BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JANUARY 21, 1927.

I.—GENERAL; PLANT; MACHINERY.

Effect of acids on the mechanical strength of timber. E. A. Alliott (J.S.C.I., 1926, 45, 463-466 T). -The tests were made mainly on pitch pine, teak, and American oak. Strips of $\frac{1}{2}$ in. cross-section and 7 in. span were broken to determine the effect of soaking in water and in sulphuric, acetic, and hydrochloric acids of various strengths for periods up to 130 days. The loss of strength after prolonged wetting is least in the case of teak (7%), and greatest in the case of pitch pine (33%). After soaking in various acids, pitch pine loses a smaller percentage of its strength as compared with its normal strength after wetting with water, though in many cases teak retains the greater actual breaking strength. Teak does not appear to resist hot acids so well as pitch pine. In general, American oak shows up unfavourably for resisting acids. Rough tests with other timbers, such as beech, kauri pine, etc., indicate that they do not approach pitch pine and teak in resistance; lignum vitæ is an exception, but at temperatures above 80° the resinous matter softens, melting at 95°, and the resistance then decreases.

Heat economy of drying with hot gases. W. GRAULICH (Chem.-Ztg., 1926, 50, 921-922).—From thermochemical data, the heat economy of drying material by means of hot gases drawn directly from the fire has been calculated (a) when the gases are passed straight over the material, and (b) when the gases are first used to preheat the material by passing them around the drying container and then backwards over the stream of damp material in such a way that the issuing gases are saturated with water vapour at 80-150°. From the results obtained the efficiency of the second procedure appears to be nearly 20% higher than that of the former. The use of a large excess of air in the furnace results in a greater fuel consumption than the use of little more than the theoretical. It is recommended that the minimum amount of air be used, and that the products of combustion be cooled to 800° immediately after leaving the furnace by addition of the A. R. POWELL. requisite amount of fresh air.

Conduction of heat through powders and its dependence on pressure and conductivity of the gaseous phase. J. ABERDEEN and T. H. LABY (Proc. Roy. Soc., 1926, A, 113, 459-477).—The thermal conductivity of "silox" powder in air, carbon dioxide, and hydrogen over the pressure range 1—760 mm. is expressed by $k = \frac{1}{2}k_0 \log_{10} p/n$, where k_0 is the conductivity, p the pressure of the gas, and n is a constant for each gas. This is not in agreement with Smoluchowski's theory of conduction of heat through powders. The thermal conductivity of silox in a vacuum was determined as 0.7×10^{-5} cal. cm.⁻¹ sec.⁻¹ deg.⁻¹. A powder such as silox appears to possess advantages for insulating liquid oxygen vessels. E. S. HEDGES.

PATENTS.

Fusion furnace. A. F. GORDON and W. H. GROVES, Assrs. to WESTERN ELECTRIC Co. (U.S.P. 1,603,762, 19.10.26. Appl., 27.4.25).—In a furnace for fusing materials, *e.g.*, specimens of refractory clay, the specimens are supported in spaced relationship from the walls of the furnace, in such manner that heat from a combustion chamber below comes in direct and even contact with the material to be fused. C. A. KING.

Vertical disc crushers. E. SYMONS (E.P. 258,471, 25.1.26).—Mechanical improvements to a gyratory disc crusher with a vertical shaft. B. M. VENABLES.

[Gyratory] crusher. B. A. MITCHELL (E.P. 258,875, 22.9.26. Conv., 24.9.25).—A gyratory crusher has the gyrating part supported from the fixed part with adjustment for the distance between the two, and the whole is supported on yielding cushions. The source of power is preferably a self-contained electric motor, and the gyrations are not positively effected, but only induced by a member rotating within the gyrating member being out of balance; the range of the gyrations is, however, positively limited. One of the crushing members may have holes formed in it for the outlet of crushed material. B. M. VENABLES.

Gyratory crusher. E. B. SYMONS, Assr. to SYMONS BROS. Co. (U.S.P. 1,600,780, 21.9.26. Appl., 17.5.24).— A circular opening in the top of a housing, including a gear-case, is provided with a circular closure formed with a bearing surface extending upwards, and lubricating passages are formed within the walls of the housing and the , closure. Unequally-spaced pins are interposed between the housing and the closure, one pin being hollow to connect the lubricating passages.

H. HOLMES.

Grinding, pulverising, or disintegrating mills. W. A. CLOUD (E.P. 260,034, 20.7.25).—The apparatuscomprises a number of rotating discs carrying pins which intercalate with fixed pins. The discs increase in size in succession, and around each grinding zone and at the back of each disc is a clearance space which is at least as great in volume as the preceding grinding zone. In the clearance space the material separates, the fine being carried forward to the next grinding zone and the coarse dropping back into the preceding grinding zone. The air current for propelling the material may be produced by self-contained fan blades. If desired, the apparatus may work drowned in liquid circulating from and to an overhead tank. B. M. VENABLES. 32

Recuperative apparatus. F. A. FAHRENWALD (U.S.P. 1,599,613, 14.9.26. Appl., 28.11.24).—The gas to be preheated or recuperated is caused to flow upwards through conduits located in each of two hollow upright chambers communicating at their upper ends, and means are provided for delivering heated gases to the bottom of one chamber and discharging them from the bottom of the other chamber. H. HOLMES.

Method and apparatus for removing large-sized particles and aggregates from certain finely divided powders and the like. G. GALLIE, B. D. PORRITT, and RESEARCH ASSOC. OF BRIT. RUBBER & TYRE MFRS. (E.P. 260,741, 7.10.25).—Substances such as carbon black are tested for gritty matter by mixing with water in a funnel-shaped vessel in the neck of which is a tensioned screen of the order of 300 meshes/linear inch. Above the screen is a jet, preferably spreading, of water which washes the powder through the screen with sufficient energy to break up aggregates. The apparatus is stated to be also applicable on a commercial scale. (Cf. B., 1926, 935.) B. M. VENABLES.

Drying devices for granular material and the like. G. HILGENBERG (E.P. 260,896, 11.6.26).—A device for drying, e.g., crystalline salts, without damage consists of a number of annular trays supported by a central rotating vertical duct through which hot gases are passed. The crystals are raked gently from tray to tray by fixed deflectors. B. M. VENABLES.

Cyclone separators or dryers. W. R. Wood (E.P. 260,776, 5.11.25).—A cyclone separator is shaped entirely as an inverted cone (*i.e.*, without a cylindrical portion at the top), and from a tangential inlet at the top the gas stream passes below a helical baffle which makes one complete turn (downwards) round the interior of the cone. An interior inverted cone may be provided leaving an annular space (which the helical baffle may completely fill) for the incoming gas. The inner cone may serve as an outlet, its lower end being shaped as a Venturi tube, with the object of gradually taking the spin out of the gas. B. M. VENABLES.

Centrifugal separators. A. W. EMPSON (E.P. 260,397, 21.9.25).—The centrifuge, which is suitable for the dehydration and purification of oil, is constructed so that the fluid is treated first in the zone of maximum centrifugal force, whereby the coarser impurities and any water are disposed of by centrifugal action. The fluid is then caused to flow inwards through a filter of superposed sheets of paper or other suitable material to eliminate colloidal substances and suspended matter.

W. G. CAREY.

Centrifugal separators. E. W. GREEN and H. OGDEN (E.P. 260,071, 15.8.25).—A centrifugal separator for two liquids, which differ only slightly in sp. gr., is constructed so that the actual overflow weirs comprise uninterrupted circular rings at very nearly the same radius, and so that the light liquid is led away from the bowl nearer the axis, and the heavy from the outermost layers, the latter passing through long narrow passages arranged round a conical cap closing the bowl.

B. M. VENABLES.

Separating substances from liquids. W. C. GRAHAM, H. S. RUMSEY, and A. U. WETHERBEE, ASSTS. to

GILCHRIST & Co. (U.S.P. 1,602,014, 5.10.26. Appl., 5.1.25).—Liquid to be clarified is delivered through a central inlet to a separating chamber, and is withdrawn at points radially remote from the inlet. The sediment is removed radially without opposing the flow of the liquid. The scums are removed through the central inlet. H. HOLMES.

Uniformly drying or processing materials. A. E. STACEY, JUN., ASST. to CARRIER ENGINEERING CORP. (U.S.P. 1,599,797, 14.9.26. Appl., 12.8.24).— The material is subjected to successive currents of air or gas of different velocities, and the currents of the highest velocity are caused to impinge on different zones of the material at different times. H. HOLMES.

Apparatus for drying and heating. W. G. GRAVES, Assr. to GRASSELLI CHEMICAL Co. (U.S.P. 1,599,467, 14.9.26. Appl., 31.1.23. Renewed 22.1.26).—A multiple closed-hearth furnace provided with revolving rabbles includes superposed closed muffles beneath and in series with the drying hearth. A passage is arranged through the furnace for preheating a gas out of contact with the material, and means are provided between the passage and the drying hearth for forcing the preheated gas over the material on this hearth and through an outlet from the drying hearth zone. H. HOLMES.

Evaporators for steam power plants. A.-G. BROWN, BOVERI & CO. (E.P. 255,050, 25.6.26. Conv., 9.7.25).—Several evaporators are used for distilling the make-up feed water, each being heated by steam tapped from a different pressure stage of the prime mover, and the vapours produced are condensed by the feed water flowing successively through corresponding separate preheaters. H. HOLMES.

Evaporators. H. HILLER (E.P. 260,387, 8.9.25).—An evaporator of the marine type has its steam coils divided into two, so that two inlet headers and one outlet may be provided (or *vice versa*) and a lower pressure of steam used. The headers may also be subdivided, so that steam from more than one source may be used.

B. M. VENABLES:

Continuous crystallising apparatus. E. C. R. MARKS. From GRASSELLI CHEMICAL CO. (E.P. 260,691, 11.8.25).—Crystallisation takes place while the liquid ascends through the space between two inverted cones. Both or either of the walls of the crystallising space may be water-cooled. Below the crystallising space is another inverted conical vessel in which the crystals are collected, and to the upper part of which the original liquor is supplied. Above the crystallising space the outer wall is expanded in the form of an enlarged cone, in which are situated heating coils, so that the motherliquor which overflows from the rim of the enlarged cone will not be saturated. B. M. VENABLES.

Separators for removing entrained oil from hot vapours. J. R. STERLING (E.P. 260,899, 14.6.26).— The separator is of the gravity type, and has hollow baffles inside the separating chamber; each baffle is subdivided by an internal partition into two compartments, and cooling water circulates in succession through the two portions of each baffle. The hot gaseous medium impinges on the cold surface, and the entrained oil is deposited in the form of droplets. W. G. CAREY. **Continuous filtration.** F. W. MANNING (U.S.P. 1,604,649, 26.10.26. Appl., 28.1.24).—A treating agent is introduced into a chamber containing a liquid, and the treated portion is immediately filtered through the agent, while that portion of the liquid yet to be filtered is treated by mixing the agent with it. W. G. CAREY.

Continuous counter-current pressure filtration. F. W. MANNING (U.S.P. 1,604,650-1, 26.10.26. Appl., [A] 3.11.24; [B] 2.4.25).-[A] An enclosed chamber is divided into two compartments by a filter wall. The liquid to be filtered is treated with an agent in one portion of the chamber, is filtered through the partition, and is then treated with another agent in the other portion of the chamber. Means are provided for forming the solids into a filter cake, and for maintaining the cake at a constant thickness. [B] A portion of a cylindrical filter wall out of contact with the liquid to be filtered is continuously coated with a treating solid, which by a spiral feed is continuously moved bodily over the wall into contact with the liquid to be treated and filtered; this liquid then passes through the coating of solids and the filter wall. W. G. CAREY.

Drying gases and vapours. W. CARPMAEL. From I.-G. FARBENIND. A.-G. (E.P. 260,914, 6.7.26).—A continuous process for drying gases with active material such as silica gel, active alumina, etc. is attained by feeding it through a revolving drum in counter-current to the gas to be dried. The active material, charged with water, is regenerated in a similar drum with direct heating on a counter-current principle, means being provided for feeding material from one drum to the other. W. G. CAREY.

Removing gases from boiler feed water by a vacuum. P. MÜLLER, G.m.b.H. (E.P. 254,707, 25.6.26. Conv., 3.7.25).—Sludge or easily-soluble salts are continuously removed from a boiler, and the steam produced from them by the reduction in pressure is passed through an air extractor of the steam-jet ejector type, which communicates with the feed water reservoir.

W. G. CAREY.

Manufacture of articles having surfaces capable of producing diffraction colour effects. F. Twy-MAN, A. B. KLEIN, and H. SAYER (E.P. 260,669, 4.8.25).— The substance, such as cellulose acetate, albumin, varnish, etc., is poured on to a suitably heated cylinder of optically polished hardened steel ruled as for a diffraction grating, and the resulting film is removed. W. G. CAREY.

Centrifuge. T. C. THOMSEN, ASST. to KOEFOED, HAUBERG, MARSTRAND, & HELWEG, AKTIESELSKABET TITAN (U.S.P. 1,603,726, 19.10.26. Appl., 9.4.25. Conv., 10.4.24).—See E.P. 232,213; B., 1925, 528.

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

Calorimetric method of determining the calorific value of fuels. W. STEUER (Brennstoff-Chem., 1926, 7, 357-359).—A historical review of the development of the calorimetric method for fuels from the earliest experiments of Lavoisier and Laplace in 1781 to the present day. W. T. K. BRAUNHOLTZ. Working with reducing gases in apparatus made of copper or its alloys. J. Y. JOHNSON. From I.-G. FARBENIND. A.-G. (E.P. 260,888, 2.6.26).—The permeability of copper to hydrogen and other reducing gases at high temperatures and pressures is overcome by fitting the copper vessel or pipe with an inner jacket of copper, so that a narrow space is formed, and filling this space with a gas which will not reduce the oxides of copper or its alloys. The gas may be circulated or may remain quiescent, but preferably it is maintained at the same pressure as the reducing gas.

W. G. CAREY.

Determination of nitrogen in coal. A. VAN STEEN-KISTE (Natuurwetensch. Tijdsch., 1926, **8**, 88—91).---Where only occasional determinations are required, the ordinary Kjeldahl method involves the least trouble, but the results are always low, probably by loss of nitrogen as such. Ter Meulen's method, which consists in mixing the powdered coal with barium carbonate, heating to bright redness in a current of hydrogen and steam, and passing the gases over finely-divided nickel at 350°, the ammonia formed being collected in the usual way, is more accurate and much more rapid once the necessary apparatus has been erected ; it is, therefore, most suitable where frequent and regular determinations must be made. S. I. LEVY.

Small chamber coke oven. A. SCHMOLKE (Stahl u. Eisen, 1926, 46, 1582-1585).—The performance of a battery of thirty regenerative coke ovens, having chambers 35 cm. wide with conical tops, is examined. Coal is not compressed before coking, and the coke produced is correspondingly more porous than that from coal which has been stamped. It has great strength, with only a slight tendency to crack. By reducing the coking period to 12 hrs. the production of coke is increased, the reduction to coke being simultaneous throughout the whole chamber, hence its superiority over coke produced in a wider type of oven in which the finished coke is exposed to the full temperature of the oven for long periods to complete the coking in other parts of the chamber. With simultaneous completion of the process the gas, otherwise expended unnecessarily and harmfully on the overheated coke, is saved. The thermal economy thus obtained is demonstrated by the small amount of heat required for coking. A large volume of gas is thus produced which is not decomposed, since, at the end of the coking period, gas collected over the mass of coke does not attain the decomposition temperature. The whole of the ammonia, benzol, and tar may, therefore, be recovered, and the high yield of by-products further increases the efficiency of the process. L. M. CLARK.

Evaluation of gas purifying material. H. ASEN-DORF (Gas- u. Wasserfach, 1926, **69**, 957—958).—The iron content of a gas purifying material is not a measure of its adsorptive power. The latter can only be usefully determined by large-scale tests. The results of such a test on a sample of "Lautamasse "from the Vereinigten Aluminium-Werke A.-G. are given. $8\frac{1}{2}$ tons of Lautamasse were used in the purification of 340,000 cub. m. of gas over a period of $4\frac{1}{2}$ months. It then contained 41.70% of free sulphur and 11.27% of Prussian blue. A. B. MANNING.

a 2

33

Adsorptive properties of an active charcoal. C. MAZZETTI (Annali Chim. Appl., 1926, 16, 430-438). --A comparison of the efficiency of adsorption of benzene vapour and of various dyes by "Roma" charcoal and other preparations. E. W. WIGNALL.

Direct determination of nitrogen in illuminating and heating gases. W. STEUER (Chem.-Ztg., 1926, 50, 860).—The apparatus used consists of two Hempel burettes, one of which contains the gas and the other a slight excess of oxygen over that required for combustion. These are connected by a quartz capillary containing palladium (cf. B., 1925, 802), which is heated first gently and finally more strongly until the volume of the mixed gases becomes constant. The carbon dioxide and oxygen in the burnt gas are absorbed by alkaline pyrogallol, any carbon monoxide liberated at this stage being removed in the usual way, and the residual nitrogen measured. F. R. ENNOS.

Influence of the aldehyde content of industrial alcohol on its use for lighting purposes. J. Deh-NICKE and W. KILP (Z. Spiritusind., 1926, 49, 280-281). -To investigate the causes of the gummy residues formed when alcohol is used in lamps, various synthetic mixtures of alcohol, benzene, acetaldehyde, and pyridine were examined. Mixtures which contained both aldehyde and pyridine yielded a resinous gummy residue, but mixtures of alcohol and benzene with aldehyde or pyridine alone yielded only a slight non-gummy residue. This was confirmed by investigating the fall in candle-power over a period of 334 hrs. in lamps using different mixtures. Alcohol which has a high aldehyde content, and has been denatured with pyridine, causes a clogging of the wick by the gummy resins, and a high acid content causes a solution of metallic salts with subsequent deposition on the wick and a consequent reduction of the candle-power of the lamp. A. C. MONKHOUSE.

Combustible liquids of high organic sulphur content as a source of anti-detonators. Y. Alt-CHIDJIAN (Compt. rend., 1926, 183, 975-978).-Distillation of the bituminous limestones of Raguse yields heavy fractions up to 350°, which may be used satisfactorily in internal-combustion engines without producing detonation. This is due partly to the presence of a high proportion of hydrocarbons which have undergone a certain amount of degradation towards the unsaturated, cyclic state, and partly to the presence of organic sulphur compounds. Since the at. wts. of the elements of the sixth group, and also the anti-detonating powers of their organic compounds, form approximate geometrical progressions, the anti-detonating powers of these sulphur compounds are evaluated. If they are present in the fraction to the extent of 8-10%, an antidetonating power equivalent to 0.1% of lead tetraethyl is produced. J. GRANT.

Analysis of small quantities of lubricating oils. [The Vogel-Ossag viscosimeter.] G. MEYERHEIM and F. FRANK (Z. angew. Chem., 1926, **39**, 1451—1454). —The Vogel-Ossag viscosimeter has been designed to test small quantities of lubricating oils. It is essentially a modification of the Ostwald capillary viscosimeter, completely enclosed in a water-bath, and so arranged that by means of a pneumatic pump provided a whole

viscosity curve, η against *t*, can be determined from one filling of 15 c.c. of oil. The results compare very favourably with those obtained by the older methods, but the apparatus is not satisfactory for testing dark or very viscous oils. A crucible is also described by the use of which reliable results for flash-points and ignition points, within the limits $\pm 5^{\circ}$, are obtainable with only 10 c.c. of oil. E. HOLMES.

Acid value of the "blown" oil and the "tarforming" value. H. VON DER HEYDEN and K. TYPKE (Petroleum, 1926, 22, 1288—1289).—The varying pro portion of free to combined acid, and the varying molecular weights of the acids formed in different oils account for the absence of a simple relationship between the acid value of the blown oil and the tar-forming value of the oil. In investigating an oil, therefore, both values should be determined. A. B. MANNING.

Drying with hot gases. GRAULICH.-See I.

Coal tars from steamed vertical retorts. PARKER. —See III.

Tars and oils from coal. SINNATT and others. —See III.

Influence of carbonising conditions on the free carbon content of tar. HOLLINGS.—See III.

PATENTS.

Heat treatment or carbonisation of coal. INTERNAT. COMBUSTION ENGINEERING CORPORATION, Assees. of W. RUNGE and E. A. PACKARD (E.P. 253,498, 17.5.26. Conv., 12.6.25. Addn. to E.P. 242,621).—Finely-ground coal is preheated to 350° in gravitating down a tower up which hot gases, preferably containing oxygen, are blown. The treated coal does not differ materially in chemical composition from the original coal; the particles, however, have lost all the angular sharpness of the original coal. The preheated coal is then carbonised at 550° by a similar process. The final product is a powdered coke, the particles of which are porous, spheroidal shells of 'ow sp. gr., and contain 8—12% of volatile hydrocarbons. S. PEXTON.

Manufacture of vegetable carbon. L. H. and A. H. BONNARD (E.P. 260,666, 4 8.25).—A carbonaceous material is carbonised with magnesium carbonate as activating medium. The magnesium oxide is extracted from the residue by carbon dioxide under pressure, and in the presence of sufficient water to dissolve the magnesium bicarbonate formed. This is recovered from the solution as magnesium carbonate by causing it to react with magnesium oxide in a fresh portion of retorted residue or by heating it. R. A. A. TAYLOR.

Production of carburetted gas. FRANKFURTER GAS-GES., F. P. TILLMETZ, and E. SCHUMACHER (E.P. 260,501, 28.4.26).—Water-gas is carburetted by the injection of a mixture of oil or tar vapour and steam. which may be superheated, into the producer at a level where the temperature lies between 700° and 900°. The same process may be used for the enrichment of coal gas. A. B. MANNING.

Production of carburetted water-gas. FRANK-FURTER GAS-GES., F. P. TILLMETZ, and E. SCHUMACHER

(E.P. 260,511, 29.5.26. Addn. to 260,501; see preceding abstract).-A gas producer comprising two parts, the upper one being of smaller diam. than the lower, is fed with coke. Each part is provided with an outlet valve through which the "blow" products pass away. The two valves are interconnected so as to fix the relative amounts of blow gases traversing the two sections of the plant, thus enabling the upper part of the charge of coke to reach a temperature of 800° when the lower part has reached 1000°. At this stage steam for making watergas is introduced into the bottom of the generator, and simultaneously oil vaporised in a current of steam is introduced into the generator between the two sections. The oil is gasified at 800° in a stream of steam and watergas in the upper part of the producer. S. PEXTON.

Manufacture of combustible gas. W. J. MURDOCK, E. E. LUNGREW, and O. B. EVANS, ASSTS. to PIER PROCESS CORP. (U.S.P. 1,602,242, 5.10.26. Appl., 23.9.24).—A gas producer for gasifying coal and producing relatively rich gas is provided with a central refractory wall. All parts of the annulus of fuel being within radiating distance of the refractory walls, no impermeable core of fuel is formed. The producer is alternately blown and steamed in a vertical direction over substantially the whole of the grate area. S. PEXTON.

