling. (D) The rise in temperature which occurs on diluting the sludge with water is compared with that given by sulphuric acid alone; by allowing for the sp. heat of the tar, and assuming that no thermal effect is due to hydrolysis, the necessary data are available if the percentage of tar is known. (E) Aniline sulphate is prepared from sludge in chloroform solution, separated by filtration, and analysed by titration or gravimetrically. It is found that there is close agreement in the results of all five methods, but that those from (B) are generally somewhat lower.

R. H. GRIFFITH.

Separation of naphthenic soaps. V. SHIPEROVICH and V. GURVICH (Nef. Choz., 1928, No. 12, 26—33).—Plant for the evaporation of kerosene alkali sludge, containing 10—25% of naphthenic soaps, is described.

CHEMICAL ABSTRACTS.

Distillation of bituminous limestones for the production of Italian mineral oil. A. LA PORTA (Giorn. Chim. Ind. Appl., 1929, 11, 109—118).—In the Ragusa district, adjacent to populous centres and to the railways, there exists sufficient readily accessible bituminous limestone to give, with a mean yield of 4%, 200,000 tons of oil annually for a century, this representing about one fifth of the whole Italian consumption of fuel oils. The nature of the asphaltic rocks, the extraction of the oils, and the thermal balance, the cost of plant and working, and the characters of the oil obtained are discussed.

T. H. POPE.

Standardisation of conditions for measuring the detonation characteristics of motor fuels. R. STANSFIELD and F. B. THOLE (Ind. Eng. Chem. [Anal.], 1928, 1, 98—105).—In these tests three types of engine have been used and the effects of alterations in operation have been studied with each. Engine 1 was similar to the Ricardo E-35, but of smaller capacity, and its compression ratio could be varied from 3.9 to 7.6;

it was fitted with devices for heating inlet air and lubricant, and for controlling water-jacket temperature, ignition advance, air-fuel ratio, etc. Engine 2 was a single sleeve-valve type of 346 c.c. capacity, and compression ratio from 5.4 to 11.9; engine 3 was a modified single-cylinder Armstrong-Whitworth "B," with compression head of special design giving ratios between 4.0 and 8.0. With engine 1 only one satisfactory procedure was developed for comparing different fuels; the number of "pinks" occurring in a given time, above a standard intensity, was counted and was found to increase very rapidly with rise of compression ratio. Curves were plotted from these observations at three compression ratios with three different ignition advances, for a standard and for a test spirit; the mean difference in the ratios giving the same result was taken in evaluating the sample. With engine 2 a series of air-fuel ratios was taken to give a knock of standard audibility with fixed ignition; the minimum compression ratio was thus determined for every spirit. With engine 3 a number of methods were available as the audibility test was suitable, and adjustment of head to give a suitable intensity of pinking was also possible. More interesting results were obtained by the use of a Midgley bouncing pin in the compression head; this depends on matching blends, and the position of maximum pinking is shown by the amount of gas collected in the bouncing-pin voltameter. For good results it was essential to run with much heavier pinking than was necessary for audibility tests, and matching had to be done with mixtures of known composition and not by additions to the sample under observation. The errors involved in these measurements were determined by extensive tests with known blends; engine 1 gave variations of 0.03 in compression ratio, equivalent to $\pm 1\%$ of benzene in *n*-heptane, whilst engines 2 and 3 gave 0.2 and 1.5%, respectively. The influence of other variables was also determined: (1) fuel-feed changes affected the results if a fixed-jet carburettor did not give maximum pinking conditions in all cases, but if the feed was adjusted to maximum audibility, no difficulties arose; (2) the choice of ignition plugs was important when pre-ignition was likely to occur; (3) engine speed and air and water temperatures affected the results to a small extent; and (4) errors were found due to unsuitable ignition setting, but more serious difficulties arose on attempting to adjust this, and the advance was kept constant throughout. The need for a new and plentiful standard pinking fuel is emphasised, as the cost of *n*-heptane is high and supplies are limited.

R. H. GRIFFITH.

Auto-ignition temperatures. III. (a) Mixtures of pure substances. (b) Gasolines. H. J. MASSON and W. F. HAMILTON (Ind. Eng. Chem., 21, 544—549; cf. B., 1928, 42, 699).—Curves showing the relationship between composition and values of the auto-ignition temperature (A.I.T.) for mixtures of carbon tetrachloride and ethyl ether with benzene show minimum A.I.T. values each below that of pure benzene. Similar curves for the addition of acetic acid and *p*-xylene to absolute alcohol show both a minimum and a maximum. A curve for mixtures of *n*-heptane and *isooctane* is perfectly regular, the relationship of the A.I.T. values being nearly additive as is the case of knock rating. The addition of

pure lead tetraethyl and of lead tetraethyl in ethylene dibromide to a reference fuel gave curves with a distinct minimum at 2 c.c., the latter having a greater effect on the A.I.T. value than the same amount of pure lead tetraethyl. The standard reference fuel gave different A.I.T. values on platinum and quartz surfaces, whereas benzene gave identical results. The A.I.T. values of a number of straight-run gasolines compared with their knock ratings gave a smooth curve approaching a minimum at approx. 510°. A similar curve is shown for a number of cracked gasolines on a platinum surface of high catalytic activity. Experiments on the effect of composition of the igniting surface on the A.I.T. value showed that gasolines which detonate readily are very sensitive to the catalytic effect of the igniting surface and *vice versa*. The effect of the addition of lead tetraethyl is to lower the A.I.T. value of benzene and alcohol towards a minimum or optimum temperature, whilst in the case of *n*-heptane it is raised. With straight-run gasolines of varying knock rating, addition of lead tetraethyl either raises or lowers the A.I.T. value towards an optimum corresponding to decreased detonation characteristics. Carbon disulphide has a similar effect.

H. S. GARLICK.

Effect of cathode rays on hydrocarbon oils and on paper. Mechanism of cable deterioration. C. S. SCHOEPFLE and L. H. CONNELL (Ind. Eng. Chem., 1929, 21, 529—537).—Three samples of vaseline, viz., (a) taken directly from the jar, (b) carefully vacuum-dried in the tubes in which it was to be tested and only exposed to air after it had cooled, and (c) as (b) except that the tubes were separated and replaced, thus exposing the oil to air, were exposed as films between concentric glass tubes to a high electrical stress. Samples (a) and (c), containing air and therefore subjected to gaseous discharge, became masses of wax after 2½ days, whilst sample (b), air-free, was unchanged after more than 2 weeks. A similar experiment was made with a petroleum oil through which a continuous stream of oxygen was bubbled. Within 24 hrs. the oil had changed colour and become rancid, but no solid wax was formed. Further experiments showed that hydrocarbon oils react readily when bombarded by cathode rays in a manner similar to that obtained by means of a silent electric discharge. Condensation of the oil takes place with the evolution of hydrogen and saturated gaseous hydrocarbons and the formation of an insoluble product resembling the wax found in electrical power-transmission cables. Tests on a large number of mineral oils of different origin showed that (1) naphthenic oils give slightly less gas than Mid-continent oils, (2) with the same degree of refinement, low-viscosity oils give more gas than those of medium viscosity, (3) oils which give the least gas are those which contain the greatest fraction of unsaturated constituents. Similar tests made with cable paper and partially purified cellulose from cotton show these react to give water, hydrogen, and carbon dioxide with smaller amounts of carbon monoxide and methane. No marked difference in results was obtained if the experiments were carried out in an atmosphere of hydrogen or nitrogen. Paper impregnated with pure mineral oil alone or containing rosin when irradiated *in vacuo* gave practically the

same amount of moisture in each case. Paper with an initial copper number of 1·13 gave values after irradiation ranging from 12·4 to 43·1, and showed distinct acid properties. Paper subjected to corona discharge gave somewhat less gas than when irradiated; the gases produced had approximately the same composition, but that from the corona-discharge test had the higher moisture content. The paper showed no marked change of structure as in the previous experiments and the copper number increased only from 2·0 to 2·3.

H. S. GARLICK.

Sulphonated oxidation products of petroleum as insecticide activators. M. T. INMAN, JUN. (Ind. Eng. Chem., 1929, 21, 542—543).—A straight gas-oil distillate of Pennsylvania crude was subjected to catalytic vapour-phase oxidation and the products, consisting of alcohols, aldehydes, ketones, and saturated and unsaturated acids, with about 25% of unchanged hydrocarbons, were condensed. This mixture was sulphonated, washed, and neutralised with sodium hydroxide. The product, containing about 40% O and 1·5% S, was a stable, clear, maroon-coloured liquid, easily emulsifiable with water. A 1% solution was toxic to various aphids, leaf hoppers, and red mites. As an activator for other poisons, 1 pt. of the oxidised gas oil products added to 200 pts. of nicotine sulphate spraying solution reduced the amount of insecticide necessary for satisfactory kills to be obtained to ½—⅓ of that commonly employed when soap is used as a spreader.

H. S. GARLICK.

Alundum crucible in the determination of carbon in crank-case oil. P. V. MCKINNEY (Chemist-Analyst, 1929, 18, 21).

Graphical methods and steam-distillation problems. BAKER and PETIBONE.—See I. Flotation practice at the Sullivan mill. OUGHTRED.—See X.

PATENTS.

Method of combustion. J. T. COOK, Assr. to A. F. HAFFENREFFER (U.S.P. 1,698,258, 8.1.29. Appl., 16.7.26).—Oil is delivered under about 30 lb./in.² in a rotating cone of rapidly dispersing particles to meet at an angle a similarly rotating cone of air under low pressure moving with low velocity. The mixture is ignited as it leaves the mixing chamber, and the spirally-moving ignited mixture is expanded and contracted laterally to complete combustion before being released for heating purposes.

R. BRIGHTMAN.

Distillation of carbonaceous materials. F. PUENING (U.S.P. 1,698,345—6, 8.1.29. App., 11.2.22).—(A) In the low-temperature distillation of bituminous coal, iron heat-transfer bodies are raised to a surface temperature of 760° in a gas furnace and discharged with the powdered coal on to a travelling belt conveyor within a closed chamber. From the conveyor the mixture drops to a second conveyor travelling in the reverse direction and is then discharged on an inclined grate through which the distilled coal passes while the iron bodies are advanced by a rotatory discharge and returned by an elevator to the furnace. The coal may be pre-heated, but preferably at least enough iron is employed to give double the amount of heat theoretically required to raise the coal to, e.g., 540°. (B) Coal is carbonised by

heat transfer in a distillation chamber divided by a series of narrow, vertical, flat, heat-storing plates, *e.g.*, of iron, into a series of narrow, vertical retort chambers which are first heated, *e.g.*, to 760° by gas contact. The gas supply is cut off and the retort chambers are filled with coal, the carbonised material being finally discharged into a hopper at the lower end of the chambers, a vertical multiple ram assisting the discharge, and the process is repeated.

R. BRIGHTMAN.

Distillation of solid carbonaceous material. H. KOPPERS (U.S.P. 1,712,082, 7.5.29. Appl., 5.6.22. Ger., 11.8.21.).—Solid heat-storing bodies are heated and then brought into contact with the material to be distilled in a suitable chamber. Distillation is brought about mainly by the sensible heat of the solid bodies, which are made to pass with the material through the chamber and are then separated from the distilled product. They may be used for preheating the material in a second chamber before being returned to the heating furnace.

A. B. MANNING.

Low-temperature distillation of bituminous coal. R. H. CARR and C. B. WATSON, Asss. to PURE OIL Co. (U.S.P. 1,698,907, 15.1.29. Appl., 15.4.26).—The coal is forced upwards by conveyors into a funnel-shaped retort, and superheated steam or oil vapour at, *e.g.*, 400–425°, introduced at the top of the retort, travels downwards in countercurrent with the column of coal. The coke is removed by gravitation through an outlet at the top of the retort, and the volatile distillation products travel downwards and escape from tuyères between the coal inlet and the hot zone.

R. BRIGHTMAN.

Low-temperature carbonisation of bituminous material. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,664, 5.5.28).—The material is passed down between internally-heated heating bodies and walls of louvre pattern, each of the former consisting of a cooled skeleton frame which supports the heating surfaces. The latter are preferably constructed of material containing silicon carbide, of pearlite or electro-cast iron, or of ceramic material of good heat conductivity. A carbonising unit comprises a number of heating bodies and louvre walls, the latter being combined in pairs, each pair enclosing a tar chamber through which the volatile products of distillation pass to the condensing system. The louvres are kept in continuous vibration by means of a striking device.

A. B. MANNING.

Production of metallurgical coke. HINSELMANN KOKSOEFENBAUGES. M.B.H. (G.P. 454,404, 4.9.23).—Coals of high volatile matter content are carbonised in two stages, at 400–500° and at 850–1000°, respectively. Direct internal heating is used, gases or vapours suitably preheated in two or more heating chambers being passed through the material.

A. B. MANNING.

Distillation retorts, gas generators, and the like. L. P. WINBY (B.P. 312,394, 25.2.28).—The lining of a tubular retort is provided with a number of ports throughout its length so that the heating gases may be by-passed when the formation of a plastic layer causes a blockage.

J. A. SUGDEN.

Water-gas generator. O. B. EVANS, Assr. to I. C. COPLEY (U.S.P. 1,699,231, 15.1.29. Appl., 13.10.24).—The generator is divided by an internal bridge of refractory material into an upper and a lower fuel chamber separated by four unobstructed vertical passages to avoid a dead or cold mass of fuel at the axis of the fuel bed.

R. BRIGHTMAN.

Manufacture of water-gas. C. DAVIES, JUN. (U.S.P. 1,712,983, 14.5.29. Appl., 18.3.22).—The operations in the manufacture are carried out in the following order: (a) the fuel bed is supplied with air which has been preheated in a heat-transfer chamber; (b) steam is passed through the latter to the fuel bed, the ash in which is maintained at a sufficiently high temperature for slagging conditions, and the hot water-gas is discharged through the first heat-transfer chamber; (c) the fuel bed is again supplied with air preheated in the last-named chamber, and the hot products are used to preheat the fuel for the generator.

A. B. MANNING.

Apparatus for manufacture of fuel gas. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,686, 29.5.28. Addn. to B.P. 214,544; B., 1924, 549).—The gas producer described in the main patent or in B.P. 279,316 (B., 1928, 79) is modified by the addition of an enlarged secondary gasification chamber where a further supply of a gasifying agent may be added. By the provision of heating devices, *e.g.*, channels of refractory material traversed by combustion gases at about 1200°, in the fuel zone the apparatus may be used for the continuous production of water-gas.

A. B. MANNING.

Production of mixed gas from tar-free illuminating gas and water-gas. J. VON JASZOVSKY (Austr. P. 108,149, 10.4.25).—Two gasification chambers forming the upper part of a generator are charged in turn with coal. They are heated externally by the combustion of part of the water-gas which is produced continuously in the lower chamber of the generator, which is also heated externally. The illuminating gas produced in one upper chamber is passed through the hot coke in the other and is then mixed with the remainder of the water-gas from the lower chamber.

A. B. MANNING.

Production of hydrogen mixed with carbon monoxide and nitrogen from coke-oven gas. COMP. NAT. DE MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES ETABL. KUHLMANN (B.P. 286,291, 23.1.28. Fr., 3.3.27).—The preheated gas, with or without the addition of air or steam, is passed into a decomposition chamber packed with a filling, *e.g.*, coke, of high heat capacity and large surface. The temperature of the chamber is at least 1200° and the gas remains therein for about 1 sec. Regenerators are arranged on both sides of the reaction chamber, whilst between the latter and each regenerator, and in direct contact with these, are combustion chambers into which open heating gas and secondary air conduits. After passing the gas through the apparatus in one direction a current of air is introduced in order to burn any deposited carbon, after which the gas is passed through in the reverse direction. Before and after the passage of the air the apparatus is swept out with an inert gas.

A. B. MANNING.

Production of hydrogen from steam and carbon. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES, Assees. of DEUTS. BERGIN-A.-G. F. KOHLE- U. ERDÖLCHEMIE (F.P. 630,330, 5.3.27. Ger., 3.12.26).—Organic materials are carbonised at low temperatures and the coke residue is treated with steam at 500–800°. A. B. MANNING.

Oil-gas apparatus. A. J. BASSETT, Assr. to BRUCE MACBETH ENGINE Co. (U.S.P. 1,698,525, 8.1.29. Appl., 12.11.25).—The apparatus comprises a converter for converting oil into gas, an air pump, an oil supply, conduits for conveying air and oil to a mixer, and means for delivering the mixture into the converter. A branch air conduit connects the main air conduit to the oil supply. Means are provided for automatically regulating the pressure in the main air conduit in accordance with the gas pressure in the oil-gas delivery pipe. A. B. MANNING.

Manufacture of oil-gas. A. J. BASSETT, Assr. to BRUCE MACBETH ENGINE Co. (U.S.P. 1,698,526, 8.1.29. Appl., 12.11.25).—The supplies of air and oil to the converter are regulated by a valve in communication with the main gas-supply pipe, which operates to reduce the air-supply pressure, and hence the production of gas, as the demand for gas diminishes, or to increase it as the demand increases. The regulator operates by diverting air continuously in amounts inversely proportional to demands for gas, from the air-supply pipes to a pressure-developing pipe. R. BRIGHTMAN.

[Bituminous] waterproof composition. F. W. GOUGH (B.P. 312,467, 4.5.28).—A bituminous emulsion containing a small amount of an aromatic sulphonic or hydrogenated phenolic product is run into a hot argillaceous suspension containing a de-emulsifying agent (e.g., acetic or propionic acid, aluminium or ferric chloride, etc.). The mixture thickens to a non-sticky plastic mass which can be thinned with cold water and does not set until allowed to air-dry. J. A. SUGDEN.

Manufacture of asphalt emulsions. A. L. HALVORSEN and P. M. TRAVIS, Assrs. to EMULSION PROCESS CORP. (U.S.P. 1,714,982, 28.5.29. Appl., 3.3.28).—Emulsions of asphalt with water are improved by adding to them 0.75% by weight, or less, of sodium phosphate. H. ROYAL-DAWSON.

Production of oxides of nitrogen from ammoniacal liquor. D. L. JACOBSON, Assrs. to KOPPERS Co. (U.S.P. 1,713,045, 14.5.29. Appl., 22.8.27).—A gas containing oxygen, nitrogen, and ammonia is produced by blowing air through the liquor and, after the oxidation of any hydrogen sulphide present, is passed over a catalyst which effects the combination of oxygen and nitrogen. A. B. MANNING.

Conversion of hydrocarbons of high b.p. into those of low b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,789, 14.12.27).—The initial hydrocarbons are heated, preferably at 400–500°, under pressures of 20–50 atm., or if necessary under higher pressures, with catalysts of the type of the "ansolvo-acids" (cf. Meerwein, A., 1927, 836). The components of the "ansolvo" acid, e.g., α -naphthoic acid and tin tetrachloride, α -naphthylcarboxylic acid

and aluminium chloride, etc., may be added separately to the material under treatment. The process may be carried out in an inert atmosphere, or in the presence of hydrogen, or of gases containing or producing hydrogen. A. B. MANNING.

Production of valuable resin-like hydrocarbons. ZEHEDE WENDEL, Assees. of E. KUGEL and H. SCHWENKE (G.P. 454,307, 25.7.22).—The wash acid from the purification of benzol is neutralised with ammonia, and as the resinous substances separate they are diluted with benzol or solvent naphtha, the liquid at the same time being stirred and cooled. After separation from the ammonium sulphate, the solvent is removed from the resins by evaporation. A. B. MANNING.

Refining of petroleum, and of oils derived from coal. F. HOFMANN and W. STEGEMANN (B.P. 292,932–3, 25.6.28. Ger., 25.6.27).—(A) Petroleum fractions are treated with 3–4% of hydrogen fluoride; metal halides, e.g., boron fluoride, are added if desired. For thick oils the treatment is advantageously carried out at higher temperatures under suitable pressure, or the oil may be dissolved in a lighter oil. (B) Oils derived from the distillation of coal are similarly treated. A. B. MANNING.

Refining of hydrocarbons. I. G. FARBENIND. A.-G., Assees. of W. PUNGS and E. GALLE (G.P. 453,883, 26.2.24).—Water-white, pleasant-smelling oils are produced by subjecting tars, low-temperature tars, or their fractions, containing phenols, sulphur, and unsaturated compounds, to the process described in B.P. 249,309 (B., 1926, 432). Although the oils still contain 0.1–0.2% S when they are passed over the catalysts, the latter retain their activity for long periods. A. B. MANNING.

Refining of hydrocarbon oils. H. J. BRODERSON, Assr. to STANDARD OIL Co. (U.S.P. 1,698,428, 8.1.29. Appl., 7.6.24).—Hydrocarbon oil distillate heated to 26–38° is mixed with sodium plumbite solution etc. and delivered continuously through a perforated pipe to the conical bottom of the agitator below the surface of a further quantity of reagent. The oil rises through the solution and flows out continually at the top of the agitator, reagent being similarly withdrawn at the bottom and pumped to the mixer. R. BRIGHTMAN.

Treating hydrocarbon oil residues. F. M. ROGERS, Assr. to STANDARD OIL Co. (U.S.P. 1,698,452, 8.1.29. Appl., 28.8.22).—Asphalt distillation residues from crude oil containing asphalt are mixed with, e.g., 70% of "miners' oil" distillate, and treated with sulphuric acid of d 1.84. The sour oil is separated from sludge, washed, neutralised, and the diluent is distilled. E.g., asphalt residuum, m.p. 43°, penetration 200 at 250°, from Mid-continent crude, gave a residual oil, d 0.9345, flash point 268°, viscosity 320 sec. (Saybolt) at 99°. R. BRIGHTMAN.

Refining of crude fuel oil. E. A. SPERRY, Assr. to SPERRY DEVELOPMENT Co. (U.S.P. 1,699,379, 15.1.29. Appl., 21.9.25).—The oil is centrifuged under pressure to prevent frothing, being heated either in the centrifuge or by previous passage through a superheater. In the latter case the centrifuge coil may be a cooling system. R. BRIGHTMAN.

Refining of cracked benzenes. H. SUIDA and H. PÖLL (Austr. P. 108,165, 9.10.26).—The crude benzenes are treated with dry zinc dust and dry hydrochloric acid and are then distilled over zinc dust. A. B. MANNING.

Manufacture of motor fuels. H. D. ELKINGTON. From G. A. CRAMER (B.P. 312,245, 17.1.28).—One or more substances (*e.g.*, potassium oxalate or citrate) capable of preventing or diminishing knocking in the cylinders of an internal-combustion engine are dissolved in a liquid solvent insoluble in the motor fuel, *e.g.*, water, and the solution is dispersed in the motor fuel, with or without the use of an emulsifying agent and/or a stabiliser. H. S. GARLICK.

Reclaiming used lubricating oil. G. L. CHERRY, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 1,698,257, 8.1.29. Appl., 5.5.26).—Used oil, *e.g.*, car-axle oil, is heated to about 200° and treated with 1–3% of dry sodium hydroxide. After settling hot for 10–14 hrs., the oil is separated from the intermediate water layer and the sludge, washed with hot water, and centrifuged. R. BRIGHTMAN.

Separation of oils from mixtures of the same with solid substances. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,383, 22.2.28).—Residues obtained from the destructive hydrogenation of coal, tars, mineral oils, etc., or other mixtures of oils with solid materials, are treated at an elevated temperature with an organic solvent in which the oil is easily soluble at high temperatures, but difficultly soluble at low temperatures. After freeing the solution from undissolved residue, the solvent and oil are separated by mechanical means, *e.g.*, centrifuging, at a temperature preferably in the neighbourhood of the settling point of the oil. H. S. GARLICK.

Centrifugal separators (B.P. 296,670). **Reaction between emulsified liquids** (U.S.P. 1,698,270).—See I. **Liquid hydrocarbons** (B.P. 311,899).—See III. **Black colouring matter from peat** (B.P. 307,861).—See IV. **Ammonium sulphate** (B.P. 308,243 and 309,852). **Catalyst** (U.S.P. 1,698,009).—See VII. **Chemical reaction in hydrocarbon gases produced electrically** (B.P. 311,352).—See XI. **Soap** (B.P. 312,405).—See XII. **Printer's ink** (B.P. 312,745).—See XIII. **Leather oil** (U.S.P. 1,715,892).—See XV.

III.—ORGANIC INTERMEDIATES.

Graphical methods and steam-distillation problems. BAKER and PETTIBONE.—See I. **Cymene from cellulose.** BÖDTKER.—See V. **Preparation of alcoholic potash solution, and test for aldehyde in chloroform.** VALJASCHKO.—See XX.

PATENTS.

Regeneration of [mercury] catalysts. H. S. HIRST, S. W. ROWELL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 309,888, 17.1.28).—The mercury (and iron) sludge from the manufacture of acetaldehyde from acetylene is dissolved in concentrated nitric acid, and sulphuric acid is added up to a concentration of 55% H₂SO₄ (*d* 1.45). All the mercury and iron are thus precipitated as sulphate, which is separated, redissolved

in 15–20% sulphuric acid, and returned to the process. C. HOLLINS.

Manufacture of liquid hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 311,899, 10.4.28. Addn. to B.P. 258,608; B., 1928, 255).—Gas mixtures containing methane and/or ethane, unsaturated hydrocarbons, especially olefines, or the higher homologues of the paraffin series, and hydrogen, in which the ratio of carbon combined with hydrogen to total hydrogen is greater than the C:H ratio in methane, are passed at 500–900° and under the ordinary pressure over the catalysts described in the main patent. High yields of benzene hydrocarbons are produced. The process may advantageously be applied to the gases resulting from the low-temperature carbonisation of carbonaceous materials. A. B. MANNING.

Concentration of volatile aliphatic acids [acetic acid]. HOLZVERKOHLUNGS-IND. A.-G. (B.P. 291,433–4, 26.4.28. Ger., 2.6.27, and Addn. B.P. 302,268–9, 27.4.28. Ger., 13.12.27).—In the manufacture of concentrated acetic acid by way of potassium hydrogen acetate, (A and C) the vapour of dilute acetic acid, or (B and D) a benzene or acetone oil extract of the dilute acid, is brought in contact with potassium acetate, preferably on the countercurrent system, using a concentrated solution of the salt. The acid salt formed (A and B) may be isolated, or (C and D) may be decomposed without isolation, the optimum temperature being about 170–220°, preferably at reduced pressure. C. HOLLINS.

Production of butadiene hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,945, 10.9.27).—In the pyrogenic production of butadienes from chain or cyclic paraffins or olefines of artificial or mineral origin, using silica, silicates, noble metals, or the catalysts of B.P. 297,398 (B., 1928, 884), improved yields result when the starting materials are diluted with at least half their volume of added steam. [Stat. ref.] C. HOLLINS.

Purification of β-phenylethyl alcohol. E. C. BRITTON, Assr. to DOW CHEM. Co. (U.S.P. 1,698,932, 15.1.29. Appl., 12.2.24).—Crude β-phenylethyl alcohol after partial elimination of alcoholic impurities by fractional distillation is esterified, *e.g.*, in toluene, with 1.1 mols. of phthalic anhydride or other dibasic organic acid; the β-phenylethyl hydrogen phthalate is distilled in steam and hydrolysed. R. BRIGHTMAN.

Preparation of tetrazoles. A. BOEHRINGER (B.P. 285,080, 3.2.28. Ger., 11.2.27. Addn. to B.P. 280,529; B., 1929, 350).—Azoidide or sodium azide reacts with esters (especially sulphonic esters) of amidoximes, R·C(NH₂):NOH, or their Beckmann transformation products, or in presence of thionyl chloride, phosphorus pentachloride, phosphoryl chloride, etc., with amidoximes themselves, to give *C*-aminotetrazoles. Phenylacetamidoxime *O*-benzenesulphonate yields with sodium azide in boiling alcohol 5-amino-1-benzyl-1:2:3:4-tetrazole, m.p. 191°. The 5-amino-1-phenyl compound, m.p. 159°, is similarly obtained from benzamidoxime *O*-benzenesulphonate. C. HOLLINS.

Manufacture of higher alkylated guanidine derivatives. SCHERING-KAHLBAUM A.-G. (B.P. 285,873,

15.2.28. Ger., 24.2.27. Addn. to B.P. 279,884; B., 1929, 163).—In the process of the prior patent the amount of solvent used may be considerably less than that required for dissolution. C. HOLLINS.

Manufacture of materials from or comprising [wax-like] chlorinated hydrocarbons. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 309,421, 4.1.28).—Wax-like chlorinated naphthalenes, toluenes, etc. are mixed with 5–20% of a fat, wax, resin, pitch, or rubber, and/or alkaline substances such as sodium phosphate, lime, magnesia, pyridine, tetrahydroquinoline, urea, etc. The irritant action on the skin is prevented. C. HOLLINS.

Manufacture of arylaminonaphthalene derivatives. I. G. FARBENIND. A.-G. (B.P. 282,111, 13.12.27. Ger., 13.12.26).—The Bucherer reaction is applied to the condensation of phenylenediamines, aminophenols, leucoindophenols, leucoindamines, etc. with naphthylamines, naphthols, aminonaphthols (other than 1:8), and polyamino- or polyhydroxy-naphthalenes, to give products useful as fur bases and for dyeing acetate silk. Examples are: *o*-aminophenyl- β -naphthylamine, m.p. 96–97°; *m*- and *p*-aminophenyl- β -naphthylamines; *p*-aminophenyl- α -naphthylamine; *p*-hydroxyphenyl- α -naphthylamine; 4-amino-4'- β -naphthylaminodiphenylamine-2(or -2')-sulphonic acid; *m*- and *p*-hydroxyphenyl- β -naphthylamines; 2:7-di-(*p*-hydroxyphenylamino)naphthalene, m.p. 249–251°; and the 1:5-isomeride, m.p. 278–280°. C. HOLLINS.

