BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 27, 1927.

I.-GENERAL; PLANT; MACHINERY.

Heat insulation. J. S. F. GARD (J.S.C.I., 1927, 46, 101-105 T).-The qualities required of a heat insulation are low heat conductivity, low specific heat, low sp. gr. to avoid strain on pipes etc., mechanical strength, noninflammability, absence of corrosive action on metals, ability to withstand alternate wetting and drying and heating and cooling without cracking. The materials giving best all-round results are : (1) For refrigeration etc., sheet and pipe sectional cork moulded from virgin cork without artificial binders. The product is waterproof, resists the action of organic solvents, can be worked with joiners' tools, is resistant to fire, and is rot- and verminproof. (2) For steam pipes, boilers, etc. at temperatures up to 700° F., 85% magnesia covering (15% of asbestos fibre and 85% of basic magnesium carbonate). (3) For superheated steam plant etc. at 700-1000° F., a modified form of the above, consisting of two layersone of a special form of magnesia lagging, resistant to higher temperatures, of $\frac{1}{2}$ in. thickness, which breaks down the temperature to 700° F.; the second a layer of 85% magnesia to the required thickness. (4) For furnace work, a honeycombed brick, consisting chiefly of kieselguhr. All these materials give a relative efficiency of over 90% against an uncovered metal surface. Methods of testing are described and examples of savings effected are worked out.

Direct determination of distribution curves of particle size in suspensions. E. M. CROWTHER (J.S.C.I., 1927, 46, 105-107 T).-During the process of sedimentation from a dilute suspension, the concentration or density at a given depth x after a time t is a measure of the amount of particles which have settling velocities equal to or less than x/t. Measurements of the density of the suspension at a given depth as a function of time can provide directly a continuous sizedistribution curve of sedimenting particles. The difference of hydrostatic pressure over a small range near the base of a column of suspension may be taken as the mean density over this range. An apparatus is described for obtaining such pressure differences by direct readings on a highly sensitive differential liquid manometer, consisting of aniline in contact with slightly alkaline water. The difference of the aniline levels in the manometer gives the proportion of material remaining in suspension at a known depth and time, and having therefore particle sizes equal to or less than a known value. Summation percentage curves are readily constructed by plotting these readings against the corresponding times or their logarithms. A simple change of scale gives the settling velocities and, in the case of the

logarithmic curve, the logarithm of the equivalent diameters, as deduced by Stokes' law.

Active silicic acid. RUFF and MAUTNER.-See VII.

PATENTS.

[Refractory heat screen for] furnaces. CARBO-RUNDUM Co., LTD. From CARBORUNDUM Co. (E.P. 266,140, 4.3.26).—In a furnace which has "radiating combustion chambers" separate from that containing the goods, the refractory walls between the combustion and goods chambers are constructed in zones having different thermal conductivities, so that a gradient in the heat transfer to different portions of the furnace may be obtained. B. M. VENABLES.

Apparatus [tunnel kiln] for burning or calcining materials. H. M. ROBERTSON (U.S.P. 1,621,222, 15.3.27. Appl., 20.6.25).—In a tunnel kiln which is provided with a preheating section and a longer burning section, the latter is heated by flames from primary combustion chambers built in the side walls, which flames enter the lower part of secondary combustion chambers, mix with more air, and emerge through ports extending the whole length of the burning section.

B. M. VENABLES.

Drying apparatus. G. W. CHRISTOPH, ASST. to AMERICAN HARDWARE CORP. and STERLING BLOWER CO: (U.S.P. 1,623,017, 29.3.27. Appl., 14.12.25).—The goods to be dried are tumbled in drying material within a drum, the drying material is removed by a concentric screen, and returned by another concentric drum to the point of entry. B. M. VENABLES.