Process and apparatus for coking coal. URBANA COKE CORP., Assees. of S. W. PARR and T. E. LAYNG (E.P. 249,886, 27.3.26. Conv., 27.3.25).-Crushed coal is heated in a state of agitation to within a few degrees of the temperature at which it becomes plastic. During preheating the cellulosic constituents, which are devoid of coking properties, decompose with the evolution of water and carbon dioxide. The hot product is transferred in bulk into retorts preheated to 750°. To produce low-temperature coke of high volatile content no additional heating of the retort setting is required. For the production of metallurgical coke it is necessary further to increase the temperature of the retorts. By this two-stage process non-coking coals having a high cellulosic content yield excellent coke. S. PEXTON.

Apparatus for carbonising wood. H. HENNE-BUTTE and E. GOUTAL (E.P. 248,376, 24.2.26. Conv., 28.2.25).—Wood is carbonised in a perforated box enclosed within a cylindrical trolley having fluid-tight doors for charging and discharging. The bottom of the perforated box forms a duct with the bottom of the trolley whereby the gases evolved during distillation can pass to the outlet at one end of the trolley. The trolley passes through a tunnel which is heated by furnace gases which enter and leave by ports situated on the opposite sides of the tunnel. The hot gases pass over the trolley, the underside being protected in order to avoid degradation of the products of distillation.

S. PEXTON.

Distilling crude oils from shale. C. A. SPOTZ (U.S.P. 1,601,777, 5.10.26. Appl. 8.6.25).—Shale is delivered by a plunger to the underside of a reticulated tray, which is totally immersed in an enclosed tank of molten tin, or like metal, maintained at 425°. The tray slopes slightly upwards to the exit end of the tank, and the reciprocating motion passes the shale in that direction. As the shale loses gas it disintegrates,

passes through the reticulations in the tray to the surface of the metal, and moves towards the exit end of the tank under the action of rotating blades. Means are provided for admitting fresh material and withdrawing spent residues without admission of air. T. S. WHEELER.

Effecting reaction between liquids tending to form tight emulsions. ARTIEBOLAGET SEPARATOR, Assees. of F. H. MCBERTY (E.P. 238,541, 7.8.25. Conv., 16.8.24).—In the purification of lubricating oils from dispersed carbonaceous material, the oil is agitated with an immiscible counter-colloid which causes agglomeration of the carbon particles. The carbonaceous material is partially agglomerated by agitation of the oil with the reagent for a short time, and the oil and the reagent separate easily. By repeating the process the oil is finally purified. S. PEXTON.

Dehydrating petroleum emulsions. H. C. EDDY and G. B. HANSON, ASSTS. to PETROLEUM RECTIFYING CO. OF CALIFORNIA (U.S.P. 1,602,190, 5.10.26. Appl., 4.1.26). —In pumping an oil well a gas containing a demulsifying agent is introduced into the oil in the well. H. MOORE.

Filtration of fluids [lubricating oil]. C. S. GAR-LAND and T. E. BEACHAM (E.P. 260,699, 18.8.25).—Oil is filtered in a pack or edge filter which is maintained at such temperature that any water will pass through with the oil as vapour, and the viscosity of the oil will be greatly reduced. The filtration may preferably be effected by vacuum, so that the water is removed from the oil as vapour, but an occasional back-flow of air under pressure is provided for cleaning the filter.

B. M. VENABLES.

Removal of sulphur compounds from petroleum oils. L. L. ODOM, ASST. to M.O.R. PRODUCTS CO. (U.S.P. 1,604,235, 26.10.26. Appl., 13.6.24).—The vapours from a still are passed through a column tightly packed with copper fibres of an even and hair-like consistency. Removal of 99.75% of the sulphur content is claimed. W. N. HOYTE.

Refining of petroleum oils. P. MCMICHAEL, Assr. to Hydrocarbon Refining Process Co., Inc. (U.S.P. 1,603,701, 19.10.26. Appl., 11.1.24).—Distillates volatile below 225° are agitated first with a solution of alkali, then with 1.5 to 15% of sulphuric acid of strength 70—83%; finally the oil is distilled with steam in the presence of alkali. W. N. Hoyte.

Refining petroleum. W. A. SCHMIDT, ASST. to INTERNATIONAL PRECIPITATION CO. (U.S.P. 1,604,424, 26.10.26. Appl., 19.7.20; cf. U.S.P. 1,204,906—7; B., 1917, 38).—The oil to be treated is sprayed into a current of hot gas, and the mixture is led through an electrical precipitator to remove tar. It is then cooled to any desired extent by introduction of cold gas, and is subjected to electrical precipitation. The process of adding gas and precipitating is repeated as required. The permanent gas finally obtained is employed partially to vaporise fresh oil, and partially for cooling purposes.

T. S. WHEELER.

Purification of mineral oil distillates and of paraffin wax. BURMAH OIL Co., and R. R. LE G. WORSLEY (E.P. 260,455, 27.1.26).—Any fraction of a petroleum or shale oil, or paraffin wax etc., is refined, by the action of fuller's earth or other hydrosilicate, while in vapour and at a reduced pressure. The temperature is not more than sufficient to maintain the oil in vapour, and the vacuum as high as practicable, particularly in treating the higher-boiling fractions. The vapours may be purified by contact with increasingly active adsorbent in successive chambers. The oil may be preliminarily washed with sulphuric acid, or with alkali, to remove phenols, and these processes may alternate with the treatment with adsorbent. Inert vapours may be mixed with the oil vapours to reduce the time of operation. H. MOORE.

Apparatus for cooling oil vapours. GRISCOM-RUSSELL Co., Assees. of J. PRICE (E.P. 240,415, 4.8.25. Conv., 29.9.24).—The oil vapour flows zig-zag downwards



as shown by the arrows in the figure, and a cooling medium flows through the banks of tubes in succession. The condensate formed in each unit may, if desired, be collected separately. B. M. VENABLES.

Treatment of acid sludge. R. A. HALLORAN, W. N. DAVIS, and G. A. DAVIDSON, ASSTS. to STANDARD OIL Co. (U.S.P. 1,604,641, 26.10.26. Appl., 7.8.22).— The sludge, together with steam and water, is continuously passed into a vessel where the mixture is maintained at such a temperature and pressure that hydrolysis takes place. Acid and tar are drawn off from the vessel at required rates. W. N. HOYTE,

Manufacture of stable iron carbonyl compositions. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 206,639, 2.5.25).—The decomposition of iron carbonyl by light can be retarded or avoided by the presence of about 0.1% of a colouring matter (e.g., azo dyes), organic acid, ester, or other organic oxy-compound of high mol. wt. which is capable of acting as a protective colloid, or a solid or liquid hydrocarbon with a b.p. above 275°. In the latter case the hydrocarbon or its mixture used as a solvent should have a much lower b.p. H. ROYAL-DAWSON.

Manufacture of iron carbonyl compositions. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 260,640, 2.5.25).—The composition consists of at least 20% by volume of iron carbonyl in a mixture with liquid hydrocarbon diluent, *e.g.*, benzol or gasoline.

H. ROYAL-DAWSON.

Supplementary charges for internal-combustion engines. H. A. KELTY (E.P. 260,676, 5.8.25).—The supplementary charge consists of the vapour of a mixture of alcohol and water which is fed to the carburettor from a container which can be heated by the exhaust gases from the engine. Feed, composition of mixture, etc. are controlled thermostatically.

R. A. A. TAYLOR. Production of hydrogen practically free from carbon monoxide from water-gas (E.P. 259,643).— See VII.

Manufacture of a manure [from peat etc.] (E.P. 260,882).—See XVI.

III.—TAR AND TAR PRODUCTS.

Coal tars from steamed vertical retorts. A. PAR-KER (J.S.C.I., 1926, 45, 408-411 T).-In the carbonisation of coal in continuous vertical retorts at high temperatures, as compared with horizontal-retort practice, the coal is submitted to a gradual rise in temperature, and the primary tar products are removed from the top of the retort without having undergone so much degradation by long exposure at high temperatures. Continuous vertical-retort tars may, therefore, be classed as intermediate, in both quantity and composition, between high-temperature and low-temperature tars. The passage of steam upwards through continuous vertical retorts also reduces the amount of secondary thermal decomposition, by removing the products more rapidly from the retort and by ensuring more uniform heating of the coal. In an investigation of the process of steaming the charges in continuous vertical retorts, it was found that with Bothwell washed nuts the yield of dry tar per ton of coal carbonised rose from 13.2 gals. when no steam was admitted to the retorts to $21 \cdot 2$ gals. when the amount of steam was equivalent to 23.7% of the weight of coal. At the same time the amount of "free carbon" in the tar decreased from 1.5 to 0.4%, indicating a reduction in the extent of thermal decomposition. Coal charged in the form of screened nuts more readily allows the flow of gases and steam than coal charged in a more finely-divided condition. In experiments in which different grades of sizes of a particular coal were carbonised under identical conditions, the yield of tar progressively decreased as the sizes of the particles of coal were diminished, and the amounts of naphthalene and "free carbon" in the tars increased, demonstrating that the tar vapours from the smaller grades of coal had undergone greater thermal decomposition. In an examination of the published results of distillation tests of tar, it is almost impossible to make satisfactory comparisons. The reasons are discussed, and a strong plea is put forward for a standard simple laboratory distillation test under specified conditions.

Vertical-retort tar for road purposes. M. BARASH (J.S.C.I., 1926, 45, 441—448 T).—The use of tars for road purposes is reviewed, and stress is laid on the anti-skid properties of tarred roads as compared with other road surfaces. To the tar producer, vertical-retort tar shows a considerably greater financial return than horizontalretort tar. A number of gas undertakings are turning out from their own stills residue tars of vertical-retort origin which satisfy the Ministry of Transport (Road Department) Specifications. The essential characteristics which distinguish present-day vertical-retort tars from tars produced in intermittent systems of carbonisation are given. To the tar refiner the preparation of road tars from vertical-retort tars presents no difficulty. In the majority of cases straight distillation gives residues of the required properties. In some cases, however, as in the case of a tar containing an excess of phenols or tar acids, "topping" has to be carried out to a higher degree than would otherwise be necessary; the residue may then have to be "cut back" to the correct consistency with a decarbolated creosote. The use of road tar prepared from vertical-retort tar has been rapidly extending during the last 10 to 15 years, and it already forms an appreciable proportion of the tars used for road construction and maintenance.

Vertical-retort tar. J. MACLEOD, C. CHAPMAN, and T. A. WILSON (J.S.C.I., 1926, 45, 401-1406 T).-The essential differences between tar produced at Glasgow Corporation's Provan Station from Woodall-Duckham continuous vertical retorts, using Lanarkshire coal of moderate coking power, and tar produced by other systems of carbonisation, are indicated. It is shown that, while the yield of motor spirit is lower, the total yield of rectified naphtha is more comparable with that from horizontal-retort tar, though the higher paraffin content of the vertical-tar products renders them useless for the preparation of dyestuffs. The yield of tar acids, tar bases, and creosote is considerably higher than from other carbonising processes, pitch production is lower, naphthalene is found in very small amount, and anthracene in traces only. A comparison of the value of products from one ton of tar, at market prices quoted in April, 1926, from Provan vertical retorts with those from Provan horizontal retorts prior to the installation of the Woodall-Duckham retorts shows the total values to be 131/10.7 and 120/3.7, or on the basis of products from one ton of coal carbonised 9/5 and 7/0 respectively. These estimates take no note of cost of production, but it is claimed that, owing to the absence of salts, less capital outlay for plant is necessary and the saving of steam for heating purposes is considerable. Trouble has been experienced in refining the tar acids, so as to yield high-boiling products (of which the amount present is largely in excess of that from horizontal tars) which do not rapidly darken on keeping, and which, when emulsified with a soap solution, will yield disinfectants capable of maintaining their white colour for an appreciable period. These vertical-tar acids invariably yield a red emulsion which is objectionable for certain specific applications. Investigations with a view to the removal of the substances causing this red colour are described, as is also the method finally adopted involving airblowing of a hot "strong soda" cresylate which has been found effective in practice. This process also renders the freshly-distilled acid more stable, and the darkening, which is extremely rapid in untreated acid, is very considerably retarded. The material of which the acid still is composed has some bearing on this darkening process; of the less expensive and easilyworked metals or alloys, copper is very satisfactory, and arrangements have been made to use copper-lined mild steel stills in the works. The high yield of creosote compensates for the low pitch, and, owing to the absence of naphthalene and anthracene and its high tar acid content, forms a good timber-creosoting oil. It can be readily washed for the removal of tar acids, yielding a spent oil which is clear and perfectly fluid at ordinary temperatures, which are essential requisites in fuel oil. The prejudice formerly existing against using verticalretort pitch for briquette making is discussed, results of examinations and comparative analyses with pitches from other sources being given. It is pointed out that the total yield of pitch from the distillation annually of 27,500 tons of vertical-retort tar is regularly disposed of at current market rates. The conclusion arrived at is that, everything considered, vertical-retort tar has many advantages over other tars from the distillers' point of view, refining being easier and cheaper (except in the case of tar acids), and the total value of the derivatives being considerably higher.

Distilled tar for roads. W. G. ADAM [with F. W. ROBINSON] (J.S.C.I., 1926, 45, 412-415 T).-The employment of distilled or refined coal tar for road-making continues to increase in spite of criticisms. The chief faults of tar in the past have been mainly due to the use of improperly refined tar, frequently of unsuitable consistency for the work in hand, and in part due to careless laying. A specification is given based upon experience, and designed to cover only such products as will give satisfactory results in practice when applied under correct conditions. The specification is more stringent than the existing Road Board Specifications, particularly in the clauses relating to the naphthalene and tar acids. It will be possible for the tar distilling industry to supply all the tar required to this specification. Information on the toxic nature of washings from tarred roads has been considerably extended since the days of the Alresford experiments, in which a material was laid down which just conformed (except for the naphthalene content, which fell widely outside the limit) to the Road Board Specification. It has now been found that the toxicity of a given tar decreases with increasing viscosity, and that soft pitches in general are non-toxic under the experimental conditions. The toxic effects of tars become far less evident as the proportion of lower boiling point constituents is reduced. Several processes are now available for preparing tars, which it is claimed are non-toxic to fish life. The real damage caused by drainage from tarred roads has been much exaggerated, and trade effluents are now being recognised to be far more important factors. The best conditions for the application of tars by spraying are considered. With reference to emulsions, it is suggested that the advantages of the ease of application are not sufficient to compensate for the extra cost of the material and the lack of permanency of the results. Figures are given to illustrate the importance of grading the aggregate for road-making to obtain a solid resilient road, free from voids. It is considered that the tar distiller should further assist the surveyor by price stabilisation, and by increased delivery service. Under the latter heading it is proposed that barrels should be replaced by road tanks conveying the hot tar direct from the distillery to the road, these delivery tanks being equipped with spraying and brushing gear.

Tars and oils produced from coal. II. F. S. SINNATT, J. G. KING, and W. H. LINNELL (J.S.C.I., 1926, 45, 385-393 T; cf. *ibid.*, 1925, 413 T).—A series of investigations on the tar obtained by the carbonisation of a medium caking British coal at temperatures between 400° and 700° are described. Medium-scale steel retorts were used having a maximum capacity of 200 lb. of coal. The yield of tar was found to increase gradually with temperature to a maximum at 550°, the yield at this point amounting to 17.65 gals. per ton of coal. The sp. gr. of the tar also increased gradually up to 550°; above this temperature the rate of increase became greater. A study of the gas yields and of the nature of the tars produced indicated that a critical carbonising temperature occurred at about 550°, e.g., the lighter fractions from the tar (up to 230°) and the middle fractions increased to a maximum at 500°, a marked acceleration in pitch formation occurred above 500°, and the phenols and unsaturateds showed an accelerated increase above 550°. The percentage of saturated hydrocarbons in the tars decreased steadily from 29.8% of the 400° tar oil to 13.4% of the 600° tar oil and to $6 \cdot 2\%$ of the 700° tar oil. Low-temperature tar obtained in the "commercial" scale distillation of Dalton main coal contained phenols 26.15, phenol 1.17, bases 2.74, neutral oils 41.6, saturated hydrocarbons 8.24, solid paraffins 0.12%. These results are in general agreement with those obtained in previous investigations (Parrish & Rowe, J.S.C.I., 1926, 45, 99 T; Brittain, Rowe, and Sinnatt, ibid., 1925, 44, 412 T). Small-scale work on the hydrogenation of coal has confirmed previous results. A liquefaction of 50-60% is possible with the average British bituminous coal. The light oils obtained by this process contain appreciable amounts of benzene, toluene, and xylene, and in this respect differ markedly from those produced by low-temperature carbonisation.

Influence of carbonising conditions on the free carbon content of tar. H. HOLLINGS (J.S.C.I., 1926, 45, 406-408 T).-The free carbon content of tar from the carbonisation of Durham coal in horizontal retorts is correlated with the weight of charge, the temperature of the combustion chamber surrounding the retorts, and the cross-sectional area of the retort. The weight of charge per cub. ft. of retort space determines the amount of free space within the retort. With temperatures between 1320° and 1376°, the free carbon content increased from $12 \cdot 1$ to $20 \cdot 8\%$ as the weight of charge was decreased from 35.5 to 29.0 lb. per cub. ft. In a slightly deeper retort a reduction in the weight of charge from 31.0 to 28.4 lb. resulted in an increase in the free carbon content from 20.8 to 24.6% notwithstanding a fall in temperature from 1348° to 1334°. When the weight of charge was maintained at about $29 \cdot 0$ lb. per cub. ft. a reduction in temperature from about 1330° to 1253° resulted in a decrease in the free carbon content from about 22 to 12.3%. With any given retort the most important factor affecting the quality of the tar is the weight of each charge of coal. Any attempt to improve an unsatisfactory tar by reducing the carbonising temperature necessitates a reduction of the temperature below that which is required for the maintenance of such throughputs of coal as are essential for economical working. An increase in the weight of charge results generally in improved working conditions. In an experimental setting retorts 24 in. by 18 in. were set alongside retorts 21 in. by 15 in.

in cross-section. Each retort was fully charged with the same coal, and was heated to the same external temperature. The tar from the deeper retorts contained $18\cdot8\%$ of free carbon, that from the smaller retorts contained $9\cdot8\%$. It was necessary to carbonise the deeper mass of coal in the deeper retort for two hours longer than was necessary for complete carbonisation in the smaller retort. During this extra time the volatile products from the centre of the large coal charge were subjected to high-temperature treatment, while the gas velocity in the retort was relatively low. This resulted in the deposition of solid decomposition residues upon the coke and in the formation of free carbon in the free space.

Consistency of "bitumen mixtures." H. M. SPIERS (J.S.C.I., 1926, 45, 399-401 T).-When consistency is measured by means of Hutchinson's tar tester, the relation between the consistency of bitumen mixtures and their bitumen content may be expressed by means of the formula dc/d B/T = kC, which on integration yields the relationship $\log C_1 - \log C_2 = K (B_1/T_1 - B_2/T_2)$, in which C_1 , B_1 , T_1 are the consistency, bitumen content, and tar content of one mixture and C_2 , B_2 , T_2 are the corresponding values for another mixture made from the same tar and bitumen. K is a constant the value of which varies at least within the range 1.4 to 3.2, and depends on the nature of the tar, on the extent to which it has been topped, and on the nature of the bitumen (or pitch) added. A nomogram based on the above relationship is reproduced in the original, and facilitates the control of the consistency of mixtures prepared industrially.

Variation of Hutchinson consistency of tars with temperature. H. M. SPIERS (J.S.C.I., 1926, 45, 396–399 T).—Consistency varies with temperature according to the relationship dc/dT = -kC, which on integration yields the formula log $C_2 - \log C_1 =$ $K(T_1 - T_2)$. In the equation, C_1 and C_2 are the consistency values at two known temperatures T_1 and T_2 , consistency being measured in seconds and temperature in degrees F., K is a constant depending chiefly on the naphthalene content of the tar, and ranging from 0.0334 in the case of tars rich in naphthalene to 0.0502 when the naphthalene content is low. The formula applies also to "bitumen mixtures." A nomogram enables the consistency of a tar at the standard temperature of 77° F. to be ascertained when the consistencies at two known temperatures have been determined, and its use avoids the necessity of cooling the tar to the standard temperature, and enables the consistency at any desired temperature within a definite range to be estimated.

Evaluation of pitch. H. F. TAYLOR (J.S.C.I., 1926, 45, 417-424 T).—*Binding power.* The tensile strength of pitch was tested in various ways, but it was found that the pitch invariably broke where the jaws of the testing machine gripped it, and the results are considered unreliable. Coal-tar pitches averaged about 80 lb. per sq. in. In the test considered most likely to give concordant results comparable with works practice, briquettes are prepared by heating 105 g. of the sand standardised for cement testing by the British Engineering Standards Association with 5% more pitch than will fill the interspace, and casting the mixture in a B.E.S.A. mould. The tensile strength of the briquette

is determined in a cement testing machine. Six pitches were tested under varying conditions. Vertical-retort pitch briquettes averaged 250-300, horizontal 350-450, low-temperature 200 lb. per sq. in. Range of plasticity and elasticity. A small mechanical apparatus is described, in which the principle of the "twist-point" test is used. The torsion is applied by a weight suspended from a thread running over a pulley and round a bobbin attached to a holder which grips one end of the block of pitch $(2 \times \frac{1}{2} \times \frac{1}{2}$ in.). A dial graduated in degrees of arc indicates the amount of the twist. A ball and ring "fusing" point apparatus is attached, and the difference between the "fusing" temperature and the temperature at which the pitch starts to twist gives an indication of the range of plasticity. Four types of coal-tar pitch gave ranges of about 40° C., soft and hard hardwood pitch 29° and 49°, and petroleum bitumen 56°. If the weight responsible for the torsion is suddenly lifted a recoil occurs, and an elasticity test is based on this. The recoils which occurred when 500 g. had twisted the pitch 360° of arc at the twist point temperature were : horizontal- and vertical-retort pitches, 10° arc, low-temperature pitch 18°, coke-oven pitch 21°, blast-furnace pitch 30°, hard hardwood pitch 88°, petroleum bitumens 120° and 130°. The effect of the length of time which had elapsed since casting the mould and of twisting the pitch a number of times was investigated. The elasticity of lowtemperature pitch can be considerably increased by treatment of tar or pitch with sulphur.