Manufacture of 2-aminonaphthalene-3-carboxylic [2-amino-3-naphthoic] acid and of intermediate compounds. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 309,516, 6.1.28. Addn. to B.P. 282,450; B., 1929, 467).—The intermediate iron compound of the prior patent is obtained by heating 2:3-hydroxy-naphthoic acid with iron turnings, ammonium chloride, and aqueous ammonia at 180–190° in a stone-lined autoclave; the pressure generated is 40–45 atm. C. HOLLINS.

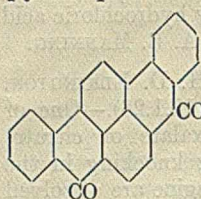
Production and use of cleaning, emulsifying, and wetting agents. ORANENBURGER CHEM. FABR. A.-G., Asses. of CHEM. FABR. MILCH A.-G. (B.P. 283,864, 17.1.28. Ger., 17.1.27).—In the process of B.P. 275,267 (B., 1929, 292) low-molecular, in place of high-molecular, fatty acids may be used. Examples are: anthracene and propionic acid, or diisopropyl-naphthalenesulphonic acid and acetic acid, treated with chlorosulphonic acid. C. HOLLINS.

Production of wetting, cleansing, or emulsifying agents and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 309,842, 15.10.27).—Sulphonic acids possessing wetting-out properties are converted into their "triethanolamine" (tri- β -hydroxyethylamine) salts. C. HOLLINS.

Manufacture of anthraquinone derivatives. BRIT. DYESTUFFS CORP., LTD., and A. SHEPHERDSON (B.P. 303,049, 15.2.28. Addn. to B.P. 271,602; B., 1927, 550).—Potassium acetate and water may replace the sodium acetate crystals in the process of the prior patent. C. HOLLINS.

Manufacture of condensation products of the pyrenequinone series. I. G. FARBENIND. A.-G.

(B.P. 287,050, 6.3.28. Ger., 12.3.27).—4-Aroylnaphthalenes are cyclised by means of sodium aluminium chloride etc. to the corresponding dibenzopyrenequinones. 4-Benzoylnaphthalene, m.p. 176°, obtained from benzanthrone-4-carboxylic chloride and benzene, gives 3:4:9:10-dibenzopyrene-5:8-quinone (annexed formula).



C. HOLLINS.

Manufacture of cyclic ketones, polycyclic ketones, and quinones. I. G. FARBENIND. A.-G. (B.P. 293,768, 11.7.28. Ger., 11.7.27).—Aromatic ketones and polyketones in general containing free *ortho*- and *peri*-positions suitably placed are cyclised by heating with sodium aluminium chloride at 110–120° in a current of air or oxygen. Examples are: 4-chlorobenzanthrone, m.p. 200°, from phenyl 4-chloro- α -naphthyl ketone, m.p. 80°; 4-cyanobenzanthrone, m.p. 234°; benzanthrone-4-carboxylic acid, m.p. 307°, from 4-benzoyl- α -naphthoic acid; 3:4:9:10-dibenzopyrene-5:8-quinone from 1:4-dibenzoylnaphthalene; 3:4:8:9-dibenzopyrene-5:10-quinone from 1:5-dibenzoylnaphthalene or 3-benzoylnaphthalene; benzanthrone-3:4-dicarboxylic anhydride, m.p. 350°, from 4-benzoyl-1:8-naphthalic anhydride; product from 3- α -naphthoylnaphthalene. C. HOLLINS.

Manufacture of arylaminoanthraquinones. K. ZAHN and P. OCHWAT, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,699,419, 15.1.29. Appl., 23.11.25. Ger., 12.12.24).—See B.P. 244,450; B., 1926, 866.

Vat dye intermediates (B.P. 307,950).—See IV. **Catalyst** (U.S.P. 1,698,009).—See VII. **Flotation agents** (B.P. 310,186).—See X.

IV.—DYESTUFFS.

Coupling of H-acid as a function of the acidity and in presence of strong electrolytes. G. R. LEVI and G. DELPONTE (Giorn. Chim. Ind. Appl., 1929, 11, 103–106).—The normal 85% yield of disazo derivative furnished by H-acid may be increased to 94–95% by replacing the neutral solution of H-acid by the freshly precipitated acid of distinctly acid reaction. The addition of the acid in solution, although it slightly accelerates the reaction, cannot be regarded as good practice as far as the yield or quality of the product is concerned. The yields of disazo compound are, in general, a function of the acid added, but phosphoric acid is an exception to this rule, as it behaves in the reaction as a weaker acid than its dissociation constant would indicate. The coupling process is retarded considerably by the presence of strong electrolytes, particularly alkali chlorides, perchlorates, and sulphates. T. H. POPE.

Coal-tar colours in foods. NICHOLLS.—See XIX.

PATENTS.

Manufacture of black colouring matter [from peat]. GOSUDARSTVENNAYA TORGOVAYA IMPORTNO-EKSPORTNAYA KONTORA GOSTORG, and S. KISLITZIN (B.P. 307,861, 15.12.27).—Raw peat mixed with solutions of

salt and alum is air-dried, distilled with tar, and the residuum acidified and boiled to precipitate a black colouring matter. C. HOLLINS.

Manufacture of vat dyes of the 2-thionaphthen-3-indoleindigo series. I. G. FARBENIND. A.-G. (B.P. 285,389, 14.2.29. Ger., 14.2.27).—6-Aminothioindoxyl is condensed with a 5:7-dihalogenoisatin, and chlorinated in the 5:7-positions in the thionaphthen nucleus by treatment with sulphuryl chloride. Alternatively, a 5:7-dichloro-6-aminothioindoxyl is condensed with a 5:7-dihalogenoisatin. 5:7-Dichloroisatin gives a brown vat dye yielding rather redder shades than the dye from 5:7-dibromoisatin. C. HOLLINS.

Manufacture of vat dyes of the anthracene series. I. G. FARBENIND. A.-G. (B.P. 296,758, 4.9.28. Ger., 7.9.27).—The vat dyes, probably carbazoles, obtainable by intramolecular condensation of 1:5- or 1:8-di-(α -anthraquinonylamino)anthraquinones ("trianthrimides") by the processes of B.P. 15,846 of 1909 (B., 1910, 939), B.P. 12,921 of 1910 (B., 1911, 531), G.P. 251,350 and 249,000, are treated with concentrated sulphuric acid below 5°, poured on ice, washed free from acid, and finally oxidised at 70–80° with hypochlorite, to give brighter vat dyes. C. HOLLINS.

Manufacture of [vat] dyes and intermediates [of the anthraquinone series]. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 307,950, 17.10.27).—3-Chloro-2-aminoanthraquinone is heated with antimony pentachloride in a nitro-solvent, or in a non-nitro-solvent with addition of nitrogen oxides, to give dichloro-flavanthron, an orange vat dye. C. HOLLINS.

Green dye of the anthraquinone series. J. OGILVIE, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,698,821, 15.1.29. Appl., 6.11.25).—1:4-Diarylaminoanthraquinones are sulphonated with 10–30% oleum in presence of boric acid below 50°. 1:4-Di-*p*-toluidinoanthraquinone with 10 pts. of 20–26% oleum at 25–50° in presence of 0.5 pt. of boric acid yields a bluish-green wool dye giving yellower shades than those obtained by sulphonation in absence of boric acid. R. BRIGHTMAN.

Manufacture of products [acid dyes] of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 282,452, 19.12.27. Addn. to B.P. 276,408; B., 1927, 809).—Other alicyclic amines are used in place of the hydroaromatic amines of the prior patent. 4-Bromo-1-aminoanthraquinone-2-sulphonic acid is condensed with cyclopentylamine or bornylamine to give a clear blue acid dye. C. HOLLINS.

Manufacture of vat dyes of the pyrenequinone series. I. G. FARBENIND. A.-G. (B.P. 287,845, 7.3.28. Ger., 26.3.27. Addn. to B.P. 287,050; B., 1929, 590).—The pyrenequinones of the prior patent are chlorinated or brominated. C. HOLLINS.

Manufacture of new azo dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 309,879, 16.12.27).—2:3-Hydroxynaphthoic 3-alkoxy- β -naphthylamides have good affinity for cotton, and the padded fibre may therefore be rinsed so as to yield ice colours fast to rubbing. These arylamides may be kept in concentrated stock solutions for dyeing in a standing bath;

no addition of formaldehyde is necessary. The 3-methoxy- β -naphthylamide, m.p. 197–198°, is coupled on the fibre or in substance with diazotised 4-nitro-*o*-anisidine (bluish-red), 5-chloro-*o*-toluidine (bluish-red), *m*-4-xylydine (red), *p*-chloroaniline (scarlet), 5-chloro-2-aminophenyl β -naphthyl ether (red), 3-chloro-4-aminobenzanilide (yellow-red), 2:5-dichloroaniline (yellow-red), 2:5:5'-trichloro-2'-aminodiphenyl ether (red), 2:3-dichloroaniline (red-orange), 2:3:4-trichloroaniline (yellow-red), *m*-nitro-*p*-toluidine (claret), 2-chloro-5-nitroaniline (yellow-red), 5-chloro-*o*-nitroaniline (yellow-red), 4-nitro-5-amino-*o*-tolyl methyl ether (blue-garnet), *o*-aminoazotoluene (garnet), 4-chloro-*o*-anisidine \rightarrow 4-chloro-*o*-anisidine (dark violet), 1:5-diaminoanthraquinone (violet). The 3-ethoxy- β -naphthylamide gives similar shades. C. HOLLINS.

Manufacture of [water-soluble] azo dyes [for lakes and wool dyeing]. I. G. FARBENIND. A.-G. (B.P. 283,897, 17.1.28. Ger., 19.1.27).—*o*-Nitroarylamines are coupled with a sulphonated acetoacetic arylamide, or the corresponding non-sulphonated dyes are sulphonated with 100% sulphuric acid or chloro-sulphonic acid. Sulphonation of *m*-nitro-*p*-toluidine \rightarrow acetoacetic *o*-chloroanilide for a greenish-yellow and of *m*-nitro-*p*-toluidine \rightarrow acetoacetic *o*-anisilide for a reddish-yellow is described. C. HOLLINS.

Azo dyes and cellulose derivatives [acetate silk] dyed therewith. W. HENTRICH, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,695,654, 18.12.28. Appl., 4.2.27. Ger., 26.2.26).—Acetate silk is dyed with water-soluble monoazo dyes obtained by coupling a diazotised dinitro-*o*-aminophenol with aminonaphthols containing no sulphonic or carboxylic groups. Examples are: 4:6-dinitro-*o*-aminophenol with 1:8-, 1:6-, 2:8-, or 1:5-aminonaphthol (black to violet-brown, giving blacks and deep browns by diazotisation and development on the fibre); partially reduced 2:4:6-trinitro-*m*-cresol gives similar dyes. 4:6-Dinitro-*o*-aminophenol \rightarrow 1:8-aminonaphthol is especially claimed. C. HOLLINS.

Vat dye derived from 3-halogenopyrazolanthrone. R. BERLINER, B. STEIN, and W. TRAUTNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,695,631, 18.12.28. Appl., 4.2.27. Ger., 3.3.26).—See B.P. 285,555; B., 1928, 362.

Azo dyes and their manufacture. H. WAGNER, H. EICHWEDE, and E. FISCHER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,694,568, 11.12.28. Appl., 22.12.25. Ger., 6.8.23).—See B.P. 256,808; B., 1926, 866.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemical examination of tissue of corn [maize] stalk. C. J. PETERSON and R. M. HIXON (Ind. Eng. Chem. [Anal.], 1929, 1, 65–67).—Maize stalks separated and cleaned by hand from bales have been examined by the general methods of Schwalbe (B., 1919, 407 A, 408 A). The experimental conditions for determination of lignin, cellulose, and pentosan are precisely described; results showed 34.3, 35.4, and 27.6%, respectively. Treatment of the stalks with 5% alkali, or with acid, and subsequent analysis of the products has been applied

to identification of the hemicellulose, and the results of both methods are shown to be of the same order, although not in exact agreement. The stalks were also separated into the outer shell, pith, and inner vascular bundles, but no large differences were found in the composition of these constituents, although variations in the physical properties of the cellulose were clearly detected.

R. H. GRIFFITH.

Determination of silk and cotton in asbestos yarns. H. SOMMER (*Gummi-Ztg.*, 1929, 43, 2103—2104).—The asbestos content is determined by removal of silk and cotton with cuprammonium solution (cf. B., 1929, 277); the content of silk is found by difference after removal with a solution of copper sulphate (10 g.), glycerol (5 g.), and potassium hydroxide (in sufficient quantity to redissolve the precipitated hydroxide) in water (100 c.c.) or by boiling with *N*-sodium hydroxide. The residue from the latter determination is then used for determining the remaining cotton by cuprammonium solution.

D. F. TWISS.

Manufacture of sulphite-cellulose from pinewood.

O. ROUTALA and J. SEVÓN (*Cellulosechem.*, 1929, 10, 97—107).—Decomposition of pine wood with the usual calcium bisulphite solutions is only possible in exceptional cases when the composition of the wood is uniform, and its rosin content not too high. By complete or partial replacement of the calcium by magnesium or alkali metals better decomposition is obtained; cooking acids containing both magnesium and sodium as bases are specially satisfactory provided that the above limitations to the composition of the wood obtain. The chief cause of trouble is the difficulty of saturating the wood with liquor due to the rapid consumption of sulphurous acid, whereby difficultly soluble normal calcium sulphite is precipitated in the pores of the wood. This separation of calcium sulphite before complete saturation of the wood may be avoided by prolonging the time taken to reach the reaction temperature or by the addition of weak acids such as acetic or phosphoric acid. The addition of the alkali salts of the chosen weak acid considerably shortens the time of cooking, and their buffering action satisfactorily regulates the p_H of the liquid during cooking. This protective action results in better yield and quality of the cellulose, and a complete and uniform decomposition of non-uniform pine wood of high rosin content may be obtained in a somewhat longer time than is required for spruce wood. The addition of these salts causes better dissolution of the rosin, but it is not possible to reduce the content considerably; the remedy for this is to be found either in pretreatment of the wood or after-treatment of the cellulose. Up to 80% may be removed by extraction with small quantities of sodium hydroxide, and the addition of 0.5—1% of sodium hydroxide on the weight of cellulose in the separator or hollander should be the simplest way of removing the rosin. Decomposition of pine wood is possible by means of nitric acid, and good cellulose is obtained with no damage to the fibre, but the high consumption of chemicals and the production of hydrocyanic acid are drawbacks.

B. P. RIDGE.

Changes in moist sulphite-cellulose on storing.

E. HOCHBERGER (*Papier-Fabr.*, 1929, 27, 282—284).—A method is described by which the isoelectric point of pulp may be determined. The isoelectric point of machine-dried, bleached pulp is changed slightly to the alkaline side by storage of the material for two months, but similar storage of moist pulp, bleached or unbleached, results in a marked change to the acid side. Moist pulp stored for this time absorbs considerably more 18% sodium hydroxide solution per unit weight under standard conditions of treatment than does fresh material in the air-dry state, and also develops greater acidity, as shown by determinations of its p_H by use of suitable indicators.

B. P. RIDGE.

Dependence of the bleaching number [of pulp] on the course of the cooking in the Ritter-Kellner process. H. NERAD (*Papier-Fabr.*, 1929, 27, 277—282).—The chemical reactions involved in the cooking process and the effect of such factors as p_H value, temperature, time to reach the effective cooking temperature, etc. on the course of the process are discussed. A formula is deduced by means of which the bleaching capacity of a pulp may be derived mathematically from factors such as the percentage sulphur dioxide content of (1) the liquor at 100°, (2) the waste liquor, (3) the waste gases, etc., which depend only on the course of the cooking process.

B. P. RIDGE.

Cymene, a by-product in the manufacture of cellulose by the bisulphite process. E. BÆDTKER (*J. Pharm. Chim.*, 1929, [viii], 9, 417—434).—The analysis, composition, and purification of crude cymene obtained in the bisulphite process for the manufacture of cellulose from wood is described. The chief impurities are terpenes, resins, fatty acids, and furfuraldehyde, and methods for their removal are discussed. Pure cymene is not oxidised on exposure to air; it has b.p. 177.1—177.2°/760 mm., d_4^{20} 0.8606, n_D^{20} 1.4920, and R_M 45.38 (calc. 44.92).

E. H. SHARPLES.

Effect of chemical agents, especially of oxidising influence, on the behaviour of cellulose. P. WAENTIG (*Cellulosechem.*, 1929, 10, 81—85).—Purified cellulose is characterised by a fibrous structure, which is distinguished by a regular arrangement of anisotropic micelles or crystallites, and which possesses comparatively great resistance to swelling. Swellable regenerated cellulose is characterised by disorientation, possibly also by amorphisation, of the micelle (which explains its increased swelling capacity), and alkali-soluble (chemically modified) cellulose corresponds with a reduction in size of the crystallite or micelle through intramolecular decomposition. In the formation of hydrocellulose and oxycellulose not only disorientation, or amorphisation, of the crystallite occurs, but also intramolecular or intramolecular decomposition. The cellulose fibre is partly physically and partly chemically decomposed, but to a greater extent physically than chemically. The decreased viscosity of viscose solutions during ripening and the increased alkali-solubility of the regenerated viscose are explained in this way, since ripening is accompanied by absorption of oxygen from the air—hence by oxidation. The accelerated ripening of viscose in the presence of metals is due to the fact that the latter act as oxygen carriers. That sodium

hydroxide has no such effect in reducing the size of the cellulose micelles is shown by the effects of mercerisation in not reducing the strength or the α -cellulose content of the material. Cuprammonium acts in the same way as concentrated sodium hydroxide; the more rapid oxidation and fall of viscosity of the cellulose on keeping are promoted by the catalytic action of the copper. Differences between the viscosities of xanthate solutions prepared from cotton linters and cellulose wadding, respectively (which have been explained by the assumption that the former has a higher "micelle mol. wt." than the latter), depend on the previous history of the materials, since not only the oxy- or hydro-cellulose content of the material, but also the specific natural compound of the cellulose, must be considered. The effects on the viscosity of the viscose or cellulose nitrate solutions of cutting up the original material into small particles, and of valency changes in the cellulose complex due to oxidation, are discussed. B. P. RIDGE.

Reaction of cellulose with phenylhydrazine acetate. C. J. STAUD and H. LE B. GRAY (Ind. Eng. Chem. [Anal.], 1929, 1, 80—81).—The reaction described by Vignon (Compt. rend., 1899, 128, 579; cf. B., 1899, 579) has been modified by treating sulphite pulp (5 g.) with 15 c.c. of phenylhydrazine in dilute acetic acid, the whole being heated in a steam bath and then washed repeatedly with aqueous alcohol. After 16 hrs. no further increase in the nitrogen content of the residue was observed; this time was accordingly used in subsequent experiments with other materials. Attempts are made to correlate the extent of this reaction with the degree of cellulose degradation, but although a qualitative agreement appears to exist, no strict numerical comparisons can be made. R. H. GRIFFITH.

Strength testing of cellulose. G. HALL (Papier-Fabr., 1929, 27, 341—347).—American proposals for the standardisation of methods of testing the strength of cellulose are examined and criticised, and a report of such attempted standardisation in Sweden is made. The relative importance of physical tests used for paper, such as tearing strength, resistance to creasing, the bursting test, porosity, etc., in relation to degree of grinding, weight, and other properties of the material is examined, and recommended conditions under which these tests should be carried out are discussed.

B. P. RIDGE.

Effect of cathode rays on paper. SCHOEPFLE and CONNELL.—See II.

PATENTS.

Production of cellulosic material. EUROMERICAN CELLULOSE PRODUCTS CORP., Assees. of B. DORNER (B.P. 283,851, 31.12.27. U.S., 17.1.27).—After mechanical disintegration, straw etc. is leached by a slowly-moving countercurrent of water, cooked with just sufficient caustic alkali to react with the whole of the silica present, freed from silicate liquor, and further cooked in the ordinary way at a temperature below that at which the pentosans present are caramelised. The pentosans may be precipitated from the waste liquor by carbon dioxide, and the caustic alkali then regenerated from the solution. F. R. ENNOS.

Treatment of flax straw and other fibrous materials. Treatment of scutched vegetable fibres. VICKERS, LTD., and O. D. LUCAS (B.P. 309,210—1, 8.12.27).—(A) Unscutched vegetable fibres are digested first with water for 1 hr. at 182°, furfuraldehyde being distilled off, and then for 4—5 hrs. with 4—5 times their weight of a 5% caustic soda solution at 150—170°. (B) Scutched fibres are heated with a 1—5% caustic soda solution at 130—170° for 20 min. to 4 hrs. In each instance a plasticiser is added during the soda digestion, and the fibre, after treatment as above, is washed in water, immersed in acid, again washed, and dried for spinning. F. R. ENNOS.

Improving the spinning properties of cotton. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 310,688, 31.5.28).—Cotton is humidified by spraying it with aqueous solutions of organic sulphonic acids possessing high wetting properties; 10 litres of a 1% solution of, e.g., the sodium salt of butylnaphthalenesulphonic acid, is a suitable quantity per 100 kg. of cotton. Organic solvents and hygroscopic agents may be added if desired. D. J. NORMAN.

Obtaining artificial threads of natural silk from colloidal solutions of natural silk. T. MUTO, S. HIDA, and KANEGAFUCHI BOSEKI KABUSHIKI KWAISHA (B.P. 312,114, 18.2.28).—After partial coagulation, the thread is elongated by means of sliding friction applied by rollers around practically the whole circumference, and the coagulation is finally completed. F. R. ENNOS.

Manufacture of mothproofing media and protection of wool, skin, material, textiles, etc. against insects. I. G. FARBENIND. A.-G. (B.P. 285,825, 21.2.28. Ger., 21.2.27).—A mothproofing medium is applied as in B.P. 173,536 and 295,742 (B., 1922, 138 A; 1928, 810) in the presence of a wetting agent which is an aromatic sulphonic acid containing one or more aliphatic groups in the nucleus, e.g., isopropyl-naphthalenesulphonic acid. F. R. ENNOS.

Artificial silk-yarn mixtures. G. DIETRICH (B.P. 309,340, 15.6.28).—Artificial silk waste or staple fibre is spun with "African wild silk" (anaphe) to give a yarn of increased strength. F. R. ENNOS.

Manufacture of cellulose esters. I. G. FARBENIND. A.-G. (B.P. 284,298, 27.1.28. Ger., 27.1.27).—Insoluble or sparingly soluble cellulose or hydrocellulose esters of higher fatty acids are mixed in an anhydrous liquid, e.g., tetrachloroethane, toluene, etc., with an organic or inorganic acid, an acid anhydride, and the salt of a strong acid with a weak base, e.g., ferric chloride, or with a mixture of any of these, and are heated at a high temperature until the desired solubility of the ester is attained. F. R. ENNOS.

Manufacture of cellulose esters and articles made therefrom. H. DREYFUS (B.P. 308,322—3 and 309,201, [A, B] 13.9. and 15.12.27, [C] 28.9. and 15.12.27).—(A) Cellulose which has been pretreated with organic acids (cf. B.P. 263,938; B., 1927, 247) is esterified by means of a fatty acid anhydride in the presence of at least 2% each of a hydrogen halide and a zinc halide, preferably 5—10% of hydrochloric acid (calc. as HCl) and 5—10% of zinc chloride on the weight of cellulose

in each case. (b) The process of (A) is applied to cellulose which has not been pretreated with organic acids. (c) Other halide salts, *e.g.*, iron, manganese, nickel, cobalt, or copper chloride, may replace the zinc halide in the above processes.

D. J. NORMAN.

Manufacture of cellulose esters. H. DREYFUS (B.P. 311,790, 17.12.27).—Cellulose which has been pretreated with organic acids is esterified by a fatty acid anhydride in the presence of one or more halides of arsenic, antimony, or phosphorus in the presence or absence of a hydrohalide acid. Suitable quantities are 10–15% of, *e.g.*, antimony or phosphorus pentachloride, and 10–15% of hydrochloric acid on the weight of cellulose in each case.

D. J. NORMAN.

Manufacture of esters of cellulose. I. G. FARBENIND. A.-G. (B.P. 285,858, 22.2.28. Ger., 23.2.27).—Cellulose esters of the higher homologues of acetic acid are prepared by warming cellulose for several hours with a halogenated acetic acid anhydride, the higher fatty acid, and a catalyst, *e.g.*, monochloroacetic acid or sulphuric acid. The ester is then precipitated by water in the usual way.

F. R. ENNOS.

Preparing partially hydrolysed cellulose acetates and articles made therefrom. S. I. VLES (B.P. 310,434, 25.11.27).—In the process of B.P. 292,398 (B., 1928, 637) an acetate of much higher viscosity is obtained if the quantity of water present in the hydrolysing solution is increased to at least 50 pts. by wt. per 100 pts. of dry cellulose over and above that required for converting any excess of acetic anhydride into acetic acid.

D. J. NORMAN.

Esterification of cellulosic materials. B. WYLAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 310,556, 26.1.28).—Pyridine sulphuric anhydride is used as a catalyst in the esterification of cellulose. *E.g.*, 15 pts. of paper cellulose are added to a mixture containing 15 pts. of pyridine sulphuric anhydride, 20 pts. of glacial acetic acid, and 10 pts. of acetic anhydride previously heated to 100°. The reaction is allowed to proceed for about 15 min. at 100°, and, after cooling, the acetate is precipitated with water. The resulting product gives viscous solutions with acetone. By varying the conditions, products are obtained which contain different amounts of combined acetic acid and are soluble in different solvents.

D. J. NORMAN.

Manufacture of cellulose aceto-esters containing higher acyl groups, and of cellulose esters containing halogen-substituted fatty acid groups. H. T. CLARKE and C. J. MALM, ASSIS. to EASTMAN KODAK CO. (U.S.P. 1,698,048—9, 8.1.29. Appl., 18.1.28).—(A) Partly deacetylated cellulose acetate, containing, *e.g.*, 34.5–37.5% of acetyl, is esterified with anhydrides of higher fatty acids, in presence of chloroacetic anhydride, methoxyacetic anhydride, or other organic anhydride containing less than 10 carbon atoms which impels esterification. *E.g.*, cellulose acetate (5 pts.) added to a mixture of 20 pts. of stearic anhydride and 40 pts. of chloroacetic acid in presence of 0.05 pt. of magnesium perchlorate at 60–65° affords a product containing 30% of stearyl. A product containing 47.5% of stearyl is similarly obtained from 5 pts. of cellulose acetate

(36.6% acetyl), 15 pts. of stearic acid, 20 pts. of chloroacetic acid, and 20 pts. of chloroacetic anhydride. With cellulose acetate (37.5% acetyl) and 15 pts. of oleic acid the same mixture affords a product which on bromination in chloroform contains 15.7% Br. Cellulose acetate (34.5% of acetyl) affords a cellulose acetodibromostearate containing 19% Br. Similar products are obtained by esterification of a mixture of 5 pts. of cellulose acetate, 20 pts. of chloroacetic anhydride, and 40 pts. of chloroacetic acid, with 20 pts. of α -dibromostearic acid and 0.5% of magnesium perchlorate trihydrate. Cellulose aceto-di- and -tetrachlorostearates containing 7.3–9% and 20–23% Cl, respectively, are similarly obtained. (B) Cellulose material is treated with a halogen-substituted fatty acid containing more than five carbon atoms in presence of an organic acid anhydride which promotes esterification, but which does not itself esterify cellulose. Thus the reaction bath obtained from 8 pts. of cellulose, 40 pts. of chloroacetic anhydride, 20 pts. of chloroacetic acid, and 0.05 pt. of magnesium perchlorate trihydrate at 60–65°, with 15 pts. of α -bromostearic acid and 4 pts. of acetic acid affords a cellulose aceto- α -bromostearate containing 11.3% Br. The preparation of cellulose acetobromostearate (23% Br), acetochlorostearate (8.5% Cl), acetotetrachlorostearate (14% Cl), and aceto- α -bromohexanoate (5% Br) is described.

R. BRIGHTMAN.

Production of viscose. I. G. FARBENIND. A.-G. (B.P. 287,492, 21.3.28. Ger., 21.3.27).—Crude cellulose xanthate is treated in one or two stages for a short time (5–10 min.) at not above 16° with aqueous alcohol—particularly methyl alcohol—which, taking into account the water content of the xanthate, contains 70–80% by vol. of anhydrous alcohol in the first stage. The mass is then pressed to remove the liquid, broken up without drying, and at once dissolved to form viscose.

F. R. ENNOS.

Production of compounds resembling celluloid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 308,395, 12.1.28).—Cellulose esters or ethers are incorporated with a neutral phosphoric ester containing at least one radical of a primary aliphatic alcohol with more than four carbon atoms, the remainder belonging to the aliphatic or hydroaromatic series, *e.g.*, diisooamyl butyl phosphate; other materials usually employed in the manufacture of artificial masses may be added if desired.

F. R. ENNOS.

Solvents for nitrocellulose and for acetylcellulose. L. E. CLÉMENT, ASSR. to DU PONT-PATHÉ FILM MANUF. CORP. (U.S.P. 1,713,512 and 1,713,997, 21.5.29. Appl., 25.11.24. Fr., 30.10.24).—A mixture of anhydrous ethyl alcohol and acetone is used, the proportions, by vol., being: (A) 4:2, and (B) 1:1.

H. ROYAL-DAWSON.

Manufacture of artificial [silk] products by dry-spinning processes. H. DREYFUS (B.P. 312,203, 18.1.28).—The spinning solution consists of a cellulose ester or ether with a volatile substance, *e.g.*, acetone, a non-solvent liquid of higher b.p. than the volatile substance, *e.g.*, xylene, and not more than 30% of the weight of the cellulose compound of a solvent of higher

b.p. than the non-solvent liquid, *e.g.*, diacetone alcohol. F. R. ENNOS.