Treatment of hygroscopic materials. E. C. R. MARKS. From CARRIER ENGINEERING CORP. (E.P. 267,215, 9.12.25).—Material which it is desired to deliver with a definite proportion of moisture is conveyed through an apparatus of three compartments, the atmosphere in each compartment being controlled by air leaving that compartment. In the first compartment the temperature is controlled by a thermostat, but the humidity is not regulated. In the second damping sprays and heating means are provided, and the humidity is regulated by wet and dry bulb thermometers. The air going to the third compartment is previously saturated and then heated (under control of a thermostat) just before entering the chamber.

B. M. VENABLES.

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Heat treatment of materials. [Ore reduction or coal distillation.] J. PRENTICE and A. P. PEHRSON (E.P. 266,402, 16.4.26).—The material constitutes a heating resistance between metallic or carbonaceous plate electrodes within a rotary or oscillatory furnace chamber provided at its opposite ends with inlet and outlet necks for maintaining a uniform level of the material in the chamber. In treating material which becomes sufficiently conductive only when heated, a resistor material, e.g., coke, carborundum, or calcined anthracite, may be preliminarily supplied to the furnace until the desired temperature is reached. The material is preferably fed continuously by a screw conveyor through a preheating chamber to the inlet neck of the furnace chamber, the residues and volatile products being continuously withdrawn and cooled. The bore of the furnace chamber between the electrodes may be made of varying cross-sectional area to obtain local variations of temperature. H. HOLMES.

Ball or tube mill. C. von GRUEBER MASCHINENBAU A.-G., and C. PFEIFFER (E.P. 262,785, 8.12.26. Conv., 8.12.25).—A ball or tube mill is divided into two grinding compartments by a transverse division comprising two walls and a narrow space between. The material from the first grinding chamber passes through concentric ring-shaped slots in one wall into the space, where it is lifted by curved vanes to a central opening in the other wall, through which it passes into the second grinding chamber. A central opening may be made also into the first chamber, and within these openings may be placed a double cone deflector which, in conjunction with the slope of the vanes, is stated to have a certain classifying effect, returning large material to the first grinding chamber. B. M. VENABLES.

Grinding or pulverising mill. GEBR. SCHLEIFEN-BAUM & Co., G.M.B.H., and A. IRMER (E.P. 267,426, 27.10.26).—A disintegrator which is specially suitable for coal and other fuel is formed with the stationary impact surface of round rods in loose contact with each other and free to revolve under the impact of the material. A number of disintegrating chambers may be assembled in line, each one having beaters of a slightly larger diameter than the preceding (coarser) one.

B. M. VENABLES.

Comminuting mill. R. C. NEWHOUSE, Assr. to ALLIS-CHALMERS MANUF. Co. (U.S.P. 1,620,982, 15.3.27. Appl., 15.4.26).—A grinding mill, of the ball or pebble type, is divided into two compartments, viz., a cylindrical primary reduction chamber of larger diameter (containing larger balls) tapering to a secondary cylindrical portion of smaller diameter, with a perforated partition between the two. B. M. VENABLES.

[Screen] separator. T. J. STURTEVANT, ASST. to STURTEVANT MILL Co. (U.S.P. 1,621,564, 22.3.27. Appl., 29.6.25).—A substantially flat screen cloth rests without being stretched upon a frame with round cross bars which is vibrated. B. M. VENABLES.

Filters [for sewage and industrial effluents]. A. C. HANDLEY (E.P. 266,857, 6.1.26).—An open tank is provided with removable filter elements extending transversely or transversely and longitudinally across it and comprising coir fibre between expanded metal sheets, which may be connected by spacing bolts.

H. HOLMES.

Edge filters. ARTIEBOLAGET SEPARATOR (E.P. 244,111, 4.12.25. Conv., 4.12.24).—Laminæ for edge filters are made of thin sheets of metal which have been

locally reduced in thickness by corrosion or erosion. The sheets are assembled with the unreduced portions in line. B. M. VENABLES.