Plea for standardisation of analytical methods in coal tar products specifications. W. G. ADAM (J.S.C.I., 1926, 45, 393-396 T) .- Although the desirability of rigidly standardising the methods of analysis of metallic and inorganic products may be debatable, its necessity can hardly be denied in the case of organic commercial products the valuation of which is almost invariably carried out by the observation of physical properties rather than by true chemical analysis. The four chief methods employed in the examination of coal tar products are the observation of (a) boiling points or distillation ranges; (b) crystallisation or setting points; (c) melting or softening points; (d) specific gravity. In some few cases fractionation is used, whilst in others, e.g., the determination of m-cresol and anthracene, true analytical methods are employed. The use of retorts for the determination of boiling points and distillation ranges is open to very grave criticism; it is suggested that the vessel used should be a standard distilling flask, the standard Engler flask used in the petroleum industry, and adopted to some extent by the B.E.S.A. Further matters requiring attention are the methods of heating and the thermometers employed. For creosote distillations the American authorities have recognised the shortcomings of the retort, and have adopted a retort flask, which has removed many of the disadvantages of the older method. The defects of many of the older methods of testing pitch have been demonstrated, and it is possible that the Kraemer and Sarnow method for determining softening point and the Brookman-Mück test for volatile matter will be adopted. It is desirable to standardise existing methods as far as possible rather than to introduce new ones.

Coal-tar disinfectants. TAIT .-- See XXIII.

Acid value of the "blown" oil and the "tarforming" value. VON DER HEYDEN and TYPKE.— See II.

PATENTS.

Manufacture of bituminous product. D. B. W. ALEXANDER (U.S.P. 1,603,502, 19.10.26. Appl., 4.5.26). —A slurry of finely-divided mineral matter and kerosene is added to a charge of petroleum, and distilled.

B. W. CLARKE.

Removal of tar acids from ammonia liquor. H. W. ROBINSON and D. W. PARKES (E.P. 260,686, 7.8.25, 9.10.25, and 29.3.26).—The liquor is neutralised by a mineral acid. Carbon dioxide (in vitiated airs etc.) may serve for this purpose. The liberated tar acids are then absorbed by active carbon, which is separated from the liquor and treated for the recovery of the tar acids. R. A. A. TAYLOR.

Production of carburetted gas (E.P. 260,501).— See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Colours in foodstuffs. DRAKE-LAW.-See XIX.

PATENTS.

Manufacture of derivatives of naphthaquinones. Soc. Anon. des Mat. Col. et Prod. Chim. de St.-Denis, R. LANTZ, and A. WAHL (E.P. 246,482, 21.1.26. Conv., 21.1.25). - 1 - Imino - 4 - arylimino - 2 - hydroxynaphthaquinones are prepared by the simultaneous oxidation by air, pure oxygen, or other oxidising agents of 1-amino-2-naphthol or its 4-sulphonic acid and arylamines, or by condensing nitroso- β -naphthol with arylamines. E.g., 52 pts. of 1-amino-2-naphthol-4-sulphonic acid, 14 pts. of anhydrous sodium carbonate, and 25 pts. of aniline are dissolved in 800 pts. of water. The cold solution is stirred and sodium hypochlorite gradually added until there is excess of chlorine and the precipitate no longer increases. The product is purified by dissolving it in cold dilute sodium carbonate, and reprecipitating the filtered solution at once by bicarbonate. The orange 1-imino-4-phenylimino-2-hydroxynaphthaquinone so obtained is identical with the product formed by dissolving 30 pts. of nitroso- β -naphthol in 300 pts. of aniline and 30 pts. of benzene, boiling for 12 min., and cooling. The former method may also be used for the preparation of the 2-hydroxy-1: 4-naphthaquinonediarylimines described in E.P. 206,142 (B., 1924, 902) from 1-arylamino-2-naphthol-4-sulphonic acids. Thus 1-phenylamino-2naphthol-4-sulphonic acid (E.P. 206,150; B., 1925, 200) when oxidised together with aniline in slightly alkaline solution by agitation with air yields 2-hydroxy-1:4-naphthaquinonediphenylimine. The action of acids on the above products replaces the group in position 1 by oxygen, thus forming 2-hydroxy-1:4-naphthaquinone-4-arylimines identical with those described by Boeniger (Ber., 1894, 27, 23). Again, by the action of arylamines, the group in position 1 is replaced by the arylimino-group with formation of 2-hydroxy-1: 4-naphthaquinonediarylimines. A. DAVIDSON.

Dyes and dyeing. [Red flavanthrone derivative.] B. WYLAM, J. E. G. HARRIS, J. THOMAS, and SCOTTISH

DYES, LTD. (E.P. 260,638, 30.4.25).—Leucoflavanthrone is treated with an alkylsulphuric halide and a tertiary base at temperatures above about 50°, or the alkylsulphuric halide may be mixed with the tertiary base in the cold, leucoflavanthrone added, and the mixture heated at above 50°. The quantity of tertiary base used may be higher than the molecular equivalent of the alkylsulphuric halide. E.g., 7.5 pts. of methylsulphuryl chloride are stirred, with cooling, into 18 pts. of dry light pyridine under carbon dioxide. 3 pts. of dry leucoflavanthrone are added, and the temperature is raised to 60° during $\frac{1}{2}$ hr., then kept at 60° for $\frac{1}{2}$ hr. more. The yellowish - red melt is poured into 200 pts. of water containing 8 pts. of ammonia solution ($d \, 0.880$), whereby a reddish solid is precipitated. This product may be used directly for dyeing in faintly alkaline solution, or it may be dissolved in 11% sodium carbonate solution, aerated to remove unchanged leucoflavanthrone, filtered, and salted out. The product can be dyed or printed on cotton or other textile fibres by hydrolysis and oxidation. Cf. E.P. 247,787 (B., 1926, 403), which describes the preparation of a red flavanthrone derivative by another process. The green flavanthrone derivative of E.P. 245,587 (B., 1926, 235) is excluded from this patent.

A. DAVIDSON.

Dyes and dyeing. [Stable water-soluble vat dye derivatives.] B. Wylam, J. E. G. HARRIS, J. THOMAS, and Scottish Dyes, Ltd. (E.P. 260,647, 4.6.25).-Stable water-soluble derivatives of vat dyes are made by treating the vat dyes with an alkylphosphoric halide or phosphorus oxychloride in presence of a metal, such as zinc dust or copper bronze, and a tertiary base such as pyridine, quinoline, or dimethylaniline. E.g., 30 pts. of methylphosphoryl dichloride are stirred, with cooling, into 150 pts. of pyridine. An intimate mixture of 30 pts. of indanthrone and 30 pts. of zinc dust is added, followed by 60 pts. of methylphosphoryl dichloride. The temperature is allowed to rise to 40°, and, after stirring for 20 min. at this temperature, 1000 pts. of water are added. The mixture is poured into 2000 pts. of water, filtered, and the paste washed with water. The product is a stable, light blue substance soluble in alkalis, and oxidisable apparently to indanthrone by acid oxidising agents. Products obtained in this manner can be dyed or printed on cotton by methods such as those described in E.P. 245,587 and 247,787 (B., 1926, 235, 403). Cf. also E.P. 248,802 (B., 1926, 403). A. DAVIDSON.

Manufacture of chromium compounds of azodyestuffs. O. Y. IMRAY. From Soc. CHEM. IND. IN BASLE (E.P. 260,830, 22.2.26).—Azo-dyes containing groups capable of being chromed are treated with chromic chromates (cf. Gmelin-Kraut, 7th Edn., III, pt. 1, p. 357, etc.). E.g., 37.8 pts. of the sodium salt of the dye obtained from diazotised 4-chloroaniline-3sulphonic acid and salicylic acid are dissolved in 1200 pts. of boiling water, and a 7% chromic chromate paste is added, made from sodium dichromate and sodium thiosulphate (loc. cit., p. 359) containing chromium equivalent to 22.8 pts. of Cr2O3. The mixture is boiled under reflux for a long time. Chromium oxide is filtered off and the dye isolated by concentration and salting out. The products obtained by this method dye wool in an acid bath very even tints fast to light and fulling.

A. DAVIDSON.

Preparations [for generating diazonium salt solutions] suitable for developing baths for use in dyeing (E.P. 260,682).—See VI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Effect of dry cleaning on silks. M. H. GOLDMAN, C. C. HUBBARD, and C. W. SCHOFFSTALL (U.S. Bureau Standards Tech. Papers No. 322, 1926, 20, 605-634).-Tin-weighted and unweighted specimens were treated with dry-cleaning agents containing benzine soap, also with acid and alkaline perspiration solutions followed by dry cleaning. No deterioration results from use of the dry-cleaning agents or from subsequent conditioning for over two months at 21° F. and 65% R.H., even after perspiration treatment. Sunlight causes greater tendering in weighted than in unweighted silks, the extent being increased by previous perspiration treatment. Damage, which is attributed to the production of acid in the fabric, is expressed in terms of the breaking load B. P. RIDGE. of the untreated material.

Adsorption of malodorous substances formed during the manufacture of sulphate-cellulose. O. ROUTALA & A. V. JÄÄTTELÄ (Cellulosechem., 1926, 7, 169-173).—The development of the sulphate pulp industry has been hindered by the extremely unpleasant odour of the exit gas, due chiefly to the presence of methyl mercaptan. This substance is adsorbed by vegetable substances such as wood, straw, etc., and this appears to be the most promising method for dealing with the nuisance (Schwalbe, B., 1922, 747 A). Wood which has been used for adsorbing methyl mercaptan does not give a higher yield of this substance in the sulphate process, as the mercaptan reacts with atmospheric oxygen in the pores of the wood. In the present research moisture is shown to have an important effect on the adsorption of mercaptan by sawdust from conifers, the best results being obtained with sawdust containing 50% of moisture. Sawdust absorbs 14 times as much as wood chips of the same moisture content, and the removal of all mercaptan from the exit gas of a sulphate pulp factory is possible by the use of the quantity of sawdust normally obtained as a by-product. The power of adsorption can be increased by treating the sawdust with aqueous solutions of various substances such as ammonium persulphate, sodium nitrite, sodium perborate, hydrogen peroxide, sulphate black liquor, ammonia, alkalis, or copper, mercury, or lead salts. Straw and peat, animal and wood charcoals are all better adsorbents than sawdust, the effectiveness of the former two materials being largely influenced by the moisture content. W. J. POWELL.

Alkali-cellulose. III. E. HEUSER and R. BARTUNEK Cellulosechem., 1926, 7, 169; cf. B., 1925, 311).—The authors disagree with Hess (Z. angew. Chem., 1925, 38, 230) regarding the constitution of the sodium hydroxide-cellulose complex. They have shown that the amount of sodium hydroxide taken up by cellulose from concentrated solutions (35-50%) only slightly exceeds that required for the formula $(C_6H_{10}O_5)_2$, NaOH. Higher results are obtained with very concentrated solutions, but these are due to precipitation of sodium hydroxide by the cellulose. The amount of potassium hydroxide taken up from the most concentrated solutions is never more than that required for $(C_6H_{10}O_5)_2$, KOH, and there is no reason to expect a difference in the behaviour of the two alkalis. It is therefore doubtful whether a compound of the formula $C_6H_{10}O_5$, NaOH can exist. Contrary to Hess, the authors find that the swelling action of sodium hydroxide, though reduced, is not prevented by the presence of alcohol, and the compound $(C_6H_{10}O_5)_2$, NaOH is still formed, unless the concentration of alcohol is high enough to prevent sufficient dissociation of the sodium hydroxide in solution, but the formation of alkali-cellulose may be masked by the increased adsorption of sodium hydroxide caused by the addition of alcohol. W. J. POWELL.

Ripening of viscose. O. FAUST, E. GRAUMANN, and E. FISCHER (Cellulosechem., 1926, 7, 165-166).-The work of Bernhardt (Kunstseide, 1926, 8, 173) and of Heuser and Schuster (B., 1926, 399) on the method for the determination of the degree of ripening of viscose, based on the reaction between iodine and sodium cellulose xanthate, is discussed, and the following procedure is recommended : 25 g. of viscose are made up to 250 c.c. with distilled water. 25 c.c. of this solution are diluted with 2 litres of water, made just acid with N-acetic acid solution (1 drop over the neutral point), and then an excess of 0.1N-iodine solution is added. These operations must be carried out quickly in view of the rapid ripening of viscose in dilute solution and the decomposition of the cellulose xanthate by dilute acetic acid. The time required (5-10 sec.) should be the same in every determination. The dixanthate formed on addition of iodine is apparently stable towards acetic acid. After $\frac{1}{2}$ hr. the excess of iodine is titrated with 0.1N-sodium thiosulphate solution. The determination is then repeated with sulphuric acid in place of acetic acid, the difference between the two results being a measure of the degree of ripening. The method gives concordant figures, and is suitable for the control of manufacturing operations. An unsuccessful attempt was made to find an acid of lower dissociation constant than acetic acid, which would decompose sodium trithiocarbonate but not cellulose xanthate. The hydrogenion concentrations required in the two reactions were of the same order, although there was a difference in the reaction velocities. W. J. POWELL.

Industrial resinates [for gumming paper etc.]. UZAC.—See XIII.

Culture of the bacillus of retting of flax. Omé-LIANSKY and KONONOV.—See XVIII.

Alloys [for jets used in spinning artificial silk]. DREAPER.—See X.

PATENTS.

Impregnating [fabrics] with rubber (E.P. 260,652). —See XIV.

Manufacturing a rubberised fibre composition (U.S.P. 1,603,318).—See XIV.

Making a rubberised fibre composition (U.S.P. 1,603,335).—See XIV.

Making cellulose esters of carboxylic acids (U.S.P. 1,604,471).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing of vat colours. G. O. MITCHELL (J. Soc. Dyers and Col., 1926, 42, 374-376).—For the hot-dyeing of yarn with such colours as Duranthrene Golden Yellow Y and 2RT, Orange 4R, Blue 3GT, GCD, CC, and RD extra, Hydron Yellow NF, Pink FF and FB, Caledon Jade Green, and Indanthrene Grey 3B and Green 2G, the dye-bath conveniently contains 1-20 lb. of colour paste, 10-20 lb. of caustic soda ($d \ 1.38$), and $1\frac{1}{4}$ -4 lb. of hydrosulphite powder in 150 gal. of water at 50-60° for 50 lb. of yarn. With cold-dyeing colours, such as Indanthrene Corinth RK, Caledon Yellow 3G, and Red FF, Duranthrene Brilliant Violet R, Olive R, Red BN, and Red Violet 2RN, only about half the amount of sodium hydroxide is required. The actual dyeing in this case is carried out at 20-25°, and for medium or heavy shades the addition of Glauber's salt is necessary. The distinction between hot- and colddyeing colours is not rigid. Thus Caledon Jade Green is best dyed at 25-30°, and Duranthrene Brilliant Violet R and Olive R, Indanthrene Brown R and G can be used as shading colours by the hot-dyeing process. Similar but more concentrated baths containing less sodium hydroxide are used for the dyeing of piece goods, and concentrated stock solution of the colour is with advantage prepared from 8 lb. of colour paste, 5 lb. of caustic soda $(d1\cdot38)$, $1\cdot75$ lb. of hydrosulphite, and 10 gal. of water at 50-55°. Precautions are necessary against over-oxidation, and with mixtures of Duranthrene Blues sodium perborate can be used for oxidising. Lists are given of the colours most suitable for hot- and colddyeing of piece goods. In the dyeing of viscose silk with vat colours, skilful and rapid manipulation of the yarn is one of the most important points. R. BRIGHTMAN.

Problems of modern calico-printing technique. J. R. HANNAY (J. Soc. Dyers and Col., 1926, 42, 369-374).—The author regards the vat dyestuffs as strongly individualistic in their behaviour on dyeing, and further information is desirable regarding their individual properties, particularly in regard to the heat generated in the formation of the leuco-compounds from the original dyes, and the effect of temperature on the reverse reaction, for the production of the best results. More effective stripping agents for vat colours are also required, and the extension of the Indigosol range of vat colours demands the development of a special technique for these colours. Reasons are given in support of the assertion that no existing range of printing colours giving a full palette can be properly issued with an unlimited guarantee. Thus the penetration of the fibre by the dyestuff, and in consequence the fastness of the latter to scouring, depends inversely on the fineness of the material. R. BRIGHTMAN.

PATENTS.

Preparations [for generating diazonium salt solutions] suitable for developing baths for use in dyeing. J. Y. JOHNSON. From BADISCHE ANILIN U. SODA FABR. (E.P. 260,682, 5.8.25).—A diazotisable amine or a dry neutral salt of it is mixed in about equal molecular proportions with a nitrite. A water-soluble neutral salt may be added as diluent. Solid compounds of acid nature such as bisulphates, naphthalenetrisulphonic acid, etc. may also be added. It is advantageous to add to the mixture a wetting agent such as Turkey-red oil or condensation products of formaldehyde with phenol- or naphthalene-sulphonic acids. Such mixtures are stable if protected from moisture, and yield solutions of diazonium salts when dissolved in water.

A. DAVIDSON.

Dyes and dyeing. [Red flavanthrone derivative] (E.P. 260,638).—See IV.

Dyes and dyeing. [Stable water-soluble vat dye derivatives] (E.P. 260,647).—See IV.

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

Modern saltpetre industry and the nitrate problem. A. SCHAARSCHMIDT (Z. angew. Chem., 1926, 39, 1457—1460).—Mainly a re-statement of the results of work already published on the interaction of the oxides of nitrogen with olefines and aromatic hydrocarbons and their derivatives, together with a discussion of possible industrial developments (see A., 1924, i, 380; 1925, i, 123, 532, 877; ii, 227). E. HOLMES.

Preparation of calcium cyanide. H. H. FRANCK and C. FREITAG (Z. angew. Chem., 1926, 39, 1430-1432) .- Following the method of Bergstrom (A., 1924, i, 949) the preparation of calcium cyanide is carried out in liquid ammonia at the ordinary pressure and at temperatures of -35° to -80° . On adding electrolytic calcium to a solution of hydrogen cyanide in liquid ammonia a vigorous reaction takes place and a blue-grey paste results. The product, purified by washing with liquid ammonia, proves to be diammonium calcium cyanide, the yield being quantitative. This compound, which also results from the use of calcium nitrate in the above reaction, is a blue-white, apparently amorphous powder. It is stable in vacuo at 100°, begins to lose ammonia at 150-160°, and by heating in vacuo for 2 hrs. at 180° yields a free calcium cyanide which appears to be purer and of greater density than that of Metzger (B., 1926, 272). E. HOLMES.

Germanium. XVII. Fused germanium dioxide and some germanium glasses. L. M. DENNIS and A. W. LAUBENGAYER (J. Physical Chem., 1926, 30, 1510-1526).-Germanium dioxide, m.p. approx. 1100° (cf. Müller and Blank, A., 1925, ii, 145), was obtained as a fused, clear, colourless, transparent glass by heating at 1450-1500° in a high-frequency induction furnace, with frequent stirring, for 30 min. The product contained traces of silicon, aluminium, iron, and copper up to 0.2%, and had $n_{\rm C}$ 1.60304, $n_{\rm D}$ 1.60696, $n_{\rm F}$ 1.61762, i.e., 10% higher than the values for fused silica (Gifford, Proc. Roy. Soc., 1910, 84, 193). The dispersive power is greater than that of fused silica, but the partial dispersion ratios are approximately the same. The effective refractivity is lower, the transmission is the same, except in the ultra-violet, when it is much lower, the density and thermal expansion are also much higher than obtains with quartz. It is less resistant to water and acids, but can be fused and freed from bubbles at lower temperatures. At high temperatures, carbon, organic compounds, and carbon monoxide rapidly reduce it, and the glaze of a porcelain crucible is attacked. Germanium light flint, crown, barium crown, and boro-crown glasses were prepared by substituting equimolecular amounts of germanium dioxide, prepared by the method of Dennis and Johnson (A., 1923, ii, 570), for silica in the corresponding silicate glasses. The fusions were made in unglazed porcelain crucibles heated in small, regenerative gas furnaces of the Hempel type at 1200-1400° for 3 hrs. Crucibles made of platinum, alundum, quartz, or glazed porcelain were unsuitable. Compared with silicate glasses, those of germanium possess higher refractivity and dispersion, a transmission which is approximately the same in the visible and infra-red portions of the spectrum, but lower in the ultra-violet, and partial dispersion ratios which are nearly equal. The densities and thermal expansions are higher, the hardness is the same, but the softening temperatures are lower. Devitrification occurs more readily. Durability and probably resistance to weathering are somewhat less than is the case with the corresponding silicate glasses. The optical properties of the germanium glasses suggest that they could be advantageously used in the construction of optical systems. Satisfactory glasses, intermediate in properties between the two types, can be made by replacing any part of the silica with germanium dioxide in the ordinary silicate L. S. THEOBALD. glasses.

Determination of carbon dioxide in carbonates. J. R. I. HEPBURN (Analyst, 1926, 51, 622-624).-From 0.15-0.3 g. of the carbonate is weighed into a test tube, covered with distilled water, and placed in a 750 c.c. filtration flask containing 50 c.c. of 0.1N-baryta solution. A separating funnel containing 3N-hydrochloric acid is fixed on the flask, which is evacuated to a pressure of 2 cm. of mercury, the clip to the air pump is then closed, and the acid cautiously run into the flask. The evolution of carbon dioxide, which is absorbed by the baryta solution, is assisted by the partial vacuum, and ordinary temperatures usually suffice. After remaining for 12-24 hrs. the remainder of the baryta is titrated with 0.1N-oxalic acid. Results were found to be within 0.5% of the theoretical value.

D. G. HEWER.

Removal of oxygen from commercial carbon dioxide. N. H. HARTSHORNE and J. F. SPENCER (J.S.C.I., 1926, 45, 474 T).—The increase in the life of chromous chloride when used as an oxygen absorbent, effected by the addition of amalgamated zinc, is discussed.

Effect of acids on the mechanical strength of timber. AllioTT.—See I.

Analysis of lime. BLUNT.-See IX.

PATENTS.

Method of making arsenic acid. H. W. AMBRUSTER (U.S.P. 1,603,308, 19.10.26. Appl., 9.4.25).—Arsenious oxide and nitric acid are gradually fed into a chamber and thoroughly agitated, the mixture meanwhile being slowly removed to another chamber.

H. ROYAL-DAWSON.

Mixtures of alkali hypochlorite and alkali chloride in solid form. A. Oppé (E.P. 249,510, 12.3.26. Conv., 23.3.25).—A highly concentrated and atomised solution of caustic soda is sprayed by means of gaseous chlorine into an empty chamber, or by blowing powdered caustic soda dust into a chamber containing chlorine, the products being ultimately dried by passing a current of air into the reaction chamber.

H. ROYAL-DAWSON.

Manufacture of chrome alums. W. CARPMAEL. From I.-G. FARBENIND. A.-G. (E.P. 260,885, 28.5.26).— The crystallisation of chrome alum is accelerated if a solution of chromium sulphate, acidified with sulphuric acid, is heated at 30—45°, the requisite amount of potassium sulphate is then added, and the solution cooled rapidly, with agitation. W. G. CAREY.