Manufacture of artificial filaments from viscose. W. HARRISON (B.P. 309,053, 1.11.27, 19.12.27, and 5.6.28).—Viscose threads of high strength are obtained, without the use of acids of high concentration as swelling agents, by spinning viscose solution, preferably prepared from unmatured or slightly matured alkali-cellulose, into an acid coagulating bath of such a type, *e.g.*, of p_H less than that of 7% sulphuric acid, that the viscose, though rapidly coagulated, is relatively slowly decomposed into cellulose, and stretching the filaments 25—100% or more during the transition stage from the coagulated viscose to cellulose. To provide more opportunity for adjusting the time of stretching, decomposition of the viscose may be delayed by passing the coagulated filaments into a bath of water containing substances, *e.g.*, neutral salts with or without the addition of glucose or the like, which, while permitting the swelling action of the water, will prevent dissolution of the filaments. D. J. NORMAN.

Manufacture of artificial threads, bands, films, etc. from solutions of cellulose esters and ethers. Manufacture of cellulose esters. RUTH-ALDO Co., Inc., Assees. of H. L. BARTHELEMY (B.P. 282,787 and 282,789, 28.12.27. Fr., 28.12.26).—(A) A solution of the cellulose ester or ether is coagulated in the desired form by means of alcohols or polyalcohols which are capable of taking up the volatile solvent of the ester or ether, *e.g.*, propyl alcohol, glycol, cyclohexanol, etc., to which a hydrocarbon such as xylol may be added. (B) After acetylation of cellulose in the presence of sulphuric acid as catalyst, the excess of acetic anhydride is destroyed by addition of an aqueous solution of formic or acetic acid, the whole being cooled to the initial temperature. A mixture of water, formic or acetic acid, and hydrochloric acid is then added to produce partial hydrolysis which is accompanied by destruction of the harmful cellulose acetosulphuric esters; the quantity of hydrochloric acid should be at least equivalent to that of the sulphuric acid used as catalyst, the total water in the mass being at the same time adjusted to 5—15% of the total acids present after the addition of the hydrolysing mixture. F. R. ENNOS.

Manufacture of [waterproof] artificial material and articles from alkyl cellulose. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 308,284, 13.10.27).—A mixture of an aqueous solution of a cellulose alkyl ether with a colloidal substance, either as such or in the form of a solution or an emulsion, *e.g.*, a drying oil or an aldehyde condensation product, is made into the desired shape and treated by suitable means such as heating to convert the latter into a sparingly soluble or insoluble state. [Stat. ref.] F. R. ENNOS.

Apparatus for spinning solutions of cellulose ethers or esters. RUTH-ALDO Co., Inc., Assees. of (Miss) M. KLEIN (B.P. 292,561, 22.6.28. Fr., 22.6.27).—Each cell is subdivided by one or more diaphragms into an upper zone containing the spinning nozzle and lower heating zones where aspiration is effected and which are traversed successively by the spun thread.

The diaphragms, which consist of a fixed portion and a central hinged portion, the latter containing a triangular notch which may be partially closed so as to leave only sufficient space for the passage of the thread, serve to prevent hot air passing into the upper zone. F. R. ENNOS.

Treatment of fibrous materials for papermaking etc. E. V. HAYES-GRATZE. From C. LEYST (B.P. 311,945, 21.5.28).—Straw, esparto grass, etc., after pretreatment, if desired, by exposure in a moist alkaline condition at 30° for 3—6 days, is cooked with water and about 30% of lime for 6—8 hrs., washed free from lime, and ground to a flour-like consistency with or without an intermediate drying. Alternatively, the material may be first ground and then cooked with lime and water. F. R. ENNOS.

Treatment of fibrous material [with synthetic resins]. A. L. BROWN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,695,912, 18.12.28. Appl., 16.3.23).—Paper or other sheet fabric is impregnated with aqueous formaldehyde containing 10% of hexamethylenetetramine, dried, and passed through a mixture of cresol and 20% of tung oil previously treated with a little formaldehyde, or through a solution of soluble phenol-formaldehyde resole. The sheet is then heated at 100°, and may afterwards be hardened as usual. C. HOLLINS.

[Discharge valve for paper] pulp strainers of the drum type. A. WHITE, W. M. WALLACE, J. and R. STEWART, Exors. of J. WHITE (B.P. 311,948, 26.5.28).

Sodium acetate (U.S.P. 1,695,742).—See VII. **Wool fat (B.P. 286,252).**—See XII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Effect of light during the bleaching of some coloured cellulose materials by hypochlorites. F. SCHOLEFIELD and C. K. PATEL (J. Soc. Dyers and Col., 1929, 45, 175—178).—When cotton or viscose, dyed with certain vat yellows and oranges, is subjected in the dark to the action of hypochlorite solutions no appreciable tendering takes place. On the other hand, if dyed material, saturated with hypochlorite, is exposed to the action of daylight or the light from the carbon arc of the Fadeometer, the cellulose is tendered when dyed with certain vat yellows, oranges, and reds. Indanthrene Yellow G produces no tendering under these conditions. The tendering action in the case of both sodium hypochlorite and hydrogen peroxide is to be explained by the absorption of rays of light by the yellow and orange dyes, which rays are not absorbed in the case of blues, violets, etc. L. G. LAWRIE.

Dyeing without colouring matters. BRUÈRE (Ann. Falsif., 1929, 22, 132—139).—Soluble and metallic salts under certain conditions of acidity in the presence of sodium nitrite will develop colours on animal fibres or, after "pseudo-animalisation," on artificial silks, with or without the addition of phenolic compounds or amines; a wide range of permanent colours may thus be produced. In order to determine in specific cases whether the colouring material has been produced in this way, the ordinary procedure according to the tables of

Green, Rota, etc. is followed, when it will be found that the yellow colour with nitric acid given by the indigo group will usually be present in this class of dye (chromium colours often showing a violet colour); sulphuric acid gives only slight colours, and hydrochloric acid nothing distinctive; the ash will furnish useful information, and a comparison of the shade with the colour possibilities of the various cations will confirm the findings. Theoretically, dissolution of the animal fibres in hot sodium hydroxide followed by saturation with sulphuric acid should give a solution of sodium sulphate and the colour metal as sulphate, but in practice only iron can thus be readily detected, as the other metals form organo-metallic complexes.

D. G. HEWER.

Action of alkalis and other swelling agents on viscose silk. A. J. HALL (J. Soc. Dyers and Col., 1929, 45, 171—175).—Viscose suffers shrinkage in length, loss of lustre, strength, softness of handle, and elasticity by treatment with caustic soda (d 1.06—1.35) and subsequent washing. The harmful effects take place during the washing, and the viscose will retain its original properties if washed in solutions of certain inorganic and organic substances. The effect of caustic potash decreases as the temperature is raised, and at temperatures above 0° it has practically no deleterious action. By the use of caustic potash instead of caustic soda it is possible to mercerise cotton goods containing viscose without the artificial silk being adversely affected. The swelling action of caustic soda, concentrated sulphuric acid, and phosphoric acid on viscose increases similarly the affinity of the viscose for direct dyes.

L. G. LAWRIE.

PATENTS.

Colouring of cellulose esters and of solutions or compositions containing such esters. W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 309,840, 11.10.27).—A pigment is incorporated with dry, ungelatinised cellulose nitrate together with a gelatiniser and/or suitable oil such as linseed oil or castor oil.

C. HOLLINS.

Use of sulphonylates for stripping dyes. C. S. HOLLANDER, Assr. to ROHM & HAAS Co. (U.S.P. 1,696,164, 18.12.28. Appl., 5.2.27).—A soluble zinc salt (sulphate, chloride, acetate) is added to sodium formaldehyde-sulphonylate to stabilise the solution.

C. HOLLINS.

Treatment [mordanting and weighting] of cellulose derivatives [esters and ethers] and products made therewith. BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. [A] 309,899 and [B] 309,876, 15.11.27. Addn. [B] to B.P. 258,874; B., 1928, 228).—(A) Mordanting and (B) weighting of cellulose ester and ether fabrics, yarn, etc. may be effected at lower temperatures in the presence of zinc salts (chloride).

C. HOLLINS.

Manufacture of [weighted] artificial silk. Soc. CHIM. DES USINES DU RHÔNE (B.P. 294,623, 13.1.28. Fr., 29.7.27).—The process of B.P. 275,553 (B., 1928, 639) is applied for preparing cellulose esters or ethers containing 2—25% of weighting agents, e.g., barium sulphate. The resulting product when spun gives silk of subdued lustre.

D. J. NORMAN.

Sizing of artificial silk. M. GUINET (F.P. 630,929, 16.3.27).—The fibres are coated with a mixture of a sulphonated substance (sodium sulphoricinate) and a colloid, the latter being produced by the action of a boiling alkaline solution on animal skins or on gelatin.

A. B. MANNING.

Treatment ["animalisation"] of cotton and artificial silk fibres. CHEM. FABR. VORM. SANDOZ (B.P. 284,358, 28.1.28. Ger., 28.1.27. Cf. B.P. 195,619 and 249,842; B., 1926, 128; 1927, 71).—Cotton, mercerised cotton, viscose silk, or other regenerated cellulose silk is treated at 110° with benzene- or toluene-sulphonyl chloride and a tertiary base (pyridine) in presence or absence of a solvent (nitrobenzene). The treated textile can be dyed with any acid dye.

C. HOLLINS.

Production of dyeings fast to light. P. RABE, H. STÖTTER, B. WENK, and W. SCHEPSS, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,694,562, 11.12.28. Appl., 15.12.26. Ger., 22.12.25).—See B.P. 283,281; B., 1928, 189.

Dyeing of cellulose derivatives (U.S.P. 1,695,654).—See IV. **Printing preparations** (B.P. 311,795).—See XIII.

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

"Poupin" system [for the lixiviation and crystallisation of caliche etc.]. H. GERLACH, WARNECKE, and F. BLANCKENHORN (Caliche, 1929, 10, 534—556; 11, 5—29).—Full technical details are given of the working of the system.

H. F. GILBE.

The Trona enterprise [at Searles Lake, California]. G. R. ROBERTSON (Ind. Eng. Chem., 1929, 21, 520—524).—The lake consists of a solid crystalline layer beneath which is a brine containing approx. 34.6% of a complex mixture of salts with the following ions: chloride, sulphate, carbonate, borate, phosphate, bromide, sulphide, sodium, potassium, and lithium, together with traces of arsenic, calcium, iron, aluminium, and antimony compounds. Potassium chloride is present to the extent of 4.75%. The principles on which the extraction of this salt is based are illustrated by phase-rule diagrams, and a brief description of the main features of the plant employed is included. The daily output of the plant is 240 tons of potassium chloride, 130 tons of borax, and 10 tons of boric acid.

H. INGLESON.

Rapid determination of nitrogen by Kjeldahl's method. H. LUNDIN and J. ELLBURG (Woch. Brau., 1929, 46, 133—137, 147—149).—Details are given of a technique employing a reaction mixture of hydrogen peroxide with sulphuric and phosphoric acids and potassium sulphate, with copper and mercury as catalysts. Distillation is performed from the digestion flasks through air-cooled condensers, and the whole process may be completed in 1 hr.

F. E. DAY.

Chlorine Institute standard valves for chlorine containers under 15 tons capacity. R. T. BALDWIN (J. Amer. Water Works' Assoc., 1929, 21, 815—819).—A standard pattern valve adopted by American chlorine manufacturers is of the internal-thread type, repackable

under pressure, and has an outlet suitable for use with present standard pipe connexions. C. JEPSON.

Lixiviation. FONTAINE.—See I. **Oxidation of ammonia from coke-oven liquors.** PERLEY and WHITE.—See II. **Rhenania phosphate and ammonia-superphosphate as fertilisers.** DENSCHE and others. **Solubility of phosphorites.** ROSANOV.—See XVI.

PATENTS.

Manufacture of beryllium oxide. H. A. SLOMAN and A. C. VIVIAN (B.P. 312,007, 27.8.28).—A concentrated solution containing beryllium sulphate is saturated with beryllium hydroxide, and basic beryllium sulphate, $\text{BeSO}_4 \cdot 4-5\text{Be}(\text{OH})_2$, is precipitated by diluting the solution with water in the presence of ammonium sulphate as coagulant. Beryllium oxide free from sulphate is obtained by heating a mixture of the product with carbon at a temperature below that at which the oxide is rendered insoluble in acids, e.g., at about 700° , excess carbon subsequently being burnt away at a low temperature. L. A. COLES.

Production of sodium acetate. F. G. RAWLING (U.S.P. 1,695,742, 18.12.28. Appl., 11.5.27).—Sodium bicarbonate is added to the sulphite in the pulping of wood, the extract, which contains only one third of the lignin and no cellulose, is concentrated, sodium hydroxide and lime are added, and the mixture is heated at $200-280^\circ$. Sodium acetate is recovered from the filtrate. C. HOLLINS.

Production of ammonium sulphate by the treatment with gypsum of an ammoniacal solution used for the scrubbing of gases. UNION CHIM. BELGE, SOC. ANON. (B.P. 308,243 and 309,852, [A] 10.4., [B] 7.6.28. Belg., [A] 20.3., [B] 16.4.28. Addns. to B.P. 307,037; B., 1929, 472).—In modifications of the prior patent, (A) ammonium sulphate is precipitated by saturating the liquor with ammonia and removed, and excess of ammonia is expelled for re-use, before the liquor enters the scrubber, which is fitted with a cooler to facilitate absorption, and (B) sufficient ammonia is added to the liquor to effect almost complete precipitation of the ammonium sulphate; added water is removed as liquor instead of by evaporation, ammonia in the withdrawn liquor being recovered by heating it with milk of lime, and the liquor, after filtration to remove calcium carbonate is heated to expel residual carbon dioxide, which is returned to the scrubber. L. A. COLES.

Production of copper sulphate. M. SPEICHERT, Assr. to HÜTTENWERKE TEMPELHOF A. MEYER (U.S.P. 1,715,871, 4.6.29. Appl., 10.1.27. Ger., 13.1.26).—A copper-tin-lead-antimony alloy is melted with sulphur, the mixed sulphides so produced are roasted, the product is leached with sulphuric acid, and the liquor is freed from insoluble residue. F. G. CROSSE.

Manufacture of metallic [aluminium] chloride. R. DE M. TAVEAU and C. B. TYGERT, Assrs. to TEXAS Co. (U.S.P. 1,698,324, 8.1.29. Appl., 17.1.22).—Bauxite, or other aluminium ore, is dehydrated in a vertical dehydrating chamber, spaced with inclined baffles to agitate and distribute the falling bauxite and heated internally by gas burners near to the base. The de-

hydrated bauxite falls through a hopper into a chlorinating chamber, heated at $370-425^\circ$ externally and provided with movable horizontal baffles, in countercurrent with an ascending stream of phosgene introduced at the base of the chamber. The aluminium chloride passes from the upper end of the reaction chamber to a series of condensers, and the excess of phosgene is destroyed by leading it to the base of the dehydrator to assist the dehydration. R. BRIGHTMAN.

Manufacture of chromium compounds. I. G. FARBENIND. A.-G., and K. and K. S. CARPMAEL (B.P. 312,097, 20.12.27. Addn. to B.P. 259,447; B., 1926, 1013).—The coarse ore used as a filler in the process described in the prior patent is, after cleaning with sulphuric acid, repeatedly returned to the process until it contains nearly 70% of impurities, when it is ground, mixed with dolomite and sodium carbonate, and roasted to recover its chromium content. A. R. POWELL.

Alkali titanate and its manufacture. L. W. RYAN, Assr. to TITANIUM PIGMENT Co., INC. (U.S.P. 1,697,929, 8.1.29. Appl., 2.6.25).—Hydrated titanium oxygen compounds, e.g., metatitanic acid or basic titanic sulphate, are heated at $100-200^\circ$ with about 1.6-2 pts. of potassium hydroxide or carbonate, giving an alkali titanate, $d\ 2.3-2.9$, soluble in organic and dilute mineral acids. As example, potassium metatitanate thus obtained dissolved in oxalic acid at 80° affords titanium potassium oxalate.

R. BRIGHTMAN.

Recovery of titanium compounds. TITANIUM PIGMENT Co., INC., Asses. of W. F. WASHBURN (B.P. 288,569, 15.3.28. U.S., 12.4.27).—Finely-ground ilmenite is mixed with 72-92% sulphuric acid and steam is injected into the lower part of the reaction vessel until the temperature reaches 120° and the mass thickens; a small quantity of water is then forced into the lower part of this thick pulp, followed by vigorous streams of air to keep the mass in agitation and to render it porous, and finally the cooled solid mass is treated with just sufficient water to dissolve the iron and titanium sulphates. The clear solution is agitated with scrap iron or zinc to reduce ferric to ferrous sulphate, and the titanium is recovered by hydrolysis in the usual way. A. R. POWELL.

Catalyst. F. W. WEBER (U.S.P. 1,698,009, 8.1.29. Appl., 1.12.24).—Asbestos, amphibolite, or other siliceous material is digested with hydrochloric acid, and, after removal of the acid liquid, impregnated with excess of 10-20% sodium silicate solution to alkalinity. Excess of solution is removed, the mass dried, heated to incandescence, and, after washing out sodium chloride, impregnated with thorium nitrate or other rare-earth salt. On raising again to incandescence to convert, e.g., the thorium nitrate into the dioxide, a porous catalyst is obtained, resistant to heat and acids except sulphuric acid. The silica-thoria-asbestos catalyst is valuable in synthetic organic oxidations, e.g., vanillin, as a heat-reflecting illuminating material, a refining agent for crude or cracked oils, and for filtration of slimy gelatinous substances. R. BRIGHTMAN.

Separation of gases [hydrogen and carbon dioxide]. J. REILLY and E. W. BLAIR, Assrs. to

COMMERCIAL SOLVENTS CORP. (U.S.P. 1,706,707, 26.3.29. Appl., 8.4.25).—Fermentation gas etc. is washed under pressure with a solvent to remove the greater part of the carbon dioxide, and the process is repeated to remove the remaining carbon dioxide. The gases evolved on releasing the pressure from the second wash-liquor are returned to the process.

L. A. COLES.

Sulphur-burning apparatus. O. BUSE, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,714,657, 28.5.29. Appl., 2.11.25).—The apparatus consists of a gas conduit having within it an inclined corrugated plate, above which are means for delivery thereon of molten sulphur.

H. ROYAL-DAWSON.

[Furnace for] generating gaseous oxide of sulphur [for preventing or extinguishing fire or for disinfecting]. CLAYTON INSTALLATIONS, LTD., and W. A. MUIRHEAD (B.P. 312,746, 30.3.28).—Rapid generation of the gases is attained by distributing an inflammable liquid over the sulphur to ignite it and to heat up the furnace; by supplying a forced or an induced draught of air over the sulphur, the change over from one to the other draught being regulated automatically according to the temperature of combustion; by mixing absorbent non-inflammable material, e.g., asbestos in the form of mats, with the sulphur to act as wicks; and by external heating of the furnace, when required.

L. A. COLES.

Oxides of nitrogen from ammoniacal liquor (U.S.P. 1,713,045).—See II. Hypochlorites (B.P. 311,218 and 311,253).—See XI. White lead (B.P. 311,986).—See XIII. Vulcanisation of rubber (B.P. 290,602).—See XIV.

VIII.—GLASS; CERAMICS.

Vitreous state. M. O. SAMSOEN (Bull. Soc. d'Encour., 1929, 128, 185—204).—Qualitative dilatometric observations with a variety of vitreous bodies show that anomalous expansion is not due to the presence of silica, but is a general property of vitreous substances. For all such substances there exists, independently of the viscosity, a temperature, or a short temperature interval, at which a physical transition occurs, and is manifested by discontinuity in the temperature-expansion curves. It is suggested that there exists for a liquid a certain viscosity above which the molecular mobility vanishes. This conception is supported especially by experiments with the binary system $\text{SiO}_2\text{-Na}_2\text{O}$.

H. F. GILLBE.

Active silica. Soluble silica in clays. A. FIOLETOVA (Trans. State Exp. Inst. Silicate, Moscow, 1927, No. 21, 111—113).—Two samples of clay, dried at 100—110°, ground, and treated for 1 hr. with constant stirring with *N*-hydrochloric acid by Dementev's method, lost 0.05 and 0.09% of silica, respectively. After cooling for 2½ hrs., heating at 530°, and again cooling for 2½ hrs. the samples similarly lost 0.25 and 0.17% of silica. The result for extraction immediately after heating was 0.29 and 0.17%. Extraction after heating at 1300° gave 0.21 and 0% loss, respectively, of silica.

CHEMICAL ABSTRACTS.

Purification of clay by electrophoresis. A. S. TAL (Trans. State Exp. Inst. Silicate, Moscow, 1927, No. 21,

115—131).—An anode of lead containing 14% Sb and an ammoniacal electrolyte, with a peat extract depolariser, were used. When heated, the material gave a lighter product; the contraction for high temperatures was greater than that for 100°.

CHEMICAL ABSTRACTS.

Durability of arc furnaces. KOTHNY.—See XI.

PATENTS.

Lehrs. C. E. FRAZIER and J. SYLVESTER (B.P. 289,017, 16.2.28. U.S., 20.4.27).—The lehr consists of a double-walled sectional tunnel, with the hollow space between the walls at the front filled with insulating material. In the middle portion, sides and bottom only are so packed, and the outer shell has a refractory roof with hinged dampers. The side-wall chambers are without packing in the rear section and are adapted for air-cooling. A horizontal metal plate supports an endless, wire-woven belt throughout the length of the lehr. A metallic combustion chamber below this plate at the front of the lehr is connected by several horizontal metal flues to a chimney header, and this method of heating may be supplemented or substituted by electrical heating units. Damper grids are provided above the heating flues for temperature control. An entry door and a series of curtains prevent draughts within the lehr.

A. COUSEN.

Manufacture of plate glass and sheet glass for window panes from pure fused transparent silica. QUARTZ ET SILICE (B.P. 302,895, 23.11.28. Fr., 23.12.27).—Ingots of opaque fused silica are heated to 2000° and squeezed out into transparent sheets under high pressure, maintained until solidification occurs.

A. COUSEN.

Heat treatment [of ceramic ware]. Tunnel kiln construction. T. G. McDUGAL, Assr. to A.C. SPARK PLUG Co. (U.S.P. 1,713,851—2, 21.5.29. Appl., [A] 12.12.25, [B] 30.7.27).—In both instances a tunnel kiln is provided with a longitudinal slot through which projects the travelling support, the mass of which is small compared with that of the heating walls. Heating is by radiation from the walls of a muffle.

J. A. SUGDEN.

Manufacture of rubber-bonded abrasive articles. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 311,104, 15.5.28).—Abrasive particles are mixed with a rubber dispersion, compounded or otherwise, the latter being preferably in a somewhat viscous but stirrable condition, produced, for example, by the addition of glue or by concentration; the mixture is then coagulated, moulded, dried, and vulcanised. Alternatively, the thickening of the latex may be effected by coagulation into a curd-like condition.

D. F. TWISS.

IX.—BUILDING MATERIALS.

Hygrometer for use in timber-seasoning kilns. E. GRIFFITHS (Proc. Physical Soc., 1929, 41, 426—427).—The hygrometer is of the wet-and-dry-bulb type using mercury-in-steel thermometers. The bulbs and capillaries are enclosed in a steel tube which can be inserted through a 3-in. hole and explorations up to 12 ft. can

be made. One dial serves both thermometers, the two pointers being arranged so that one moves over the top face of the dial and the other is brought round from beneath on to the outside edge. An electrically-operated fan draws air past the two bulbs. The moistening of the covering over the wet bulb is effected by a reservoir and by connexion to an external water supply.

N. M. BLIGH.

Influence of the electric current on wood. B. F. SCHWARZ (Bull. Inst. Pin, 1928, 215—220; Chem. Zentr., 1928, ii, 2762).—An apparatus for the determination of the electrical resistance of wood is described, and the effect of the current is discussed.

A. A. ELDRIDGE.

Road tar. MALLISON.—See II.

PATENTS.

Ageing of calcined gypsum. S. G. McANALLY (U.S.P. 1,713,879, 21.5.29. Appl., 4.10.26).—The pulverised product is agitated, while to each ton of it are added 70 lb. of water.

F. G. CROSSE.

Slowly-setting hydraulic materials with high initial resistance. E. GOFFART (B.P. 294,111, 13.7.28. Belg., 16.7.27).—The setting of a highly aluminous cement (so quick-setting as to be unusable) is delayed by the addition of a small amount of a colloid such as casein, albumin, gum, gelatin, etc.

J. A. SUGDEN.

Treatment of concrete surfaces. G. B. ELLIS. From CHEM. FABR. GRÜNAU, LANDSHOFF & MEYER A.-G. (B.P. 312,216, 22.2.28).—The surface of the mould is treated with a solution of the products of the alkaline hydrolysis of albumins or proteins (*e.g.*, alkali salts of lysalbic and protalbic acids). This treatment delays the local setting of the binding agent, so that the surface layer of the material may be washed off when the mould is removed.

J. A. SUGDEN.

Manufacture of cellular building materials. L. DESMARQUEST (B.P. 312,764, 18.4.28).—A mixture of clay etc. previously baked at 200—1100°, cement, unbaked clay, and, *e.g.*, powdered aluminium, made to a paste with water, is dried in moulds rendered waterproof, *e.g.*, by greasing, and the moulded blocks are calcined at about 1300°.

L. A. COLES.

Treatment of wood. L. P. CURTIN, Assr. to WESTERN UNION TELEGRAPH Co. (U.S.P. 1,698,622, 8.1.29. Appl., 16.4.27).—Wood or other cellulose material is rendered resistant to fire or fungus or insect attack by impregnation with a solution of ammonia and arsenious acid, containing upwards of 5% As_2O_3 , which deposits arsenious oxide on exposure to air, or with an emulsion of such a solution and a liquid hydrocarbon.

R. BRIGHTMAN.

Waterproof compositions (B.P. 312,467).—See II.
Laminated products (B.P. 299,441).—See XV.

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

Single crystals of iron. H. GRIES and H. ESSER (Arch. Eisenhüttenw., 1928—9, 2, 749—761; Stahl u. Eisen, 1929, 49, 879—880).—To obtain large single crystals of iron, the metal must be as pure as possible, and in any case the carbon content should not exceed

0.02%. The necessary deformation required to produce rapid crystal growth increases rapidly with an increase in the size of the original crystal grains, but rapid growth is obtained by prolonged annealing at 880° of metal containing 100—140 grains/mm.² after rolling or extending in a tensile machine to produce a deformation of 3—3.25%. At 880° annealing for 48 hrs. usually suffices, but at slightly lower temperatures a much longer time is required to produce a single-crystal rod of the dimensions used in the usual tensile test. When the original deformation is produced in a tensile machine there is a tendency for coarse secondary crystals to form on the surface of the single crystal; these may be removed by etching with a mixture of 1 vol. of concentrated hydrochloric acid and 9 vols. of concentrated nitric acid. The presence of less than 0.05% O in the metal has little effect on the production of single crystals, whereas with more than 0.055% O no single crystals can be obtained; these results indicate that the solid solubility of oxygen in iron is of the order of 0.05%. In recrystallisation tests on rolled single crystals, new crystals began to form on the faces of the rhombic dodecahedron after 16%, on octahedron faces after 21%, and on cube faces after 24.3% deformation. The new crystals formed on the cube faces were of much greater size than those on the dodecahedron.

Influence of alloying elements on the polymorphism of iron. F. WEVER (Arch. Eisenhüttenw., 1928—9, 2, 739—748; Stahl u. Eisen, 1929, 49, 839—840).—The elements which form solid solutions with iron may be divided into four groups, viz.: (I) those which lower the A3 point and broaden the γ -field, *e.g.*, the elements of the eighth group of the periodic system and manganese, which immediately precedes iron; (II) those which give an enclosed γ -field in the equilibrium diagram, *e.g.*, beryllium, aluminium, silicon, phosphorus, titanium, vanadium, chromium, arsenic, niobium, molybdenum, tin, antimony, tantalum, and tungsten; (III) those that give an equilibrium diagram in which the transformation lines lead out from one another into regions of heterogeneous equilibrium, *e.g.*, carbon, nitrogen, copper, zinc, and gold; and (IV) those that narrow the γ -field, *e.g.*, boron, sulphur, zirconium, and cerium. It thus appears that elements isomorphous with γ -iron increase the stability of the γ -phase, and those isomorphous with α -iron tend to repress the $\alpha \rightarrow \gamma$ transformation. All the elements of groups I and III, *i.e.*, those that increase the γ -range, occur at the lowest points of the atomic radius—atomic number curve, whereas the elements of groups II and IV occur chiefly on the descending portions of the curve, and the elements that are insoluble in iron generally on the ascending portions and at the peaks.

A. R. POWELL.

Specific volume of white pig iron. L. ZIMMERMANN and H. ESSER (Arch. Eisenhüttenw., 1928—9, 2, 867—870; Stahl u. Eisen, 1929, 49, 912—913).—The sp. vol. of white pig iron containing 3.5—3.9% C, 0.13—0.67% Si, and 0.06—0.1% Mn has been determined at temperatures up to 1300° by heating the metal, *in vacuo*, in a porcelain crucible. The mean value of the volume change during fusion is about 1.33—1.41% of the maximum value of the sp. vol. in the solid state.

A. R. POWELL

Adherence of thin sheets [of iron during hot-rolling]. W. TITZE (*Stahl u. Eisen*, 1929, 49, 897—903).—For the manufacture of thin iron sheets for galvanising or tinning the carbon content should preferably be between 0.08 and 0.16%, and the metal must be thoroughly deoxidised to prevent the sheets sticking together when doubled over in the last stages of hot-rolling. The most satisfactory tests for these conditions are the bending test on the quenched material and the flattening test on the white hot metal; in the latter test, the usual dip sample from the furnace is flattened under the hammer at a welding heat and then examined for unsoundness. For the production of sheets thinner than 0.55 mm., the addition of a small amount of copper or, better, ferrocobalt-titanium to the metal in the ladle is recommended. Deoxidation with titanium yields a better quality sheet for deep drawing, and to a great extent prevents segregation of sulphur and phosphorus; nevertheless, the upper portion of the ingot should always be cut off and used where a lower grade of metal is required. A. R. POWELL.