[Fritted glass] filter. SCHOTT & GEN., Assees. of M. HERSCHKOWITSCH and P. PRAUSNITZ (U.S.P. 1,620,815, 15.3.27. Appl., 12.6.24. Conv., 27.6.26. Cf. E.P. 218,286 and 226,182; B., 1925, 230; 1926, 423).—A filter comprises a porous plate of fritted glass fused into a funnel made of glass having the same coefficient of thermal expansion. T. S. WHEELER.

Distilling apparatus [bubbling column]. W. E. BROWN, ASST. to UNITED IRON WORKS, INC. (U.S.P. 1,620,593, 8.3.27. Appl., 6.8.24).—The gases pass upwards through a casing which is provided with a number of liquid holders or trays, each comprising a number of parallel troughs, through which the liquid passes zig-zag horizontally in each tray, then zig-zag downwards from tray to tray. The openings between the troughs are covered by hoods, which cause the gases to bubble through the liquid. B. M. VENABLES.

Evaporator. C. W. AIKEN (U.S.P. 1,621,862, 22.3.27. Appl., 5.4.23).—A quantity of liquid contained in a casing is heated by a steam drum or nest of vertical tubes in the lower part, the steam being admitted through the upper tube plate into a telescopic nozzle tube, which has discharge nozzles in the lower part of the drum.

B. M. VENABLES.

Method of emptying containers. I. G. FARBENIND. A.-G., Assees. of FARBENFABE. VORM. F. BAYER & Co. (E.P. 249,475, 8.1.26. Conv., 23.3.25).—A container, as described in E.P. 247,744 (B., 1926, 345), is provided, immediately below the nozzle, with a constricted channel shaped as a solid of revolution and directed downwards towards one or more discharge passages. Preferably the bottom of the container is fitted with a discharge funnel having a cylindrical portion into which projects a conical member forming a bearing for the rotary pipe carrying the nozzle. Removal of coarse or moist material is facilitated. H. HOLMES.

Apparatus for the mechanical production of colloidal material. SUDENBURGER MASCHINENFABR. & EISENGIESSEREI A.-G., and W. OSTERMANN (G.P. 436,368, 28.3.25. Addn. to G.P. 421, 318).—The outer casing and grinder inside it are both provided with grooves parallel to the axis, which decrease in depth towards the outlet, a short length at the end being ungrooved so that the material has to pass between the grinder and the casing. There are also one or more grooves cut at right angles to the axis. L. A. COLES.

Production of impervious [graphitic] material. W. F. BLEECKER (U.S.P. 1,620,940, 15.3.27. Appl., 28.12.21. Cf. U.S.P. 1,462,003; B., 1923, 832 A).—Artificial graphite is heated *in vacuo*, and molten asphalt, tar, or the like is then admitted so as to impregnate the graphite. The material is then baked out of contact with oxygen, and the process is repeated if necessary. The product is of value as a chemically-resistant material.

T. S. WHEELER.

Method of heating furnaces. F. HELBIG (U.S.P. 1,625,082, 19.4.27. Appl., 21.3.23. Conv., 17.5.21).— See E.P. 211,548; B., 1924, 382. Absorption refrigerating apparatus. B. C. VON PLATEN and C. G. MUNTERS, ASSTS. to ELECTROLUX SERVEL CORP. (U.S.P. 1,620,843, 15.3.27. Appl., 4.8.23. Conv., 18.8.22).—See E.P. 202,650; B., 1925, 975.

Comminuting mill. ALLIS-CHALMERS MANUF. Co., Assees. of R. C. NEWHOUSE (E.P. 268,264, 29.12.26. Conv., 15.4.26).—See U.S.P. 1,620,982; preceding.

Method and apparatus for grading solid materials. G. R. BAKER, W. E. PRESCOTT, and C. W. GILDERDALE, ASSIS. to ROWNTREE & Co., LTD. (U.S.P. 1,623,040, 5.4.27. Appl., 31.1.24. Conv., 26.2.23).— See E.P. 220,671; B., 1924, 895.