Recovering vanadium compounds from iron ores containing vanadium and titanium. B. PER F. KJELLBERG (E.P. 260,661, 30.7.25).—The finelyground ore, or concentrate, is mixed with a halogen compound of an alkali metal or of calcium or magnesium, formed into briquettes, and roasted for some hours at 950—1050°, at which temperature the vanadium compounds are not fully oxidised to the V_2O_5 stage, thus avoiding the formation of sparingly soluble compounds. The roasted mass is treated with dilute hydrochloric acid, which extracts 90% of the vanadium compounds. The vanadium from the solution is precipitated by the addition of lime. M. E. NOTTAGE.

Treating [recovering] vanadium compounds. H. BLUMENBERG, JUN., ASST. to STOCKHOLDERS SYNDICATE (U.S.P. 1,604,630, 26.10.26. Appl., 25.11.25).—Crude phosphate rock containing up to 0.5% V₂O₅ is treated with ammonium nitrate and sulphur dioxide, and the solution obtained is freed from all base metals except alkali metals, and agitated for 6—24 hrs. with oxycellulose, when a vanadium compound of the carbohydrate separates. The vanadium is recovered by ignition. T. S. WHEELER.

Simultaneous recovery of nitric oxide, and the production of alkali nitrate and hydrochloric acid. W. H. Ross and A. L. MEHRING (U.S.P. 1,604,660, 26.10.26. Appl., 7.10.25).—A stream of nitrogen peroxide mixed with air is brought into contact with a saturated solution of an alkali chloride.

H. ROYAL-DAWSON.

Production of hydrogen practically free from carbon monoxide from water-gas. G. CICALI (E.P. 259,643, 15.6.25).-Water-gas is freed from carbon dioxide and hydrogen sulphide, and then further purified to remove the bulk of the carbon monoxide. A volume of free nitrogen equal to the volume of carbon monoxide removed is added to the purified gas, and the mixture is compressed, cooled, and passed into a liquefying machine provided with a fractionating column. The most readily liquefiable component, viz., the carbon monoxide, liquefies in the greatest proportion. The hydrogen and nitrogen contain smaller and smaller quantities of carbon monoxide in the upper parts of the rectifying column. Superimposed upon the rectifying column are a series of tubes surrounded by a mixture of liquid carbon monoxide and nitrogen, supercooled by evaporation at approximately atmospheric pressure. The hydrogen and nitrogen containing traces of carbon monoxide pass through these tubes, in which the bulk of the nitrogen and the last traces of carbon monoxide condense. The hydrogen, free from carbon monoxide but containing some nitrogen, leaves the refrigerating machine by a coiled pipe surrounding the tube by which the gas to be purified enters. S. PEXTON.

Continuous crystallising apparatus (E.P. 260,691). Drying devices for granular material and the like (E.P. 260,896).—See I.

Iron carbonyl (E.P. 206,639 and 206,640).-See II.

VIII.—GLASS; CERAMICS.

Determination of soluble alkali in glass. E. FISCHER and W. TEPOHE (Glastech. Ber., 1926, 4, 137— 142; Chem. Zentr., 1926, II, 2010).—A modification of Mylius' method in which methyl-red is used in place of iodoeosin.

Constitution of porcelain. R. SCHWARZ and H. MERCK (Z. anorg. Chem., 1926, 156, 1-16).-The literature relating to the nature of the crystalline aluminosilicate formed on heating clays to high temperatures is reviewed (cf. also Hyslop, Trans. Ceram. Soc., 1925, 24, 402). Analytical data are presented in support of the assumption that mullite, rather than sillimanite, is the silicate formed in fired porcelain. The method used to isolate mullite closely resembled that previously applied by Bowen and Greig (B., 1924, 512). Analyses of unglazed porcelains, fired from one to six times in a porcelain oven, showed that the percentage of mullite increased from an average of 3.7 to 13.0%. Similarly, in Zettlitz kaolin, fired for 2 hrs. at 1400° and for 4 hrs. at 1500°, the amount of mullite increased from 30.2% to 44.7%. The resistance to the action of chemicals was not appreciably affected by repeatedly firing porcelain. Porcelain fired four times was somewhat more resistant to abrupt changes of temperature than that once fired. Six-fire porcelain showed slightly less resistance than that fired four times, owing to the development of larger mullite crystals, which rendered the material less homogeneous. Microscopical study of thin sections showed clearly the development of mullite by repeated firings. Once-fired porcelain contained numerous quartz fragments surrounded by a glassy matrix and fine, felty, acicular crystals of mullite. In the six-fire porcelain the glassy matrix was filled with well-developed mullite crystals. Hence the examination of thin sections in conjunction with physical tests is the best method of testing the quality of finished porcelain. Felspar is capable of dissolving mullite to the extent of 9%; quantities exceeding 13% were no longer taken into solution. The solvent action of felspar on quartz has an effect on mullite formation, since mullite appeared to be less soluble in a felspathic glass rich in silica. Homogeneous fusion of quartz and felspar is complete in porcelain after the sixth fire, and mullite can then crystallise out. Experimental evidence supports the theory that alumina and silica are dissolved partly as such by felspar and partly in the form of mullite. The amorphous silica and finally the quartz also go into solution, and the highly acidified glass thus formed becomes less solvent towards mullite. Simultaneously, however, the glass becomes more viscous and crystallisation is retarded. When the viscosity is reduced (e.g., by repeated firing)

44

the mullite readily crystallises out. Support of this theory was obtained by experimentation with a felspathic glass rich in silica, directly prepared to resemble the matrix formed in ordinary porcelain containing 25% of felspar. It follows that the quartz, depending upon its grain-size and quantity, has a determining influence upon the quantitative and qualitative development of mullite in porcelain. F. SALT.

Notes on antimony yellows. I. A. CHAMBERS and J. F. RIGG (Trans. Ceram. Soc., 1926, 25, 101—107). —Trials were made with red lead, tin oxide, and antimony oxide, every possible mixture within the 20% limit being examined. The colours were calcined at cone 09A. The colours within the range of 40-60% of lead oxide, 20-40% of tin oxide, and 20-40% of antimony oxide gave the best results, but as underglaze colours, whether in lead or leadless glazes, they were not successful. As on-glaze colours the mixtures gave satisfactory results when mixed with four pts. of No. 8 flux (3 pts. of red lead, 2 pts. of borax, and 1 pt. of flint). The yellow colour was deepened and developed by the addition of from 1 to 5% of iron oxide. The colours were also improved by slow calcination. F. SALT.

Formulæ for slip calculations. S. R. HIND (Trans. Ceram. Soc., 1926, 25, 108—110).—A collection of formulæ is given for use in calculations in connexion with slips of clays, various ground materials, and mixtures thereof. F. SALT.

Refractories for the pottery industry. W. EMERY (Trans. Ceram. Soc., 1926, 25, 111—126).—A general survey is made of the requirements of refractories used in the construction of intermittent, continuous, and frit kilns, and in the manufacture of supports for ware in the kiln. F. SALT.

Tunnel kilns for burning firebricks. I. S. R. HIND (Trans. Ceram. Soc., 1926, 25, 154—170).—The relative merits of tunnel kilns and other continuous kilns for burning firebricks are discussed. The special requirements and the chief features of design of such tunnel kilns are also outlined. F. SALT.

Some changes taking place in the low-temperature burning of Stourbridge fireclay. II. C. E. MOORE (Trans. Ceram. Coc., 1926, 25, 127-149).-Further data are put forward in support of conclusions presented in a previous paper (cf. B., 1923, 774 A). Coefficient of expansion determinations on clay bars showed a decrease at the dehydration point, an increase at 690°, a reversal up to about 800°, followed by a continuous increase with reversals at 905° and at about 1000°. Certain small changes were also observed at approximately the same temperatures on reheating a previously fired clay, indicating that some of the changes are reversible. With rapid heating, dehydration takes place in two distinct stages; with very slow heating the water evolved in the second stage is given off progressively after the first loss is complete. The first stage represents the removal of SiO₂-water; the second, that of the Al₂O₃-water. Rehydration of fired clay was effected by prolonged treatment with a 40% caustic soda solution. The results suggest that the caustic soda solution hydrates the nonpolymerised clay, decomposes it into the products normally found in the intermediate stage of the isomeric

change, and then hydrates these products. Analysis of the supernatant liquor obtained by suspending powdered clays in distilled water for 24 hrs. indicates that the clays consist of a mixture of $6Al_2O_3$, $15SiO_2$, $10H_2O$ with silica and other impurities. Contraction experiments were repeated, and the three critical ranges were again observed. The complete series of changes taking place in clays on heating is : (1) loss of hygroscopic and colloidal water ; (2) loss of SiO_2 -water ; (3) loss of Al_2O_3 water ; (4) isomeric change involving decomposition and re-formation ; (5) polymerisation in two or three stages. The results are all explained on the basis of the hexite-pentite theory. F. SALT.

Further note on the storage of silica refractories. W. J. REES (Trans. Ceram. Soc., 1926, 25, 150—153).— Four types of silica bricks, varying in texture and degree of firing, were subjected to the action of water and to "weathering" tests. The results of mechanical strength tests on these and on fresh bricks indicate that highlyfired, close-textured bricks are more resistant to the action of rain, frost, etc. than less thoroughly burned bricks. Immersion for 7 days in distilled water reduced the mechanical strength of the bricks considerably, owing to partial solution of the lime bond. Rapid drying after exposure caused greater loss in strength than slow drying. F. SALT.

Refractory materials, their testing and behaviour in foundry work. E. H. SCHULZ (Stahl u. Eisen, 1926, 46, 1667—1678).—A general discussion of the properties required in refractory materials for the various purposes of the iron industry, from the points of view, respectively, of the user and manufacturer. A plea is put forward for closer co-operation between the two, particularly in the standardisation of laboratory tests for refractories.

A. B. MANNING.

Conduction of heat through [silox] powders and its dependence on pressure and conductivity of the gaseous phase. Aberdeen and LABY.—See I.

Germanium glasses. DENNIS and LAUBENGAYER.— See VII.

PATENTS.

Glass, sand, or flint paper, emery cloth, or like abrasive. C. A. KLEIN and R. S. BROWN (E.P. 260,704, 26.8.25).—Linseed oil and paraffin or other wax is applied as a waterproofing agent to a suitable base, followed by an adhesive consisting of a drying oil with a gum, resin, or synthetic resin, and an abrasive material, the successive applications being made at temperatures above 100°. B. W. CLARKE.

Coating for [non-glare] mirror. G. F. and W. H. COLBERT (U.S.P. 1,603,936, 19.10.26. Appl., 30.4.26).— A mirror, which will reflect bright light without glare, has the reflecting surface formed of lead sulphide, produced by applying to the glass a mixture of lead acetate (1 pt.), sodium hydroxide (2 pts.), thiocarbamide (4 pts.), and water (264 pts.). Copper, nickel, and cobalt sulphides can also be employed. T. S. WHEELER.

Fusion furnace (U.S.P. 1,603,762).-See I.

IX.—BUILDING MATERIALS.

Analysis of commercial lime. T. P. BLUNT (Analsyt, 1926, 51, 625).—Caustic lime, present in commercial lime, absorbs carbon dioxide, and may be determined by grinding 0.4-0.5 g. of the sample with successive portions of distilled water up to 400 c.c. in all, pouring the extracts into a flask, adding the residue of the sample, closing the flask, and shaking for 30 min., rapidly filtering the contents into a known quantity of sulphuric acid, washing the filter once, and titrating the unneutralised acid. D. G. HEWER.

Effect of acids on the mechanical strength of timber. Alliott.—See I.

Vertical-retort tar for road purposes. BARASH.— See III.

Distilled tar for roads. ADAM and ROBINSON.— See III.

PATENTS.

Plastering walls. G. E. HEYL and O. KUNZE (E.P. 260,833, 26.2.26).—A preparation for direct application to rough brick or concrete walls, thus eliminating the usual plaster layer, is made by mixing with water, *e.g.*, 40 pts. of finely disintegrated paper or wood pulp, 10 pts. of plaster of Paris or Portland cement, 20 pts. of glue, and 30 pts. of china clay, chalk, or other lubricant to promote free flow of the fibrous particles. Pigments may also be incorporated with the mixture.

A. DAVIDSON.

Manufacture of cement, lime, and the like, and kilns therefor. E. P. C. GIROUARD (E.P. 260,684, 7.8, 25.9, and 19.11.25).—A kiln for sintering, roasting, or calcining calcareous, argillaceous, or the like materials in dry powdered form, *e.g.*, for cement making, is formed by sinking a shaft in the natural bed available in the locality, thereby minimising external heat-radiation losses. The kiln is provided with a cooling shaft in which the air for combustion is preheated by the hot clinker, and a retort situated above the kiln for the low-temperature carbonisation of powdered coal, heated by the waste gases from the kiln, the products of carbonisation being burnt in the lower part of the kiln. B. W. CLARKE.

Manufacture of quick-setting lime products. M. E. HOLMES and G. J. FINK, ASSTS. to NATIONAL LIME Assoc. (U.S.P. 1,604,574—5, 26.10.26. Appl., [A] 16.5.23; [B] 28.5.23).—[A] Commercially pure hydrated lime is mixed with a soluble sulphate of a metal which has an at. wt. between 52 and 59. [B] A metal carbonate which readily hydrolyses is added to hydrated lime, producing a cementitious material having quick initial setting properties. B. W. CLARKE.

Manufacture of quick-setting lime. J. W. STOCKETT, JUN., ASST. to NATIONAL LIME ASSOC. (U.S.P. 1,604,577, 26.10.26. Appl., 17.8.25).—Quicklime is mixed with substantially completely hydrated lime in the presence of water, and the mixture is poured immediately into a mould, where reaction occurs without agitation of the mould. B. W. CLARKE.

Production by carbonation and the addition of sulphates of plastic materials having an initial quick set. F. C. MATHERS and R. L. HARDY, ASSTS. to NATIONAL LIME ASSOC. (U.S.P. 1,604,576, 26.10.26. Appl., 25.7.23).—A plastic material having quick initial setting properties consists of the reaction product of carbonated hydrated lime and a soluble compound having a sulphate radical. B. W. CLARKE.

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

Cementation of soft steel by cyanogen and cyanamide. E. Pérot (Compt. rend., 1926, 183, 1108-1110).-The influences of time, temperature, pressure, concentration, and renewal of the gaseous atmosphere on the cementation of a steel containing 15% C in cyanogen gas have been studied. The results obtained when the gaseous atmosphere is not renewed are equivalent to those found when ethylene and methane are used in continuous circulation, and indicate that, other conditions being equal, cyanogen is a superior medium for cementation. Except so far as concerns the hypereutectoid zone, the total penetration is less with cyanogen in an atmosphere which has not been renewed than when the sample is heated in contact with tricalcium cyanamide. The micrographical structures are similar, especially at low temperatures.

J. GRANT.

Determination of silicon in pig iron and cast iron. H. PINSL (Chem.-Ztg., 1926, 50, 924-925).—The results obtained for silicon in pig iron and cast iron by the method of Rubricius (Stahl u. Eisen, 1905, 25, 1444; cf. also B., 1905, 988) are usually a little low unless the filtrate from the first precipitate is again evaporated. The longer the heating with the strong sulphuric acid the more easily does the precipitate filter; 1.5 hrs. gentle boiling is recommended. More accurate results with only one filtration are obtained if 20 c.c. of 1:1 hydrochloric acid are added after the first evaporation and evaporation is repeated, followed by heating for 45 min. at 130-140°. A close-textured filter paper fitted closely into the funnel should be used for the filtration; subsequent ignition of the precipitate leaves a residue of almost pure silica, which is usually 0.05%less than the correct weight, the small amount of impurity present compensating for part of the loss of silica in the filtrate; for commercial work recovery of the traces of silica in the filtrate may be dispensed with, and no evaporation with hydrofluoric acid is necessary. The sulphuric acid method is quicker and more accurate than the usual nitric-hydrochloric acid procedure.

A. R. POWELL.

Equilibrium between iron, carbon, and oxygen. Reduction of iron ores, cementation, and gas occlusion of iron and steel. K. Iwasé (Sci. Rep. Tôhoku Imp. Univ., 1926, 15, 511—529).—Existing data are used to construct a ternary equilibrium diagram of the system iron-carbon-oxygen. The use of this to determine the mechanism of the reduction of iron oxides is described. The reduction of the magnetic iron oxide by carbon takes place with the formation of the following successive phases :—

 $\operatorname{Fe_3O_4} \longrightarrow [\operatorname{Fe_3O_4}] \longrightarrow [\operatorname{FeO}] \longrightarrow [\operatorname{Fe}] \longrightarrow [\operatorname{Fe_3C}]$, where the brackets indicate a solid solution based on the particular component. A small region at the iron end of the system has no gaseous phase, and here simple occlusion (*i.e.*, absorption without alteration in the composition of the gas) can take place with particular mixtures of carbon monoxide and dioxide. The mechanism of cementation in the presence of absence of oxygen is also discussed. With a particular gas mixture the equilibrium degree of cementation is lowered with rise of temperature, but the velocity of cementation is increased. W. HUME-ROTHERY.

Nitrogenisation of steels. L. GUILLET (Compt. rend., 1926, 183, 933-935).-Brinell tests have been carried out on a tempered case-hardened steel, and on a chromealuminium steel which had been nitrogenised by means of ammonia for 90 hrs. at 510°, so as to produce a nitrogenised layer 0.8 mm. thick. The latter steel had the greater initial hardness, and this was retained to a greater extent than in the case of the former, when the steels were maintained at gradually increasing temperatures (from -180° to 600°) for various periods of time. This difference was more marked when the hardness tests were carried out at 20°. After a period at the temperature of liquid air there was a distinct increase in the hardness at 20° of the nitrogenised steel, but none in the case of the other. In general, the hardness decreased as the temperature rose. J. GRANT.

Diamond cone indentation hardness tests. G. A. HANKINS (Proc. Inst. Mech. Eng., 1926, 823-842) .---Accurate and reliable indentation tests can be made on hard materials with diamond cones. The indentations are easily measured and the results expressed on a rational scale. Surface lubrication had no effect on the size of the indentations. Tests made with cones of various angles gave results which were independent of the load, but dependent on the angle of the cone, the effect of the cone angle being less on hard material than on soft. The coefficient of adhesion or friction for hard steel was found to be 0.10. The corrected contact pressure $P/\pi r^2(1 + \mu \cot \theta)$ was independent of the angle of the cone from 78° to 120°, but 142° cones gave low values. Results expressed in this way were numerically the same as Brinell numbers up to 440. Above this value the Brinell numbers were lower owing to flattening of Tests with diamond pyramids had fair the ball. agreement with the cone tests up to a hardness of 500 with the latter, but fell away above this figure, and the shape of the pyramid impressions was not constant. The Rockwell test, based on depth of indentation, showed no general agreement with the pressure scales. The most suitable diamond cone has an angle of 120°, the standard load being 100 kg. The impressions must be greater than the diameter of the spherical tip. For thin sheets the point must be accurately ground to permit the use of lower loads. A fairly good surface on the specimen is desirable, but a high polish unnecessary. The 120° diamond cone is recommended for use on materials above 300 Brinell hardness, and can be used on softer materials when the values of the coefficient of adhesion are known. T. H. BURNHAM.

Comparison between Rockwell and Brinell hardness. R. C. BRUMFIELD (Trans. Amer. Soc. Steel Treat. 1926, 9, 841—856).—Hardness determinations were made using the Rockwell hardness tester on various metals, and the results plotted against the respective Brinell hardness numbers. The Brinell test is the more sensitive except for soft metals. Equations were established for the conversion of the various hardness figures. T. H. BURNHAM.

Durometer-an instrument for testing hardness. A. SAUVEUR (Trans. Amer. Soc. Steel Treat., 1926, 9, 929—932).—A hardened steel ball of 10 mm, diameter is allowed to drop on the specimen held at 45° and rebounds on to a horizontal graduated paper. The rebounding impulse increases with the hardness of the sample, and the scale reading of the position of fall is called the durometer number. T. H. BURNHAM.

Apparatus for measuring Hertzian hardness. ESNAULT-PELTERIE (Rev. Mét., 1926, 23, 553–566).

Measurement of hardness by the pendulum. P. LE ROLLAND (Rev. Mét., 1926, 23, 567-574).

Measurement of hardness by the Rockwell machine. M. NICOLLET (Rev. Mét., 1926, 23, 575– 580).

Magnetostriction. L. W. McKEEHAN (J. Franklin Inst., 1926, 202, 737-773).—A review of recent work.

Refractory materials, their testing and behaviour in foundry work. SCHULZ.—See VIII.

PATENTS.

Treating solid ferrous material. S. WESTBERG (E.P. 260,646, 2.6.25).—For the removal of impurities contained in iron or its alloys the material is embedded in an alkaline-earth oxide and heated to a temperature between 800° and the m.p. of the alloy in a mixture of hydrogen and an inert gas. In the elimination of sulphur, the hydrogen sulphide formed combines with the surrounding oxide, and is removed from the gaseous phase. Other materials, *e.g.*, aluminium silicide, which form stable compounds with the impurities and at the same time regenerate hydrogen, may be used to pack the metal or alloy. C. A. KING.

Manufacture of wrought iron. G. G. GEDDA (E.P. 260,789, 28.11.25).—In the ordinary process of manufacturing wrought iron, nickel-bearing material may be part of the charge of the blast furnace, thus producing pig iron containing nickel; or nickel or a nickel-iron alloy is introduced into the refining furnace together with pig iron. Cobalt may be substituted wholly or in part for nickel, and the wrought iron may contain also a small quantity of copper for the further protection from rusting. C. A. KING.

Alloy steels. H. B. KINNEAR (E.P. 260,835, 2.3.26). —An alloy steel, made by melting an ordinary carbon steel of the composition 0.2-0.6% C, 0.6-0.9% Mn, 0.2-0.6% Si, and under 0.05% of S and P, with 0.5-5% of copper, is poured into an ingot or cast, normalised by heating it at about 850° for a short time, and then slowly cooled in air. The treatment is repeated at about 550°. By varying the heat treatment it is possible to produce castings meeting different specifications from a single grade of metal. M. E. NOTTAGE.

Producing pure iron from its ore. C. E. PARSONS and S. PEACOCK, ASSTS. to METAL RESEARCH CORP. (U.S.P. 1,603,710, 19.10.26. Appl., 17.10.23).—Pure iron may be produced from iron oxide free from carbonaceous material by roasting it, in the presence of a reducing gas free from sulphur, at 800—1000°.

M. E. NOTTAGE.

Reduction of rare metal oxides. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,602,542, 12.10.26. Appl., 6.1.21).—A compound of the zirconium, titanium, thorium, uranium, tungsten, molybdenum. etc. groups is heated with magnesium in the reducing atmosphere of a closed vessel, producing a refractory metal powder. H. ROYAL-DAWSON.