Third report on heterogeneity of steel ingots. I. Introduction. II. Liquidus and solidus ranges of some commercial steels. III. Solubility of iron and manganese sulphides in steel. J. H. ANDREW and D. BINNIE. IV. Interim report on the density of molten steel. C. H. DESCH and B. S. SMITH. V. Effect of latent heat on solidification of steel ingots. N. M. H. LIGHTFOOT (*Iron and Steel Inst.*, May, 1929. Advance copy. 71 pp.; cf. B., 1928, 409).—II. The phase changes in straight carbon, nickel, nickel-chromium, and nickel-chromium-molybdenum steels were compared with those given by Arco iron and four crucible steels by a thermal method. The liquidus and solidus curves for the commercial steels conform with respect to carbon content and temperature with the iron-carbon diagram, provided the manganese content is below 0.45%. The liquidus for nickel and nickel-chromium steels lies below, whilst the solidus practically coincides with, that of plain carbon steels. A method is given for calculating the freezing and melting ranges of low-nickel and low-chromium steels. The addition of molybdenum to nickel-chromium steels depresses the solidus by a proportional amount; the liquidus is unaffected. It is suggested that the decreased tendency to segregate shown by nickel and nickel-chromium steels is due to the narrower range between the liquidus and solidus and to the obliteration of the peritectic transformation. The addition of molybdenum to nickel-chromium steels widens the liquidus-solidus range on account of the segregation of molybdenum carbide. It is inferred that metals which form a solid solution with the iron phase depress the liquidus only, whilst those which form a carbide depress the solidus also. The peritectic reaction in carbon steels is discussed and its importance emphasised, and micrographs are shown of various sections from a specimen differentially heated and melted at one end. The liquidus and solidus curves derived from carbon steels run almost parallel with those of Carpenter and Keeling.

III. To determine whether the sulphides were soluble at all in solid steel the effect of these compounds on the magnitude and temperature of the Ar₃ change in iron

was determined. The results show definitely that sulphur may be retained in solution in an iron-sulphur alloy by rapidly chilling the melt, and that deposition of iron sulphide occurs on reheating the alloy to a temperature above 900°. The results with manganese sulphide were indefinite, but it appears probable that this compound is only very slightly, if at all, soluble in solid iron.

IV. The Archimedean principle was adopted. Preliminary results are given which are lower than those given by Berlin (B., 1927, 487), but in good agreement with those published by Benedicks, Berlin, and Phragmén (*Iron Steel Inst.*, Carnegie Schol. Mem., 1924, 13, 129).

V. Mathematical.

M. E. NOTTAGE.

Influence of nitrogen on special steels and experiments on case-hardening with nitrogen. S. SATOH (*Rev. Mét.*, 1929, 26, 248—258).—The increase in surface hardness due to absorption of nitrogen on heating in ammonia at 560—580° steels containing aluminium, titanium, manganese, zirconium, molybdenum, tungsten, or uranium, with and without chromium, has been determined. In all cases the added element produced a considerable increase in hardness, the maximum effects being obtained with aluminium and titanium. Boron, magnesium, copper, and cerium have only a relatively small action in nitrogen case-hardening. The use of nickel coatings on arc-welding electrodes has practically no effect in preventing the absorption of atmospheric nitrogen by the weld, although it tends to retard the separation of the characteristic nitride needles owing to their greater solubility in the nickel-iron solid solution.

A. R. POWELL.

Influence of the method of manufacture and the annealing atmosphere on the surface structure of tool steels. E. ZINGG, P. OBERHOFFER, and E. PIVOVARSKY (*Stahl u. Eisen*, 1929, 49, 721—725; 762—768).—Cementation of steel at 980—1000° in wood charcoal converts the surface austenite into free cementite which with plain carbon steels decomposes on prolonged annealing and slow cooling into temper carbon and γ -solid solution, the crystals of which are totally enclosed in a film of carbon so that further formation of austenite from gaseous carburising agents is prevented. The presence of 0.5% Cr, W, or V in the steel prevents decomposition of the cementite and carburising can continue up to 3% C. Prolonged annealing of high-carbon steels and high-speed tool steels without decarburisation or further cementation at temperatures up to 1300° may be effected in an atmosphere of 2 vols. of carbon monoxide and 1 vol. of hydrogen such as is produced by the incomplete combustion of acetylene with oxygen. This gas mixture is also suitable for salt-bath annealing as it prevents oxidation of the steel and also retards considerably the decarburising action of the fused salts; hence, hardened tool steels may be case-hardened at 580—600° in salt baths under an atmosphere of this gas mixture without surface etching or internal softening taking place. Diffusion, carburisation, and decarburisation experiments on a wide variety of steels indicate that the solubility of carbon in α -iron decreases from 0.035% at 720° to 0.006% at the ordinary temperature, and that at just below the A₁ point the rate of diffusion of carbon through α -iron is relatively great.

This rate is not affected by 0.5% Cr, 0.85% W, or 0.5% V alone, but when two or more of these elements are present together diffusion is much slower. The presence of impurities along the grain boundaries also retards diffusion, so that, by observing the behaviour of a steel during annealing above the A3 point and during subsequent decarburisation in moist hydrogen in the temperature zone between the A₁ and A₁ points, important conclusions as to its freedom from impurities which deleteriously affect its mechanical properties may be drawn. A. R. POWELL.

Prolonged bending tests with steels. E. HOUDEMONT and R. MAILÄNDER (Stahl u. Eisen, 1929, 49, 833—839).—The fatigue strength of steel, S , under alternate bending stress may be expressed by the equation $S = 0.25(\sigma_S + \sigma_B) + 5$, where σ_S is the yield point and σ_B the ultimate strength. The influence of the composition, mechanical and heat treatment, and grain size of numerous steels on the ratio S/σ_B and $S/(\sigma_B + \sigma_S)$ have been investigated, and the results are recorded in a series of tables and graphs. A. R. POWELL.

Tensile tests [of steels] at elevated temperatures. R. GUILLET, J. GALIBOURG, and M. SAMSOEN (Compt. rend., 1929, 188, 1205—1208).—A modified Martens elasticimeter is described by which the extension of steel at 450° under the conditions of the usual tensile test may be measured. The results obtained for four samples of mild steel are tabulated; for plain carbon steels, after forging, the first elastic limit increases with the carbon content and is also increased by the presence of excess of silicon over the normal. A. R. POWELL.

Properties of manganese in the basic open-hearth process. I. KOTAIRA and M. MAEDA (J. Study Ferrous Met., 1928, 103, 110—133).—The effect of ferrous and manganous oxides on the m.p. and fluidity of the slag has been studied. Increase in the concentration of ferrous oxide causes decrease in the concentration of manganese in the steel. With increase of lime, the liberation of manganous oxide is favoured, and hence the amount of manganese in the steel is increased. Desulphurisation, and the effect of manganese on dephosphorisation, are discussed. A. R. POWELL.

CHEMICAL ABSTRACTS.

Determination of molybdenum in steel. W. F. MURRAY (Chemist-Analyst, 1929, 18, 10).—The steel (5 g.) is dissolved in *N*-sulphuric acid (200 c.c.), ammonium persulphate (3—4 g.) is added, and the excess is removed by boiling. The solution, after dilution to 400 c.c. and saturation with hydrogen sulphide at 80° for 45 min., is filtered, the residue being washed with a dilute solution of hydrogen sulphide and ignited, the resulting oxide being dissolved in 20% sodium hydroxide (10 c.c.). The solution is boiled, filtered, diluted to 400 c.c., and slightly acidified with hydrochloric acid. After boiling (5 min.), 5% lead acetate solution (15 c.c.), 25% ammonium acetate solution (25 c.c.), and acetic acid (5 c.c.) are added with stirring. After being kept near the b.p. for 20 min., the solution is filtered and the precipitate is washed with hot water, ignited, and weighed as PbMoO₄. Tungsten, if present, is first determined in the usual way, the filtrate being evaporated to fuming, diluted, boiled, and treated with persulphate.

CHEMICAL ABSTRACTS.

Determination of vanadium in steel. J. KASSLER (Z. anal. Chem., 1929, 77, 290—298).—The steel turnings (3.75 g.) are dissolved by boiling with 40 c.c. of 1:5 sulphuric acid whereby the greater part of the vanadium remains in the carbide residue. The dissolved vanadium is recovered by diluting the solution with 100 c.c. of cold water and adding a suspension of zinc oxide in water until a little oxide remains undissolved. The solution is filtered, the residue washed twice with cold water and dissolved in hydrochloric and nitric acids, and the tungstic acid collected and washed. The filtrate is neutralised with sodium hydroxide, boiled with hydrogen peroxide, and poured in a thin stream (after addition of 1 g. of ferric chloride, if chromium is present) into 130 c.c. of hot 30% sodium hydroxide solution. The solution is boiled, cooled, diluted to 500 c.c., and 400 c.c. are filtered through a dry paper, acidified with hydrochloric acid, and treated with 20 g. of ammonium chloride and 25 c.c. of ammonia; the vanadium is separated from molybdenum by precipitation with a solution of 10 g. of manganese chloride and 5 g. of ammonium chloride in 50 c.c. of water. The precipitate is collected, washed, dissolved in sulphuric acid, and, after reduction with sulphur dioxide, the vanadium is titrated with permanganate. The small quantity of vanadium retained by the tungstic acid is determined colorimetrically with hydrogen peroxide after dissolving the precipitate in sodium hydroxide and acidifying with sulphuric acid. A. R. POWELL.

Separation of aluminium as hydroxide from steels. W. J. SHANEMAN and J. J. GALLAGHER (Chemist-Analyst, 1929, 18, 6).—The solution of steel in hydrochloric acid, oxidised with nitric acid, is evaporated to a syrup and added dropwise to an excess of boiling 20% sodium hydroxide solution. After boiling, the cooled solution is diluted to a definite volume, of which half is treated with ammonium carbonate to precipitate the aluminium. A. R. POWELL.

CHEMICAL ABSTRACTS.

Determination of sulphur in plain and alloy steels, pig iron, ferro-alloys, etc. K. SWOBODA (Z. anal. Chem., 1929, 77, 269—277).—The metal (1 g.) in the form of turnings or powder is heated at 1150—1200° in a stream of oxygen for 3—6 min. and the gases are passed through silver nitrate solution whereby nitric acid is liberated and silver sulphite precipitated. Passage of the oxygen is continued for 7 min. after combustion is complete to remove carbon dioxide from the absorption flask, and the solution is titrated with 0.005*N*-sodium hydroxide using methyl-red as indicator. For the analysis of ferrochrome, high-speed steels, and high-chromium steels, 0.5 g. is used and the combustion effected at 1300°; to prevent sintering, some crushed electrolytic iron is mixed with the sample. A. R. POWELL.

Crystalline changes in copper due to annealing. F. C. HOWARD and E. T. DUNN (Ind. Eng. Chem., 1929, 21, 550—553).—Photomicrographs of sections of cold-drawn copper rod have been taken, using a strong etching agent and relatively high magnification after the metal had been annealed at 700° for periods varying

from 3 to 40 hrs. The constantly recurring regularity and uniformity of the lamination in the etched crystals may be due to some physical property of the substance.

H. INGLESON.

Precipitation of copper from mine waters at Britannia Mines, B.C. F. EBBUTT and W. E. SELNES (Trans. Canad. Inst. Min. Met., 1929, 31, 290—309).—Water from the Britannia Mines contains from 0.6 to 1.8 g./litre of copper as sulphate derived from the oxidation of pyrites and bornite in the crushed upper zones of the mine. This copper is recovered by running the water through an adit into settling tanks to remove silt, thence through a number of launders provided with perforated false bottoms on which is placed scrap tinplate to precipitate the copper, which falls as a sludge through the perforations. To ensure efficient contact and to prevent accumulation of copper on the scrap, air is blown through the water from below the false bottom. The launders are used in series of two, in the first of which 66—75% of the copper is recovered and in the second 25—17%, giving a total recovery of just over 90%. The sludge from the first launder averages 65% Cu and that from the second 45% Cu; both sludges contain about 30% of moisture. The consumption of iron is about twice the theoretical owing to the presence of free acid and ferric sulphate in the water. The cost of recovering the copper is about 7.2 cents/lb.

A. R. POWELL.

Effect of mixing small quantities of cobalt with brass. D. IITSUKA (Mem. Coll. Sci. Kyoto, 1929, 12A, 179—198).—The ternary equilibrium diagram for the system copper—zinc—cobalt has been established in the region of copper 50—100%, zinc 0—50%, and cobalt 0—10%. Mechanical and corrosion tests were carried out on a number of alloys. β -Brass is improved in all its mechanical properties by addition of cobalt; α -brass is improved in strength but not in elongation.

C. W. GIBBY.

Influence of grain size on the corrodibility of brass and bronze. W. KÖHLER (Korrosion u. Metallschutz, 1928, 4, 227—230; Chem. Zentr., 1928, ii, 2748).—The influence of heating is examined.

A. A. ELDRIDGE.

Analysis of speculum metal. L. SCHWEITZER (Chem.-Ztg., 1929, 53, 457—459).—For the determination of copper and lead 0.5 g. of the powdered alloy is heated on the water-bath with 15 c.c. of a mixture of 1 vol. of bromine and 9 vols. of hydrobromic acid, d 1.38. The solution is evaporated to dryness and the residue again evaporated with 10 c.c. of the same mixture, whereby arsenic, antimony, and tin are volatilised. The residual bromides are boiled with 40 c.c. of 1:1 nitric acid to expel bromine, and the solution is diluted and electrolysed for copper and lead dioxide. Tin may be determined in a second 0.5 g. sample, if less than 12% Cu and 1% As are present, by direct dissolution in bromine and hydrochloric acid, reduction with iron, filtration to remove copper etc., and titration with iodine. An alternative and generally applicable procedure comprises dissolution of the alloy in nitric acid, collection of the metastannic acid, fusion with sodium peroxide, dissolution of the mass in hydrochloric acid, reduction of the solution with iron, and titration of the tin with iodine.

Antimony is determined in alloys containing less than 0.8% (As + Fe) and 15% Cu by titration with permanganate following dissolution of the alloy in sulphuric acid; in other cases the antimony must be isolated as the trisulphide in the usual way, and this is dissolved in sulphuric acid for permanganate titration. Arsenic is determined by distillation as bromide, sulphur by treatment of the alloy with concentrated hydrochloric acid and collection of the hydrogen sulphide in cadmium acetate solution, and nickel, iron, and zinc by the usual methods after separation of the hydrogen sulphide group.

A. R. POWELL.

Flotation practice at the Sullivan mill. C. T. OUGHTRED (Trans. Canad. Inst. Min. Met., 1929, 31, 310—322).—The ore consists of 13.6% of galena, 14.4% of zinc blende, 1% of iron pyrites, 61% of pyrrhotite, and 9.8% of rock matter, all in a very finely-disseminated form, so that it is necessary to crush the ore to 88% through 200-mesh before treatment in the flotation plant. The ore pulp from the grinding circuit containing 25% of solids is fed to lead rougher cells where the galena is floated using 0.85 lb./ton of a mixture of 60% of water-gas tar, 20% of coal-tar creosote, and 20% of cresylic acid as flotation agent; flotation of other minerals is prevented by addition of 3 lb. of sodium carbonate and 0.09 lb. of sodium cyanide per ton. The tailings from the lead cells pass to the zinc rougher cells where the blende is floated at 30° after adding 0.65 lb. of copper sulphate and 0.09 lb./ton of sodium dichromate together with 0.4 lb./ton of the above oil mixture. Cleaning of both lead and zinc concentrates is effected in separate series of clean-up cells. Complete flow-sheets and analyses of the various products obtained are included.

A. R. POWELL.

Development of the chlorine process of extraction of platinum metals from ores. R. A. COOPER and F. W. WATSON (J. Chem. Met. Min. Soc. S. Afr., 1929, 29, 220—228).—Froth flotation of South African norite ores effects an 87% recovery of the platinum metals in a sulphide concentrate weighing about 5% of the ore and assaying 7.5—10 oz./ton. After roasting to remove sulphur the concentrates are mixed with 15—20% of sodium chloride and heated at 500—600° in an atmosphere of chlorine for 4 hrs. The best results are obtained with a layer of ore 4—5 in. thick and without rabbling; the chlorine consumption is about 120—150 lb./ton. The product is only slightly caked, and is readily leached to give a solution containing over 90% of the platinum and 80% of the copper and nickel. The gold in the residue together with some of the remaining platinum and palladium may be recovered by cyanide leaching. The chloride solution is agitated with powdered limestone (20 lb./ton of solution) to precipitate copper carbonate containing about 2 oz./ton of platinum metals which may be recovered by smelting the precipitate to copper and electrolysing. The filtrate is agitated with 1 lb./ton of zinc dust and a little hydrochloric acid whereby a high-grade precipitate of platinum metals is obtained. The filtrate is treated with bleaching powder to recover nickel.

A. R. POWELL.

Röntgenographic study of the improvement of aluminium alloys. G. SHINODA (J. Jap. Min. Met.,

1928, 44, 514—562).—X-Ray studies were made of aluminium-silicon alloys to which was added potassium hydrogen fluoride, sodium fluoride, sodium peroxide, sodium carbonate, potassium carbonate, borax, or a calcium compound. The effect of the addition of sodium or calcium to aluminium-nickel alloys was also examined.

CHEMICAL ABSTRACTS.

Determination of the tin deposit on tin plates. F. PETER (Chem.-Ztg., 1929, 53, 438—439).—A piece of metal 100×200 mm. is cleaned in alcohol, dried, weighed, rolled into a coil, and dropped into boiling hydrochloric acid, *d* 1.08. When the tin coating has disappeared and the iron begins to be rapidly attacked, the coil is removed, washed first with warm water, then with alcohol, dried, and weighed. The loss in weight represents all the tin and some iron; the amount of the latter is determined in an aliquot part of the solution by oxidising with potassium chlorate, reducing with stannous chloride, and titrating with permanganate by the Zimmermann-Reinhardt method. The iron so found is increased by 0.4% to compensate for the impurities in the metal and the tin found by subtracting this weight from the loss in weight caused by the acid treatment. The results are correct to $\pm 1\%$.

A. R. POWELL.

Determination of sulphur in metallic antimony. C. G. SNYDER (Chemist-Analyst, 1929, 18, 6).—The sample (2—3 g.) is fused with sodium peroxide in a nickel crucible; the mass is extracted with water, and the solution heated until clear with concentrated hydrochloric acid (100 c.c.) and tartaric acid (10 g.). After dilution with hot water (400 c.c.), the solution is treated with barium chloride. CHEMICAL ABSTRACTS.

Rapid detection of tungsten in ores. A. PETROVSKY (Z. anal. Chem., 1929, 77, 268—269).—The finely-ground ore (0.1—0.2 g.) is boiled with 1—2 c.c. of concentrated hydrochloric acid and a small piece of lead foil for 1—2 min. A blue solution which on dilution with water gives a blue, flocculent precipitate indicates the presence of tungsten in excess of 0.5%. Niobium gives a similar blue solution, but the colour disappears on dilution.

A. R. POWELL.

Tensile strength and fracture of rolled strips of molybdenum. T. FUJIWARA (Mem. Coll. Sci. Kyoto, 1929, 12A, 165—178).—The tensile strengths of rolled strips of molybdenum have been investigated with reference to the direction of rolling. The tensile strength measured in the direction parallel to that of rolling is about 20% greater than when tension is applied at an angle of 45° or 90° to it. X-Ray examination of the fractures shows that they take place at the (211) and (110) planes of the cubic crystals of molybdenum.

C. W. GIBBY.

Hardness and abrasion testing of metals. G. A. HANKINS (Inst. Mech. Eng., June 30, 1929. Advance copy. 57 pp.).—A synopsis of the present state of knowledge of the subject, with special reference to the work done during 1921—27. Suggestions for further work are included.

Corrosion and metal protection in gasworks' practice. MAAS.—See II. Developers and metal tanks. ROSS and CRABTREE.—See XXI.

PATENTS.

Cupola furnace. J. ROBINSON (B.P. 312,401, 27.2.28).—A cupola furnace of the ordinary cylindrical type is provided with an offset chimney in its upper part and a central charging opening at the top of the shaft, which is closed with a plug which acts as a counterbalance to a charging bucket or elevator, and is so arranged that it is automatically removed as the bucket moves over the charging opening and replaced as the empty bucket swings away.

A. R. POWELL.

Furnace for melting metals. A. W. MACHLET (U.S.P. 1,713,543, 21.5.29. Appl., 15.4.26).—The furnace comprises a melting chamber in which a crucible is supported so that it almost closes the upper aperture which leads into a preheating stack in which is a concentrically arranged charging column for the crucible terminating in a feed hopper.

A. R. POWELL.

Condenser for zinc vapours from electric furnaces. F. THARALDSEN (U.S.P. 1,715,960, 4.6.29. Appl., 26.6.23. Norw., 3.11.21).—A block of deep, relatively narrow, separate, parallel channels is used, which are spaced apart and inclined downwards from the furnace chamber to a common container for collecting the products of condensation.

F. G. CROSSE.

Briquetting or consolidation of sponge-iron granules. F. L. DUFFIELD (B.P. 312,834, 25.6.28).—To obtain iron from ores containing large amounts of sulphur and phosphorus compounds, the ore, mixed with carbonaceous material and with or without lime, is passed through a reducing chamber whereby sponge-iron granules are obtained containing sulphur and phosphorus as impurities; the granules are then mixed with lime or limestone, pressed into briquettes, and melted, whereby the impurities are removed.

M. E. NOTTAGE.

Production of wrought iron. A. J. BRIGGS, ASST. to ONONDAGA STEEL CO., INC. (U.S.P. 1,713,887, 21.5.29. Appl., 8.12.26).—Discrete particles of iron are mixed with slag-forming materials in the proportion to produce the desired fibrous structure of the iron, and the mixture is heated to a welding temperature in a rotary electric furnace from which air is excluded.

F. G. CROSSE.

Solder for cast iron and other metals for surfacing them. J. BUTLER, ASST. to I. P. WALTON (U.S.P. 1,697,714, 1.1.29. Appl., 30.12.27).—A melt of 3 pts. of ground steel, 13.5 pts. of lead, and 3.5 pts. of tin is used as a solder for cast iron in conjunction with a flux consisting of 1 pt. of steel, 1 pt. of emery, 1.5 pts. of Tobin bronze, 1 pt. of plaster of Paris, 1 pt. of borax, 0.5 pt. of Epsom salts, and 0.5 pt. of salt.

R. BRIGHTMAN.

Treatment of silicon steel. A. F. MURPHY and W. JONES, ASSTS. to AMER. ROLLING MILL CO. (U.S.P. 1,714,038, 21.5.29. Appl., 15.4.27).—In the preparation of silicon-steel sheets for electrical purposes, the sheets are pickled, washed to remove acid, annealed, cold-rolled, and finally re-annealed.

F. G. CROSSE.

Ferrous alloy. L. W. MALLASEE, ASST. to W. H. MALLASEE (U.S.P. 1,714,177, 21.5.29. Appl., 2.9.27).—Tin and aluminium are added to the molten ferrous metal at a pouring temperature.

F. G. CROSSE.

Manufacture of nickel-iron alloys. GEN. ELECTRIC CO., LTD., and R. W. W. SANDERSON (B.P. 312,411, 29.3.28).—Nickel and iron in a friable condition are simultaneously deposited electrolytically from a solution containing compounds of the two metals, a high current density being employed. Thus the metals may be deposited from a solution containing, per litre, 120 g. of nickel sulphate, 31 g. of ferrous sulphate, 13 g. of ammonium chloride, 6 g. of boric acid, 5 g. of quinol, and 2 g. of ammonium citrate, using two anodes, respectively, of iron and nickel, and a current density of 40 amp. per sq. in. of cathode. The magnetic properties of the deposited material, consisting of a mixture of nickel, iron, and nickel-iron alloy, are developed by heat treatment of the material in an inert gas.

J. S. G. THOMAS.

Alloy. G. G. MARSHALL and H. S. BOOTH (U.S.P. 1,713,766, 21.5.29. Appl., 27.2.23).—The alloy contains 2–12% Be, 46–90% Fe, and 4–42% Cr.

H. ROYAL-DAWSON.

Vanadium alloy. B. D. SAKLATWALLA, Assr. to VANADIUM CORP. OF AMERICA (U.S.P. 1,715,867, 4.6.29. Appl., 2.2.28).—A ferrovanadium alloy containing 85–95% V is claimed.

H. ROYAL-DAWSON.

Alloys and their manufacture. P. C. CHESTERFIELD, Assr. to CHESTERFIELD METAL CO. (U.S.P. 1,698,934 and 1,698,936, 15.1.29. Appl., 1.12.24).—(A) Alloys for high-speed cutting tools, which consist essentially of carbide crystals in a matrix of nickel and cobalt, are obtained by melting 10–45% of cobalt, 7–30% of nickel, and 0.5–3.5% of carbon, preferably added as carbide, with 25–35% of chromium and 15–35% of tungsten, the total amount of nickel and cobalt being between 30 and 70%. (B) The tungsten is replaced by 4–30% of vanadium or other metal, m.p. 1000–2100°, e.g., titanium or niobium, which forms hard carbides; the alloy may contain 15–55% Co, 7–30% Ni, 20–45% Cr, 10–25% V, and 0.5–3.5% C.

R. BRIGHTMAN.

High-speed alloy. P. C. CHESTERFIELD, Assr. to CHESTERFIELD METAL CO. (U.S.P. 1,698,935, 15.1.29. Appl., 1.12.24).—The alloy comprises 15–50% Co, 7–30% Ni, 20–45% Cr, 7–35% Mo, and a small amount of carbon.

F. G. CROSSE.

Metallic alloy. H. L. COLES and J. G. DONALDSON, Assrs. to GUARDIAN METALS CO. (U.S.P. 1,698,212, 8.1.29. Appl., 5.8.24).—Heat- and oxidation-resistant alloy containing upwards of 60% W or a metal of the tungsten group, 10–25% Ni, and 2–5% C, obtained, e.g., by fluxing with calcium sulphate and fluoride a mixture of nickel sulphide or molybdenite and tungsten concentrates, is encased in copper or other metal of higher heat conductivity.

R. BRIGHTMAN.

Manufacture of alloys. W. M. GROSVENOR and V. P. GERSHON (U.S.P. 1,716,050, 4.6.29. Appl., 3.2.25).—The alloy contains about 20% of zinc, 70% or more of nickel, and up to 9% of copper.

F. G. CROSSE.

Metal [copper alloy]. F. J. READ, Assr. to CALAVERAS IRON & STEEL CO., and S. H. DEMAREST (U.S.P. 1,714,729, 28.5.29. Appl., 28.9.27).—The alloy contains 0.1–1% Ce and 5–10% Al, the remainder being chiefly copper.

F. G. CROSSE.

[Anti-friction metal] alloy. D. C. LEE (U.S.P. 1,714,679, 28.5.29. Appl., 17.11.26).—An alloy (4:1) of brass and Babbitt metal is claimed.

H. ROYAL-DAWSON.

Soldering of aluminium. P. ODAM (B.P. 300,969, 21.11.28. Fr., 21.11.27).—The solder consists of an aluminium alloy containing 5–12% Si, and is mechanically treated in the hot condition by working it into wires, rods, or thin plates; subsequent separation of silicon when melting the alloy is thereby avoided.

M. E. NOTTAGE.

Treatment of tin-bearing materials. D., M., S. R., and S. GUGGENHEIM, J. K. MACGOWAN, and E. A. C. SMITH (GUGGENHEIM BROS.) (B.P. 306,108, 30.5.28. U.S., 16.2.28).—Impure tin concentrates are ground to pass 200-mesh and, with or without a preliminary roast, are agitated with sulphuric acid, *d* 1.45, at 200° to remove iron, copper, etc. The residue is washed and digested with 20% sodium chloride solution to remove lead and silver, then with an acid sodium chloride solution to dissolve bismuth and any remaining arsenic and antimony. Tungstic acid is removed by leaching with 5% sodium hydroxide solution, and the purified cassiterite is smelted in an electric furnace with coal, sodium carbonate, and lime to produce metallic tin and a slag having a composition corresponding with that of the eutectic of Na₂SiO₃ and CaSiO₃. Owing to its low iron content, this slag is relatively free from tin, and the metal contains very little hardhead. The latter is removed by centrifuging the tin at 300°, and, after roasting at 600–700° until completely oxidised, is returned to the leaching circuit with further quantities of concentrate.

A. R. POWELL.

Flotation agents. E. C. R. MARKS. From AMER. CYANAMID CO. (B.P. 310,186, 26.4.28).—The dithiophosphates obtainable by the action of phosphorus pentasulphide on alcohols or phenols, e.g., on *isopropyl* alcohol or crude cresol, are used as flotation agents especially for extraction of sulphide ores.

C. HOLLINS.

Controlling the action of pickling acids on metals. J. H. GRAVELL and A. DOUTY (B.P. 287,912, 28.3.28. U.S., 29.3.27).—A substance, e.g., a thiocyanate of an alkali metal or ammonium, which will combine with the nascent hydrogen, is added to the bath so that hydrocyanic acid is liberated within the bath. A foam-producing material, e.g., cellulose pulp waste liquor, evaporated or otherwise, is also added to prevent the liberation of acid spray into the air.

M. E. NOTTAGE.

[Metallically] coating metal. F. SMITH, Assr. to AMER. MACHINE & FOUNDRY CO. (U.S.P. 1,710,747, 30.4.29. Appl., 17.12.25).—Vaporised metal is ionised and electrically deposited and condensed on the metal to be coated.

J. S. G. THOMAS.

Electrolytic production of metals and apparatus therefor. I. G. FARBENIND, A.-G. (B.P. 285,824, 21.2.28. Ger., 21.2.27).—Electrolyte is caused to flow through the bath from below upwards, and the outflow is distributed along opposite sides of the cell so that movement of the electrolyte is substantially uniform with respect to all the electrodes.