Casting molten metal. A. KADOW (E.P. 260,743, 7.10.25).—Molten metal enters a partially evacuated mould through openings having a maximum width of 3/32 in. in the bottom of the mould. Increase in surface tension due to the chilling effect of the mould allows the mould when full to be lifted away from the molten metal without any further metal escaping from the narrow openings. C. A. KING.

Treating aluminium to facilitate its welding and soldering. A. KIRCHHOF (U.S.P. 1,604,698, 26.10.26. Appl., 8.10.25. Conv., 7.3.24).—Pulverised calcium carbonate is mixed with molten aluminium from which the scum is removed. H. ROYAL-DAWSON.

Alloys [for jets used in spinning artificial silk]. W. P. DREAPER (E.P. 260,672, 5.8.25).—An alloy of platinum and gold, containing 20—32% Pt, with or without small additions of other metals of the platinum group, is specially suitable. M. E. NOTTAGE.

Zinc blende roasting kiln. G. BALZ (U.S.P. 1,603,442, 19.10.26. Appl., 9.3.25. Conv., 21.3.24) .---A zinc blende roasting kiln consists of superposed parts forming compartments, the bottom of every upper one of which forms the cover or ceiling of the next lower one, every second part in the downward direction being rotatory. The zinc blende is introduced into the uppermost compartment, and then conveyed, alternately, radially outwards and inwards through the successive compartments. It is provided with an air-heating chamber below the lowest compartments, and with means for supplying air to this chamber and discharging it into the discharge passage for the roasted zinc blende. Pipes extend radially inwards from the outside tonear, the centre of one of the intermediate hearths upon which the blende is moved radially inwards and delivered into the compartment below through a central aperture near which the pipes terminate. Means are provided for introducing cold air into these pipes. M. E. NOTTAGE.

Sintering plant. J. E. GREENAWALT (E.P. 260,895, 27.5.26).—Ore sintered in a rotating pan is dropped into a hopper beneath the pan, the bottom of which hopper consists of a perforated table on which the sinter is cooled by a current of air. The table has preferably a reciprocating motion which delivers the material on to a suitable screen for the separation of fines, and delivery of the coarser material into trucks. If necessary, the hopper is lined with cast iron, and is provided with a chimney for removing floating dust. C. A. KING.

Machine for ore separation. J. F. REILLY (U.S.P. 1,603,213, 12.10.26. Appl., 18.12.23).—An oscillatory table contains a number of compartments with a porous covering. A horizontal pipe with a line of perforations on its upper side passes through each compartment. An upward flow of air is thus directed through the porous covering, and means are provided for independently controlling the supply of air to each compartment, for retarding the flow of material over the surface of the table, and for feeding granules of crushed ore over the table. M. COOK.

Vanadium compounds (E.P. 260,661).-See VII.

XI.—ELECTROTECHNICS.

PATENTS.

Precipitating apparatus. I. HECHENBLEIKNER (U.S.P. 1,604,553, 26.10.26. Appl., 7.2.23).—In an apparatus for electrical precipitation, parallel vertical flues of non-conducting material and polygonal in section are situated in a chimney stack. Electrode wires are arranged centrally in each flue, with conducting supports for the upper ends of the wires, and weights at the lower ends, with a device for spacing the wires and preventing their movement. W. G. CAREY.

Rendering liquids radio-active. J. G. VAUGEOIS (E.P. 250,935, 9.4.26. Conv., 14.4.25).—The radioactive material is separated from the liquid to be treated by an air chamber and by a porous partition permitting the passage of emanations to the liquid. The container in which the radium salt or solution is placed passes through the stopper of the vessel containing the liquid, and opens into the interior of this vessel in the free space above the liquid. W. G. CAREY.

Ionic-concentration meter. E. A. KEELER, Assr. to LEEDS & NORTHRUP Co. (U.S.P. 1,604,584, 26.10.26. Appl., 7.5.20).—The method of measuring $p_{\rm H}$ values, in which a deflecting galvanometer is employed to determine the voltage between a hydrogen and a calomel electrode immersed in the solution under test, is modified by impressing on the galvanometer a voltage equal and opposite to the voltage between the electrodes, when the solution has the maximum hydrogen ion concentration. The whole scale of the galvanometer is then available for graduation in $p_{\rm H}$ values. T. S. WHEELER.

Method for and means of separating electrolytes. J. B. SPEED, Assr. to WESTERN ELECTRIC Co. (U.S.P. 1,603,298, 19.10.26. Appl., 17.12.24).—Pressure is applied to a mixture of electrolytic solutions contained in an enclosure, two walls of which are formed of porous inert conducting plates, while a potential difference, insufficient to effect electro-deposition, is applied between electrodes. The applied pressure forces a considerable portion of the liquid through the plates.

J. S. G. THOMAS.

[Cathode] for electrodeposition. INTERNAT. COPPERCLAD Co., Assees. of T. ROBINSON (E.P. 255,114, 4.8.25. Conv., 5.8.24). — A cathode comprises two electrically connected body portions, separated by insulating material. They are secured to a supporting bar, and are of conducting material covered with a thin film of metal different from that to be deposited on the cathode. J. S. G. THOMAS.

Manufacture of electrical insulators and other moulded articles and the like. A. H. BROWN and THE SILUMINITE INSULATOR CO., LTD. (E.P. 260,653, 8.7.25).—An intimate mixture of a binding material, e.g., Portland cement, with less than 10% of incompletely condensed synthetic resins, together with, if desired, one or more fillers, is moulded without the addition of water, and, after removal from the mould, is subjected to heat treatment. The moulded article, after it has set and hardened, may be impregnated with a liquid synthetic resin and subjected to final heat treatment.

J. S. G. THOMAS.

Making electric-insulation bodies. D. M. SUTHER-LAND, JUN. (U.S.P. 1,604,728, 26.10.26. Appl., 2.2.26).— A small proportion of acaroid resin and a relatively large proportion of gilsonite are added to a pulp composed of cellulose and oil soap. The mass is shaped, dried, compressed while heated under pressure, and cooled while still under pressure. J. S. G. THOMAS.

Battery electrode. D. PEPPER (U.S.P. 1,603,291, 19.10.26. Appl., 30.7.24).—Preformed lead material is mixed with a paste of sulphuric acid and zinc sulphate and applied by pressure to a conducting support and dried. J. S. G. THOMAS.

[Depolariser for] dry batteries. Soc. ANON. LE CARBONE (E.P. 255,426, 1.6.26. Conv., 17.7.25. Addn. to 228,872; cf. B., 1926, 550).—The depolariser consists of powdered charcoal distributed throughout an immobilised material, and covered by a film rendered impermeable to liquids prior to its incorporation in the mass containing the electrolyte by being mixed with paraffin oil. J. S. G. THOMAS.

XII.—FATS; OILS; WAXES.

Plant for fat extraction. L. J. SIMON and J. W. HINCHLEY (J.S.C.I., 1926, 45, 252-259 T).-Extraction of oil-seeds in a 3-4 ton vessel leads to loss of time due to the long time occupied by the flow of solvent through a large mass of meal, involves distillation of weak solutions of oil, and further involves much labour and loss of time between each extraction during operations of discharging and re-charging with crushed meal. A long period of steaming the meal for removal of solvent results in a high percentage of moisture being present, and the length of time during which meal and oil are subjected to heat and moisture may lead to deterioration of the oil. These objections are overcome in a plant holding only 4 cwt. of material at one time, though the output is 3-4 tons in 8 hrs. Total time of extraction is about 30 min., and total time of "steaming off" of the meal to remove solvent is 4-6 min., these results being achieved by preheating the meal to the temperature of steam, by the fact of steam having to percolate through only a few inches of material, and all meal being in contact with solvent for the same length of time. The distillation of the oil solution is taking place continuously, only solvent well saturated with oil entering the stills. In the plant described only about 1 ton of solvent is needed to treat 3 tons of meal, as against 7 tons when 3 tons of meal are treated at one charge. The cage holding the meal consists of a perforated drum carried on a hollow shaft through which the solvent and steam are allowed to enter-the meal being charged into the cage by removal and replacement of one of the end plates-and the cage is inserted into a cylinder carrying the gear for rotating it. Each machine carries three such cylinders with their cages, each operated separately. After the operation of charging and discharging, automatic hydraulically - controlled valves operated by means of a timed " cam " shaft which itself determines the course of all the operations from the entry of the solvent until the extraction and "steaming off " are completed. This timed shaft makes one complete revolution in 32 min., by which time an extraction

is completed and the cage containing the meal is ready for removal. The solution tanks and solutions are collectively dealt with, the complete plant having in addition the usual condenser and separator, solvent tank, six solution tanks, one solvent measuring tank, and a distilling apparatus. The operation of the machine consists of (1) a preliminary treatment of the dried meal with solvent vapour; (2) a washing of the material with a concentrated solution of oil and solvent to obtain a rich solution for distillation; (3) a second treatment with solution which is used for the next charge for operation 2; (4) a third treatment, which is used in the next charge for operation 3; (5) a final treatment with pure solvent; (6) a drying period, in which most of the solvent is expelled from the meal by centripetal force, the material being warmed by indirect steam; and (7) steaming off with direct steam to remove the last traces of solvent from the meal. This method of working ensures a highly concentrated oil solution for distillation in a regulating still which, while continuously evaporating the solvent by means of a closed steam coil, converts the intermittent flow which it receives into a regular stream, which enters a set of six small stills coupled in two equal sets in parallel. During periods 1 to 5 the cage is rotating slowly, ensuring constant agitation of the meal, and in periods 6 and 7 rapidly so as to form the meal into a cylinder with a wall of even thickness which allows steam to pass through quickly, ensuring effective removal of the last traces of solvent. Only the final flush is clean solvent, and the total solvent used for each charge is only 16-20 gallons. The claims for the system are (1) more complete extraction, (2) less use of solvent, (3) lower steam consumption, and (4) dryer meal after extraction. H. M. LANGTON. (Cf. E.P. 255,923; infra.)

Phytosterols of rice-bran fat. F. P. NABENHAUER and R. J. ANDERSON (J. Amer. Chem. Soc., 1926, 48, 2972 -2976).—Extraction of rice-bran with light petroleum yielded about 10% of an oil, consisting largely of free fatty acids. The unsaponif. matter (about 5%) contains myricyl alcohol (cf. Jamieson, J. Oil Fat Ind., 1926, 3, 256), dihydrositosterol (Anderson, Anderson, and Nabenhauer, B., 1924, 691, 729), stigmasterol (Windhaus and Hauth, A., 1907, i. 129), and a phytosterol, probably not the homogenous sitosterol of Weinhagen (A., 1918, i, 56). No substance resembling the latter's saturated hydrocarbon (*loc. cit.*) was identified. Some oily constituents (b.p. 100—260°/1mm.) were not fully identified. F. G. WILLSON.

Phytosterols of corn [maize] oil. R. J. ANDERSON and R. L. SHRINER (J. Amer. Chem. Soc., 1926, 48, 2976—2986).—The crystalline unsaponif. matter present in maize oil is a mixture of dihydrositosterol, stigmasterol (cf. Anderson and Moore, B., 1923, 937 A), and three isomerides, α -, β -, and γ -sitosterol, of which only the last was obtained pure. The crystals have the formula $C_{27}H_{45}OH,H_2O$, m.p. 145—146°, $[\alpha]_D$, in chloroform solution, $-42 \cdot 43^\circ$; γ -sitosteryl acetate has m.p. 143—144°, $[\alpha]_D$, in chloroform solution, $-46 \cdot 09^\circ$. Dibromo- γ -sitosteryl acetate, m.p. 136—137°; dihydro- γ -sitosterol, from the parent hydrocarbon by reduction with hydrogen in presence of platinum-black, m.p. 144—145°, $[\alpha]_D$, in chloroform solution, $+ 17 \cdot 82^\circ$, and dihydro- γ -sitosteryl acetate, m.p. 143°, $[\alpha]_D$, in chloroform solution, $+ 8 \cdot 98^\circ$, are also described. γ -Sitosterol is possibly identical with the *p*-phytosterol of Likiernik (A., 1891, 606). It appears that the sitosterol previously described by Anderson and Moore (*loc. cit.*) is a complex mixture of isomerides. F. G. WILLSON.

Phytosterols of wheat-germ oil. R. J. ANDERSON, R. L. SHRINER, and G. O. BURR (J. Amer. Chem. Soc., 1926, 48, 2987—2996).—The crystalline sterols in American-grown wheat-germ oil contain no pure homogeneous sitosterol, but constitute a mixture of α -, β -, and γ -sterols (cf. preceding abstract; Burian, A., 1898, i, 72; Ritter, A., 1902, i, 446) and dihydrositosterol, with possibly other constitutents. As with maize oil (cf. preceding abstract) only γ -sitosterol could be obtained pure. American-grown wheat appears to contain different sterols from wheat grown in Europe. F. G. WILLSON.

Methods of saponification in the light of modern saponification theory. C. BERGELL (Z. deuts. Oel- u. Fett-Ind., 1926, 46, 737-738, 753-754, 769-770).-The first two sections of this paper deal mainly with the empirical development of soap-boiling processes during the last century. It is then shown that the velocity of saponification $= v = KC_1C_2$, where C_1 and C_2 are the concentrations of esters and alkali respectively, and since C_1 and C_2 decrease in geometric progression, v also decreases. This equation would hold for a molecularly homogeneous solution, but in the case of emulsions in soap boiling a modification results from the fact that the surface of the fat globules does not vary directly with the decrease of weight of fat as the saponification proceeds. Experiments are then described in which specimens of coconut oil and tallow are saponified at varying temperatures, and with varying amounts of alkali in excess of that theoretically required, the results of acid value determinations being tabulated and plotted. When the amount of saponification per unit time is plotted against time a minimum occurs in the first third of the curve in each case, this being the point at which the lye in oil emulsion changes over to an emulsion of oil in lye, the actual position varying with conditions of temperature, concentration of lye, and the kind of fat. In one case separation occurred half-way through the experiment, and the rate of saponification fell off steeply. The general conclusion is that the maintenance of a good emulsion is more important than a high saponification temperature, and the author is of opinion that the cold saponification process merits much wider application in the soap industry. E. HOLMES.

Saponification of olive oil. G. I. FINCH and A. KARIM (J.S.C.I., 1926, 45, 469-472 r).—Olive oil was saponified with equivalent quantities of aqueous caustic soda solutions of various strengths under various conditions of stirring. An optimum alkali concentration of 29.25% was found, leading, with suitable stirring conditions, to a 98% saponification within 30 min.

Acetin and dichromate methods [for glycerin analysis]. O. SACHS and K. RIEMER (Z. deuts. Oel- u. Fett-Ind., 1926, 46, 739-740).—Following the communication of Prager (B., 1926, 923), the authors tabulate the results of numerous analyses of glycerin by these two processes, the differences in no case being in excess of 0.11%. They consider the acetin process

equal in accuracy to the dichromate process, over which it has the advantage of quicker manipulation, and are strongly in favour of its retention. H. M. LANGTON.

Rate of polymerisation of perilla oil. M. Toch (J. Oil Colour Chem. Assoc., 1926, 9, 309-318).-The thickening of heated perilla oil was studied both in the laboratory and under factory conditions, changes in sp. gr., refractive index, acid value, iodine value, and mol. wt. being recorded. Very little change occurs at low temperatures, but at 304° the iodine value decreases rapidly, while the remaining constants show a corresponding increase, the rates of these changes falling off with the time the oil is maintained at 304°. The changes are compared with the very similar ones for linseed oil, perilla oil being shown to lag at lower temperatures, but to polymerise faster than linseed oil at the high temperature. The mechanism of oil polymerisation is briefly discussed. S. S. WOOLF.

Iodine value of paprika oil. L. C. MITCHELL (J. Assoc. Off. Agric. Chem., 1926, 9, 477–482).—The oil extracted from paprika is unstable, and special precautions are necessary to prevent oxidation, or low iodine values will be obtained. A simplified method, which yields results in close agreement with the anhydrous ether method, and is also much quicker, consists in extraction with chloroform, and determination of the quantity of iodine absorbed and of the weight of oil used on aliquot portions of the chloroform solution.

F. R. Ennos.

Tertiary bromine-iodine values of drying oils. W. VAUBEL (Farben-Ztg., 1926, 32, 514).—To the primary and secondary bromine-iodine values previously described by the author (cf. B., 1926, 887) a tertiary value is added, obtained by the action of excess of bromine for three days (in the secondary value the excess is immediately determined). The calculated bromine-iodine values for a number of drying and non-drying oils, and of oil films at various ages, are tabulated. The tertiary values, which are much in excess of previous highest iodine values, sometimes rise on the ageing of oil films, whereas primary and secondary values generally fall. The significance of this new value and its effect on the oil analyses previously given (*ibid*.) are discussed. S. S. WOOLF.

Importance of soya-bean oil for softening and increasing the stretch of cold-vulcanised rubber. DITMAR.—See XIV.

PATENTS

Extraction of oils, fats, waxes, greases, and the like. L. J. and A. SIMON, and SIMON BROS. (ENGINEERS), LTD. (E.P. 255,923, 20.4.25 and 17.6.25).—Improved results are achieved in a machine which treats small quantities of meal at a time, leading to a high rate of solution of fatty oil and thorough removal of solvent from meal. Solvent is conveyed by large entry pipes on to a few inches depth of meal contained in a rotating cage, whereby continuous agitation with solvent occurs, and the amount of liquor left in the meal on drainage is a minimum. The cage consists of two end plates on a hollow, perforated, central shaft with gauzes, and/or with filter cloth fixed to the periphery of the plates as well as around the shaft. The charged cage is placed in a cylinder carrying the gear for rotating it, several such cylinders being contained in each machine. Coupled with the machine are a large tank A, which has a constant supply of solvent, and two smaller tanks, B, C, one for preliminary extraction, and one for the final flush and steam, and these adjoin and open into A, from which



they can be isolated. A cam-operated hydraulic contrivance controls the whole cycle of operations, at the start of which a charged cage is placed in the small tank B, in which it is subject to a preliminary solvent flush, transferred to the large tank A, capable of holding several such cages, and subsequently to the small tank C



for giving the material a final flush of solvent and for steaming off solvent from the meal. Means are provided for supplying solvent or steam to the centre of the cages through the perforated shaft thereof. The tanks are provided with the usual coils, and stills, condensers, and solvent and solution storage tanks form part of the installation. (Cf. Simon and Hinchley, *supra*.)

H. M. LANGTON. cooling oil vapours (E.P. 240 415)

Apparatus for cooling oil vapours (E.P. 240,415).— See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Noxious solvents and thinners used in paints and adhesives. A. BEYTHIEN (Farben-Ztg., 1926, 32, 567— 569).—The harmful effects of the vapours arising in industrial operations involving the use of special paints and adhesives are indicated, and the ingredients of several technical preparations are discussed from this viewpoint. S. S. WOOLF. Colloid-chemical studies on chrome yellow. L. BOCK (Farben-Ztg., 1926, 32, 459-460).—The improvement in the tone of pale chrome yellows by co-precipitation with colloidally-dispersed inorganic substances, *e.g.*, aluminium hydroxide, lead sulphate, aluminium phosphate, lead arsenite, etc., is also effected by the use of organic substances such as animal or vegetable glues. Whilst these protective colloids maintain or increase the degree of dispersion of the precipitated pigment, the latter is still subject to the same darkening under the action of light as is pure (normal) lead chromate. S. S. WOOLF,

Cobalt blues and allied colours. J. J. Fox (J. Oil Colour Chem. Assoc., 1926, 9, 304—308).—An account of the nature, details of preparation, uses, and methods of analysis of various cobalt pigments. S. S. WOOLF.

U.S. Government master specification for turpentine (gum spirits of turpentine and steamdistilled wood turpentine) (U.S. Bureau of Standards, 1926, Circular No. 86, 1–10).—The requirements for either type of turpentine are as follows: $d_{15.5}^{15.5} 0.860 - 0.875$; $n_D^{\circ0} 1.465 - 1.478$; after polymerisation with 38N-sulphuric acid, not more than 2%of a viscous, straw- or darker-coloured residue, having $n_D^{\circ0} 1.500$ (minimum) shall be obtained; on distillation under specified conditions, the initial b.p./760 mm. shall be 150-160°, whilst at least 90% shall distil below 170°/760 mm. Details of apparatus and methods for sampling and testing are given. The specification does not cover destructively distilled wood turpentine.

S. S. WOOLF.

Fluorescence- and capillary-analysis of resins. H. WOLFF and W. TOELDTE (Farben-Ztg., 1926, 32, 294— 296).—To avoid errors in fluorescence analysis of resins due to the condition of the latter, it is proposed to examine, under the analytical quartz lamp, the "adsorption pictures" obtained under standard conditions by Stock's method of capillary analysis (cf. B., 1926, 679). A full description of the characteristic phenomena exhibited by various resins and resin mixtures when treated in this way is given. S. S. WOOLF,

Industrial resinates. R. UZAC (Rev. gén. Colloid., 1926, 4, 257-268; cf. A., 1925, i, 1295).-Resin soaps contain an acid resinate which can be extracted by means of toluene. In the gumming of paper an aluminium resinate, containing free resin, is formed by addition of aluminium sulphate to the resin. Resin alone fixes on the fibre, but the gum is not permanent. On the other hand, treatment of the paper with alcohol causes a loss in adhesive power, due to dissolution of free resin. It is shown that a colloidal complex is formed between aluminium resinate and resin, and it is suggested that, since cellulose readily adsorbs aluminium hydroxide, a colloidal complex of aluminium resinate, resin, and cellulose is formed at the surface of the paper. A study is made of the viscosities of preparations of the resinates of lead, zinc, calcium, and manganese, and it is concluded that colloidal complexes of basic resinates and free resin are formed, rather than complexes of metallic oxides and resin. E. S. HEDGES.

PATENTS.

Preparation of paint filler. T. G. GAUDRY, Assr. to A. HOOLAHAN (U.S.P. 1,604,904, 26.10.26. Appl., 12.5.23).—An aqueous solution of oxalic acid, alum, and washing soda is added to a methylated spirit solution of rosin under steam pressure, and after successive additions of aqueous solutions of potassium hydroxide, zinc.sulphate, lead acetate, gum arabic, sodium hydroxide, and sodium silicate, the whole is stirred in a large volume of water. S. S. WOOLF.

Treating paints [to improve flexibility]. R. MEZGER, Assr. to P. LECHLER (U.S.P. 1,604,930, 26.10.26. Appl., 7.9.23. Conv., 1.8.22).—Paints containing nonaqueous vehicles are rendered more flexible on hardening by adding a small amount of a soluble salt of an irongroup metal, and precipitating the insoluble hydroxide in the paint by adding an alkali hydroxide, the amount of water so introduced being less than the amount of non-aqueous vehicle. S. S. WOOLF.