J. S. G. THOMAS.

Electrodeposition of metals. ELECTRO BLEACH & BY-PRODUCTS, LTD., J. HOLLINS, and D. JEPSON (B.P. 312,395 and 312,403, 25.2.28).—(A) Anodic material is supported at the base of the anode compartment by glass or similar balls which are unaffected by the electrolysis, and provide spaces through which the sludge produced percolates. The balls are supported on a grid around which air under pressure is injected. (B) Anodic plates or strips are enclosed within graphite-impregnated sleeves or covers protecting them from the abrasive action of anodic material.

J. S. G. THOMAS.

Purification of magnesium and its alloys. G. MICHEL, Assr. to H. O. BERG (U.S.P. 1,698,647, 8.1.29. Appl., 5.2.25. Fr., 4.10.24).—See B.P. 261,528; B., 1927, 80.

Melting furnace (U.S.P. 1,715,678). **Magnetic material** (U.S.P. 1,715,541 and 1,715,543).—See XI.

XI.—ELECTROTECHNICS.

Durability of the structure of direct-arc furnaces under different working conditions. E. KOTHNY (Feuerfest, 1929, 5, 1—3, 75—80).—Data were collected by means of a questionnaire on the efficiency and durability of one acid and twenty-three basic arc furnaces operating under varying industrial conditions. In all cases the furnace roofs were constructed of silica bricks, the normal thickness being 250 mm. for furnaces up to 7.5 tons capacity. The method of setting up (wet or dry) had no effect on the durability of the roof. In most cases the thickness of the insulating layer in the hearth walls ranged from 100 to 125 mm.; the thickness of the whole wall varied considerably. Basic, tamped walls were usually more durable than walls made of bricks. Pauses during the working day had an unfavourable effect on the hearth walls. Insulation of the furnace hearth varied from 50 to 240 mm. in thickness. In nearly all basic furnaces the hearth proper was made of magnesite bricks covered with a magnesite or dolomite tamping mixture. No direct relation was found between the durability of the hearth and its total thickness or the proportion of brick and tamped material. The data collected indicated that, in general, the durability of the roof, hearth wall, and hearth of basic arc furnaces depends mainly on the quality of the refractory material used and the care and attention applied in the construction and maintenance of the furnace.

F. SALT.

Electrical heating by the container-resistance method. R. A. CARLETON (Ind. Eng. Chem., 1929, 21, 525—529).—The method of heating in which an electric current of high amperage at low voltage is passed through the walls of the vessel to be heated can be applied to large numbers of processes, *e.g.*, heating of drums, rotary roasters, ovens, pipes, and tubing used in the transfer of viscous liquids. It may be applied in nearly all processes in which the container used is a good electrical conductor. A scheme is suggested for using the method in the varnish industry. The costs and efficiency of conversion of the heat energy derived from coke, oil, and gas are compared with those of the container-resistance method of heating.

The chief advantages of the method are great flexibility automatic temperature control, even heating, and its ready application to vessels of different shapes.

H. INGLESON.

Cathode rays and cable deterioration. SCHOEFFLE and CONNELL.—See II. **Light sources for weathering systems.** SCHMUTZ and GAMBLE.—See XIII.

PATENTS.

Electric furnace. F. A. J. FITZGERALD, Assr. to HARPER ELECTRIC FURNACE CORP. (U.S.P. 1,714,081, 21.5.29. Appl., 13.5.27).—A resistor chamber containing an inert atmosphere, and a heat-treating chamber containing a gas having a pyrochemical and physical action on substances treated, are separated by a septum which is unaffected during operation of the furnace.

J. S. G. THOMAS.

[Electrical] melting furnace. S. SCHNEIDER, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,715,678, 4.6.29. Appl., 13.9.26. Ger., 14.12.25).—Gas under pressure is supplied to an electrically-heated melting pot having a removable cover in which is mounted the upper end of a discharge conduit extending through the cover from near the bottom of the pot. A recess in a second cover member is adapted to receive the first cover, and a discharge passage in this member receives the upper end of the discharge conduit.

J. S. G. THOMAS.

Electric dry cell. H. SHIMIDZU (B.P. 312,828, 19.6.28).—The whole operative content of the cell is contained and maintained automatically in compression within a vulcanised india-rubber casing. Excess of electrolyte is removed by pressure. J. S. G. THOMAS.

Diaphragms and the like for electrolytic cells. DR. A. WACKER GES. F. ELECTROCHEM. IND. G.M.B.H., and H. MÜLLER (B.P. 312,713, 9.3.28).—Finely-divided, slightly soluble material, *e.g.*, barium sulphate, quartz, glass, purified slag powder, corundum, asbestos, mixed with a glutinous binder, *e.g.*, solutions, emulsions, or suspensions of rubber, gutta-percha, balata, cellulose, is spread in a thin layer on a carrier, *e.g.*, of metal or fabric gauze, and dried at atmospheric temperature.

J. S. G. THOMAS.

Manufacture of electrolytes. R. SCHUSTER (U.S.P. 1,695,667, 18.12.28. Appl., 13.11.23. Renewed 8.8.28. Ger., 22.7.25).—A solution of sodium dichromate and sulphuric acid to which a soluble silicate has been added is evaporated in a revolving drum, and the dry product is sealed in air-tight containers for storage.

C. HOLLINS.

Electrolytic production of sterilising agents, germicides, etc. [e.g., hypochlorites, chloroamines, etc.], and application of such agents. UNITED WATER SOFTENERS, LTD., and E. B. HIGGINS (B.P. 311,218 and 311,253, [A, B] 6.2.28, [B] 30.11.28).—(A) Solution to be electrolysed, *e.g.*, an aqueous solution of sodium chloride or ammonium chloride, flows from one or both electrodes arranged so that the distance between them is small relatively to the cross-section of either electrode. If desired, electrolyte flowing through the anode may flush away products of electrolysis from the cathode. (B) An aqueous solution of an alkali chloride, *e.g.*,

sodium chloride, is electrolysed, as described, in the presence of ammonia or a soluble ammonium salt.

J. S. G. THOMAS.

[Stabilising the output of a tantalum-lead-sulphuric acid] electrolytic rectifier. C. W. BALKE, Assr. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,710,806, 30.4.29. Appl., 18.7.27).—The current density is controlled at about 1 amp. per 0.5–1.5 in.² of active tantalum surface exposed.

J. S. G. THOMAS.

Manufacture of storage battery plate. L. J. PEARSON, Assr. to PHILADELPHIA STORAGE BATTERY CO. (U.S.P. 1,716,320, 4.6.29. Appl., 22.5.20).—A predetermined volume of air is circulated over storage battery negative plates containing metallic lead and sulphuric acid, and is then dried and heated prior to acting again on the plates.

J. S. G. THOMAS.

Manufacture of secondary battery plates. F. GARACA, Assr. to LUTHY RES. LAB. (U.S.P. 1,713,825, 21.5.29. Appl., 18.11.27).—A paste consisting of sulphated active material and a dilute solution of sodium acetate is applied to supporting grids, and sulphates and acetates are removed from the paste before the forming process.

J. S. G. THOMAS.

[Filling for plates of] electric accumulators. B. HEAP, and CHLORIDE ELECTRICAL STORAGE CO., LTD. (B.P. 312,851, 26.7.28).—Finely-divided barium sulphate is produced throughout the active material of storage battery negative plates by the action of sulphuric acid on a lead-barium alloy or on barium hydroxide solution.

J. S. G. THOMAS.

Manufacture of Röntgen ray fluorescent screens. C. HAIDER (B.P. 297,037, 11.9.28. Ger., 12.9.27).—A substance which fluoresces under the action of Röntgen rays, *e.g.*, zinc sulphide, calcspar, magnesite, is spread between two flat plates, *e.g.*, of celluloid; the pressure of air between the plates is then reduced and the edges of the plates are cemented together.

J. S. G. THOMAS.

Production of electric radiating elements, particularly electron-emitting elements for electric discharge devices. E. HARSANYI (B.P. 287,098, 14.3.28. Ger., 14.3.27).—A metal, metallic oxide, or other compound is deposited upon a metallic body, *e.g.*, a roughened platinum wire, by electrophoresis from a colloidal suspension, electrolytic decomposition of the bath being prevented during the process by employing a sufficiently low voltage. On glowing, the deposited material yields a metallic oxide.

J. S. G. THOMAS.

Light-sensitive discharge device. [Photoelectric cells.] ELECTRICAL RES. PRODUCTS, INC., Assees. of G. R. STILWELL (B.P. 288,539, 14.1.28. U.S., 6.4.27).—Light-sensitive material, *e.g.*, potassium, deposited upon a specially or critically positioned auxiliary member, *e.g.*, an axial, re-entrant, tubular, closed stem, within a tube, is re-vaporised and redeposited upon the inner surface of the outer walls of the cell. The method is applicable to the construction of photoelectric cells.

J. S. G. THOMAS.

Effecting chemical reactions in [hydrocarbon] gases by means of electrical discharges. P. H. HULL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P.

311,352, 10.1.28).—Hydrocarbon gas, *e.g.*, methane, is supplied to the arc in a number of adjustable streams arranged so that the arc is distended solely by one stream. If desired, the supplementary gas streams supplying gas to the arc may have a higher velocity than the primary stream, and may impinge on one another to produce a broad, flat disc of gas, or may impart a whirling motion to the gas.

J. S. G. THOMAS.

Manufacture of electric lamps, discharge tubes, and the like. A. LIERENFELD (B.P. 312,659, 27.2.28).—The lamp filament, during pretreatment, is subjected to the action of an intermittent electric current, traversing the filament in the opposite direction to the direction of movement of the filament through the pretreatment zone.

J. S. G. THOMAS.

Electric incandescence lamp. F. ECKHARDT and M. HOHNEKAMP (U.S.P. 1,713,752, 21.5.29. Appl., 5.5.27. Ger., 27.9.26).—A shunt composed of a sulphide ore, which is non-conducting when the normal operating voltage of the lamp is applied to it, but which conducts when a higher voltage is applied, is permanently connected as a shunt to the incandescence filament of the lamp.

J. S. G. THOMAS.

Manufacture of arc lamp electrodes. COMP. LORRAINE DE CHARBONS POUR L'ELECTRICITE (B.P. 289,042, 20.3.28. Fr., 21.4.27).—In order to stabilise the arc flame, an amount of non-oxidised or very slightly oxidised silicon, boron, or zirconium, up to about 2%, is incorporated in an arc lamp core containing carbon, rare earths, and alkali metals, or alkaline earths. The percentage of silicon, boron, or zirconium in the shell may be slightly increased to compensate for loss by lateral combustion.

J. S. G. THOMAS.

Electrical condenser. S. RUBEN (U.S.P. 1,715,789, 4.6.29. Appl., 10.10.24).—The space between aluminium electrodes coated with aluminium sulphide contains cupric sulphide making contact with the aluminium sulphide and supplying free sulphur ions under electric pressure.

J. S. G. THOMAS.

Magnetic material. Magnetic core. G. W. ELMEN, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,715,541 and 1,715,543, 4.6.29. Appl., [A] 19.9.27, [B] 20.6.28).—(A) A magnetic material of very constant permeability over a range of magnetising forces including that employed in continuous loading of electrical signalling conductors contains iron, nickel, and cobalt, and at least one of the elements molybdenum, chromium, tungsten, manganese, vanadium, tantalum, zirconium, copper, and silicon to increase its resistivity. (B) A loading coil comprises a core composed principally of material containing 8–80% Ni, 5–80% Co, and 10–45% Fe.

J. S. G. THOMAS.

Manufacture of cores for electromagnets or the like. GEN. ELECTRIC CO., LTD., W. SINGLETON, and G. C. MARRIS (B.P. 309,394, 9.1.28).—Cores for electromagnets, loading coils, etc. are made of magnetic material, *e.g.*, powdered or laminated iron or nickel-iron alloy, and coated with silicon or material containing silicon. Thus the magnetic material may be heated in a stream of an inert gas, *e.g.*, nitrogen, and silicon tetrachloride.

J. S. G. THOMAS.

Treatment of substances used for impregnation of cables. FELTEN & GUILLEAUME CARLSWERK A.-G. (B.P. 308,748, 5.6.28. Ger., 30.3.28).—The impregnating substance is treated under vacuum or in a chemically inert atmosphere, e.g., nitrogen or carbon dioxide, during the mixing, boiling, and impregnation so that an airtight covering preventing oxidation of the impregnator is formed on the cable. J. S. G. THOMAS.

Non-corrodible battery terminal. C. T. HIXSON, Assr. to W. M. CAMPBELL and H. E. PISCHEL (U.S.P. 1,715,599, 4.6.29. Appl., 5.7.27).—Powdered glass is stirred into a molten mass of lead and antimony at red heat, and the whole is moulded. F. G. CROSSE.

Manufacture of moulded bodies [dynamo brushes] from carbon or mixtures of carbon and metal, with metal insertions. K. F. and H. VON SIEMENS and A. FRANKE (GEBR. SIEMENS & Co.) (B.P. 285,081, 7.2.28. Ger., 11.2.27).

Nickel-iron alloys (B.P. 312,411). Coating of metal (U.S.P. 1,710,747). Electrolytic production of metals (B.P. 285,824, 312,395, and 312,403).—See X.

XII.—FATS; OILS; WAXES.

The butyro-refractometer. F. BOLM (Z. Unters. Lebensm., 1929, 57, 91—93).—The liquids supplied for standardisation of the Zeiss butyro-refractometer vary in refractive index, and care must be taken to use the table referring to the particular standard liquid used. The n value of the liquid varies with age, so that a standard is not usable after a period of a year. The value of n in absolute Zeiss degrees can readily be found by means of the special thermometer. A table for converting Zeiss degrees into n values, and a formula for calculating n for any given temperature are provided. W. J. BOYD.

"Values" of fat from preserved-milk products and its mixtures with cacao butter. H. FINCKE (Z. Unters. Lebensm., 1929, 57, 9—13).—The "values" of mixtures of cacao butter with milk fat, i.e., the saponification and iodine values and the m.p. of the fats and fatty acids, agree sufficiently with the values calculated from the actual milk fat contents and from the milk fat contents derived from the Reichert-Meissl values to permit conclusions to be drawn as to the presence or absence of foreign fats. W. J. BOYD.

Luminescence of sound Dutch lard in ultra-violet light. A. VAN DRUTEN (Z. Unters. Lebensm., 1929, 57, 60—62).—Contrary to hitherto accepted explanations of luminescence phenomena in lard it is shown that perfectly sound Dutch lard rendered on the water-bath at a temperature not above 60° may show blue or blue-violet fluorescence. It is therefore not permissible to draw definite conclusions from such phenomena as to the nature, origin, and method of manufacture of the product. W. J. BOYD.

Determination of detergency of soap products. L. T. HOWELLS (Oil & Fat Ind., 1929, 6, [6], 23—29).—The Detergents Sub-Committee of the American Oil Chemists' Society report on the experimental machines designed for laboratory testing of the detergency of soaps.

The standard washing tests proposed proved not altogether satisfactory, the readings being less consistent and the end-points (number of washings required for complete removal of soil) more drawn-out than in practical laundry tests. It is clearly indicated that the last traces of soil are the most difficult to remove; testing procedure must be modified to accomplish this in a reasonable time. E. LEWKOWITSCH.

Application of the hydrogen value to unsaturated fatty acids. H. I. WATERMAN, S. H. BERTRAM, and H. A. VAN WESTEN (J.S.C.I., 1929, 48, 50—51 r).—The hydrogen values of elaidic, linoleic, and stearolic acids were determined by the method described previously (B., 1929, 102); the results obtained from the first-named are considered as standard and confirm the accuracy of the method. $\Delta^9:12$ -Linoleic acid was shown to contain two ethylenic linkings, 2 mols. of hydrogen being absorbed in the conversion into stearic acid. Stearolic acid, which behaves towards thio-cyanogen as though fully saturated, and absorbs 1 mol. of iodine, also absorbs 2 mols. of hydrogen to yield stearic acid, proving the presence of a triple linking. E. LEWKOWITSCH.

Composition of α -elæostearic acid, the most important component of Chinese wood (tung) oil. J. BÖESEKEN (J.S.C.I., 1929, 48, 71—72 r).—The author criticises the quotations of the work of himself and others on the constitution of elæostearic acid made by Steger and van Loon (B., 1929, 103). It is noted that Böeseken and Hoogland (A., 1928, 1169) demonstrated that the ethyl ester absorbed exactly 3 mols. of hydrogen, and the work establishing the constitution of elæostearic acid as $\text{CH}_3 \cdot [\text{CH}_2]_3 \cdot [\text{CH} : \text{CH}]_3 \cdot [\text{CH}_2]_7 \cdot \text{CO}_2\text{H}$ is reviewed. E. LEWKOWITSCH.

Unsaturated fatty acids of chrysalis oil. W. KIMURA (Chem. Umschau, 1929, 36, 185—190).—The oily bromides, insoluble in light petroleum, obtained from the unsaturated fatty acids (separated by the lead salt-alcohol method) of chrysalis oil are shown to consist principally of tetrabromolinoleic acid, admixed with small amounts of liquid bromides of linolenic and oleic acids: not more than 60% of hexabromostearic acid was obtained. The composition of the liquid unsaturated fatty acids of chrysalis oil is computed as 21.3% of linolenic, 48.9% of linoleic, and 29.8% of oleic acids. Oxidation with alkaline permanganate yielded linusic (m.p. 204°), isolinusic (m.p. 174°), dihydroxystearic (m.p. 132°), and two sativic (m.p. 156°, 173°) acids. E. LEWKOWITSCH.

Preparation of neutralised olive oil. M. MALMY (J. Pharm. Chim., 1929, [viii], 9, 521—524).—The difficulties occasioned in the technical production of neutral olive oil and suggested modifications of the official specifications are outlined. E. H. SHARPLES.

Fachini's reaction for detection of "residue" olive oils. R. MARCILLE (Ann. Falsif., 1929, 22, 163—166).—Fachini's reaction with acetic anhydride (B., 1926, 592) gives a strong cherry-red colour with olive residue oils; with the finer oils the colour is paler, and it is shown that a dark coloration may be due to the quality of the oil being tested, and may not necessarily indicate admixture with residue oil. Assuming that all

olive oils give some colour, and that kernel oils do not, Fachini's reaction is useful for identifying olive oil in mixtures, and also for determining whether or not olive oils of low acidity contain refined inferior oils.

D. G. HEWER.

Palm oil from the Belgian Congo. G. S. JAMIESON and R. S. MCKINNEY (Oil & Fat Ind., 1929, 6, [6], 15—17).—The oil, on analysis by the lead salt-ether separation method, followed by fractionation of the methyl esters, had d_{20}^{25} 0.9146, n_D^{20} 1.4578, acid value 20.65, saponif. value 197.9, unsaponif. matter 0.39%, iodine value (Hanus) 53.7, acetyl value (André-Cook) 15.27, Reichert-Meissl value 0.10, Polenske value 0.29, saturated acids (corr.) 44.3%, unsaturated acids (corr.) 50.6%, iodine value of unsaturated acids 99.9. It contained the glycerides of oleic (47.2%), linoleic (5.6%), myristic (0.5%), palmitic (40.8%), stearic (5.2%), and lignoceric (0.1%) acids, and 0.39% of unsaponifiable matter. Lignoceric acid is thus reported as a constituent of palm oil for the first time. E. LEWKOWITSCH.

Cacao oil. BODINUS (Pharm. Ztg., 1929, 74, 647—648).—Samples of fat extracted from cocoa dust, a by-product in the manufacture of cocoa and chocolate, and sold as pure cacao butter, have inferior colour and odour, a lower m.p., and higher iodine value and refractive index than normal cacao butter. The differences are due to the presence of about 1.6% of the fat extracted from the pods, which yield about 2% of a pasty fat of unpleasant odour, and very high acidity, refractive index, and iodine value. S. I. LEVY.

Oil from seeds of *Erucastrum elongatum*. N. BELIAIEV (Oil Fat Ind. [Russia], 1928, No. 8, 26—28; Chem. Zentr., 1929, i, 166—167).—The oil has acid value 4.52, iodine value (Hübl) 116.3, saponif. value 176.02. It is a semi-drying oil. A. A. ELDRIDGE.

Hydrogenation of oils. H. I. WATERMAN and S. H. BERTRAM (J.S.C.I., 1929, 48, 79—80 T).—Soya-bean oil was hydrogenated in the presence of a nickel-kieselguhr catalyst; in contrast to the results of similar experiments by Kaufmann and Hansen-Schmidt (B., 1927, 226) on sunflower-seed and arachis oils, it was found that the decrease in iodine value (76%) was accompanied by a decrease (35%) in the thiocyanogen value, although the hydrogenation of those unsaturated compounds, which are not saturated by thiocyanogen, but are saturated by iodine chloride, predominates. E. LEWKOWITSCH.

Influence of temperature on hydrogenation [of oils]. A. MARKMAN and V. VASSILIEV (Oil Fat Ind. [Russia], 1928, No. 8, 23—24; Chem. Zentr., 1929, i, 166).—The hydrogenation of oils is reversible. For sunflower oil the optimum temperature is 250—270°. A. A. ELDRIDGE.

Tung oil. Possibilities of production within the British Empire, with a bibliography. L. A. JORDAN (J. Oil & Col. Chem. Assoc., 1929, 12, 113—153).

Colloid-chemical changes in fatty oils. AUER.—See XIV.

PATENTS.

Production of neutral fats and oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,523, 2.7.28).—Fats and oils containing free fatty acids are treated at

100—200° with ethylene oxide or its homologues. The process is accelerated by working under pressure or by the use of catalysts, e.g., metal acetates or oxides such as titanium oxide. E. LEWKOWITSCH.

Manufacture of water-soluble or emulsifiable products from wool fat. I. G. FARBENIND. A.-G. (B.P. 286,252, 29.2.28. Ger., 1.3.27).—Semi-solid, water-soluble or emulsifiable products are obtained by sulphonating wool fat (or its fatty acids) in the presence of a phenol. Emulsions of these compounds are stable even in acid solution, form clear solutions on the addition of alkali, but are salted out by the addition of neutral salts. E. LEWKOWITSCH.

Manufacture of soap and saponaceous materials. J. Y. JOHNSON. From FARBENIND. A.-G. (B.P. 312,405, 8.6.28).—The oxidation products of paraffin hydrocarbons are saponified with about the theoretical amount of concentrated caustic alkali and the solution is cooled to 10—5°, causing the unsaponifiable matter (about 20%) to separate. E. LEWKOWITSCH.

Extraction of oils. J. W. BECKMAN (U.S.P. 1,698,294, 8.1.29. Appl., 19.5.24).—Animal or vegetable material containing oil is macerated with water and treated with a culture of lactic acid bacteria at 50°, preferably in darkness or shadow and with at least partial exclusion of air. The lactic acid produced is periodically neutralised, and when the cellular structure is destroyed the mixture is diluted with water and the oil separated. Addition of 10% of sodium chloride solution accelerates the decomposition. E.g., 1000 pts. of macerated coconut (copra) are treated for 100—125 hrs. with 1 pt. of malt and 1 pt. of magnesium carbonate at 50°, neutrality being maintained by further additions of magnesium carbonate as required. R. BRIGHTMAN.

Extraction of oil from fish liver. W. P. WILLIAMS. From SOC. FRANÇ. DES PROD. ALIMENTAIRES AZOTÉS Soc. ANON. (B.P. 312,768, 20.4.28).—Fresh or salted fish livers are pulverised and subjected to autolysis for 10—24 hrs. at 42—45° until a semi-liquid mass is obtained from which the bulk of the oil, which is pure and of good odour, may be decanted; the remaining oil is removed by filtration or centrifuging, the residue constituting a rich degreas for leather dressing. E. LEWKOWITSCH.

Manufacture of emulsions. E. C. R. MARKS. From CHEM. FABR. STOCKHAUSEN & Co. (B.P. 312,799, 24.5.28).—Stable oil emulsions are produced by the aid of the sulphuric acid compounds of fats or fatty acids described in B.P. 293,480 and 293,717 (B., 1928, 678, 718) as emulsifying agents. E. LEWKOWITSCH.

Manufacture of hair toilet oil. Y. SHIMURA and K. TAKAGI (B.P. 312,568, 23.10.28).—Non-drying vegetable oils (castor, tea-seed, etc.) are agitated and warmed with equal quantities of ethyl or methyl alcohol and a small quantity of ether for a considerable time (e.g., 7 days); the product on being washed and distilled under reduced pressure yields a pale non-oxidising oil of low viscosity. E. LEWKOWITSCH.

Extraction of candelilla wax. J. T. GARCIA (U.S.P. 1,715,194, 28.5.29. Appl., 15.9.25. Mexico, 30.10.24).—The candelilla plant is crushed and subjected

to a tearing and beating action followed by screening to separate the wax and non-waxy matter from the plant fibres. E. LEWKOWITSCH.

Wax-like chlorinated hydrocarbons (B.P. 309,421).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Microstructure of paint films and proof of internal stresses. A. V. BLOM (Farben-Ztg., 1929, 34, 2127—2130).—Transverse sections were cut from old paint films and examined under the microscope after suitable preparation—staining, etching with solvents, etc. A distinct laminated microstructure was observed, due to the alternation of layers relatively rich in pigment or fillers with layers rich in resins, but practically devoid of pigment: this segregation of pigment during the drying process cannot be due to gravity settlement as the paints examined were stripped from vertical surfaces. Good or defective adhesion of successive coatings is evidenced by smooth or irregular transverse fractures respectively. The considerable distortion produced in a red lead undercoating by a hard bituminous top-coat gave evidence of the magnitude of the stresses evoked during drying, and the conception of "compressibility" of a paint film is introduced in connexion with the development of stresses (and consequent breakdown) in the film by the shrinkage of the support with falling temperatures. E. LEWKOWITSCH.

Testing [ageing] of paints. P. NETTMANN (Farben-Ztg., 1929, 34, 2181—2183).—The ageing of paints is discussed from a theoretical point of view on the basis of energy changes in the coating: it is suggested that, during ageing, and as a result of the various influencing factors (irradiation, temperature, etc.), the paint film may accumulate potential energy, which is chiefly responsible for the changes in the inner structure of the film, even after the lapse of years. E. LEWKOWITSCH.

New accelerated [weathering] test for paints. W. H. DROSTE and M. WERNER (Farben-Ztg., 1929, 34, 2131—2133).—Accelerated durability tests for paints are discussed and criticised. E. LEWKOWITSCH.

Destructive light sources for use in accelerated weathering systems. F. C. SCHMUTZ and D. L. GAMBLE (Ind. Eng. Chem. [Anal.], 1929, 1, 83—86).—The action of vigorous artificial light sources has been studied with mixtures of lithopones with lacquer or with house paints. The specimens were mounted on porcelain palettes and dried at 50°, and then subjected to rays from carbon and mercury arcs or to sunlight. The carbon arcs were operated so as to give accentuation of different parts of the spectrum, and it was found that intensification of the infra-red and visible part had little effect, but that in the far ultra-violet the differences between the pigments was lessened. Much information is summarised as to the operation of mercury arc lamps, particularly with a view of obtaining a constant total intensity of radiation, and the use of special glass filters is also discussed. The problem of comparison of these sources with natural sunlight is complicated by large variations in the intensity of the latter. R. H. GRIFFITH.

Examination of pigments in ultra-violet light. M. J. SCHOEN and J. RINSE (Chem. Weekblad, 1929, 26, 321—322).—Pigment value cannot be determined from the fluorescence colours in the cases of lithopone and zinc white. Natural and precipitated chalk can, however, be distinguished, and the presence of zinc oxide (25%) in titanium white changes the violet fluorescence to greenish-yellow. Admixture of organic colouring matter in mineral pigments can usually be detected. S. I. LEVY.

Pigment and oil. E. KLUMPP (Farben-Ztg., 1929, 34, 2130—2131).—The theoretical conclusion that oil absorption varies inversely with particle size is challenged: pigments that had been very thoroughly pulverised in a mortar gave very much lower values than those obtained for the raw pigment. E. LEWKOWITSCH.

Determination of oil absorption of pigments. RUCHTI (Farben-Ztg., 1929, 34, 1954).—It is pointed out that oil absorption depends not only on the degree of subdivision or specific surface, but also on the chemical nature of the pigments; the oil absorption should only be taken as an analytical "value" of a pigment and the convenient Wolff method (cf. B., 1929, 103) is recommended to obtain reproducible results in technical practice. E. LEWKOWITSCH.

Determination of arsenic in antimony oxide pigment. G. SIROIS (Chemist-Analyst, 1929, 18, 14).—Small quantities of arsenic are distilled off as trichloride and weighed as trisulphide. For larger quantities the pigment (1 g.) is treated with concentrated sulphuric acid (15 c.c.) and potassium sulphate (5 g.), the mixture is heated to eliminate sulphur, then cooled, diluted with 20 c.c. of water, heated to dissolve the salts, treated with hydrochloric acid (20 c.c.), and saturated with hydrogen sulphide. Treatment of the arsenic trisulphide with carbon disulphide is recommended.

CHEMICAL ABSTRACTS.

Thickening of oils for use in the varnish industry. C. DORN and J. BURDIN (Oil Fat Ind. [Russia], 1928, No. 7, 29—31; Chem. Zentr., 1928, ii, 2682).—Restricted action of sulphur monochloride on oil affords a suitable product; experiments with aluminium chloride were fruitless. A. A. ELDRIDGE.

Enrichment of poor gums. M. TOMEO (Anal. Fis. Quim. [tecn.], 1929, 27, 77—106).—The effect of the addition of turpentine during the preparation, decantation, and distillation of various gums has been studied. With several classes of resins considerable advantage is to be gained by addition to the gum of about one half the quantity of turpentine, although the exact proportion varies with the material to be treated.

H. F. GILLBE.

Electrical heating [in varnish manufacture]. CARLETON.—See XI.

PATENTS.

Preparations for printing, painting, coating, or impregnating surfaces. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 311,795, 14.2.28).—The products comprise solutions of cellulose esters or ethers in water-soluble esters of formic acid and polyhydric alcohols, e.g., glycol mono- or di-formate, together with water and, if desired, dyes etc. L. A. COLES.