Making ["non-breaking"] varnish oil. A. SCHWARCMAN, Assr. to SPENCER KELLOGG & SONS, INC. (U.S.P. 1,604,425, 26.10.26. Appl., 27.6.25. Cf. U.S.P. 1,407,952; B., 1922, 301 A).—Raw linseed oil is emulsified with a solution of zinc sulphate (0.1%) Zn on the oil treated), and an equivalent quantity of sodium hydroxide is added. The mixture is heated under reduced pressure to evaporate the water present and filtered. The product can be heated to 260° without clouding ("breaking"). Other freshly-precipitated, hydrated oxides, which do not catalyse the drying of the oil, *e.g.*, hydrated alumina or lime, can also be employed. T. S. WHEELER.

Method and apparatus for removing large-sized particles and aggregates from certain finely divided powders and the like (E.P. 260,741).—See I.

Manufacture of articles having surfaces capable of producing diffraction colour effects (E.P. 260,669). —See I.

Separators for removing entrained oil from hot vapours (E.P. 260,899).—See I.

Manufacture of electrical insulators and other moulded articles (E.P. 260,653).—See XI.

Photographic medium [or varnish] (U.S.P. 1,604,674).—See XXI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Smoked sheet rubber prepared with dinitro-ocresol. H. P. STEVENS (Bull. Rubber Growers' Assoc., 1926, 8, 594—596).—Experiments with smoked sheet rubber in the preparation of which 0.2 g. or 0.5 g. of 3:5-dinitro-o-cresol was introduced with the coagulant into 3000 c.c. of latex, confirm the earlier finding with unsmoked sheet that this substance prevents the formation of mould, and does not appreciably influence the vulcanising properties of the rubber. D. F. TWISS.

Importance of soya-bean oil for softening and increasing the stretch of cold-vulcanised rubber. R. DITMAR (Gummi-Ztg., 1926, 41, 535-536).— "Dipped" goods containing up to 20% of soya-bean oil (calculated on the rubber) and vulcanised by immersion in a solution of sulphur chloride in carbon disulphide show a high stretch. If vulcanised in sulphur chloride vapour, or if containing a higher proportion of the oil, the products deteriorate rapidly. D. F. Twiss. Preparation of a standard plantation rubber. F. B. JONES (Trans. Inst. Rubber Ind., 1926, 2, 180– 200).—The ideal plantation rubber should be as uniform as possible in plasticity and rate of vulcanisation, in order to reduce the tendency to pre-vulcanisation and "scorching" during manufacture; relatively slow vulcanisation is, however, an advantage. For the increase of uniformity, latex should be bulked as far as possible, and the conditions of coagulation and aftertreatment carefully standardised. D. F. Twiss.

Connexion between constitution and accelerator action of diarylthioureas and diarylguanidines. W. J. S. NAUNTON (J.S.C.I., 1926, 45, 376-384 t).-For the study of the connexion between chemical constitution and vulcanisation accelerator action the substances to be tested should be free from impurities which could possibly enhance the action (*i.e.*, that could give the "two-accelerator effect"), and the greatest care is necessary in making the vulcanisation tests. Standardisation of every operation, from milling of the raw rubber to temperature control of the tensile tests, is essential. The work necessitated the characterisation of the hitherto undescribed di-m-tolylguanidine, m.p. 116-117°, di-o-xylylguanidine, m.p. 247-248°, di-pxylylguanidine, m.p. 165-166°, di-β-naphthylguanidine, m.p. 200-200.5°, di-p-nitrophenylguanidine, m.p. 222° and tetramethyldiaminodiphenylguanidine, m.p. 162-163°. It was found both in the diarylthiourea and diarylguanidine series that groups which tend to increase the aromatic nature of the molecule tend to increase the accelerator activity, whereas groups which intensify the aromatic nature (i.e., electro-negative groups) decrease the potency; thus the dinitrodiphenyl guanidines have practically no accelerator action. Hydroxyl groups decrease the activity in a zinc oxide mix, but the metallic salts of such compounds are more active in a pure rubbersulphur mix than the parent substances. The nitroderivatives are capable of vulcanising rubber in the presence of litharge and the absence of sulphur, but have no interest as accelerators in either zinc oxide or litharge mixes. The basic derivatives, especially in the thiourea series, exhibit marked superiority over the parent substances when used in mixes containing substitute, and also confer better ageing properties, as demonstrated by rapid ageing at 70° upon vulcanisates containing them. The introduction of a second thiourea group in the same molecule results in a less active accelerator instead of a more active ; the increased insolubility of such substances may, however, partly account for this effect. A simple attachment for recording automatically the loads at given extensions of the dumb-bell in the testing machine is also described.

Analysis of vulcanised rubber. Direct determination of free carbon. E. KAHANE (Caoutchouc et Gutta-Percha, 1926, 23, 13331—13334).—Cautiously treat 1 g. of the rubber in small pieces with 6—8 c.c. of fuming nitric acid; after the initial vigorous action, boil the mixture for 5 min., dilute to 50 c.c., and keep hot until decantation is possible. Decant into a Gooch crucible, the bottom of which is covered by a disc of filter paper, wash with warm dilute nitric acid, and then repeatedly with a mixture of 20 c.c. of boiling water, 2 c.c. of acetic acid, and 5 c.c. of concentrated ammonia solution. Drain with the aid of suction until the solid in the crucible can be detached and returned (together with the paper) to the beaker. Repeat the previous treatment, but using 5 c.c. of fuming nitric acid and washing with dilute nitric acid and with boiling water after the ammoniacal solution. Dry 2 hrs. at 120° and weigh with a similarly-treated disc of filter paper on the other pan of the balance. Finally oxidise the carbon at a dull red heat. The loss in weight represents 110% of the content of free carbon.

D. F. Twiss.

Physical properties of caoutchouc. M. LE BLANC and M. KRÖGER (Kolloid Z., 1926, 40, 143-144).--Explanatory in reply to van Rossem and van der Meyden (cf. B., 1926, 502). E. S. HEDGES.

PATENTS.

Accelerator for the vulcanisation of rubber and method of making same. L. B. SEBRELL, Assr. to GOODYEAR TIRE AND RUBBER Co. (U.S.P. 1,604,199, 26.10.26. Appl., 4.4.24).—A mercaptobenzothiazole is produced by heating a mixture of an aryldithiocarbamate with sulphur to between 100° and 400° under a pressure of 100—3000 lb./sq. in. D. F. Twiss.

Method of vulcanising caoutchouc and product obtained thereby. C. M. CARSON, ASST. to GOODYEAR TIRE AND RUBBER CO. (U.S.P. 1,603,317, 19.10.26. Appl., 15.2.24).—Rubber is mixed with a vulcanising agent and an aminothiophenol and then vulcanised.

D. F. Twiss.

Rubber-vulcanisation accelerator. W. Scott, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,606,321, 9.11.26. Appl., 17.12.24).—A mixture of plasticised rubber and guanidine polysulphide is vulcanised. S. S. Woolf.

Liner for rubberised material and method of treating same. H. A. ENDRES, ASST. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,603,334, 19.10.26. Appl., 4.11.22).—A liner is treated with a composition containing free sulphur and a material capable of wetting the sulphur. D. F. TWISS.

Making a rubberised fibre composition. J. M. GILLET, ASST. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,603,335, 19.10.26. Appl., 31.7.22).—Rubber is dissolved in a mixture of toluene and alcohol above a temperature y indicated by the equation: 7y - 33x = -563, where x represents the percentage of alcohol; precipitation of the rubber is then effected by reducing the temperature of the solution below the level represented by the equation 7y - 33x = -605.

D. F. Twiss.

Manufacturing a rubberised fibre composition. J. T. CHARLESON, ASST. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,603,318, 19.10.26. Appl., 31.7.22).—A solution of rubber containing a quantity of fibres is heated; the depolymerised rubber is subsequently precipitated on to the fibres, which are then pressed and vulcanised. D. F. TWISS.

Concentrating [rubber] latex. K.D.P., LTD. (E.P. 244,727,13.11.25. Conv., 20.12.24. Cf. E.P. 243,016; B., 1926, 956).—In the concentration of heated latex (stabilised or vulcanised, if desired) by exposure to a

current of air in a rotating drum, an unsupported cylinder, present inside the rotating drum and rolling continuously in the latex, aids the exposure of the latex to the drying gases, and also ensures uniformity in the concentrated product. D. F. Twiss.

Impregnating leather and other materials with rubber. N. J. S. NUNN (E.P. 260,652, 3.7.25).—Leather, fabrics, or paper can be impregnated with a solution containing, *e.g.*, rubber, benzene, naphtha, carbon tetrachloride, isoprene, and acetone at 63°; the drained and dried material is then subjected to any desired finishing process. Chrome-tanned leathers treated in this way are improved in their waterproof qualities, durability, and pliability. D. F. TWISS.

Method and apparatus for removing large-sized particles and aggregates from certain finelydivided powders and the like. (E.P. 260,741).— See I.

XV.—LEATHER; GLUE.

Standardisation of hide powder [for tannin analyis]. II. H. G. BENNETT (J. Soc. Leather Trades' Chem., 1926, 10, 355-374. Cf. J.S.C.I., 1907, 455).-Recently suggested criteria for judging hide powder, viz., the alkalinity of the ash (B., 1925, 558) and the $p_{\rm H}$ value (B., 1926, 454), do not distinguish between powders B8 and B11, which were so widely different in use, and therefore the tests cannot be useful in revealing similar future differences. The adoption of an official adsorption test is recommended using the different hide powders under examination in conjunction with a standard solution of gallic acid. The author's previous method (Collegium, 1917, 56, Lon. Edn.) gives comparative adsorptions for B3, B4, B8, and B11 as 100, 92, 104, and 96, respectively, thus indicating those powders showing bad adsorption. To meet criticisms, the author has modified the adsorption test. A 0.6% solution of gallic acid is used, and a tannin analysis done on this solution. The adsorption ratio, A, is the ratio of the gallic acid adsorbed by the powder to the unadsorbed portion. An alternative method is to carry out the test with two solutions containing 0.6 and 0.7% respectively, churn with the chromed powder for 30 min., and use the equation $y = ac + \tilde{b}$, where y = amount adsorbed and c the amount unadsorbed, to find the constants a and b, and thus determine the adsorption ratio for the standard solution. The current official hide powder B13 is very variable in quality, and it is suggested that the calculated mean variations of the adsorption ratio, the test for soluble matter, and the percentage of mineral ash should also be used as official criteria for hide powders. The adsorption ratio and the mean variations of this and other determinations should be used by the Society of Leather Trades' Chemists in the adoption of an official hide powder, and also by the hide powder manufacturers in controlling the manufacture. The "relative specific surface" of B8 powder is practically the same now as it was two years ago, so that its absorbency has not changed. The adsorption ratio for B8, a satisfactory powder, is $1 \cdot 30 \pm 0 \cdot 04$. A =1.30 as a minimum, and the mean variation +0.04 as a maximum may be considered the desiderata of a good hide powder. D. WOODROFFE.

CL. XV.-LEATHER; GLUE.

53

Characteristics of hide- and sinew-collagen and their behaviour towards ferments. W. S. SSADIKOV (Collegium, 1926, 512-518).-Alkaline solutions of tryptase or collagenase-free trypsin hydrolyse fibrin into amino-acids, but not collagen. Acid solutions of α -glutinase-free pepsin dissolve collagen, forming non-glutinous peptones, without formation of aminoacids. Papayotin dissolved collagen in neutral solution at 80°, forming decomposition products similar to aminoacids. Collagenase, free from trypsin, peptises collagen in alkaline solution without forming amino-acids. a-Glutinase, in acid solution, converts collagen into a jelly-like glutin, resistant to acids and a-glutinase. β-Glutinase, in alkaline solution, transforms collagen into a jelly, which is resistant to the action of caustic soda and β-glutinase. The above properties are general for collagens of different origins and different preliminary treatments. A sample of sinew-collagen treated with a 0.25% solution of caustic soda, was preserved in alcohol for 20 years, and was found to differ from fresh sinew-collagen. It was not attacked by an alkaline solution of trypsin and collagenase, an acid solution containing 5% of pepsin, or by papayotin. It required an 8% solution of pepsin to decompose it. Animal hide differs from hide powder and sinew in that it is almost completely dissolved by commercial pepsin. Commercial trypsin has no decomposing action, whilst sinew is partially attacked. Papayotin has no action on hide. A table is given showing the comparative behaviour of sinew, hide, and fibrin towards various fractions of the ferments obtained by treating the latter with charcoal or kaolin and determining the action of the absorbent and the clear liquor respectively. Hide is much more resistant to enzymes than sinew. Collagenase has no action on hide either in acid or alkaline solution, and pepsin treated with kaolin has only a slight swelling action. After calf-skin has been finely divided and treated with weak caustic soda solution, like sinew, the product, hide collagen, can be dissolved by all the above-named enzymes, viz., trypsin, pancreatin, pepsin, collagenase. The differences between sinew- and hidecollagen are apparent only, being due to differences in preliminary treatment, e.g., hydration, maceration. Experiments have shown that bates of the "Oropon" type contain tryptase and collagenase, which do not dissolve the hide provided the latter has preserved its original properties. Hide powder, though only slightly changed chemically, is definitely attacked by "Oropon" bates. D. WOODROFFE.

Sulphato-hydroxo-chromi-collagen compound. K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1926, 21, 559-570) .- Samples of delimed calf-skin were pickled with sulphuric acid and sodium sulphate, also one-bath chrome tanned and washed, respectively. Pieces of each were cut from corresponding parts of the samples and analysed, and portions shaken with distilled water. The diffused acid was neutralised with 0.1Nsodium hydroxide solution at frequent intervals. Equilibrium was established after 48 hrs. treatment, and the pickled stock had then yielded up all its acid. From the titration figures of alkali consumed, the rates of hydrolysis of the collagen-sulphuric acid compound in pickled and tanned stock were shown to differ. If the

total acid sulphate in chrome leather existed as protein sulphate, the rate of hydrolysis should have been the same as for pickled pelt. Chrome leather which had been washed for 48 hrs. showed the acidity of the chromium-collagen compound to be 32.9 and 31.4%. The $p_{\rm H}$ values of the solutions obtained by treating 5 g. of the pickled skin and chromed skin respectively with 200 c.c. of water for 24 hrs. were 2.81 and 3.71. As the concentration of the chrome tanning liquor increased, so the acidity of the neutralised tanned leather increased at first, reached a maximum, and then diminished slightly. The increase in acidity follows from $Cr[(H_2O)_6]^{+++} \longrightarrow Cr[X(H_2O)_5]^{++}$ where the formation of a heterogeneously substituted internal sphere is favoured by increase in concentration. The decrease with higher concentrations is probably due to the transference of cationic chromium complexes into the anionic state. Portions of hide powder were tanned for 48 hrs. with a 63% chrome liquor containing sodium sulphate, and the samples washed to remove protein sulphate ; the acidity of the neutralised product increased from 37.9 to 49.1% with increasing amount of sodium sulphate, whereas the acidity of cationic chromium complexes fixed by permutit from the same chrome liquors showed an increase with small addition of sodium sulphate and a decrease with subsequent larger additions. Evidently electro-chemical changes take place in the chromium salt in the presence of large amounts of sodium sulphate. Present methods of controlling chrome tanning liquors, e.g., p_H figure and precipitation figure, have only a limited application, the composition of the internal sphere being the important factor. Its acidity may show variation from 20-50%. D. WoodROFFE.

Action of neutral salts on hide protein. G. D. McLAUGHLIN and E. R. THEIS (Collegium, 1926, 431-436).-Fresh corium and hide powder prepared therefrom were treated with saturated solutions of sodium chloride and sodium sulphate respectively. Fresh corium was dissolved equally well by each solution, but sodium chloride proved a better antiseptic than sodium sulphate. In the absence of bacteria, hide powder prepared from fresh corium is dissolved to a less extent by sodium sulphate than by water or sodium chloride. The latter exercises a greater hydrolytic effect than water. The researches of others (cf. B., 1925, 292; 1926, 23, 503) have shown that the difference between the solvent action of sodium sulphate and chloride, respectively, increases with increasing change in the hide proteins, e.g., in hide powder for analysis and in gelatin. When a tissue or an albuminous substance contains more than one protein, and when one or more of these are characterised by different solubilities in salt solutions, then the nitrogenous compounds dissolved by the latter must differ. The effect of neutral salts depends on the non-coagulable portion or on the hydrolysed proteins in D. WOODROFFE. the hide corium.

Comparison of the tanning action of extracts and crude tanning materials. P. PAVLOVITSCH (Colle-gium, 1926, 441-445).-Comparative analytical tests have been made on pieces of hide which had been coloured in the same weak tan liquors and subsequently tanned in ordinary "layers," in "layers" dusted with sterilised

materials, in clear extract liquors, and in turbid extract liquors respectively. There was no difference between the results obtained from clear or turbid liquors nor between sterilised and natural dusting materials. Extract liquors were not inferior to "layers." There was no evidence to support the view that enzymes exercise a favourable effect on the tannage (cf. B., 1923, 367). D. WOODROFFE.

Comparison of boric and hydrochloric acids in the determination of nitrogen in leather. [Report of Committee of American Leather Chemists' Association.] R. W. FREY and R. M. HANN (J. Amer. Leather Chem. Assoc., 1926, 21, 583—587).—Various samples of chrome- and vegetable-tanned leathers have been used for comparative determinations of the nitrogen content by Kjeldahl method, distilling the ammonia into N/5-hydrochloric acid and 4.5% boric acid respectively. The results agreed very well. In some cases the boric acid yielded slightly higher figures. The adoption of boric acid for collecting the ammonia is recommended. D. WOODROFFE.

Absorption of acid and basic dyes by cationic and anionic chrome-tanned hide powder. К. Н. GUSTAVSON (Collegium, 1926, 437-441).-Cationic chromium appears to combine with the acid groups in collagen, whereas the anionic chromium combines particularly with the basic groups. Samples of hide powder separately tanned with each kind were tested with acid and basic dyestuffs respectively. Acid dyes are taken up by hide protein on the acid side of the isoelectric point only, whilst basic dyes are absorbed on the alkaline side only. Acid dyes showed a much greater affinity for hide powder tanned with cationic chromium than for anionic chrome-tanned powder or for powder tanned with anionic and cationic chromium, whereas basic dyes were absorbed more readily by anionic chrome-tanned powders. Vegetable-tanned hide powder behaves towards acid dyestuffs in the same way as powder tanned with anionic chromium compounds, which confirms Procter's view (" Principles of Leather Manufacture," 1922, 491) that tanning is mainly a function of the condition of the basic groups in the collagen. The dyeing of unmordanted chrome leather with acid dyes depends on the hydrogen-ion concentration and on the condition of the inactive protein groups. The different behaviour of one- and two-bath chrome-tanned leathers towards acid dyes can be attributed to the dual mechanism of chrome tannage ; two-bath leather comprises anionic chromium complexes only. D. WOODROFFE.

Noxious solvents and thinners used in paints and adhesives. BEYTHIEN.—See XIII,

PATENTS.

Method of moulding and hardening algincontaining material. C. C. LOOMIS and A. L. KEN-NEDY, ASSTS. to KELP PRODUCTS CO. (U.S.P. 1,603,783, 19.10.26. Appl., 17.8.21).—The algin in the material is converted into an insoluble alginate, and a relatively small amount of softening agent is added (insufficient to bring the total moisture content above 25%). The softened material is moulded by pressure, and hardened by the removal of sufficient water. S. S. WOOLF.

Preparation of japanned leather goods. G. E. HALDINSTEIN (E.P. 260,803, 23.12.25).

Impregnating leather with rubber (E.P. 260,652). —See XIV.

XVI.—AGRICULTURE.

Stimulation of plant growth by potassium iodide. O. LOEW (Z. Pflanz. Düng., 1926, A7, 233—234).—In cylinder experiments with barley, using increasing dressings of potassium iodide, a maximum yield was obtained with a dressing equivalent to 500 g./hectare. The increase over the control for this dressing was 34%. With millet, 376 g./hectare of potassium iodide produced an increase of 28%. With sodium fluoride a maximum increase with barley was obtained using a dressing equivalent to 5 kg./hectare. Similarly, with millet a 40% increase was obtained with 940 g./hectare.

G. W. ROBINSON.

Mitscherlich's method for determining the manurial requirements of soils. GERLACH (Z. Pflanz. Düng., 1926, B5, 489—505).—In considering the quantitative relationship between plant growth and growth factors, the increase in production of dry matter over the control should be used and not the actual production. It is further suggested that a given factor should be represented in the equation by the amount actually used by the plant. The effect constant for a given factor ("Wirkungswert") probably varies for different plants. In investigating the effect of any one factor, the others should be in favourable amount or intensity. The general applicability of Mitscherlich's equation is questioned. G. W. ROBINSON.

Application of the Neubauer seedling method to tropical soils. H. VAGELER (Z. Pflanz. Düng., 1926, B5, 506—509).—The author gives data for the assimilability of potassium and phosphorus, respectively, in certain Colombian soils, using the Neubauer seedling method. The corresponding amounts soluble in 10% hydrochloric acid are also given. There is a rough correlation between potassium soluble in hydrochloric acid and potassium available to seedlings, the acidsoluble potassium being about twice or three times the amount taken up by seedlings. In the case of phosphorus the correlation is less obvious, and a much lower availability is indicated. The number of experiments is too small to suffice for any trustworthy conclusions.

G. W. ROBINSON.

Influence of manuring etc. on the strength of straw of cereals. P. STUCH (Z. Pflanz. Düng., 1926, A7, 257–290).—Strength of straw in cereals is mainly influenced by the supply of nitrogen to the plant. A suitable nitrogen supply results in a strong development of the tissues. A superabundance of nitrogen causes a thinning of the cell wall and a general slackening in the exterior stratified cells; plants over-stimulated with nitrogen are prone to fungal diseases, which still further weaken the cellular structure. Phosphatic fertilisers invariably increase straw strength by thickening the cell walls and consolidating the external layers. The mechanical tissues develop earlier, and the straw is shown to withstand both increased tension and pressure. In some cases over-manuring with phosphate favours the incidence of fungal diseases, which may so weaken the cells as to outweigh the strengthening effect of the fertiliser. Potassic fertilisers both increase the strength of straw and add to the plant's resistance to fungal attacks. A. G. POLLARD.

Relation between physiological acidity of fertiliser salts and soil acidity. H. KAPPEN and W. BERGEDER (Z. Pflanz. Düng., 1926, A7, 291-317).-Comparison is made of the effects of physiologically acid fertilisers on the "hydrolytic" and "exchange" acidity of soils (B., 1925, 140), their $p_{\rm H}$ values and buffer capacities (Jensen). The physiologically acid fertilisers increase both types of acidity, and decrease $p_{\rm H}$ values and buffer capacities. Ammonium sulphate is the most active in this respect; calcium superphosphate and potash salts do not increase acidity. The physiologically alkaline fertilisers-sodium nitrate, basic slag, and some potash salts-have the opposite effects. Slag is the most effective in this respect. Plant injury due to acidity produced by the use of acid fertilisers is not the result of the activity of free acids, but is due to enhanced "exchange" acidity. Soils exhibiting con-siderable "exchange" acidity may be treated with acid fertilisers without any appreciable increase in $p_{\rm H}$ values. For this reason lime-requirement determinations based on $p_{\rm H}$ measurements must fail. In soils of similar mechanical composition, values obtained for the neutralising power of soil for acids are comparable with the values for "hydrolytic" and "exchange" acidity. Comparison is not possible of soils with different physical composition. This conforms with the theory that the intensity of acidity is dependent on the quantity and state of disintegration of zeolitic silicates and humates in the soil. It is recommended, therefore, that determinations of the neutralising power of soil for acids should be supplemented by measurements of the "ex-change" and "hydrolytic" acidity. A. G. POLLARD.

Nature and significance of the physiological soil reaction. B. DIRKS (Z. Pflanz. Düng., 1926, A7, 318-338).-It is shown that alkaline humates in soil can exert a definite alkaline reaction, and can neutralise free hydrogen ions up to $p_{\rm H}$ 6. Within the range $p_{\rm H}$ 6-7, in humus soils free from zeolitic material, plants may suffer from excessive alkalinity derived from the calcium humates. Calcium carbonate can be decomposed by humic acids within this range. In zeolitic soils no appreciable amount of calcium humates can exist, since the humate base can be withdrawn from solution into the insoluble zeolite complex. Only when there is an insufficiency of zeolitic material present does the plant suffer from alkali-toxicity. Calcium humates are decomposed by carbon dioxide within the range $p_{\rm H}$ 7-8.3. Under still more alkaline conditions begins the formation of "soda" soils. In these toxicity may be reduced owing to the formation of calcium carbonate by decomposition of the humate. Calcium humates are decomposed by boiling the soil extract for a short time, and the base may be directly titrated. Aqueous extracts are preferable to neutral salt extracts for this purpose, and give higher results, more closely paralleled by plant experiments. The optimum lime dressing for acid soils can be determined by an electrometric titration of a N-potassium chloride extract of the soil with lime water. The $p_{\rm H}$ value of the resulting liquid after boiling for 1 min. and filtering should not exceed 6.0 for zeolitefree soils. The end-point of the latter part of the experiment may, alternatively, be determined by titration with 0.01N-hydrochloric acid, using methylred as indicator. A. G. POLLARD.

Absorption of plant nutrients and formation of dry matter by varieties of millet under different manurial conditions. W. SCHLEUSENER (Z. Pflanz. Düng., 1926, A7, 137-165).-The course of absorption of plant nutrients by different varieties of millet during the growing period is similar to that of summer cereals, particularly barley. Production of dry matter lags behind the intake of plant nutrients, which is largely completed by the time of flowering. During the first three fortnightly periods of the growing season the course of absorption is not markedly influenced by the manuring. During the later periods the growth on manured plots was limited by drought. Only in the case of nitrogen was the course of absorption affected by manuring, and the greater the amount of nitrogen given the more did absorption precede formation of dry matter. The plant nutrient requirements of millet appear to be less than those of barley; nevertheless, this crop removes considerable amounts of plant food from the soil. On account of drought it was impossible to decide as to the response of millet to manuring. G. W. ROBINSON.

Translocation of potassium before and during the death of leaves of *Populus nigra* (black poplar) and *Hedera helix* (ivy) in autumn. T. SABALITSCHKA and A. WIESE (Z. Pflanz. Düng., 1926, A7, 166—173).— During the dying of the leaves of *Populus nigra* and *Hedera helix*, a 60—70% reduction of the potassium content occurs. The decrease is most marked shortly before and during coloration, and in the case of *Populus* begins in August. In the case of *Hedera*, leaves which remain green and do not fall showno decrease in potassium content. It is probable that a translocation of potassium to the permanent parts of these plants occurs in autumn. It is suggested that potassium may behave differently in other plants, and that the conclusions for *Populus* and *Hedera* may not be generally applicable.

G. W. ROBINSON.

Preparation and solubility of magnesium phosphates compared with those of calcium and aluminium, and their utilisation by cats and barley. E. UNGERER (Z. Pflanz. Düng., 1926, A7, 352—364).— The extremely variable constitution of laboratory preparations of magnesium phosphate is demonstrated. The phosphates of magnesium and calcium are all more soluble in carbon dioxide solutions than in water. The solubility of aluminium phosphate in water is decreased by the presence of carbon dioxide. Plant experiments indicate that the fertiliser value of these phosphates is of the same order as their increased solubility in the presence of carbon dioxide. A. G. POLLARD.

Manuring of fruit trees. I. II. T. WALLACE (J. Pomology, 1925, 4, 117—140; 5, 1—33).—A detailed series of nutritional experiments on apples, gooseberries, black currants, raspberries, and strawberries, extending over several ycars, is described. The plants were grown in sand cultures supplied with nutrient solutions. Deficiency of any one of the elements, nitrogen, potassium, phosphorus, calcium, magnesium, and sulphur, produced characteristic effects on the various plants, influencing especially the type of foliage produced, the blossom formation, and the time of defoliation. Other data recorded relate to the effect of the treatments on the nature of the root systems, the amount of shoot growth, and the yield of fruit. The ratio of nitrogen to potassium and of potassium to magnesium in the food supply may be important. Some of the effects of deficiency of essential nutrients are so characteristic that they may prove useful for diagnostic purposes in the field. C. T. GIMINGHAM.

Determination of $p_{\rm H}$ by means of the quinhydrone electrode. T. ARND and W. SIEMENS (Z. Pflanz. Düng., 1926, A7, 191–204).—For the determination of the $p_{\rm H}$ of well buffered solutions, including soil extracts, commercial quinhydrone is as satisfactory as quinhydrone freshly prepared by the oxidation of quinol. In the absence of buffering substances, freshly prepared and recrystallised quinhydrone should be used. With soils having $p_{\rm H} > 5.0$, carbon dioxide should first be removed either by a current of air or, where the $p_{\rm H}$ is less than 8.5, a current of hydrogen.

G. W. ROBINSON.

Importance of hydrogen-ion concentration in the study of soils. G. WIEGNER and H. GESSNER (Kolloid-Z., 1926, 40, 209-227).-The importance of hydrogenion concentration and methods of measurement in soils are discussed. Soils appear to be well buffered in general, their $p_{\rm H}$ value lying between 4 and 8. The chief sources of acidity are carbonic acid, humus, clay, and the hydrolysis of salts. Calcium carbonate and clay act as strong buffers towards acids, whilst humus acts as a strong buffer towards bases. Also, clay has a weak buffering effect towards bases, and similarly humus towards acids. Humus which is poor in lime has $p_{\rm H}$ $3 \cdot 5 - 4 \cdot 2$, but the $p_{\rm H}$ is raised by increasing the lime content. The limits of $p_{\rm H}$ favourable to different types of plants are considered, and the effect of manuring is discussed. E. S. HEDGES.

Influence of water containing sulphuric acid on the germinating power of steeped grain. J. DEHNICKE (Z. Spiritusind., 1926, 49, 336—337).—The prevention of mould during malting by the use of sulphuric acid is dependent upon both the concentration of the acid and the time it is in contact with the grain. After alternate steeping and aeration, the grain was soaked in water containing different amounts of sulphuric acid for varying periods, and the percentage of germinating grains compared with that obtained in a control experiment in which no sulphuric acid was used. Soaking for 30—60 min. in acid containing 0.049 g. of sulphuric acid/100 c.c. caused no diminution in the germinating power, but stronger acid caused a distinct falling off. F. R. ENNOS.

Nitrifying power of puzzuolana. II. C. SERONO and L. GUERCI (Annali Chim. Appl., 1926, 16, 452—456; cf. B., 1925, 858).—When air is passed through a sodium hydroxide solution containing suspended puzzuolana, or when moist air is passed slowly over the earth, nitric acid is sometimes formed. Very few samples have this property, which is lost after a few days.

E. W. WIGNALL.

Acrid and poisonous qualities of seeds and cakes from Cruciferæ. G. JORGENSEN (Ann. Falsif.,

1926, 19, 454-459).-Two varieties of Crucifera, Brassica juncea and Brassica niger, yield the harmful allylthiocarbimide and the cake is first examined microscopically for seeds of these. If a qualitative test shows the presence of acrid mustard oil, determinations are made of its amount by formation of the thiosinamine and also of the percentage of nitrogen in the latter. When the nitrogen content of the thiosinamine exceeds 22% the material is harmful to cattle, even though it yields no more than 0.35% of mustard oil; when between 21% and 22% it is doubtful, but if less than 21%, and Brassica juncea is absent, no poisonous qualities are to be expected. When this value approaches 21% and Brassica juncea is present, it is advisable to try the effect of the cake on rats. F. R. ENNOS.

Action of potassium chloride on soils free from calcium. A. DEMOLON and E. NATIER (Compt. rend., 1926, 183, 1121—1122).—The adsorption of potassium from potassium chloride by clay soils free from alkaline earths shows that even with soils showing a slightly acid reaction, sufficient calcium is retained adsorbed on the clay for an interchange of ions to take place (cf. Demolon, A., 1926, 678); and when this is the case the ratio (Ca displaced)/(K fixed) is approximately 0.5. When this ratio has a lower value the soil is deficient in calcium, and in this case an interchange of potassium and hydrogenions occurs, so that after washing, the soil is less acid, the final $p_{\rm H}$ value being higher than the initial value. No change in the $p_{\rm H}$ is found when sufficient calcium is present. J. W. BAKER.

PATENTS.

Insecticidal composition and the like. H. B. GOODWIN, ASST. to THE LATIMER-GOODWIN CHEM. CO. (U.S.P. 1,604,774, 26.10.26. Appl., 6.5.24).—A small amount of casein is added to the insecticidal compound, which is mixed with water when required, a sufficient quantity of lime being included in the spray mixture to form calcium caseinate. H. ROYAI-DAWSON.

Manufacture of a manure. P. RIPPERT (E.P. 260,882, 26.5.26).—Peat, brown coal, or the like is made feebly alkaline in order to induce a conversion of the organic nitro-compounds into ammonia. The mass is then treated with oxygen, particularly nascent oxygen, e.g., a 0.1% solution of potassium permanganate with addition of hydrogen peroxide. When the oxygen development is complete, cultures of aerobic yeasts which form protein in presence of ammonia are added. E. H. SHARPLES.

Manufacture of phosphate fertilisers. E. L. LARISON, ASST. to ANACONDA COPPER MINING CO. (U.S.P. 1,604,359, 26.10.26. Appl., 24.3.26).—Ground phosphate rock, sulphuric acid, and phosphoric acid are mixed in such proportions that the thin mobile slurry thus formed will, on keeping without artificial drying, form a mass which is easily disintegrated into a granular powder suitable for use in fertiliser distributing machinery. E. H. SHARPLES.

XVII.—SUGARS; STARCHES; GUMS.

Chemical and physical de-liming of sugar solutions and syrups. H. A. C. VAN DER JAGT (Chem. Weekblad, 1926, 23, 566—572).—Lime present in sugar juices is distinguished as "combined" when in the form

of salts other than the saccharate; this form is not precipitated by carbonatation, unless sodium or potassium hydroxide be present. The hydroxides may be added as such, or produced by the action of lime or alkali salts already present ; sodium carbonate is to be preferred. The addition of lime and subsequent carbonatation causes no diminution of the combined lime content of artificially prepared sugar solutions, but produces a very marked diminution with cane juice ; it is suggested that during flocculation of the colloidal particles, the non-sugars and especially calcium compounds are selectively adsorbed and carried down. The same explanation is suggested for the more complete purification of alkaline syrups by treatment with lime and carbon dioxide; if such syrups are first neutralised with acetic acid, de-liming is less complete. S. I. LEVY.

Influence of peptic digestion in the determination of total carbohydrates in cereal products. HARTMAN and HILLIG.—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Culture of the bacillus of retting of flax. V. OMÉLIANSKY and M. KONONOV (Compt. rend., 1926, 183, 919—921).—Growth of the bacillus of retting of flax, *Granulobacter pectinovorum*, in presence of air, is favoured by addition of aerobic bacteria. A sterile suspension of flax fibres in water is inoculated with well retted material and heated for 15 min. at 115° (20 min. at 120° destroys the spores). It is then incubated at 32° in presence of *B. fluorescens liquefaciens*. Retting commences in 2—3 days, and is completed in a further 5—9 days. The same results are obtained with other aerobic bacteria, and also with pure cultures of *Granulobacter* under anaerobic conditions. H. E. F. NOTTON.

PATENT.

Manufacture of a manure (E.P. 260,882).-See XVI.

XIX.—FOODS.

Detection of sulphites in foodstuffs. A. E. PARKES (Analyst, 1926, 51, 620-622).-10 g. of solid material mixed with about 10 c.c. of water in a 50-100 c.c. conical flask, or 10 c.c. of liquid are taken, 10 c.c. of dilute hydrochloric acid and 2 or 3 small pieces of marble are added, and the flask is closed by a rubber stopper carrying a thistle funnel of 3-4 mm. internal diam. bent twice, and having on each limb a bulb capable of holding about 2 c.c.; 2 or 3 drops of 0.01Niodine solution and 1 drop of barium chloride solution are placed in the funnel as a seal through which all liberated gases must pass. When the action of acid on the marble has moderated the liquid is heated to boiling, and immediately the first drop of condensed liquid passes over into the funnel the colour of the iodine is discharged if sulphur dioxide is present, and a white opalescence due to barium sulphate is formed. Some fruit products contain certain volatile substances which discharge the colour of iodine, but do not give any reaction with barium chloride. It may be necessary to add an antifrothing agent such as amyl alcohol. If the boiling be continued for 1-2 min. most of the sulphur dioxide will have been driven over (more 0.1N-iodine solution being added if necessary), and if the reagent and precipitate are washed out they may be matched against a known

amount of sulphuric acid and barium chloride in a nephelometer and the process made partly quantitative. D. G. HEWER.

Variations in the composition of milk. J. F. TOCHER (Analyst, 1926, 51, 606-613).-The percentage of total solids-not-fat of the bulked milk of one herd of cows from daily samples is much less variable (3 times less) than the percentage of total solids-not-fat in the bulked milk of different herds, so that a high percentage of butter fat is associated, in general, with a lower percentage than average of solids-not-fat. There is a high correlation between yield per milking and solidsnot-fat per milking, and that between total butter fat per milking and yield per milking is also fairly high. Yield can be used as a measure of the total amount of solid matter in milk with a fair degree of accuracy. Although there is a correlation between solids-not-fat and butter fat percentages in the bulked milk of different herds, due to the high positive correlation between percentage of casein and of butter fat, the relationship is negative for the daily samples of bulked milk from one herd, owing to the small variation in the total solids from day to day. The percentage of casein in milk, lowest after calving, gradually rises to a maximum at the end of the lactation period. The correlation between butter fat and lactose is negative, accounting largely for the negative correlation between non-fatty solids (other than casein) and butter fat. Friesian cows were found to give a higher quantity of solids-not-fat in general, and of lactose in particular, per milking than Ayrshire or other cows, although Ayrshires showed higher percentages of butter fat and of solids-not-fat D. G. HEWER. per milking.

Resistance of fat-soluble vitamins to hydrogenation. L. RANDOIN and R. LECOQ (Ann. Falsif., 1926, 19, 518-523) .- Commercial cod liver oil (iodine value 134.39) was hydrogenated with reduced nickel catalyst at 180-190° for 6 hrs. to an iodine value of $28 \cdot 69$, and also at $120 - 130^{\circ}$ for 8 hrs. to an iodine value of 64.93, and fed to rats which had been on a Sherman and Pappenheimer No. 84 diet and exhibited typical rachitic signs. A slow rate of growth was maintained, but in neither case were ophthalmic lesions inhibited, and the oil hydrogenated at the lower temperature was, if anything, more prejudicial to the symptoms than the harder one. Butter, particularly summer butter, was found to be less wanting in anti-rachitic properties than has been supposed, and margarines made with hydrogenated oils are inferior as sources of fat-soluble vitamins to those prepared with natural oils and fats.

D. G. HEWER.

Cellulose sausage casings. W. F. HENDERSON and H. E. DIETRICH (Ind. Eng. Chem., 1926, 18, 1190-1194). -Details are given for the conversion, on a manufacturing scale, of high-grade purified cotton linters, by means of the viscose process, into thin-walled seamless cellulose tubes which are mechanically and chemically suitable for use as sausage casings. The new material has distinct advantages over the animal casings hitherto used. W. J. POWELL.

PATENTS.

Liquid-treating apparatus [for milk]. H. H. MILLER, Assr. to H. H. MILLER INDUSTRIES CO. (U.S.P. 1,603,970, 19.10.26. Appl., 26.10.22).—A vat for storing large quantities of milk without deterioration is fitted with a lining of German silver, surrounded by a heat insulating material, such as wood, and contains a series of pipes through which water at a constant temperature is circulated. Propellers are also provided for causing a gentle agitation of the milk in the vat. T. S. WHEELER.

Preparing flaked cereal food products. H. J. HEINZ Co., Assees. of E. R. HARDING (E.P. 245,474, 4.1.26. Conv., 3.1.25).—The grain is cooked with finelydivided pure cellulose and the mass dried incompletely, and then the original kernels are separated, dried, and flaked. E. H. SHARPLES.

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

Ethyl phthalate test. H. WALES (J. Assoc. Off. Agric. Chem., 1926, 9, 476—477).—In testing for ethyl phthalate in drug products it is necessary first to remove derivatives of naphthalene, anthracene, and phenanthrene, as these may be oxidised to phthalic acid, and will consequently show a positive test with resorcinol. The precipitant used for the purpose is basic lead acetate, excess of which is removed by sodium carbonate. After extraction of the ester with light petroleum, hydrolysis, and conversion of the phthalic acid into anhydride, the latter is condensed with resorcinol. The fluorescence obtained if ethyl phthalate is present persists indefinitely without fading. If a positive test is obtained, it is advisable to confirm by distilling some of the original product and applying the test to the distillate.

F. R. ENNOS.

Analysis of Quinosol (2-hydroxyquinoline) preparations. R. BERG (Pharm. Ztg., 1926, 71, 1542— 1543).—The material is dissolved in water and titrated with a bromide-bromate solution, using indigo-carmine as indicator. The active quinoline compound is quantitatively brominated to 5:7-dibromohydroxyquinoline, any excess of bromine reacting with the colour indicator. As a check, a slight excess of the bromine solution may be added, then potassium iodide, and the liberated iodine determined by means of thiosulphate. S. I. LEVY.

Determination of morphine in opium. E. C. M. J. HOLLMAN (Pharm. Weekblad, 1926, 63, 1337-1349, 1370-1380, 1393-1408).-The standard methods have been subjected to critical examination, and none is found satisfactory. The variation in opiums from different sources makes it impossible to recommend any one simple or composite method. The acidity of the opium varies, whilst the composition may be affected by ferments and oxidation; the solvent to be used for the extraction must be selected with due regard to the acidity; lime water is usually best, but the solution filters badly, and the crystals obtained are impure. Acid solutions extract the related alkaloids, and alcohol acts similarly. The $p_{\rm H}$ values and morphine contents of a large number of opiums from various sources show variations from 4.00 to 5.05 and from 5.99% to 15.00% respectively, the variations being entirely arbitrary; determinations by the methods of the Dutch and German pharmacopœias in the same samples

gave differences in the morphine content up to 1.58%. The most favourable temperature for separation of the crystals is about 16°; lower temperatures considerably retard crystallisation. Related alkaloids and the usual impurities, besides adulterants and diluents, seriously affect both extraction and crystallisation; the influence of albumins on the separation is very unfavourable. Results are given of tests of the influence of time, agitation, etc. on the rate of separation. Trials on the most favourable $p_{\rm H}$ values for separation gave the best value as 8.84 for pure aqueous solutions. An apparatus for the determination of $p_{\rm H}$ values by means of the calomel electrode is described, also the results of a large number of examinations of opium from various sources tabulated with the morphine contents as determined by the Dutch and German methods; the results are discussed and some conclusions drawn, the most important being that the $p_{\rm H}$ determinations afford no help in deciding the best conditions for extraction and The use of alcohol for extraction and separation. crystallisation is discussed; it is found to promote contamination with lime, except where the procedure of the Japanese pharmacopœia is followed. The determination of the morphine finally obtained, whether this is weighed or titrated, may introduce errors ; these are considered for the two methods. The use of adsorbent charcoal introduces considerable errors, which are less in acid than in alkaline solutions, but never negligible. The sources of error in the lime water and ammonia methods respectively are discussed in detail; the colorimetric, refractometric, and polarimetric methods are also considered. S. I. LEVY.

New colour reaction for procaine [novocaine] and some other local anæsthetics, and its application to the determination of procaine. E. R. RIEGEL and J. F. WILLIAMS (J. Amer. Chem. Soc., 1926, 48, 2871-2878).-A weighed sample, representing 10-15 mg. of procaine, in water (10 c.c.) is treated with 10% hydrochloric acid (0.5 c.c.), 2% sodium nitrite solution (1 c.c.), and concentrated ammonium hydroxide solution (1 c.c.), and, after 30 sec., the mixture is diluted to 100 c.c. 1, 1.25, 1.5, 2, and 2.5 c.c. of this solution, diluted to 50 c.c. in Nessler tubes, are compared with a standard tube, prepared by diluting a 0.1% solution of potassium dichromate (8.5 c.c.) to 50 c.c., the colour standard so obtained representing a total content of 0.25 mg. of procaine. No colour is obtained in the solution under test if the amount of procaine present in 10 c.c. is less than 0.05 mg. A concentration of 10-15 mg. in 10 c.c. gives the maximum colour ; with concentrations below 10 mg./10 c.c., there is an absence of turbidity during the development of colour, indicating that the concentration is too low. The accuracy of the method is within 10%, provided that the temperature is maintained at 20° throughout. The reaction can also be used as a qualitative test for local anæsthetics, tutocaine, butyn, butesin, propaesin, benzocaine, and orthoform ; saligenin gives a yellow colour with hydrochloric acid and sodium nitrite without the addition of aqueous ammonia. The method can also be applied to the determination of tutocaine. For the determination of mixtures of procaine and cocaine, synthetic mixtures must first be employed to establish a standard, since cocaine, although it gives no colour in the test, removes some of the yellow substance from the solution when precipitated by the ammonia. Alternatively, the reaction is carried out in alcoholic solution, in which the cocaine does not interfere, but it is then necessary to establish colour values for procaine in alcohol.