Painting process. E. FRENKEL (H. FRENKEL) (B.P. 288,624, 16.3.28. Ger., 14.4.27).—Paints which dry throughout the mass, and thereby permit the application of successive coats before the preceding coats are dry, contain binding material consisting of fatty oils pre-treated with sulphur chloride, with or without the addition of high-boiling hydrocarbons, esters, amines, etc. to retard the time of drying. Ordinary oil paints, varnishes, nitrocellulose lacquers, etc. may be used for the final coat. L. A. COLES.

Manufacture of non-lead face powder or paint. M. FUKUI and T. MIYAGUCHI (B.P. 312,562, 3.10.28).—Zinc oxide, powdered talc, titanium oxide, starch, etc. is treated with a solution in a volatile solvent of transparent material insoluble in water, *e.g.*, nitrocellulose or caoutchouc, the solution being coloured, if desired, and the solvent removed by evaporation. L. A. COLES.

Manufacture of [white] pigments. J. B. PIERCE, JUN. (U.S.P. 1,715,384, 4.6.29. Appl., 10.11.24).—Hydrogen sulphide is passed into a solution containing 200–500 g. of zinc sulphate per litre and having precipitated barium sulphate or blanc fixe suspended in it. F. G. CROSSE.

Production of white lead. METALLBANK & METALLURGISCHES GES. A.-G., and G. SITZ (B.P. 311,986, 17.7.28).—Lead chloride, obtained, *e.g.*, by the digestion of waste plumbiferous material with hot sodium chloride solution and subsequent precipitation by cooling and/or dilution, is treated in aqueous suspension with sodium carbonate, sodium hydroxide and carbon dioxide, or sodium carbonate and hydroxide, these being added gradually so that the solution does not exhibit a definitely alkaline reaction until conversion of the lead chloride into basic carbonate is complete. L. A. COLES.

Preparation of printer's ink. R. and I. M. MACLAURIN (B.P. 312,745, 30.3.28).—The use is claimed of low-temperature or semi-low-temperature tars, and of products derived from them. The tar may be thickened by adding resinous constituents precipitated from other portions of the tar by the addition of petroleum oil and/or dilute acids, or thinned by the addition of the oil remaining after removal of the resins, or it may be treated with ferrous hydroxide and/or mixed with linseed oil, sodium hydroxide, pigments, etc. L. A. COLES.

Composition for lithographing ink. O. E. HARDER (U.S.P. 1,714,166, 21.5.29. Appl., 11.6.27).—A mixture of petrolatum oil, paraffin, and magnesium carbonate is used with printers' ink. H. ROYAL-DAWSON.

[Non-explosive and fireproof] cellulose lacquers. INTERNAT. FIREPROOF PRODUCTS CORP., Assees. of F. S. VIVAS (B.P. 286,724, 8.3.28. U.S., 11.3.27).—Products are claimed comprising a solution of a gum and boric acid in a mixture of butyl and ethyl alcohol, a solution of nitrocellulose in a mixture of an aromatic hydrocarbon (toluol) and ethyl or butyl acetate, and a mixture of a vegetable oil, *e.g.*, castor oil, with alcoholic calcium chloride solution. A chlorinated hydrocarbon, *e.g.*, carbon tetrachloride, is added to each solution and to the mixture, and a mixture of alcohol and carbon tetrachloride may be used as diluent. L. A. COLES.

Driers for varnishes, lacquers, oil paints, etc.

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,716, 14.1.28).—The use is claimed of metal compounds insoluble or sparingly soluble in water of crude or purified oxidation products of paraffin wax etc., alone or mixed with salts of high-molecular acids of natural origin, *e.g.*, resinates, linoleates, stearates, naphthenates, etc. L. A. COLES.

Application of lacquers containing cellulose esters. IMPERIAL CHEM. INDUSTRIES, LTD., N. STRAFORD, E. E. WALKER, and W. J. JENKINS (B.P. 312,204, 14.2.28).—The surfaces to be coated are primed with a lacquer containing a phenol-formaldehyde resole and a resin of the "glyptal" type, with or without the addition of an acid catalyst, *e.g.*, sulphuric acid, before applying the cellulose ester lacquer. L. A. COLES.

Removal of [non-vitreous] enamel. H. O. LANG (U.S.P. 1,714,879, 28.5.29. Appl., 19.5.24).—The enamelled metal article is immersed in a bath of molten material consisting chiefly of an alkali salt of nitric or nitrous acids. H. ROYAL-DAWSON.

Manufacture of pure colophony and other resinous products from resinous plants or woods. D. GARDNER (B.P. 289,774, 23.4.28. Fr., 30.4.27).—Wood etc., after removal of volatile constituents by treatment with steam, preferably at 1 atm. pressure, followed by drying, is treated in a finely-divided form with sodium hydroxide solution, *d* 1.015–1.02, in the presence of a reducing agent, *e.g.*, sodium bisulphite or hyposulphite, and an oleate. The treatment may be effected at about 30° in a colloid mill, or, when the wood is not so finely divided, at 75–80° in closed apparatus. After removal of the cellulose by filtration, the solution is acidified to precipitate colophony, which is removed and treated with a solvent, *e.g.*, turpentine. L. A. COLES.

Refining of rosin. HERCULES POWDER Co., Assees. of A. LANGMEIER (B.P. 298,214, 10.1.28. U.S., 6.10.27).—A high-grade rosin, suitable for soap and varnish making or for white paper sizes, may be prepared from low-grade wood rosin by passing the molten material through a small-bore tubular distiller and then through a similar vaporiser in which it is heated in countercurrent by means of oil to temperatures of 240–260° and 260–302°, respectively. At the same time superheated steam is passed through the molten rosin, and the whole system is maintained at a pressure of 6–127 mm. of mercury. E. HOLMES.

Manufacture of artificial [resin] materials. SOC. CHEM. IND. IN BASLE (B.P. 281,717, 5.12.27. Switz., 4.12.26).—A preliminary condensation product of urea and formaldehyde, condensed beyond the methylol-urea stage, is subjected to the action of an acid or a non-basic salt polymerising agent, in sufficient quantity to cause the whole mass, including the solvent still present, to gelatinise; the resulting material hardens without the application of external heat. The polymerising agent is first neutralised and then washed out, leaving a product of sp. gr. as low as 0.4. Filling materials, plasticisers, and dyes may be incorporated during manufacture. E. HOLMES.

Resin-like hydrocarbons (G.P. 454,307).—See II. **Treatment of fibrous materials** (U.S.P. 1,695,912).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber and its fractions. R. PUMMERER (Kautschuk, 1929, 5, 129—135).—Gel and sol rubber separated from various types of commercial rubber show a difference in the proportion of iodine chloride or benzoyl peroxide with which they react. The gel constituent possesses the greater reactive power, but with iodine chloride yields more hydrogen chloride. This difference in chemical behaviour may be due to cyclic formation in one of the constituents, to *cis-trans* isomerism, or to difference between the structure of the micelles. Sol rubber appears to be convertible partially into the gel type by heating with exclusion of air. A benzene solution of sol rubber on partial crystallisation yields mixed crystals; this renders cryoscopic measurements for rubber in benzene untrustworthy, and may explain the discrepancies between the results of cryoscopic and capillary determinations of the mol. wt.

D. F. TWISS.

Rubbers with low nitrogen content. A. D. CUMMINGS and L. B. SEBRELL (Ind. Eng. Chem., 1929, 21, 553—557).—Purified rubber prepared from latex by treatment with sodium hydroxide by the method of Pummerer and Pahl (A., 1927, 1193) still contained 0.004% N even after intensified treatment. Unlike the rubber from ordinary latex, the rate of vulcanisation of rubber from this purified latex was very little affected by the p_H of the alcohol used as coagulant. Vulcanisation yielded products of good quality comparable with controls containing the normal proportion of nitrogen. Such nitrogen-poor rubber was not easily electro-deposited from the latex. The small proportion of residual nitrogen was not present as protein, but was removable by acetone; the extracted rubber thus obtained vulcanised only slowly and the products were physically less satisfactory.

D. F. TWISS.

Isoprene and rubber. H. STAUDINGER (Kautschuk, 1929, 5, 94—97, 126—129).—A review of the author's investigations of the constitution of rubber mainly by way of hydrocaoutchouc and of polymerisable substances such as vinyl acetate and styrene. The properties of rubber are dependent on a high mol. wt., possibly approaching 100,000. The difference between rubber and gutta-percha or balata rests probably on a different spatial arrangement of the groups at the double linking, *i.e.*, on *cis-trans* isomerism.

D. F. TWISS.

Colloid-chemical changes in rubber and fatty oils. L. AUER (Trans. Inst. Rubber Ind., 1929, 4, 499—520).—The colloidal condition of iso-colloids is discussed. The isolated sol and gel constituents of rubber are regarded as mixtures representing only products containing increased proportions of the respective true constituents. In the iso-colloidal fatty oil system the disperse phase may increase at the expense of the chemically similar dispersion medium by the action of light, heat, and aggregators, *e.g.*, electrolytes and gases such as oxygen. Formic acid acts as a coagulant on the iso-colloidal fatty oil system, effecting separation of the dispersed phase. Gases such as oxygen also have a coagulant effect on lyophobic colloidal systems, similar to that of electrolytes, *e.g.*, on latex or oils. Sulphur resembles oxygen in its

vulcanising action on oils, increasing the concentration of the disperse phase and coagulating the system; if the sulphur is present in sufficient excess it may subsequently combine chemically with the fatty acids, but this is not primarily necessary for the vulcanisation process. The gelation of oils by heat can be accelerated or retarded by the addition of suitable electrolytes. By vulcanising oils which have been solidified with the aid of electrolytes new types of "rubber substitutes" can be prepared. Similarly, by heating rubber containing electrolytes, thermoplastic conversion products are obtainable.

D. F. TWISS.

Ageing tests for sponge rubber. H. P. STEVENS (Trans. Inst. Rubber Ind., 1929, 4, 486—492).—By compressing a piece of sponge rubber between plates in an ageing oven at 70° for 3 days a compact sheet is formed from which rings can be punched for testing in the usual manner. The ageing can be prolonged as desired, but 2 weeks is the useful limit. Tested in this way, and also by hand tests and chemical examination after ageing uncompressed, different samples not only showed wide variations between themselves, but irregularities in vulcanisation could be detected in individual samples.

D. F. TWISS.

Physical characteristics of sponge rubber. H. F. CHURCH (Trans. Inst. Rubber Ind., 1929, 4, 533—542).—A wide range of samples of sponge rubber sheeting has been examined as to general physical properties. "Cellularity," *i.e.*, the ratio of trapped air to the total air space, was estimated by enclosing a sample in a space of known volume and pressure and observing the effect of alterations in pressure on the total volume. Other characteristics examined included weight per unit volume, compressibility, *e.g.*, between two parallel plates or by an indentation instrument, and permanent set. Cellularity had a very marked influence on compressibility; in the case of true sponges with inter-connecting cells, compressibility was mainly dependent on cell size, and was relatively insensitive to alteration in the physical properties of the rubber or its composition.

D. F. TWISS.

Oxidation of rubber mixings. W. C. DAVEY (Trans. Inst. Rubber Ind., 1929, 4, 493—498).—Examination of progressively aged (70°) samples of vulcanised rubber as to increase in weight and acetone-soluble matter before and after extraction with acetone or with acetone and chloroform successively indicates three stages of oxidation: in the first, oxidation is slight and yields acetone-soluble products; in the second, increase in weight is much more rapid than the production of soluble matter; and in the third, the acetone-soluble matter increases after oxygen absorption has ceased. It is suggested that three successive reversible changes occur, *viz.*, vulcanised rubber = depolymerised rubber (chloroform-soluble) = resinous matter (acetone-soluble) = oxidised matter (insoluble). Of these, the middle stage is technically of greatest importance.

D. F. TWISS.

Distribution of ingredients in rubber mixings. H. PAGE (Trans. Inst. Rubber Ind., 1929, 4, 521—525).—Evidence is adduced that, contrary to a recent opinion

(Reiner, B., 1928, 341), compounding ingredients can be uniformly dispersed in rubber by mixing under the ordinary conditions of the factory mixing operation.

D. T. TWISS.

Chemical reactions in rubber compounds.

I. Reactions between pine tar and litharge. W. H. REECE (Trans. Inst. Rubber Ind., 1929, 4, 526—532).—When litharge is added to a rubber "compound" containing pine tar, a hardening soon occurs. That this change is due to a chemical reaction between litharge and pine tar comparable with that which occurs between them when heated without rubber is confirmed, *e.g.*, by the approximate agreement in the amount of water formed under the two sets of conditions. The pine tar used contained 46.9% of acids which reacted with litharge yielding 2.45% of water. D. F. TWISS.

Temperature-recording micropress for studying the course of vulcanisation. J. C. WALTON (Ind. Eng. Chem. [Anal.], 1929, 1, 106—108).—The micropress consists of an electrically heated upper part, a central portion containing a thermocouple, and a container for the sample undergoing vulcanisation; readings obtained on a millivoltmeter are interpreted by measurements with crystals of different organic substances. Continuous observation of specimens at temperatures between 30° and 170°, with the addition of various curing agents, has shown that the changes produced in the rubber can be critically followed in this way. R. H. GRIFFITH.

Compressibility of rubber. R. ARIANO (Nuovo Cim., 1928, 5, 77—101; Chem. Zentr., 1929, i, 153).—A study of the compressibility curves of vulcanised rubber, and of the effect of duration of vulcanisation on the mathematical constants. A. A. ELDRIDGE.

Power consumption in [rubber] crêpeing mills. R. RIEBL (Med. Proefstat. Rubber No. 41; Arch. Rubberecultuur, 1929, 13, 219—238).

PATENTS.

Treatment of rubber. SOC. ITAL. PIRELLI, and U. PESTALOZZA (B.P. 284,608, 30.1.28. Italy, 31.1.27).—Latex is treated with an appropriate proportion of a suitable coagulant, especially salts of bi- or ter-valent metals, *e.g.*, calcium sulphate, so as to render it capable of local thickening and subsequent formation of a compact layer of coagulum when brought into contact with a heated surface, *e.g.*, at 75—95°. Rubber solvents such as benzol or benzine may be used in conjunction with such coagulants. Articles are manufactured from such latex, which may be natural, preserved, concentrated, compounded, or otherwise, by bringing it into contact for a short period with a heated mould or former of the desired shape and size. D. F. TWISS.

Vulcanisation of rubber. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of B. W. NORDLANDER (B.P. 290,602, 15.5.28. U.S., 16.5.27).—"Stabilised amorphous sulphur," for the vulcanisation of rubber, is prepared by fusing sulphur with selenium or tellurium and grinding the brittle product or by allowing hydrogen sulphide to react with selenious acid in a suitable liquid medium; vulcanisation may be aided with an organic accelerator.

The selenium or tellurium maintains the sulphur in an amorphous form which is exceptionally active in vulcanisation. Sulphur so treated may also be mixed colloiddally into rubber latex for electrodeposition purposes. D. F. TWISS.

Preparation of products of latex-like character. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 312,201, 19.12.27 and 1.6.28).—Butadiene and its homologues or analogues are dispersed in aqueous media with emulsifying agents reducing the surface tension of the water; polymerisation is then effected in the presence, if desired, of buffers giving a p_H range of 4—8.5. The addition of compounds supplying oxygen and of agents which combine with acids further simplifies the formation of a latex-like product. D. F. TWISS.

Manufacture of rubber compositions. DUNLOP RUBBER Co., LTD., A. E. T. NEALE, and F. THOMAS (B.P. 311,930, 5.5.28).—Condensation products of an aldehyde and an aminophenol prepared in neutral or acid solution, *e.g.*, of acetaldoal or crotonaldehyde and *p*-aminophenol, are employed as antioxidants for rubber. D. F. TWISS.

Incorporation of colloidal substances in rubber. FELTEN & GUILLEAUME CARLSWERK A.-G. (G.P. 453,628, 20.1.23).—Ingredients such as sulphur are dissolved colloiddally in a rubber solvent and then mixed with the rubber. Uniformity is thus ensured. D. F. TWISS.

Method of compounding caoutchouc. R. P. DINSMORE, Assr. to GOODYEAR TIRE and RUBBER Co. (U.S.P. 1,712,333, 7.5.29. Appl., 22.11.24).—An emulsion of rubber is mixed with a suspension of compounding ingredient, the rubber being then coagulated on the latter. The product is freed from water-soluble material by washing, and a rubber softener is added to the still pasty mass, which is then incorporated into a rubber mixture by milling. D. F. TWISS.

Direct production of thread or tubes from concentrated compounded latex. DUNLOP RUBBER Co., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 311,844, 23.2. and 5.9.28).—Concentrated and/or compounded aqueous dispersions of rubber or similar materials are caused to flow through orifices of the desired shape into a bath, such as a solution of common salt (150 pts.), ammonium acetate (150), and water (450), capable of effecting dehydration and setting at least partly by penetrative osmotic action. The cross-sectional area of the threads or tubes can be varied by altering the distance of the orifice below the surface of the bath. If a number of threads, on issuing from the bath, are allowed to come into contact and are then dried, they unite to form a tape with longitudinal grooves on its wide surfaces. D. F. TWISS.

Deodorisation of articles produced by the electrophoresis of latex mixings. DUNLOP RUBBER Co., LTD., and E. W. MADGE (B.P. 312,443, 29.3.28).—Articles produced by electrophoretic deposition from latex are treated with formaldehyde or an aqueous solution of paraformaldehyde, *e.g.*, by immersion for 2—3 hrs. in a 20% solution of the former. D. F. TWISS.

Rubber-bonded abrasive articles (B.P. 311,104).—See VIII.

XV.—LEATHER; GLUE.

Biochemistry of soaking and liming [of animal skins]. III. Influence of gaseous environment on the soaking of heavy hides. E. R. THEIS and J. M. MILLER (J. Amer. Leather Chem. Assoc., 1929, 24, 290—314; B., 1928, 827).—The gases which cause the greatest protein degradation of "domestic" hides are nitrogen and hydrogen, whilst in the case of "Frigorificos," hydrogen causes the greatest decomposition and nitrogen the least. A greater amount of volatile fatty acids is obtained in a nitrogen atmosphere than in one of oxygen; these acids appear to be derived from the hydrolysis of the skin proteins. The greatest hydration of the skin is obtained in atmospheres of carbon dioxide and oxygen, respectively, and the least in nitrogen. The soak water is rendered alkaline by using atmospheres of air or oxygen. D. WOODROFFE.

Hydrolysis of hide powder in saturated sodium chloride solutions at various p_H values. A. W. THOMAS and M. W. KELLY (J. Amer. Leather Chem. Assoc., 1929, 24, 280—282).—Weighed portions of hide powder were treated with saturated solutions of sodium chloride of p_H values 2.0 to 11.0, known volumes of the solutions were withdrawn at intervals up to 224 days, the nitrogen content was determined, and the percentage hydrolysis calculated for each time interval. The p_H values of the solutions were determined at the end of the experiment. The hydrolysis of hide powder in saturated sodium chloride solutions is shown to increase with increasing alkalinity, and the hydrolysis of hide powder as a function of the p_H value is affected by the presence of sodium chloride. The addition of sodium carbonate to preclude salt stains causes a loss of hide substance. D. WOODROFFE.

Action of sodium chloride on hides and on animal tissues. A. PONTE (Boll. Uff. Staz. Sperim. Ind. Pelli, 1929, 7, 97—119).—The differences in the solubilising actions of sodium chloride and sodium sulphate on the derma proteins seem to become accentuated as the degree of alteration undergone by these proteins increases. Experiments on prepared hides ready for tanning and freed from water of capillarity show that dilute sodium chloride solutions (5.8%) effect only slight changes due to a feeble swelling action. Increasing salt concentration is accompanied by a progressive deswelling effect, a saturated or almost saturated solution reducing the moisture content of the hide to about 50%. Similar dehydration doubtless occurs when beef is salted. T. H. POPE.

Measurement of the properties of sole leather and the effects of sulphuric acid. D. BURTON (J. Soc. Leather Trades' Chem., 1929, 13, 178—191).—The various physical properties of sole leather are enumerated and the work which has been done in that connexion is summarised. The usual chemical analysis is considered to have very little relation to the durability of the leather and actual practical requirements. The sp. gr. of leather is important in judging quality and cutting value, but it is difficult to obtain a representative figure since it varies with the position of sampling. The degree of tanning indicates the quality of a leather and the yield likely to be obtained on currying or retanning.

Some leathers which contain 1.1% of free mineral acid as determined by the Procter-Searle method are quite sound after 5 years' keeping; others are entirely disintegrated. Raw hide treated with sulphuric acid will keep indefinitely. Innes' method (B., 1928, 721) fails if oxalic acid and syntans are present in a leather. Deterioration of leather appears to be caused by free, but not combined, sulphuric acid. The yield of sole leather and the amount of tan fixed by the hide is affected by the method and the degree of liming. The greater the hydrolysis of the collagen, the greater is its combining capacity. Methods for sterilising anthrax-infected hides, preventing the formation of deposits from myrobalan extracts, and overcoming the difference between tan liquors prepared from extracts and those made by leaching are considered. D. WOODROFFE.

Influence of hydrogen-ion concentration on the colour of vegetable-tanned leather. R. O. and A. W. PAGE (Ind. Eng. Chem., 1929, 21, 584—585).—Two series of tan liquors ranging in p_H value from 1.0 to 9.0 were prepared from wattle bark and chestnut wood extracts, respectively. Pieces of bated ox hide were brought to the p_H value of the tan liquor with which they were to be tanned, then placed in that tan liquor for 24 hrs., removed, and their colours compared. The tan liquors showed a progressive darkening in colour with increasing p_H value, but the variation in colour of the leather followed closely the swelling curve. It was palest at p_H 5, darkest at p_H 2, brightened toward p_H 1, but with an increased reddish tint, darkened gradually from p_H 5 to p_H 7, and more so at p_H 8—9, especially with the chestnut liquors. Pretreatment of the hide pieces with a buffer solution of p_H 5 followed by tannage first in wattle-bark liquors at p_H 5 and finally in liquors varying in p_H from 1 to 9, gave leather pieces in which there was a regular gradation in colour from the lightest at p_H 1 to the darkest at p_H 9. Pretreatment of the hide pieces with buffer solutions of high or low p_H values caused a darkening in colour of the subsequently tanned leather. It follows that the colour changes on the tanned leathers are due to actual changes in hide structure brought about by variation in the hydrogen-ion concentration. D. WOODROFFE.

Temperature factor in vegetable tannin fixation. A. W. THOMAS and M. W. KELLY (J. Amer. Leather Chem. Assoc., 1929, 24, 282—289).—Portions of hide powder were tanned with wattle bark, oak bark, gambier, and quebracho liquors, respectively, at 4°, 7°, 25°, and 37.5°, and the amount of fixed tannin was determined at intervals up to 117 days. The rate of tanning was markedly greater at 37.5° than at 25°, and then gradually fell with decrease in temperature. Fixation of tannin occurred at low temperatures, but the amount was very small. D. WOODROFFE.

Determination of moisture in tanning extracts. Report of Committee of the American Leather Chemists' Association. H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1929, 24, 314—321).—Comparisons have been made of the direct method of determining moisture, *i.e.*, by drying, and the indirect method, in which it is obtained by difference from the total solids figure in the official method of tannin

analysis, using a solid quebracho extract. Under some conditions identical results were obtained by the two methods, and under others 0.7—1.2% more water was found by the direct method. The effect of time of drying, size of sample, and thickness of the film on the results has been studied, and also the method of drying in both methods. Identical results were obtained by both methods in determining the moisture in wattle and larch extracts. It is recommended that the direct method for determining moisture in tanning extracts be used only on materials which do not give a uniformly turbid solution. D. WOODROFFE.

Qualitative analysis of [vegetable] tanning materials by means of ultra-violet rays. V. KUBELKA and V. NEMEC (J. Soc. Leather Trades' Chem., 1929, 13, 113—117).—Ethyl ether or ethyl acetate (10 c.c.) is shaken for 5 min. with 20 c.c. of an aqueous infusion of the tanning material of analytical strength, and the ethereal or acetate layer is separated and examined by ultra-violet light. The solutions have been obtained for a number of different tanning materials and their fluorescence alone, after rendering alkaline and acid respectively, on cotton wool, and after rendering alkaline and placing on cotton wool have been observed. Extracts of the dry tanning materials with 50% alcohol have been examined in ultra-violet light, then rendered alkaline, the colour change noted, and the colours of each observed on cotton wool. To identify gambier, 5 c.c. of the tannin solution is shaken for 1 min. with 5 c.c. of alcohol; a greyish-violet fluorescence in ultra-violet light is produced. Addition of 1 c.c. of sodium hydroxide solution produces a faint greenish fluorescence. When 10 c.c. of petroleum spirit are added and the mixture is shaken in a separating funnel and allowed to separate, the spirit layer will show a brilliant pale-green fluorescence even in presence of very small quantities of gambier. D. WOODROFFE.

One-bath chrome-tanning process in the light of Werner's co-ordination theory. C. H. SPIERS (J. Amer. Leather Chem. Assoc., 1929, 24, 246—270).—The addition of neutral salts to a chrome-tanning liquor causes an increase in the precipitation figure and diminishes the amount of chromium absorbed by pelt. Boiling diminishes the p_H value of the liquors, increases the precipitation figure, diminishes the amount of chromium absorbed by the pelt, and increases the time required for tannage. A definite time is required for the chrome liquor to reach equilibrium after it has been diluted or boiled, or salts, acids, or alkalis have been added. The precipitation figure and the amount of chromium absorbed by pelt are both diminished by dilution of a chrome liquor. These various effects are attributed to the formation and existence of chromium complexes of the Werner type. Their stability depends on the nature of the central co-ordinating atom and of the co-ordinated groups. Thus hydroxyl groups are more firmly retained than is chlorine and will replace it. Bivalent ions give more stable complexes than univalent ions and will replace them. Units with two co-ordinatable groups, e.g., oxalate ions, form very stable ring structures, hence the extraction of chromium from chrome-tanned leather by oxalic acid. In chrome

tanning the carboxyl of the collagen combines with the chromium by co-ordinating itself with it, but this would be impossible if the complex ions were too stable. Chromium cations should contain water and hydrolyse in order to show good tanning properties. The work of Stiasny and his collaborators is reviewed, and their theories of "ol" and "oxo" compounds, which are most active in tanning, is expounded. The more complicated the "ol" and "oxo" compounds, the larger are the molecules, and the more they approach colloidal dimensions. Dilution promotes hydrolysis with consequent formation of "ol" and "oxo" compounds, thus producing coarser dispersions. Additions of alkali or pelt to a chrome liquor disturbs the equilibrium in it, causing the production of complex ions containing more "hydroxo," "ol," and "oxo" groups, and it may require time to restore equilibrium, hence the subsequent changes on ageing. Stiasny and Gustavson's theories of the mechanism of tanning are reviewed and compared. D. WOODROFFE.

Distribution of chromium in one-bath chrome-tanned leathers. W. SCHINDLER and K. KLANFER (Collegium, 1929, 121—153).—Calf pelts were tanned with different chrome alum solutions rendered basic with sodium carbonate, and the chromium was determined in the different layers of the tanned leather. The chromium content of all layers increased with increase in basicity of the tan liquor. The middle layer contained most chromium for tan liquors of basicity $> 40\%$, and the grain layer for liquors of basicity $> 40\%$. "Ageing" did not affect the chromium distribution for basicities of 22%, neither did variations in the precipitation figure of the tan liquors used. The grain of leathers tanned with fresh tan liquors of 45% basicity contained more chromium than the middle layer, whereas the reverse was found with "aged" liquors. The composition of chrome-tanned leathers was affected more by the composition of liquors of 45% basicity than by that of tan liquors of lower basicity. The grain contained more chromium than the middle layer when the precipitation figure of a chrome-tanning liquor fell below an "isostatic" limit (about 2.0—3.0) which depended on the kind of tan liquor used and the pre-treatment of the pelt. A smaller amount of chromium was fixed by the grain than by the middle layers with increased amount of sulphuric acid in the pickle used on the pelts before chrome tanning, whilst there was decreased fixation of chromium in the middle layers when a similar increased amount of formic acid was used. Small variations in the concentration of the chrome-tanning liquor, within narrow limits, did not affect the fixation of chromium. At 11° and 26° the middle layer fixed more chromium than the grain, but at 35° the grain fixed slightly more than the middle layer. Large additions of sodium carbonate to the chrome-tanning liquors towards the end of the tannage considerably increased the chromium absorbed by the grain, but had little effect on the flesh and middle layers. D. WOODROFFE.

Proposed official method for analysis of one-bath chrome[-tanning] liquors. Report of Commission of the Society of Leather Trades' Chemists. R. F. INNES (J. Soc. Leather Trades' Chem., 1929, 13,

111—112).—An aliquot portion of the liquor is diluted to 150 c.c., an excess (10 c.c.) of *N*-sodium hydroxide and 10 c.c. of 10-vol. hydrogen peroxide are added, and the mixture is boiled in contact with a clean piece of iron and either filtered or diluted to 400 c.c., then mixed with 5 c.c. of cold saturated potassium permanganate solution, made up to 500 c.c., and filtered; the chromium is determined iodometrically using a 5 c.c. excess of concentrated hydrochloric acid and 15 c.c. of 20% potassium iodide solution. The acidity is determined as before (cf. *ibid.*, 1924, 504) and the p_H value obtained electrometrically.

D. WOODROFFE.

Production of tannin extracts from crude tanning materials. P. JAKIMOV (J. Chem. Ind. [Russia], 1928, 5, 507—509; Chem. Zentr., 1928, ii, 2767).—The process (of Smetkin and Jakimov) depends on the fact that the colloidal tannic acid solutions diffuse more slowly through a membrane than the other water-soluble substances. The cell walls of the extracted material serve as the dialysing membrane.