F. G. WILLSON.

Critical inspection of the directions for the examination of essential oils and perfumes. W. TREFF (Z. angew. Chem., 1926, 39, 1306-1309).---Xylol-musk exists in two modifications, a stable form, m.p. 112-113°, and a labile form, m.p. 105-106°. Formation of the stable form is influenced by inoculation of the melted musk or a concentrated alcoholic solution with a small particle of the stable form; also by crystallisation from ether, benzene, toluene, or light petroleum. The labile form exists only within narrow limits. Rearrangement is possible in the melted state, but the melt usually solidifies to crystals of the higher-melting modification. These facts explain the discrepancies in the recorded melting points of xylol-musk. The specified density of benzaldehyde (d^{15} 1.050—1.055), is considered to be too high. Chlorine-free benzaldehyde only oversteps the lower limit by reason of its benzoic acid content; a more suitable value is 1.049. The catalytic effect of small amounts of impurities and the sensitivity of many oils at high temperatures are shown in the distillation of pure linalool with the addition of small pieces of porous clay. Over 30% was converted into terpenes and resins. Small pieces of glass capillaries are much more suitable, and should always be used in the distillation of essential Specified b.p. should define the velocity of disoils. tillation. In a series of distillations with a pure sample of cassia oil, different rates of distillation gave residues varying from 6-30%. A steam-distilled oil under similar conditions gave varying amounts of residue, and for this to be a minimum it is necessary to distil as rapidly as possible. In the saponification of acetylated alcohols an excess of alkali is essential for complete saponification and the prevention of the formation of ethyl acetate.

E. H. SHARPLES.

PATENTS.

Apparatus for producing acetic acid from acetaldehyde. E. G. THORIN, ASST. to STOCKHOLMS SUPERFOS-FAT FAB. AKT. (U.S.P. 1,601,891, 5.10.26. Appl., 27.4.23. Conv., 19.9.21).—Acetaldehyde is oxidised in an upright tube filled with small pieces of solid material, the upper part of the tube being provided with a cooling jacket to condense the acetic acid and acetaldehyde, and the lower part with a heating jacket to drive off the acetaldehyde from the acetic acid. An acetic acid solution of the catalyst is admitted to the top of the tube, and between the two jackets there is an inlet for liquid acetaldehyde. At the bottom of the tube is an inlet for the gas containing oxygen and an outlet for the acetic acid formed. Waste gases are withdrawn from the top of the tube.

E. H. SHARPLES.

Manufacture of alkyl carbonates. J. A. S. HAMMOND, Assr. to U.S. INDUSTRIAL ALCOHOL CO. (U.S.P. 1,603,689, 19.10.26. Appl., 23.3.25).—Phosgene and an aliphatic alcohol are heated under reflux. The carbonate is recovered from the mixture by addition of an inert aliphatic solvent, which in the presence of water (which is added to form separate liquid layers) acts as a non-solvent for the alcohol. B. FULLMAN.

Manufacture of alkyl carbonates. H. G. MITCHELL, Assr. to U.S. INDUSTRIAL ALCOHOL CO. (U.S.P. 1,603,703, 19.10.26. Appl., 26.3.25).—Phosgene vapour is brought into contact with an aliphatic alcohol, the latter being in 100% excess. The mixture is allowed to remain until the product is formed. B. FULLMAN.

Making acetylsalicylic acid. D. A. NIGHTINGALE, Assr. to KETOID Co. (U.S.P. 1,604,472, 26.10.26. Appl., 22.7.24. Renewed 18.3.26).—Salicylic acid is treated with keten. B. FULLMAN.

Production of acylresorcinols. SHARP AND DOHME, Assees. of A. R. L. DOHME (E.P. 250,893, 6.7.25. Conv., 4.16.25).—Acyl resorcinols of the formula $C_6H_3(OH)_2 \cdot COR$ (R = alkyl), free from tarry byproducts, are prepared by condensing fatty acids with about one-third of their weight of resorcinol, in the presence of zinc chloride (from less than half to less than one-fifth by weight of the fatty acid), at 125—145° (preferably 130—140°). The resorcinol is gradually added to the heated mixture of fatty acid and zinc chloride, heating continued for about 3 hours, and the product washed and distilled *in vacuo*. (Cf. A., 1926, 838.) B. FULLMAN.

Manufacture of solid polymers of formaldehyde. J. Y. JOHNSON, From I.-G. FARBENIND, A.-G. (E.P. 260,908, 26.6.25).-Water-soluble solid polymerides of formaldehyde, of any consistency from a grease to a dust, are obtained from gaseous mixtures of formaldehyde and water vapour (possibly also containing other gases and vapours) by removing the water vapour, preferably by rapid cooling, but also by other means. When the cooling method is used, the bulk of the water vapour and some formaldehyde condense and form a formaldehyde solution, which is removed, while the uncondensed formaldehyde passes on and is separated as a solid polymer. Examples are given of the catalytic oxidation of anhydrous and of 80% methyl alcohol, the cooling being effected in the two cases respectively by blowing the hot vapour obliquely on to the surface of strongly cooled 30% formaldehyde solution, and by passing the vapour from below into an inclined cooler.

B. FULLMAN.

Making cellulose esters of carboxylic acids. D. A. NIGHTINGALE, ASST. to KETOID CO. (U.S.P. 1,604,471, 26.10.26. Appl., 22.7.24).—Cellulose is treated with keten in the presence of a neutral liquid in which keten is soluble. B. FULLMAN.

Manufacture of alkaline-earth salts of the carboxylic acids of aromatic sulphonhaloalkali amides. "ESSEFF" CHEM. IND. & HANDELS A.-G. (E.P. 248,339, 30.1.26. Conv., 25.2.25).—The calcium salt of 4-calciumchlorosulphonamidobenzoic acid, which is obtained by the action of calcium hypochlorite on 4-sulphonamidobenzoic acid, is treated in aqueous solution with a sodium salt, whereby the calcium salt of 4-sodiumchlorosulphonamidobenzoic acid is formed. A. DAVIDSON.

Manufacture of medicaments containing bismuth. CHEM.-PHARM. A.-G. BAD-HOMBURG, Assees. of A. LIEBRECHT (E.P. 237,912, 29.7.25. Conv., 29.7.24).-Products having high therapeutic qualities for the treatment of syphilis in both early and late stages are prepared by the action of one or more bismuth compounds on phosphatides such as lecithin. Reaction may be effected in the presence of organic solvents, and liquids suitable for injection are obtained by the addition of olive oil either before or after removal of the solvent. For example, 10 pts. of bismuth quinine iodide and 10 pts. of lecithin dissolved in ether are thoroughly mixed. On removal of the solvent an orange powder remains which is soluble in chloroform, carbon disulphide, and benzene. E. H. SHARPLES.

Methods of preparing a remedy for tuberculosis in men and animals. K. F. O. GRONSTEDT (E.P. 248,319, 1.9.25. Conv., 28.2.25).—Goats, dogs, or other animals are rendered immune to tuberculosis by injections of dead or living tubercle bacilli, or metal salts or the like, and killed by blood letting. The bone marrow is removed and, after drying at moderate heat, is prepared into tablets or powder permitting its use as a remedy. Extracts may be obtained by dissolving the finely ground marrow in water rendered faintly alkaline. E. H. SHARPLES.

Manufacture of anæsthetic and antiseptic compounds. E. RITSERT (E.P. 260,493, 15.4.26).—By interaction of esters of aminobenzoic acids, or their salts, and iodine- or bromine-substituted hydroxyaromatic or hydroxyheterocyclic sulphonic acids, or their salts, in the presence of solvents or blood serum, there are formed the corresponding difficultly-soluble sulphonates of the aminobenzoic esters, having anæsthetic and antiseptic properties. The following are described : the *di-iodophenol-p-sulphonate* of ethyl *p*-aminobenzoate, m.p. 225° (decomp.), and the corresponding *dibromo-p-sulphonate*, m.p. 193—200°; the 7-*iodo-8-hydroxyquinolinesulphonate* of propyl *p*-aminobenzoate, m.p. 225—228°; and the corresponding *di-iodophenol-p-sulphonate*, m.p. 224° (decomp.); and the *di-iodophenol-p-sulphonate* of *iso*butyl *p*-aminobenzoate, m.p. 222—224° (decomp.).

B. FULLMAN.

Product for dissolving essential oils. E. G. THOMSSEN, ASST. to J. R. WATKINS Co. (U.S.P. 1,602,183, 5.10.26. Appl., 24.7.25).—The product consists of a flavouring extract of a flavouring material dissolved in the ester of a hydroxy-acid, or of a flavouring extract consisting of a flavouring material dissolved in ethyl lactate and containing no alcohol. B. FULLMAN.

Promoting catalytic reactions. C. R. DOWNS, Assr. to THE BARRETT Co. (U.S.P. 1,604,739, 26.10.26. Appl., 5.12.21).—An apparatus for carrying out catalytic reactions comprises a number of tubes containing the catalyst, surrounded by a liquid boiling under a pressure corresponding to the temperature it is desired to maintain. The refluxing portion of the liquid is employed to preheat the material passing to the catalyst. All overheating is avoided, even in carrying out strongly exothermic reactions, such as the oxidation of naphthalene by air to form phthalic anhydride. T. S. WHEELER. Solvent recovery [from adsorbent carbon]. A. B. RAY, ASST. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,604,481, 26.10.26. Appl., 14.11.24).—The aqueous portion of the condensate, obtained by treating adsorbent carbon containing recovered solvent (e.g., amyl acetate) with steam, is employed to cool the reactivated carbon, when the solvent dissolved in the aqueous liquid is adsorbed. The carbon is then employed in the usual way to recover fresh solvent.

T. S. WHEELER.

Separating volatile chemicals [aniline from water]. W. E. LUMMUS (U.S.P. 1,603,851, 19.10.26. Appl., 27.8.20).—A mixture of aniline vapour and steam is passed into a fractionating column, from the bottom of which water free from aniline is withdrawn, and from the top a mixture of aniline (1 pt.) and steam (3 pts.). These vapours are partially condensed, and the solution of aniline in water, thus formed, is returned to the column. The remaining vapours are completely condensed, and run into a gravity separator, in which aniline and a solution of water in aniline are obtained. The solution is returned to the column. T. S. WHEELER.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Effects of dilution and stirring of a photographic developer. A. H. NIETZ and A. WHITAKER (Brit. J. Phot., 1926, 73, 630-632, 645-647, 660-661, 676-677).-Time of development with a given exposure is not proportional to the dilution of the developer. The curve of density against logarithm of dilution is in general concave to the dilution axis. The following factors to which this may be due are considered in detail: (1) Penetration effects due to surface development (tanning) and to oxidation products in the emulsion layer; (2) penetration effects due to swelling; (3) effect of excess reducing agent; (4) aerial oxidation of the more dilute solutions; (5) possible variation of reducing energy with concentration; (6) hydrolysis; (7) effect of more vigorous stirring. All these points are tested experimentally, and it is considered that the effects of dilution are caused, in order of importance, by (1) the effect of hydrolysis in lowering the concentration of the active anion of the phenolate. No satisfactory explanation is suggested for the action of amines; (2) imperfect diffusion outwards of the bromide and other reaction products formed in the emulsion layer as development proceeds; proper stirring eliminates this effect and gives a differential increase of density, favouring dilute solutions; (3) aerial oxidation accounts for part of the falling off in density with the more dilute solutions; this only occurs with developers which oxidise rapidly.; (4) penetration effects caused by surface development and tanning not explained by (2). These conditions would affect only the concentrated solutions. W. CLARK.

Relation between time and intensity in photographic exposure. F. C. Toy (Brit. J. Phot., 1926, 73, 704).—Emulsions were exposed to light at differing intensity levels, but for the same *It* value. With a certain low intensity level it was not possible to make more than about 80% of the grains developable even with considerable increase in exposure. At higher intensity levels, however, it was very easy to get 100% changed. Using a slow, pure bromide emulsion, exposed to wave-length 3650 Å., the average number of development centres per grain increased rapidly with increasing exposure to a high intensity level, tending to infinity at a certain exposure. With corresponding exposure at a lower intensity, the average number of centres per grain tended to a low maximum with increasing exposure. The results fit in with recent observations of Jones and Hall (B., 1926, 1030). W. CLARK.

Panchromatisation of photographic plates for use in the ordinary spectrum. E. VITERBI (Gazzetta, 1926, 56, 612-620) .- Of a number of different compounds and mixtures of compounds tried with respect to their ability to render ordinary photographic plates panchromatic, solutions having as basis a mixture of pinachrome and pinachrome-violet gave the best results. The effect is improved by addition of ethylcyanine; and a bath containing 220 c.c. of water and 2 c.c. each of 1:1000 solutions of pinachrome, pinachrome-violet, and ethylcyanine is recommended. The plate is immersed in this bath for 3-4 min., washed for 1-2 min. in flowing water, and dried in an ordinary draining rack. If the 220 c.c. of water are replaced by 150 c.c. of water and 70 c.c. of 95% alcohol the washing may be omitted. T. H. POPE.

PATENTS.

Photographic medium. M. C. BEEBE and H. V. HERLINGER, Assrs. to WADSWORTH WATCH CASE Co. (U.S.P. 1,604,674, 26.10.26. Appl., 22.4.25).—A stable light-sensitive varnish is produced by introducing a free halogen into a varnish consisting of hydrophobic colloids, metal salts capable of combining with free halogen, and a solvent, the salts being present to the point approaching saturation. The proportion of free halogen is such as to combine with a substantial portion of the salts and to avoid the presence of free halogen after the metallic halides have formed. W. CLARK.

Reducing the intensity of photographic silver images. K. E. TAESCHNER (E.P. 260,892, 7.6.26).— Silver images are reduced by immersion in a preliminary bath containing an alkali chromate or dichromate and potassium ferricyanide, followed by a second bath in acid sodium thiosulphate. The first bath may contain sugar. W. CLARK.

Photographic [photomechanical] processes. H. WADE. From THE WADSWORTH WATCH CASE Co. (E.P. 260,460, 4.12.26).—A photographic medium consists of a phenol condensation resin with a halogen source as sensitiser. Suitable sensitisers are halogen-liberating compounds such as halogen-substituted hydrocarbons, e.g., iodoform, bromoform, magenta (hydrochloride), or rhodamine alone or in combination with iodine, metal alkyls with a slight excess of iodine, or metallic halides. Hydrogen iodide or any compound liberating a halogen under the action of light is available. As solvent for the resin it is preferable to use one which will give a mobile varnish-like medium. Development is by immersion in a solvent such as alcohol and water. W. CLARK. Utilisation of biochemical light-sensitising extracts in the art of photographic materials. S. E. SHEPPARD, Assr. to EASTMAN KODAK CO. (U.S.P. 1,602,590, 12.10.26. Appl. 21.1.26).—See E.P. 235,210; B., 1925, 694.

Photographic light-sensitive material containing [A] tellurium, [B] selenium. S. E. Sheppard, Assr. to EASTMAN KODAK Co. (U.S.P. 1,602,591-2, 12.10.26. Appl., 6.6.24).—See E.P. 235,211; B., 1925, 694.

XXII.—EXPLOSIVES; MATCHES.

Properties of initiating explosives. H. KAST (Z. ges. Schiess- u. Sprengstoffw., 1926, 21, 188-192).-Tests carried out in 1909 showed that the effect of mercury fulminate increased when it was compressed. The initiating effect was measured by modifying Esop's test by mixing crystalline T.N.T. with increasing amounts of paraffin and noting the amount of paraffin necessary to prevent any considerable compression (1 mm.) of a lead cylinder by the initiation of the phlegmatised T.N.T. by the initiator under test. It was found that detonators containing a layer of fulminate compressed at 1500 kg./ sq. cm. had a greater initiating effect than those with fulminate compressed at 600 kg./sq.cm. Similar results were obtained with lead azide. No experimental support was found for statements that the greater effect of aluminium detonators was due to a thermo-chemical action of the metal. Increased power obtained by adding potassium chlorate to mercury fulminate is not due to increased brisance, as the brisance of the mixture remains practically the same as that of the fulminate alone. The favourable effect must be attributed to an increase in the ease of ignition and quicker establishment of the detonation wave. Storage of technical mercury fulminate in atmospheres of 50%, 80%, and 100% relative humidity showed that it is not hygroscopic, but in admixture with commonly occurring impurities such as mercurous chloride or in presence of potassium chlorate or gum arabic it shows considerable hygroscopicity. The hygroscopicity of fulminate in presence of potassium chlorate, which is itself also non-hygroscopic, is due to reduction of chlorate by impurities present in technical fulminate. The sublimate formed when cyanuric triazide is heated is found to be unchanged cyanaric triazide. Several errata in a paper by H. Kast and A. Haid are corrected S. BINNING. (see B., 1925, 153).

XXIII.—SANITATION; WATER PURIFICATION.

Testing of disinfectants by the Rideal-Walker method. Q. MOORE, JUN. (J.S.C.I., 1926, 45, 472– 474 T).—The standardisation of the broth was found to be an extremely important factor in the test. Variation in the method of neutralising the broth to phenolphthalein, by titration in the cold or at the b.p., altered the final reaction of the broth and resulted in differences in the phenol coefficient values of disinfectants. An increase of, approximately, one-third in the phenol coefficient values of disinfectants was obtained when tests were made using $p_{\rm H}$ 7.6 broth as compared with $p_{\rm H}$ 6.9 broth. The $p_{\rm H}$ 7.6 broth gave results for disinfectants comparable with the presently accepted Rideal-Walker values.

Coal-tar disinfectants. A. C. TAIT (J.C.S.I., 1926, 45, 415—417 T).—A brief review. The method of Chick and Martin for the determination of the bactericidal coefficient is recommended.

Potassium permanganate absorbtion, "chlorine number " and chlorination of water. W. OLSZEWSKI (Z. angew. Chem., 1926, 39, 1309—1310).—Determinations of the "chlorine numbers" and permanganate absorbtions of a series of artificially contaminated waters confirm the results of Froboese and Keiser (cf. B., 1926, 222). For the examination of drinking waters the importance is emphasised of the ratio of the permanganate absorbtion to the chlorine number in conjunction with the bacteriological examination for the detection of contamination by protein degradation products. For the control examination of swimming-bath waters the permanganate absorption and chlorine number should both be determined. E. H. SHARPLES.

Automatic hydrogen-ion control of boiler feed water. H. C. PARKER and W. N. GREER (J. Amer. Water Works Assoc., 1926, 16, 602-616).-A description is given of the apparatus used for electrometrically determining and automatically recording the $p_{\rm H}$ of feed water after treatment with caustic alkali, and for automatically controlling the supply of chemical required to bring the feed water to the desired $p_{\rm H}$. With automatic hydrogen-ion control the average alkalinity of boilers may be lowered with safety, and more uniform control is obtained of the alkalinity of individual boilers and of the average alkalinity of boilers as a whole. It is cheap, and requires little attention; is the only method which automatically compensates for condenser leakage, adds chemicals automatically in proportion to the flow, and in such proportion as to compensate approximately for the acidity or salt content of the feed water, and provides a method for the prevention of corrosion in the preheating sections and the feed lines.

W. T. LOCKETT.

Determination of colon bacterium. C. J. LAUTER (J. Amer. Water Works Assoc., 1926, 16, 625-630).— A review of research work carried out at Washington, D.C. Filtration Plant laboratory on brilliant-green-bile medium. Results indicate that the presence of *B. coli* in raw and filtered water can be detected by the bile medium with a high degree of accuracy, and in a shorter time than that required for the performance of the lactose presumptive and confirmatory tests. As a confirmatory medium, in place of eosin-methylene-blue plates, the brilliant-green-bile medium has given good results. (Cf. B., 1926, 302.) W. T. LOCKETT.

Diagnostic value of neutral-red-lactose-peptone media for the coli-aerogenes group. F. R. GEORGIA and R. MORALES (J. Amer. Water Works Assoc., 1926, 16, 631-641).—The neutral-red-lactose-peptone medium proposed by Stokes and developed by Chamot and Sherwood as a presumptive medium for the coli-aerogenes

group would appear to be better than any medium so far proposed, and results indicate that when a typical contrast reaction is obtained further confirmation is unnecessary. No differentiation of coli and aerogenes sections of the group can, however, be effected by the medium. In tests on samples from a variety of sources, 96.8% of tubes showing a typical reaction were confirmed as containing members of the coli-aerogenes group. Of all the tubes confirmed, $76 \cdot 5\%$ gave typical reactions, $18 \cdot 1\%$ were completely reduced, and $5 \cdot 4\%$ showed no reduction. From tubes completely reduced, in addition to a member of the coli-aerogenes group, other organisms capable of completely reducing neutral-red were isolated, and these when present prevent the typical contrast reaction being obtained. Absence of any reduction may be due to attenuated forms or to the presence of certain of the more unusual members of the coli-aerogenes group that do not have the ability to reduce neutral-red, or which do so very slowly. The medium has no inhibitory action. W. T. LOCKETT.

Alkalimetric determination of the hardness of industrial waters. C. BELCOT (Bull. Soc. chim., 1926, [iv], 39, 1648-1652).-The values obtained for the permanent hardness of a water not containing alkali carbonates or hydrogen carbonates by the method of Treadwell or of Pfeifer (Z. angew. Chem., 1902, 15, 9, 198) often disagree with those obtained by difference. The discrepancy is due to the partial solubility of magnesium carbonate, which influences the determination of the temporary hardness. Hence, in a water containing alkaline-earth and alkali hydrogen carbonates, magnesium sulphate and chloride, and sodium carbonate, the total carbonate and hydrogen carbonate is determined by titration. The total hardness is then determined, and the permanent hardness determined after boiling the water and neutralising the alkali hydrogen carbonates with hydrochloric acid. S. K. TWEEDY.

PATENTS.

Dewatering, compressing, and drying industrial wastes and sewage solids. F. W. MANNING (U.S.P. 1,604,652, 26.10.26. Appl., 17.6.25).—The filtered solids are built up upon a spiral filter wall, and are moved along the wall. W. G. CAREY.

Water softener. H. A. KERN (U.S.P. 1,604,124, 26.10.26. Appl., 24.4.24).—A permanent liquid product containing sodium aluminate and caustic soda. H. ROYAL-DAWSON.

Process for clarifying water. H. A. KERN (U.S.P. 1,604,125, 26.10.26. Appl., 26.5.24).—Calcium hydroxide mixed with a solution of sodium aluminate and caustic soda is added to water. H. ROYAL-DAWSON.

Solidified water-softening compound. H. A. KERN (U.S.P. 1,604,126, 26.10.26. Appl., 24.9.24).— A sufficient amount of soda ash is added to a solution of sodium aluminate to take up the solvent as water of crystallisation. H. ROYAL-DAWSON.

Removing gases from boiler feed water by a vacuum (E.P. 254,707).—See I.

62