A. A. ELDRIDGE.

Alkaloid test for tannins. C. M. FEAR (Analyst, 1929, 54, 316—318). The addition of 2 c.c. of 1% gallo-tannin solution to a large number of alkaloid hydrochlorides showed that the only ones giving appreciable precipitates were brucine, caffeine, cinchonine, cinchonidine, quinine, and strychnine. The reaction appears not to be due to precipitates of unchanged alkaloid hydrochlorides, since the intensity of the precipitates does not vary from 1% to 10% solutions, but is due rather to an interaction, either physical or chemical, of alkaloid and tannin.

D. G. HEWER.

Glue testing. II. Report of the Commission of the German Association for testing technical materials. O. GERNGROSS (Collegium, 1929, 119—121; 191—207; cf. B., 1928, 496).—The water content, ash, p_H value, viscosity, jelly strength, "adhesibility" values of Rudeloff and of Bechhold, and glutin content were determined by various members of the Commission for 15 different glues and gelatins, of which five were bone glues. The m.p. and setting point were determined for gelatins. A glue recovered from chrome leather showed high jelly strength, glutin content, m.p., and setting point. The above short method of testing gave a false valuation for bone glues which did not coincide with their adhesive properties. Bone glues of the same viscosity as hide glues possessed a considerably higher "adhesibility" (Rudeloff). Attempts to evaluate glues and gelatins by precipitation with sulphosalicylic acid were unsuccessful.

D. WOODROFFE.

PATENTS.

Leather oil and its manufacture. A. E. BECKER and A. B. BOEHM, ASSRS. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,715,892, 4.6.29. Appl., 27.7.25).—A mineral oil (lubricating grade) and an oil-soluble sulphonate containing free alkali, which is neutralised by a saponifiable fish oil, are compounded.

E. LEWKOWITSCH.

Waterproofing vegetable protein-base glue. G. H. OSGOOD (U.S.P. 1,706,674, 26.3.29. Appl., 11.8.26).—A solution of relatively small quantities of gum dammar, asphalt, and sulphur in carbon disulphide is mixed with an aqueous solution of vegetable meal with

a high protein content, calcium hydroxide, sodium carbonate, and sodium silicate.

L. A. COLES.

Compressed laminated products and their manufacture. SPRUCOLITE CORP., ASSEES. OF L. J. OLLHEIMER (B.P. 299,441, 29.2.28. U.S., 28.10.27).—Sheets of wood are bound together under compression by a glue containing water, e.g., an aqueous solution containing casein, lime, sodium silicate, and a metal salt, the original wood or the finished block being dried to such an extent that the moisture content of the final product, including the glue, is less than that of ordinary dry wood.

L. A. COLES.

Tanning material. O. SPENGLER and A. THURM (U.S.P. 1,698,659, 8.1.29. Appl., 25.2.27. Ger., 23.2.26).—See B.P. 266,697; B., 1928, 206.

Leather oil (B.P. 312,768).—See XII.

XVI.—AGRICULTURE.

Soil microbiology. IV. Degradation of cellulose in soil. S. WINOGRADSKY (Ann. Inst. Pasteur, 1929, 43, 549—633).—The organisms concerned in the decomposition of cellulose in soil are classified and their characteristics described. Cellulose undergoes a rapid oxidation in soil and the product closely resembles oxycellulose. The biological oxidation of cellulose is differentiated from the purely chemical process in that the product does not reduce Fehling's solution. Assimilable nitrogen, preferably inorganic, is essential to this process, the consumption amounting to approx. 2 pts. of nitrogen per 100 pts. of cellulose decomposed. The consumed nitrogen is transformed into organic combination, but there is evidence of some partial reduction to ammonia even with unrestricted access to the air. The optimum reaction for cellulose decomposition is p_H 7. Slight changes of reaction occur during the process depending on the form in which the nitrogen is supplied. Colloidal acids affect the reaction of the medium to a small but definite extent. There is no production of fatty acids or of volatile matter, and the presence of these substances may be taken as an indication of the presence of anaerobic organisms.

A. G. POLLARD.

Soil actinomyces. I. Introduction. K. SUBRAHMANYAN and R. V. NORRIS (J. Indian Inst. Sci., 1929, 12A, 53—56). **II. Mode of occurrence in soil.** K. SUBRAHMANYAN (*Ibid.*, 57—68).—I. The literature of the subject is reviewed.

II. Actinomyces occur in soil only in the conidial form. The usual micro-organic nutrients and stimulants do not accelerate the vegetation of actinomyces in soil.

A. G. POLLARD.

Colorimetric determination of nitrates in soil and water. L. U. DE NARDO (Giorn. Chim. Ind. Appl., 1929, 11, 107—109).—Use is made of a 2.5% pyrogallolsulphonic acid solution, prepared by dissolving 5 g. of pyrogallol in 10 c.c. of concentrated sulphuric acid, heating for a few moments at 80—90°, allowing the acid formed to crystallise, and dissolving in water to 200 c.c. A sample of the fresh soil (100 g.) is shaken for 6 hrs. with 200 c.c. of water, and 80 c.c. of the filtered liquid are shaken and heated to boiling in a 100-c.c. flask with 1—3 c.c. of saturated baryta solution. When

the precipitate has settled, 0.5—1 c.c. of 50% basic lead acetate solution is added, excess of lead and any barium being precipitated after 2—3 min. with about 5 c.c. of saturated sodium sulphate solution, and the cold liquid made up to 100 c.c. and filtered. Ten c.c. of the filtrate are placed in a flat-bottomed porcelain dish (about 50 c.c.) and, if the soil contains nitrites in excess of 0.1 mg. of N_2O_3 per kg., are treated with a drop of saturated carbamide solution and, with stirring, with about 1 c.c. of concentrated sulphuric acid. After 10 min., 0.5 c.c. of the pyrogallolsulphonic acid solution is added and also slowly and with stirring, 20 c.c. of concentrated sulphuric acid. After the lapse of 1 hr. the coloration is compared with those obtained similarly with nitrate solutions of known strengths. If the 10 c.c. of solution tested contains more than 0.1 mg. of KNO_3 , pyrogallol should be used instead of the sulphonic acid. If the latter fails to detect nitrate, smaller proportions may be tested for by suitably concentrating the alkaline defecated solution before applying the colour test. The method may be applied also to aqueous vegetable extracts, foodstuffs, etc.

T. H. POPE.

Dependence of soil reaction on fertilisation and season. K. BORESCH and R. KREYZI (Fortschr. Landw., 1928, 3, 963—968; Chem. Zentr., 1929, i, 284).—In pot experiments with potatoes, stable manure and calcium cyanamide reduced the faintly acid reaction of the soil; carbamide, ammonium sulphate-saltpetre, and ammonium sulphate increased it considerably, whilst sodium nitrate was almost without effect. Variations in the hydrogen-ion concentration of the aqueous extract of the soil represent its varying carbon dioxide content.

A. A. ELDRIDGE.

[Fertilising] action of "ammonia-superphosphate" compared with ammonium sulphate and superphosphate. DENSCH, HUNNIUS, and STEINFATT (Landw. Jahrb., 1928, 68, Suppl. I, 21—22; Chem. Zentr., 1929, i, 130).—"Ammonia-superphosphate" undergoes conversion from ammonium sulphate and superphosphate into ammonium phosphate and calcium sulphate, giving a favourable soil reaction for plant growth.

A. A. ELDRIDGE.

Rhenania phosphate [fertiliser]. DENSCH, HUNNIUS, and STEINFATT (Landw. Jahrb., 1928, 68, Suppl. I, 20—21; Chem. Zentr., 1929, i, 130).—Comparative experiments with basic slag and superphosphate are described.

A. A. ELDRIDGE.

Solubility of phosphorites at different hydrogen-ion concentrations, and their assimilability by plants. S. N. ROSANOV (Landw. Jahrb., 1928, 68, 559—581; Bied. Zentr., 1929, 58, 253—255).—The solubility in the usual solvents of Russian phosphorites was compared with that of apatite and of basic slag. The solubility in various buffer solutions of all materials examined increased with acidity over the range p_H 3.0—8.0. Differences in the solubility of phosphorites due to fineness of grinding were examined by shaking with buffer solutions for 2 hrs. Such differences were only apparent in acid solutions, and these disappeared when the extraction period was increased to 2 weeks and over. Pot experiments with buckwheat indicated a

similar order of assimilability of the phosphorites by plants as was shown by extraction with buffer solutions.

A. G. POLLARD.

Effect of nitrates on the growth of flax. V. ILLUVIEV and K. GALUNOVA (Ber. landw. Versuchsstat. Engelhardt, 1928, No. 3, 59 pp.; Chem. Zentr., 1929, i, 283—284).—In a loess soil sodium nitrate retards the development of flax; the cultural state of the soil, and particularly its nitrogen content, influence this effect, as also does the time of application. Sodium dihydrogen phosphate is somewhat compensatory. The p_H of the soil is raised by sodium nitrate and phosphate, and lowered by ammonium nitrate and potassium sulphate. Variations in the p_H of the soil caused by the flax were followed.

A. A. ELDRIDGE.

Effect of nitrogen in increasing yield [of plants]. GERLACH and SEIDEL (Landw. Jahrb., 1928, 68, Suppl. I, 299; Chem. Zentr., 1929, i, 130).—The effect of a given quantity of nitrogen in a particular fertiliser depends on the amount of water it receives, and on the kind of plant treated.

A. A. ELDRIDGE.

Cause of the sensitiveness to lime of yellow lupins. DENSCH (Landw. Jahrb., 1928, 68, Suppl. I, 33—34; Chem. Zentr., 1929, i, 131).—The action is specific, and is more marked with calcium sulphate than with the carbonate. The availability of the phosphate appears to be diminished, an arrest of protein formation and an accumulation of sugars resulting.

A. A. ELDRIDGE.

Stimulation of mustard seeds with carbon disulphide. MUTH and VOIGT (Landw. Jahrb., 1928, 68, Suppl. I, 428—429; Chem. Zentr., 1929, i, 132).—Stimulation of growth was observed.

A. A. ELDRIDGE.

Composition of mangolds grown in Mid-Wales. T. W. FAGAN and J. E. WATKIN (Welsh J. Agric., 1928, 4, 102—113).—Five varieties were compared. Nitrogenous fertilisers increased the protein in the dry matter. Application of ammonium chloride or heavy dressings of ammonium sulphate reduced the percentage of dry matter and sugar. Potash narrowed the ratio of true to crude protein. Storage of mangolds in clamps reduced the dry matter and sugar content, but largely increased the percentage of true protein in the dry matter. When mangolds were allowed to run to seed the dry matter and sugar content were reduced. Mangold leaves contain: crude protein 21.64, silica-free ash 19.00, CaO 2.19, P_2O_5 0.98, K_2O 5.61%.

CHEMICAL ABSTRACTS.

Laboratory method for measuring relative adhesive qualities of fungicidal dusts. R. C. WILLIAMS (Ind. Eng. Chem. [Anal.], 1929, 1, 81—82).—Microscope slides are coated with shellac, placed under a bell jar, and treated with fungicide dust from an atomiser. The amount of dust on each is determined by weighing, and its resistance to washing and drying is measured by moistening in steam, drying in air, and dipping ten times into water; the loss from this treatment is shown by another weighing. Similar tests can be made with apple leaves of standard size, and the results are in general agreement with those of field experiments.

R. H. GRIFFITH.

Petroleum products as insecticides. INMAN.—
See II.

PATENT.

Plant disinfectant and its manufacture. R. ZELLMANN and D. LAMMERING, ASSTS. to CHEM. FABR. VON HEYDEN (U.S.P. 1,694,497, 11.12.28. Appl., 26.12.25. Ger., 4.10.24).—Metallic copper is distributed on a finely-divided carrier; e.g., lime water is added to a solution of ferrous and copper sulphates to give reduced copper on a ferric hydroxide-gypsum carrier.

C. HOLLINS.

XVII.—SUGARS; STARCHES; GUMS.

Diffusion experiments with dried beet slices. V. STANEK and K. SANDERA (Z. Zuckerind. Czechoslov., 1929, 53, 525—534).—Beet slices dried at Eynsham by the Oxford process were extracted in a laboratory diffusion battery comprising 16 water-jacketed cells, designed to reproduce the conditions of large-scale working. The slices were about 1 in. long on the average, and contained 6.9% of moisture, 66% of sucrose (polarisation), and 1.01% of invert sugar. The diffusion operation presented no great technical difficulties. The charge of dry slices per cell was 200 g. (corresponding to 80 kg. of fresh slices per hectolitre), and successive cells were charged at intervals of 8 min., so that the slices were in battery for about 128 min. Circulation of juice was maintained by pressure, the fresh water being introduced at 85°, whereby the temperature of the end five cells was kept at 80—83°. With a juice draw-off of 180 c.c. per cell the density attained was 52.9° Brix. The purity quotient of the juice was 88.7—88.9%. Its composition, calculated on 100 pts. of sucrose, showed no striking difference from that of ordinary diffusion juice in respect of ash, total nitrogen, or nitrogen as ammonia, amides, or betaine. The amount of protein nitrogen was relatively low, representing about 3—5% of that in the original slices, whilst in the diffusion of fresh slices about 20% of the total passes into the juice. The juices from the dried slices were rather acid, 100 c.c. requiring 5.2 c.c. of N-sodium hydroxide, and consequently some inversion occurred during diffusion so that the final juices contained about 2 pts. of invert sugar per 100 pts. of sucrose. About 4% of the sucrose remained in the exhausted slices, but this abnormally high loss may be attributable to the thickness of the slices, which in the swollen state ranged up to 7 mm.

J. H. LANE.

Treatment of beets by De Vecchis' process. J. PROCHÁZKA (Z. Zuckerind. Czechoslov., 1929, 53, 453—456).—Juices obtained by extraction of dried beets are not of higher purity than those from fresh beets. In climates where beets deteriorate rapidly in the ground after a certain stage, desiccation of the beets at this stage might avoid serious losses of sugar, but the necessity of drying the whole crop within a short time involves a heavy outlay in drying plant, which would probably outweigh the advantages of the process in other directions.

J. H. LANE.

Spontaneous decomposition of sugar-cane molasses. C. A. BROWNE (Ind. Eng. Chem., 1929, 21, 600—606).—Periodical analyses of two samples of sugar-

cane molasses, which had undergone spontaneous deterioration during 14 years, showed progressive lowering of polarisation, constant decrease in sucrose, slow irregular increase in invert sugar, constant decrease in total sugars after inversion, and constant increase in organic non-sugars. Considerable quantities of formic and acetic acids and of organic colloidal substances both nitrogenous and non-nitrogenous were present. These organic colloids had a carbon content of approx. 52%. No micro-organisms could be detected in the molasses and the spontaneous decomposition is believed to be a retarded "froth" or "hot-room" fermentation purely chemical in nature. The chief type of reaction appears to be the formation of unstable compounds (glucic acid etc.) by the action of the lime used in clarification on the reducing sugars of the juice, but the reaction of reducing sugars with the amino-acids present undoubtedly also plays a part.

W. J. BOYD.

Syrup filtration in making white sugar using kieselguhr. T. VAN DER LINDEN (Archief Suikerind. Nederl.-Indië, 1928, Afl. No. 4, 170—175).—Syrup from the evaporators was heated to 85—90° and passed through Kroog filter-presses the cloths of which had been pre-coated with "Hyflo-supercel" grade of kieselguhr. A clear filtrate, lighter in colour than that obtained by the Bach process, was obtained; the consumption of kieselguhr was about 0.6% of the cane.

J. P. OGILVIE.

Effect of activated carbon and of sulphur dioxide combined on juices etc. G. MEZZADROLI and E. VARETON (Ind. Sacch. Ital., 1928, 21, 491—501, 545—554).—Heavy sulphitation of cane and beet juices even at 85° followed by the addition of "Norit" does not cause inversion. Such combined treatment is advantageous, especially with dilute juices, the surface tension, e.g., being diminished to a greater extent than when they are used separately. Decolorisation is permanent, and is not diminished when the juice is made alkaline.

J. P. OGILVIE.

[Cane] milling-diffusion process in Egypt. F. MAXWELL (Internat. Sugar J., 1929, 31, 186—189).—Juice extracted by two 3-roller mills is measured, tempered, and heated; it then passes up through a "meichage" cell filled with bagasse from the mills, and from thence into the Naudet battery of diffusion cells, which it finally leaves in a clear condition ready for the evaporators. Bagasse discharged from the final cells is further expressed in mills, and finally sent to the furnaces. An extraction of juice from the cane of about 98 is obtained in this process employing only four 3-roller mills.

J. P. OGILVIE.

Keeping quality of white sugars. P. HONIG (Internat. Sugar J., 1929, 31, 214).—Sugar should leave the centrifugals with at most 0.6% of water, otherwise it must be passed through a dryer. On leaving the granulators, it should be allowed to cool in a sieving installation so that it comes into contact with the atmosphere at its prevailing water content. Drying and sieving plants should be installed apart from the factory, not in an atmosphere saturated with water. In this department the control of the moisture of the air is as important in the store. The effect of micro-organisms in sugar

deterioration has been over-emphasised, the contents in water and invert sugar, and the p_H being the three main factors.

J. P. OGILVIE.

PATENTS.

Purification of sugar juice. AKTIEBOLAGET SEPARATOR (B.P. 287,526, 16.3.28. Swed., 23.3.27).—The sugar juice is clarified in the usual manner (heat and chemical precipitation) and then most of the sediment is allowed to separate by gravity settling. Clarification of the juice is completed by treatment in a centrifuge of the non-perforated bowl type, action being discontinued at intervals for the removal of sediment from the bowl.

E. B. HUGHES.

Removal of hydrochloric acid from sugar solutions. A. CLASSEN (B.P. 309,896, 17.1.28).—For the removal of hydrochloric acid, particularly residual traces, from sugar solutions such as those obtained in the acid saccharification of wood, the solutions are distilled, preferably under reduced pressure, in presence of solid grains or pieces of quartz or other natural siliceous matter, so that by the end of the distillation the syrupy liquid is distributed over the surface of the grains.

J. H. LANE.

Treatment of saccharate scums. RAFFINERIE TIRLEMONTAISE SOC. ANON. (B.P. 290,265, 21.1.28. Ger., 12.5.27).—Carbonation scums obtained in ordinary sugar manufacture or saccharate scums obtained in the desaccharification of molasses are separated from their respective liquors by centrifuges in which the centrifugal force is increased to 1500 or more times the force of gravitation. The moisture content can thus be reduced to 15–20%, compared with 50% attainable by means of filter presses.

J. H. LANE.

XVIII.—FERMENTATION INDUSTRIES.

Phosphatases of malt. H. LÜERS and L. MALSCH (Woch. Brau., 1929, 46, 143–146, 153–157).—Four phosphatases are present in barley, and during malting increase about tenfold in amount. Glycerophosphatase and phytase are markedly, and saccharophosphatase slightly, restricted during kilning, nucleotidase being little affected. Their temperature and reaction optima are: glycerophosphatase 36°, p_H 5.2; nucleotidase 49°, p_H 5.6; phytase 48°, p_H 5.2–5.3; saccharophosphatase 41.5°, p_H 6.0. Only in the case of phytase and at low concentration is the velocity of hydrolysis proportional to the amount of enzyme. Relatively long flooring at low temperature increases the phosphatase content of malt; high floor temperatures have the reverse effect. The enzymes may be separated by fractional adsorption on alumina.

F. E. DAY.

Biological condition of "bottich"-sediment yeast. Usefulness of pumping-off wort from the "bottich." F. WINDISCH (Woch. Brau., 1929, 46, 183–187, 197–200).—The practice of some bottom-fermentation breweries of removing the fermenting wort from the yeast which first settles, on account of the supposed impurity and lack of vitality of the yeast, is shown to be without justification. In a test, 44% of the yeast present at pumping-off after 1 day was in the sediment and would be rejected. Comparison, with several races, of the sediment-yeast with that centrifuged

from suspension in the fermenting wort showed that the former fermented cane sugar or wort more rapidly than the latter, and gave a slightly higher degree of fermentation. Both small-scale and brewery fermentation was checked by pumping-off the wort, and the final degree of fermentation was lowered. The bearing of wort turbidity and pumping-off on fermentation is discussed.

F. E. DAY.

Importance of hop-tannin for wort and beer. H. BILGRAM (Woch. Brau., 1929, 46, 137–141).—Three brewings, employing hops, hop extract, and tannin-free hop extract, respectively, are compared. It is concluded that the tannin assists and increases precipitation of the more complex proteins and imparts a distinctive hard bitterness which is preferred by some. The addition of hop-tannin to beer precipitates a considerable amount of protein, but though 0.05 g./litre reduced the nitrogen in a beer by 10.9 mg./100 c.c., it did not reduce the liability to turbidity on pasteurisation.

F. E. DAY.

Artificial acidification of mashes and worts. F. EMSLANDER (Woch. Brau., 1929, 46, 157–159).—Laboratory mashes and fermentations confirm the conclusions of Windisch, Kolbach, and Banholzer (B., 1929, 69) that increase in acidity during mashing leads to higher nitrogen in the resulting beer. The technical application is discussed.

F. E. DAY.

Phosphoric acid and iron contents of Rheingau musts and wines. MUTH and VOIGT (Landw. Jahrb., 1928, 68, Suppl. I, 422–426; Chem. Zentr., 1929, i, 160).—The iron content of the musts was 10.2–20.9 (average 13.93) mg. per litre, falling after 6 weeks to 11.12 and after 12 weeks to 7.85 mg. per litre. The phosphoric acid content of the must and wine, about 20% of the total ash, was 0.48–0.7 (average 0.62) g. per litre. The decrease during the principal fermentation was 17%, and after 12 weeks 20.7%.

A. A. ELDRIDGE.

Occurrence of invertase in must and wine. C. VON DER HEIDE and H. MÄNDLEN (Z. Unters. Lebensm., 1929, 57, 13–36).—Small quantities of invertase occur in grapes and must. The invertase produced by the yeast passes into the wine, which contains relatively much invertase when new. The invertase content decreases gradually and is usually absent from wine over five years old.

W. J. BOYD.

Sweet musts. KOCHS (Landw. Jahrb., 1928, 68, Suppl. I, 364–366; Chem. Zentr., 1929, i, 160).—Apple must contains alcohol 0.16, extract 11.89, sugar 8.71, sugar-free extract 3.28, malic acid 0.714%. Grape must contains alcohol 0.19, extract 17.13, sugar 13.53, sugar-free extract 3.66, acid 0.873%. The musts contain more acid and sugar-free extract than the wines.

A. A. ELDRIDGE.

Use of alkali sulphites in wine manufacture. J. H. FABRE and E. BRÉMOND (Ann. Falsif., 1929, 22, 262–271).—Fermentations were conducted on musts prepared from dried grapes to which were added (1) sulphurous acid alone, (2) ammonium bisulphite, (3) sulphurous acid and diammonium phosphate, (4) sulphurous acid and disodium phosphate. The quantities added corresponded to 25 g. of sulphurous acid, 6.02 g. of ammoniacal nitrogen, and 6.65 g. of phosphorus per

hectolitre. Those with added ammonia (samples 2 and 3) fermented most rapidly, the yeast utilising the added ammonia but not the phosphate. With fresh musts fermented on an industrial scale, with similar additions, except that the amount of phosphorus was doubled by the use of monophosphates, there was little difference in the fermentation rates, sample 2 fermenting most slowly. As before, the added nitrogen was utilised but not the phosphorus. It is concluded that ammonium sulphite is suitable for use in wine manufacture, being without ill effect on yeast or wine. Phosphates appear to be of no value as yeast nutrients, and in this case appeared to favour the development of "casse ferrique" which subsequently appeared in (3) and (4) of the second series, which contained about 0.04 g./litre of iron.

F. E. DAY.

General action of very hard frosts on the chemical composition of wines. ASTRUC and CASTEL (Ann. Falsif., 1929, 22, 272—275).—Wines which were partially frozen during the severe frost of the early part of this year have suffered an appreciable loss of acidity and extract, presumably by abnormal separation of potassium bitartrate. Analyses, of which three sets are quoted, show that this occurred mainly after December 26, 1928, and attention is drawn to the resulting raising of Blarez' and the alcohol/extract ratios and lowering of the Halphen and Roos ratios. Since these ratios are used to form an opinion on the genuine or other character of wines, the possibility of their alteration by freezing must not be overlooked.

F. E. DAY.

Test for cider and other fruit juice in wine. J. WERDER (Ann. Falsif., 1929, 22, 260—261). **Conversion of dibenzylidenesorbitol into sorbitol hexaacetate.** C. ZACH (*Ibid.*, 261—262).—Sorbitol is present in most fruits of the natural order *Rosaceæ* and absent from grapes. The suspected wine is fermented as completely as possible, treated with animal charcoal, concentrated to a syrup, and a few drops of benzaldehyde and 1 c.c. of 1:1 sulphuric acid are added. After well shaking, the test is stood aside for at least 10 hrs., when dibenzylidenesorbitol separates if cider is present, and on dilution remains insoluble as a white flocculent precipitate. For further identification this may be hydrolysed by sulphuric acid, the benzaldehyde removed by ether and the sulphuric acid by baryta. The sorbitol obtained by evaporation is treated with 0.5 c.c. of acetic anhydride and a drop of pyridine for 1 hr. on the boiling water bath, and the hexa-acetyl derivative crystallised from hot water. If necessary, it may be purified by washing its solution in ether with 10% soda and water.

F. E. DAY.

Examination of damaged wines. L. ROOS (Ann. Falsif., 1929, 22, 166—169).—Wines attacked by *Mycoderma vini* may exhibit the characteristics of watered wine, for both the alcohol content and acidity are lowered. A red wine with a surface of 30 cm.²/litre was inoculated with a trace of *Mycoderma vini* and on analysis for alcohol content, and for acidity on the original wine and before and after 7 and 11 days, respectively, had alcohol 8.6, 7.7, and 6.65; total acidity 5.0, 4.8, 4.7; volatile acidity 1.28, 1.18, and 1.18.

D. G. HEWER.

Detection of isopropylalcohol in brandy by Griebel's micro-beaker method. F. WEISS (Z. Unters. Lebensm., 1929, 57, 45—48).—The alcohol is distilled from 1—5 c.c. of the brandy and 0.1 c.c. of the distillate is placed in a flask containing 5 c.c. of water and 8—10 drops of 50% chromic acid solution. 3 c.c. of the liquid are slowly distilled into a Reichert-Meissl flask containing 2.5 g. of 50% silver nitrate solution and 1 g. of a 30% sodium hydroxide solution in 10 c.c. of water. The mixture is boiled for 4 hrs. under reflux, cooled, and kept for 2—3 hrs. Then 1 c.c. of the liquid is distilled off and tested for acetone by Griebel's micro-beaker method (cf. B., 1929, 121), using *m*-nitrophenylhydrazine. If the brandy is free from isopropyl alcohol no crystals are formed in the drop of reagent unless the last traces of acetaldehyde have not been removed by the silver oxide, but the acetaldehyde derivative is easily distinguishable from the acetone compound by its crystalline form. If 5% or more of isopropyl alcohol is present in the brandy the final distillate must be diluted in order that the characteristic form of the crystals may be seen. No genuine brandy was found to give a positive test for isopropyl alcohol, 1% of which can be satisfactorily detected by this method.

W. J. BOYD.

Composition of fusel oils which have been separated as vapour. B. LAMPE (Z. Spiritusind., 1929, 52, 190).—The percentages of amyl alcohol obtained by fractional distillation from seven samples of fusel oil are quoted, and comparison of these with the results of rough commercial tests shows the latter to be misleading. Since two samples which had been separated as vapour contained 42.6 and 46.6% of amyl alcohol, against 50—63.75% in five samples separated as liquid, there appears to be no ground for supposing that the former separation yields fusel oil richer in amyl alcohol.

F. E. DAY.

Determination of nitrogen. LUNDIN and ELLBURG.—See VII. **Inoculation of milk.** WOLFF.—See XIX.

PATENTS.

Recovery and drying of solid matter from distillery wash and other liquids. B.W.A. and J. L. WATNEY (B.P. 312,433, 22.3.28).—The solid matter is separated from the liquid by a horizontally inclined oscillating or revolving sieve and discharged into a tank in which it is agitated with water. The sludge is pumped into a filter press and the separated solid matter conveyed to a steam-heated dryer consisting of a number of horizontally inclined units which are alternately oppositely inclined, and enclosed in a perforated metal or wire-gauze cover.

C. RANKEN.

Separation of gases (U.S.P. 1,706,707).—See VII. **Extraction of oils (U.S.P. 1,698,294).**—See XII.

XIX.—FOODS.

Colorimetric determination of ergot in flour. F. S. OKOLOV (Z. Unters. Lebensm., 1929, 57, 63—71).—The colorimetric method described is based on that of Hofmann (Arch. Pharm., 1885, 223, 828). The disturbing effect of the colouring matter of the flour is avoided by concentrating the ergot by flotation on a mixture of 500 c.c. of chloroform and 60 c.c.

of alcohol. The colouring matter is extracted from the ergot fraction by shaking it with a mixture of ether and 35% sulphuric acid, and again removed from the ether by shaking with sodium bicarbonate solution. The coloured solution so obtained is compared with standard carmine solutions in a Walpole comparator (cf. Michaelis, "Praktikum der physikal. Chem."), using tubes containing alkaline methyl-orange solution to compensate for the varying quantities of yellow pigment derived from the flour. In the case of wheaten flour concentration by flotation on chloroform is inapplicable, but a modified method is described in which the flour is extracted with a larger quantity of sulphuric acid present. The standard carmine solutions must be specially prepared for the particular method used or corrections must be applied. Prolonged storage of the flour does not influence the results to an important extent.

W. J. BOYD.

Serological determination of ergot in flour. F. S. OKOLOV and I. G. AKINOV (Z. Unters. Lebensm., 1929, 57, 72—76).—Rabbits were immunised to aqueous ergot extracts prepared by extracting 1 pt. of ergot with 10 pts. of normal saline. An antigen without marked poisonous properties was prepared by extracting the material first with water for 24 hrs., drying it, and then extracting it with alkaline normal saline for 24 hrs., the extract being finally neutralised and filtered. By the method of Uhlenhut slightly modified a titre of 1:2000 was obtained which was further raised to 1:20,000 by Raishi's method of repeating the immunisation after two months or more. The precipitin reaction was applied by means of the ring test, using 0.2 c.c. of immune serum to which 0.5 c.c. of the extract under examination was added. The tubes were incubated for 25 min. at 37° and 35 min. at room temperature. The extract of the flour was prepared by extracting 20 g. of the material with 100 c.c. of normal saline for 24 hrs., a control extract being made at the same time from ergot-free flour. The specificity of the reaction in relation to the seeds of various weeds was studied, but although non-specific reactions were obtained with certain of these they were not detectable with flour containing only 3% of the seeds. The development of the reaction as a quantitative method is being continued.

W. J. BOYD.

Detection of oxygenated salts in flour. NEUMANN and SCHEYER (Landw. Jahrb., 1928, 68, Suppl. I, 330—331; Chem. Zentr., 1929, i, 164).—For the detection of per-salts the benzidine reaction is employed; for bromates, reduction to bromide and treatment with chlorine; and for oxidising agents generally the oxidation of hydrogen iodide.

A. A. ELDRIDGE.

Distinguishing wheaten flour and bread from those of rye. M. WAGENAAR (Z. Unters. Lebensm., 1929, 57, 37—45).—Microscopical methods involving the use of polarised light are described.

W. J. BOYD.

Rice husks in bran and sharps. A. J. AMOS (Analyst, 1929, 54, 332—333).—Rice husks (the glumes and palea of the fruit) are light brown, stiff, and hard, with dull outer and shiny inner surfaces. A normal bran has a fibre content of 7—10% and sharps 4.5—6.5, but rice husks contain about 40% of fibre. Rice

husks may readily be detected under the microscope after boiling the sample with chloral hydrate solution, for the cells of the outer epidermis are arranged in longitudinal rows, are square in general outline, and have very sinuous side-walls. Dagger-like hairs or hair scars are also to be seen.

D. G. HEWER.

Milk of East Friesian milch sheep during lactation. A. BURR (Landw. Jahrb., 1928, 68, Suppl. I, 178—179; Chem. Zentr., 1929, i, 163).—The milk of a 4-year-old sheep contained 6.44%, and of a 1-year-old sheep 5.35%, of fat. The milk was high in dry matter; the acidity, density, and refractive index of the fresh milk were higher than for cows' milk. The lactose was at first 5%, but fell to 4% shortly before drying-off.

A. A. ELDRIDGE.

Relation of food to the composition of milk. R. O. DAVIES and A. L. PROVAN (Welsh J. Agric., 1928, 4, 114—121).—Grazing generally increases the total protein, casein, total phosphorus, inorganic phosphorus, and calcium in the milk; the changes depend on the previous winter feeding. The flow of milk increased simultaneously.

CHEMICAL ABSTRACTS.

Inoculation of pasteurised milk [with lactic acid bacteria]. A. WOLFF (Milch. Zentr., 1928, 57, 277—283, 293—297, 341—346, 357—364; 58, 105—110, 157—160).—The bacterial flora of pasteurised milk can be favourably influenced by addition of lactic acid bacteria before or after pasteurisation. Pasteurised milk in practice contains abundant lactic acid bacteria, but pure lactic acid fermentation seldom develops as the surviving strains are attenuated. Addition of raw, sour milk cultures before, or of heated cultures after, pasteurisation produces a suppression of the spore formers, but the best results are obtained by breeding a resistant strain of the common lactic acid bacteria and adding it in pure culture to the milk which is to be pasteurised. Such a strain must be of proved purity and resistance to temperature changes in milk fermentations of definite age. Addition of raw cultures after pasteurisation is dangerous, as the bacteria develop too quickly.

W. J. BOYD.

Gerber's acid-butyrometry as a scientific method of examination of milk. G. KOESTLER and W. LOERTSCHER (Z. Unters. Lebensm., 1929, 57, 48—59).—The values obtained by means of Gerber's acid butyrometer agree much better with the values obtained by exact methods such as the Gottlieb-Röse and Schmid-Bondzynski methods, when the reading is assumed to be g. of fat/100 c.c. of milk instead of g. of fat/100 g. of milk. Corrections for use when the latter reading is adopted are given. The method of operation is described in detail.

W. J. BOYD.

Hoyberg's method for determining fat in milk and cream. A. BURR (Landw. Jahrb., 1928, 68, Suppl. I, 179; Chem. Zentr., 1929, i, 164).—The advantages over Gerber's more rapid method are discussed. The accuracy attainable is: for milk 0.1%, for cream 0.5%.

A. A. ELDRIDGE.

Refraction of milks low in solids-not-fat. G. D. ELDON and J. R. STUBBS (Analyst, 1929, 54, 318—320).—A continuation of the work on milk analysis (cf. B., 1928, 346) shows that of 2850 samples examined in 1928, in every case except 2 (which were sour milks) a low

value for solids-not-fat corresponds with a low refraction, so that all such low values are due (a) to watering, (b) to the fact that milks naturally low in solids-not-fat do not give a normal refraction of 38 or above, or (c) to a combination of the two causes. D. G. HEWER.

Water content of cheese. A. BURR (Landw. Jahrb., 1928, 68, Suppl. I, 172—173; Chem. Zentr., 1929, i, 164).—Values (%) for fresh and ripe cheese, respectively, are: Limburg 48.47—61.22, 48.90—60.93; Romadur 46.53—63.02, 54.82—61.58; Camembert 56.00—69.55, 54.30—68.12; Brie 42.80—69.82, —; Tilsit 43.73—58.70, 35.90—55.08; Gouda 46.97—56.83, 33.58—56.27; Edam 44.80—56.53, 38.00—45.17; Holstein 35.80—58.00. A. A. ELDRIDGE.

Analytical differences between acid and rennet caseins. C. PORCHER and J. BRIGANDO (Ann. Falsif., 1929, 22, 153—163; cf. B., 1928, 543).—The large number of "acid caseins" found in commerce are produced owing to deviations from the method of preparing pure casein which calls for a slow precipitation to bring about controlled but total solubility of the di- and tri-calcium phosphates and of the casein-calcium, a low temperature, and the use of very dilute acid to keep the reaction at p_H 4.6. Lactic caseins result from acidity induced by lactic acid, and acid caseins by the use of mineral acids. Rennet casein is a double calcium salt—calcium paracasein with di- and tri-calcium phosphates—the mineral ash of which should, in practice, be kept at 7.2—7.5%. It results from the action of ferments on the calcium caseinate, the paracaseinate formed carrying down at the same time the insoluble calcium phosphates. D. G. HEWER.

Formation of acid and alcohol in fruit residues and its significance in the nutrition of utility animals. E. CRASEMANN (Mitt. Lebensm. Hyg., 1928, 19, 371—378; Chem. Zentr., 1929, i, 455).—The formation of acetic acid is wasteful, that of lactic acid desirable; the formation of alcohol, for which the material is suited, is not undesirable. The residues are best preserved with sodium benzoate. A. A. ELDRIDGE.

Detection of regenerated preserved peas. O. LÜNING and E. BEYER (Z. Unters. Lebensm., 1929, 57, 76—85).—Fresh green peas which have been preserved directly by heat sterilisation in closed containers can be distinguished from dried fully-ripe peas which have been swelled by soaking in water before being introduced into the containers, by their moisture content. The moisture content of the former lies between 79.0% and 85.6%; that of the latter between 72.7% and 77.6%. The observation of Froidevaux (B., 1927, 91) that the former have a lower content of water-soluble nitrogenous substance calculated on the dry weight was not confirmed. W. J. BOYD.

Determination of the degree of ripeness of peas. E. LASAUSSE, B. GUÉRINHAULT, and PELLERIN (Bull. Sci. pharmacol., 1928, 35, 575—579; Chem. Zentr., 1929, i, 455).—An attempt to discover chemical differences in the preserved products. A. A. ELDRIDGE.

Examination of marzipan. J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1929, 57, 85—90).—Different kinds of almond oil give the Bellier and the Kreis reactions in different degrees. The intensities of these reactions diminish with ageing of the material, a

negative result being obtained for both after six months. The Kreis reaction is, however, useful in detecting apricot-kernel oil in marzipan. The latter should be considered adulterated when the Kreis reaction is equal in intensity to that given by a mixture of 15% of apricot-kernel oil in an almond oil which gives a negative result by itself. W. J. BOYD.

Detection of prohibited vegetable and coal-tar colours in foodstuffs. J. R. NICHOLLS (Analyst, 1929, 54, 335; cf. B., 1927, 922).—Test 16 has been found to be fallacious and should be deleted, and test 17 has been modified so that the excess of permanganate is reduced after the oxidation by the addition, drop by drop, of sodium sulphite solution. D. G. HEWER.

Packaging and quick-freezing perishable flesh products. II. Packaging flesh products for quick freezing. C. BIRDSEYE (Ind. Eng. Chem., 1929, 21, 573—576).—Causes of deterioration of flesh products during cold storage, requirements of the manufacturer, distributor, and consumer, and relative merits of various kinds of packaging materials and shipping cases are discussed. The advantages of packaging flesh goods before freezing are emphasised. W. J. BOYD.

Relative vitamin-A content of four oriental foods. Relative content of water-soluble vitamin-B in thirty oriental foods. Relative water-soluble vitamin-C content of nine oriental fruits and vegetables. Certain proteins added to mung beans, or to white or red *Sorghum vulgare*, increase the fertility of mice. H. E. SHERMAN (Philippine J. Sci., 1929, 38, 1—7, 9—36, 37—46, 47—67).

Chemical analysis of thirty-seven oriental foods. Calcium, iron, and magnesium content of sixteen Chinese foods. H. E. SHERMAN and T. C. WANG (Philippine J. Sci., 1929, 38, 69—79, 81—82).

Preserved-milk products etc. FINCKE.—See XII.
Composition of mangolds. FAGAN and WATKIN.—See XVI.

PATENT.

Production of frozen food compositions HOMPFEZE CORP. (B.P. 302,883, 7.6.28. U.S., 23.12.27).—In the ice-cream mix (particularly water-ices) a small quantity of agar-agar is used in order that overrun may be obtained on freezing. The agar is boiled in about 200 times its weight of water until it darkens, is then mixed with the flavour and a boiling solution of sugar, and the mixture is maintained at about 82° for some time. E. B. HUGHES.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Nitrobenzaldehyde as reagent for organic medicinal substances. H. W. VAN URK (Pharm. Weekblad, 1929, 66, 429—435).—The colorations obtained by evaporating mixtures of the commoner organic medicinal substances with alcoholic solutions of *o*-, *m*-, and *p*-nitrobenzaldehydes in presence of sulphuric acid are tabulated. The three isomerides show little difference in behaviour in general, though in some cases only the *o*-compound reacts. S. I. LEVY.

Preparation of alcoholic potash solution and a test for aldehyde in chloroform using potassium hydroxide. N. VALJASCHKO (Pharm. J. [Russia],

1928, 34—35; Chem. Zentr., 1929, i, 457).—The development of a yellow colour by a particle of potassium hydroxide may be due to the presence of alcohols in the chloroform. A. A. ELDRIDGE.

Detection of carbon tetrachloride in chloroform. J. SIVADJIAN (J. Pharm. Chim., 1929, [viii], 9, 434—437).—Two c.c. of chloroform and 0.5 c.c. of concentrated sodium hydrate solution are carefully added to a few c.c. of a 1% alcoholic solution of pyrocatechol. A pinch of copper powder is added and the mixture is heated rapidly and boiled for 8—10 sec. It is then cooled, 1 c.c. of concentrated hydrochloric acid and 1 c.c. of water are added, and, after shaking, the liquid is filtered. If the chloroform contains not more than 0.25% of carbon tetrachloride the colour of the filtrate is pale yellow to yellowish-brown, with 0.25—0.50% yellowish-orange, and with more than 0.50% it is reddish-purple. Pure chloroform gives no colour. E. H. SHARPLES.

Colour reactions of sesamin. H. KREIS (Mitt. Lebensm. Hyg., 1928, 19, 385; Chem. Zentr., 1929, i, 458).—Sesamin (0.1 g. in 100 c.c. of petroleum, b.p. 50—100°) gave the following colorations: nitric acid, with or without sulphuric acid, yellow; acetic anhydride and sulphuric acid (Bömer and Winter), red, then green; pyrogallol and hydrochloric acid (Tocher), violet; vanadium and sulphuric acid (Bellier), dark green; formaldehyde and sulphuric acid (Bellier), violet; stannous chloride solution (Soltsien), red; sulphuric acid and hydrogen peroxide (Kreis), green. A. A. ELDRIDGE.

Preparation and probable constitution of dermatol. I. TELETOV (Pharm. J. [Russia], 1928, 19—23; Chem. Zentr., 1928, ii, 2353—2354).—Bismuth hydroxynitrate is employed for the preparation of dermatol; the constitution proposed by Fischer and Grützner (A., 1894, i, 416) is confirmed. A. A. ELDRIDGE.

Macedonian opium. A. VRGOČ (Arch. Pharm., 1929, 267, 352—370).—The culture, harvesting, and marketing of Macedonian opium is considered in detail. Analyses of representative samples from twelve districts indicate that the morphine content is usually above 10% and may reach 16%. A few samples contained adulterants consisting of dried, baked, and ground-up bread, the fleshy parts of apricots or melons, egg shells, boiled starch, or, less frequently, ground dates. S. COFFEY.

Essential oil of *Salvia officinalis*, L. V. MORANI (Nuovi Ann. Agric., 1928, 7, 25—40).—The oil of *Salvia officinalis* grown in the littoral regions of the Mediterranean exhibits marked differences in composition, according as it is obtained in spring and summer from the flower heads alone or from the whole flowering plant, or in winter from the non-flowering plant. The oil from the flowering plant contains cineole 32—35, sesquiterpenes about 30, borneol 9—14, camphor and thujone 5—10, esters 2%, whereas the winter oil shows cineole 13—20, sesquiterpenes about 20, borneol 7.5—12, camphor and thujone 20—32, esters 2.2—3.7, terpenic hydrocarbons about 15%. These oils differ from those of other origins by their comparatively low solubility in alcohol and their higher refractive indices. As with the oil from the Eastern Mediterranean attributed

to *S. triloba*, this Italian oil is levorotatory, and thus differs from the oil of *S. officinalis* from Dalmatia, Spain, France, Germany, etc.; the camphor in the Italian oil is optically inactive. During the winter the oil changes little in composition or properties, but the yield is less in the morning than in the evening and at the same hour is greater in clear than in cloudy weather. T. H. POPE.

Essential oils of *Magnolia grandiflora*, L. G. TOMMASI (Riv. Ital. Ess. Prof., 1928, 10, 156—160; Chem. Zentr., 1929, i, 401).—Oil from the leaves (two specimens), flowers, young fruiting plants, and bark gave the following values: d_{15}^{20} 0.915, 0.92, 0.902, —, —; $[\alpha]_D^{20} + 1^\circ 32'$, $+ 1^\circ 46'$, $+ 3^\circ 20'$, $+ 1^\circ 35'$, $+ 1^\circ 42'$; $[\alpha]_D$ after acetylation $+ 5^\circ 16'$, $+ 5^\circ 36'$, $+ 6^\circ 50'$, —, —; n_D^{20} 1.5014, 1.5020, 1.5250, 1.5080, 1.5060; f.p. -16° , -16° , -16° , —, —; b.p. 170—265°, 165—275°, 155—260°, —, —; acid value 2.10, 1.90, 1.20, 1.75, 1.80; ester value 30.24, 32.20, 54.20, 32.65, 32.55; acetyl-saponif. value 51.00, 55.50, 75.50, 53.70, 52.20. A. A. ELDRIDGE.

Report of the Essential Oil Sub-Committee to the Standing Committee on Uniformity of Analytical Methods. II. Physical constants (Analyst, 1929, 54, 335—338).—Apparatus and methods of procedure for f.p. and m.p. are given, with a special method for otto of roses. For determining the b.p. the use of a standardised apparatus and conditions is considered necessary. The apparatus is illustrated and dimensions are given, together with formulæ for temperature corrections. (Cf. B., 1927, 506.) D. G. HEWER.

Adulterations of essential oils, perfumes, and drugs. SCHIMMEL & Co. (Ber. Schimmel, 1928, 39, 41, 45, 50, 55, 61, 71, 83, 104, 113, 114, 118, 129; Chem. Zentr., 1929, i, 311—312).

Alkaloid test for tannins. FEAR.—See XV.

PATENTS.

Preparation of monoalkyl ethers of proto-catechuic aldehyde in addition to vanillin. F. BOEDECKER (B.P. 309,929, 15.12.27. Cf. B.P. 285,156; B., 1928, 360).—*iso*Chavibetol, obtained with vanillin from safrole according to the process of the prior patent, is ethylated, and the resulting 4-methoxy-3-ethoxypropenylbenzene, m.p. 50—51°, b.p. 145—148°/14 mm., is demethylated by heating with methyl-alcoholic or alcoholic sodium hydroxide at 140—150° in a closed vessel. The product is mainly 4-hydroxy-3-ethoxypropenylbenzene, b.p. 145—150°/13 mm. (benzoate, m.p. 89°), which is oxidised in the usual way to the aldehyde. C. HOLLINS.

Preparation of 1-phenyl-2-methylaminopropanol-1 [β -methylamino- α -phenyl-*n*-propyl alcohol]. W., K., L., W., and F. MERCK (E. MERCK) (B.P. 284,644, 12.1.28. Ger., 2.2.27).— α -Bromopropiophenone reacts with aqueous methylamine solution, and the resulting α -methylaminopropiophenone, isolated as hydrochloride, m.p. 188°, or hydrobromide, m.p. 144°, is catalytically reduced to the secondary alcohol (hydrochloride, m.p. 188—189°; hydrobromide, m.p. 200°). C. HOLLINS.

Manufacture of basic derivatives of substituted quinolinecarboxylic acids [anæsthetics]. Soc.

CHEM. IND. IN BASLE, and K. MIESCHER (B.P. 310,074, 22.12.27. Addn. to B.P. 270,339; B., 1928, 730).—The following compounds, prepared by the process of the prior patent, show increased anæsthetic properties: (a) ethers of 2-hydroxyquinoline-4-carboxylic β -diethylaminoethylamide: *cyclohexyl*, m.p. 19°; *p-methoxyphenyl*, m.p. 108°; *n-butyl*, m.p. 64°; *n-propyl*, m.p. 63°; *allyl*, m.p. 57°; *n-amyl*, m.p. 72°; *isoamyl*, m.p. 35°; *n-heptyl*, m.p. 66°; *benzyl*, m.p. 119°; β -*phenylethyl*, m.p. 90°; (b) ethers of 2-hydroxyquinoline-4-carboxylic *N*-ethyl-*N*-(β -diethylaminoethyl)amide (prepared from the 2-*chloro*-compound, b.p. 165°/0.015 mm.); *ethyl*, b.p. 158—160°/0.02 mm.; *isoamyl*, b.p. 165—168°/0.01 mm.; *methyl*, b.p. 150°/0.008 mm.; *n-propyl*, b.p. 155°/0.008 mm.; *n-butyl*, b.p. 163°/0.01 mm.; *n-amyl*, b.p. 175°/0.02 mm.; *cyclohexyl*, b.p. 185°/0.015 mm.; *benzyl*, b.p. 192°/0.01 mm.; (c) ethers of the 4'-methylpiperazide (from 2-*chloroquinoline-4-carboxylic 4'-methylpiperazide*, m.p. 208°): *ethyl*, m.p. 183°; *n-butyl*, m.p. 145°. From 2-*chloroquinoline-4-carboxylic bis*-(β -diethylaminoethyl)amide, b.p. 165—170°/0.01 mm., prepared from the carboxylic chloride and *bis*-(β -diethylaminoethyl)amine, b.p. 105—110°/8 mm., there are obtained the 2-*n-butoxy*- and 2-*ethoxy*-compounds, b.p. 172°/0.008 mm. and 165°/0.01 mm., respectively. 2-*Chloroquinoline-4-carboxylic ϵ -diethylaminoamylamide*, m.p. 55°, gives a 2-*ethoxy*-compound, m.p. 74°; 2-*chloroquinoline-4-carboxylic β -N-piperidylethylamide*, m.p. 147°, gives a 2-*n-butoxy*-compound, m.p. 93°. C. HOLLINS.

Agar product. J. BECKER (U.S.P. 1,712,785, 14.5.29. Appl., 7.12.26).—The liquor obtained by cooking with water at constant temperature and pressure previously soaked and washed seaweed (1 lb. per gal. of water) is filtered, agitated, concentrated to remove excess water, and congealed at about 15°; the product is sized and frozen, after which the ice is melted, excess water is drawn off by suction, and the residue is dehydrated.

L. A. COLES.

Reducing the nicotine content of raw tobaccos, semi-manufactured and finished tobacco products. R. E. ELLIS. From GENERAL-DIREKTION DER OESTERR. TABAKRÉGIE (B.P. 290,204, 2.5.28).—Previously dried tobacco is heated rapidly to and maintained at above 150° until the required quantity of nicotine has been expelled from it. Apparatus is described.

L. A. COLES.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Method of increasing the printing speed of dichromated gelatin. F. J. TRITTON (Phot. J., 1929, 69, 281—285).—The sensitisation produced by the addition of certain metallic salts to the sensitising baths used in conjunction with dichromate-colloid mixtures is due to the tanning action of the insoluble metal hydroxide or chromate formed in the colloid film, and is therefore largely controlled by the p_H at which the metal hydroxide or chromate is precipitated and the p_H of the colloid. The best results are obtained with a metal, the chromate or hydroxide of which is precipitated at a p_H value slightly above that of the colloid, since under these conditions no general precipitation and

consequent hardening of the colloid film can take place. Practical applications, especially with regard to the use of cerium salts, are described. J. W. GLASSETT.

Hypersensitising for extreme speed of exposure. K. JACOBSON (Brit. J. Phot., 1929, 76, 314—315).—Methods of hypersensitising and its practical limitations are outlined (cf. B., 1928, 212). J. W. GLASSETT.

Physical development and nature of the latent image. R. E. OWEN (Phot. J., 1929, 69, 278—280).—The results of A. and L. Lumière and Seyewetz on post-fixation physical development (cf. B., 1924, 579, 732) are confirmed, but their deduction that the latent image cannot therefore consist of metallic silver is shown to be invalid, since these results may be explained by the fact that during fixation the latent-image silver becomes coated with silver sulphide formed from the silver-sodium thiosulphate complex, and will therefore react as silver sulphide in any later treatment. Experiments in confirmation of this showed that the conditions necessary for the deposition of silver upon a fine suspension of silver sulphide in gelatin were the same as those required for post-fixation physical development of the latent image. J. W. GLASSETT.

Developer fog from metal of tanks. J. F. ROSS and J. I. CRABTREE (Brit. J. Phot., 1929, 76, 346—349).—The fogging properties of three tank developers, one of which contained sodium bisulphite, were studied after storage in contact with a number of metals and alloys under the following conditions: in open containers; in closed containers; in open containers with the metals coupled with metallic silver. Metallic zinc differed from the other metals causing fog in that a fogging effect was only produced with the bisulphite developer and was probably caused by the formation of sodium hyposulphite by reaction between zinc and the bisulphite. Copper and its alloys caused strong aerial fogging properties, whilst tin and its alloys caused strong chemical fog. The fogging effects were minimised when closed containers were used, and coupling the metals with silver produced no effect. J. W. GLASSETT.

Magnesium light as a standard source in photographic sensitometry and its relation to the international sunlight standard. J. M. EDER (Z. physikal. Chem., 1929, 141, 321—331).—Light from burning magnesium leaf filtered through solutions of cobalt and copper salts is recommended for photographic sensitometry in the case of both negatives and positives. When the solutions are made up in the proportions given, such light possesses the same spectral energy distribution as that of a full radiator at a "black body" temperature of 5000° Abs. A pure carbon arc closely approximates to magnesium as regards its black-body temperature, and is in practice more convenient. A table is given showing the relation of the colour range of other light sources of different temperatures to that of burning magnesium: this comparison is of importance in determining times of exposure for panchromatic plates and in three-colour photography. Figures are given to show the relative colour sensitivity of a number of different types of paper. F. L. USHER.

Tentative hypothesis of the latent image. II. A. P. H. TRIVELLI (Z. wiss. Phot., 1929, 26, 381—392).—See B., 1928, 212.

PATENTS.

Manufacture of light-sensitive materials. S. SOKAL. From KALLE & Co. (B.P. 311,196, 7.1.28).—An aqueous solution of methyl cellulose is used as the vehicle for light-sensitive substances of the diazo-type. A slightly acid solution of the diazotised amine together with a coupling agent or a diazo compound which bleaches in light is admixed with the aqueous solution of methyl cellulose and coated on a suitable base. In the first case gaseous ammonia may be used as the developer, and in the second an alkaline alcoholic solution of a coupling agent. J. W. GLASSETT.

Photomechanical printing. F. H. HAUSLEITER (B.P. 280,220, 3.11.27. Ger., 4.11.26).—By using a hardening developer the dots or lines of the screen negative are embedded in hardened gelatin, thereby controlling the action of the subsequently applied photographic reducer which is used to etch the dots or lines for correction of tone or colour value. Alternatively, a positive is made by general blackening of the dry plate, resensitisation with potassium dichromate, exposure behind the screen negative, and, finally, treatment with the reducing agent, the action of which is modified as in the first case. J. W. GLASSETT.

Production of natural-colour [photographic] pictures. R. GSCHÖPF (B.P. 311,833, 27.4.28. Addn. to B.P. 279,381; B., 1928, 838).—An ordinary positive of the first part-negative is produced in an emulsion coated on a non-permeable, white, opaque celluloid film, and is converted into any suitable mordant and dyed. The second colour-image is superimposed by the iron-silver process, and the third added in a similar manner. Alternatively, the third image may be produced on a separate stripping emulsion, weakly dyed, transferred in register on to the others, and then further dyed until correct colouring is obtained. J. W. GLASSETT.

Colour photograph and its production. W. V. D. KELLY, Assr. to M. B. DU PONT VITACOLOR CORP. (U.S.P. 1,712,439, 7.5.29. Appl., 24.5.20).—A dye-absorbing image and a reduced silver image are formed in the same colloid film; the former is then dyed and the latter tinted in different colours. J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

Permitted explosives in coal mines. NATHAN.—See II.

PATENT.

Quick-burning delay powder. W. T. INGRAHAM (U.S.P. 1,716,313, 4.6.29. Appl., 8.11.23).—The powder contains a mixture (9:1) of mercury fulminate and black powder. H. ROYAL-DAWSON.

XXIII.—SANITATION; WATER PURIFICATION.

Changes in sulphur compounds during sewage treatment. A. L. ELDER and A. M. BUSWELL (Ind. Eng. Chem., 1929, 21, 560—562).—The sulphur present in sewage is shown to be mainly of non-fæcal origin. During treatment under aerobic conditions there is little change in the total sulphur content, though the immediate oxygen demand observed during experiments on pure compounds indicates that sulphides and thio-sulphates are completely oxidised to sulphates within five days. Under anaerobic conditions sulphates are

reduced to hydrogen sulphide, with possibility of aerial nuisance unless the sewage is kept alkaline and at as low a temperature as possible. C. JEPSON.

Activated carbons and their use in removing objectionable tastes and odours from water. J. R. BAYLIS (J. Amer. Water Works' Assoc., 1929, 21, 787—814).—Chlorine and phenolic substances, or both, may be adsorbed by activated carbon, and filtration through this material is suggested as a final treatment for drinking water in order to prevent the occurrence of any objectionable tastes or odours, of which they are a common cause. The cost of such treatment is estimated to be about \$1.5 per million gallons. Very highly polluted water may be rendered potable by treatment which would include super-chlorination and removal of residual chlorine by carbon, in those regions where, owing to scarcity of supply, the cost of treatment is not a vital factor. C. JEPSON.

Copper from mine waters. EBBUTT and SELNES.—See X. **Determination of nitrates in water.** DE NARDO.—See XVI.

PATENTS.

Sewage-disposal apparatus. M. B. TARK, Assr. to LINK-BELT Co. (U.S.P. 1,714,739, 28.5.29. Appl., 30.3.27).—A settlement tank is divided into a series of narrow compartments, the dividing walls of which are submerged and tapered at the top to facilitate collection of solid matter on the floor of the tank, whence it is removed by a travelling scraper into a hopper situated at the end of each compartment. C. JEPSON.

Sewage-treatment plant. J. T. TRAVERS, Assr. to TRAVERS-LEWIS PROCESS CORP. (U.S.P. 1,715,438, 4.6.29. Appl., 25.3.27).—The plant consists of detritus and sedimentation tanks, the latter being suitable for chemical precipitation, and drainage beds for the disposal of the sludge produced. C. JEPSON.

Manufacture of [veterinary] disinfectants. E. A. GARBE (B.P. 311,920, 25.4.28).—Tobacco is extracted with salt solution, preferably in the presence of sulphur, and, after fermenting for 8—14 days, the extract is mixed with glacial acetic acid and sodium cresolate solution, turpentine oil etc. being added, if desired. The product, diluted with 5—7 pts. of water, is sprayed, alone or together with sulphur dioxide, into stables to prevent the spreading of anthrax, glanders, whitlow, etc. L. A. COLES.

Destruction of vermin and their brood, also injurious germs, parasites, etc. A. VONDRAN (B.P. 311,668, 8.2.28).—Verminous garments, hides, foodstuffs, etc. are treated with a humid atmosphere at a temperature about that of human blood heat, and subsequently with dry air or other gases at 80°.

L. A. COLES.

[Boiler] scale solvent and scaling composition. J. M. COUGHLIN, Assr. to SCALE SOLVENT AND PRODUCTS Co. (U.S.P. 1,715,337, 28.5.29. Appl., 21.1.27).—A solution (*d* 1.40—1.52) containing soluble glass, coffee extract, and a diluent is used.

H. ROYAL-DAWSON.

Oxides of sulphur for disinfecting (B.P. 312,746).—See VII. **Germicides etc.** (B.P. 311,218 and 311,253).—See XI.