Condensing apparatus. A. O. H. PETERSEN, ASST. to N. V. NEDERLANDSCHE INSTALLATIE MAATSCHAPPIJ THERMA (U.S.P. 1,624,066, 12.4.27. Appl., 20.5.25. Conv., 17.1.24).—See E.P. 243,982; B., 1926, 113.

Hammers for grinding, crushing, and pulverising mills. Bosserr' CORP., Assees. of W. J. CLEMENT (E.P. 260,269, 20.10.26. Conv., 20.10.25).

Cleaning high-pressure boiling apparatus. G. ULLMANN (E.P. 257,600, 19.8.26. Conv., 28.8.25).

Producing foam for fire extinguishing purposes. Excelsion Feuerlöschgeräte A.-G., Assees. of Minimax A.-G. (E.P. 254,285, 1.6.26. Conv., 29.6.25).

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

Coke in relation to some of its industrial and domestic uses. H. HOLLINGS and N. E. SIDERFIN (J.S.C.I., 1927, 46, 76-84 T).-A rapid method for determining reactivities of coke is described, which is designed to reveal small differences of reactivity and the progressive changes which occur in any one coke during prolonged heating at 950° in contact with carbon dioxide. The gas is passed continuously at constant rate over the coke sample at 950°. The reactivity at any given time during the test is obtained by noting the velocity of the gas after its passage over the coke relative to its initial velocity, and calculating therefrom the change in volume which has occurred due to carbon monoxide formation. The reactivity is expressed as percentage of carbon dioxide converted. The cokes tested, arranged in order of their reactivities, were as follows :-- Coke from blended coals ; verticalretort coke treated with sodium carbonate solution; horizontal-retort coke similarly treated; vertical-retort coke; horizontal-retort coke; beehive oven coke. The general properties of "hard" and "soft" cokes are discussed. Six different fuels were tested in closed stoves for combustibility in air, the basis for comparison being the minimum quantity of air required to maintain combustion during a 10-hr. run. Two stoves with grates 24 in. and 8 in. diameter, respectively, were used, and it was found that whereas "hard" coke was most suitable for the large stove, the "soft" cokes were preferable for the small stove because their higher combustibility promoted easier control. The control is rendered easy on the large stove, not by good combustibility, but by the insulating effect of the coke surrounding the central core of burning fuel. It is suggested

that by carbonising a suitable blend of coals in suitable plant (e.g., coke ovens) it should be possible to produce a hard coke which is also highly combustible and reactive. The efficiency of water-gas manufacture as influenced by quality of coke and air-blast velocities is discussed with reference to experimental data published in the Carbonisation Committee's Report to the American Gas Association. It is shown that there is less loss of heat in the blow gases when using a coke of low reactivity than when using a more reactive fuel, and that with any given fuel the efficiency of the blow is increased by increasing the velocity of the blast.

Coke formation. R. A. MOTT (J.C.S.I., 1927, 46, 85-92 T).-The results are recorded of an experimental study of coke formation from lump coal and crushed fine coal for a series of 17 representative British coals. The coals chosen cover a range of carbon contents of 92 to 79% for bright coals (clarains) and 88 to 81% for hard coals (durains). The standard for coking power chosen is the ability to produce in practice a metallurgical coke from crushed fine coal. It is shown that coals which produce a serviceable coke in practice have considerable swelling power when heated at fast or slow rates of heating, whilst inferior coking coals swell less considerably, and non-coking coals show no marked swelling power. It is argued that this swelling power (which is due to the evolution of volatile matter during the plastic stage of coke formation) is an important factor in conferring mechanical strength to the resultant coke in coke-oven practice. It is suggested that coke formation is due to vitrification when the molecules of the solid surfaces gain under mechanical disturbance (due to the swelling power) a temporary and local mobility approaching that of the liquid state. It is also suggested that in coke formation from crushed coal, wetting phenomena are important, and aggregation of the various particles into a strong coherent residue can only occur if liquid hydrocarbons extruded from the particles remove the air films from their surfaces. If swelling also occurs at this stage, the molecular force of cohesion can act and aggregate the particles into a coherent residue. In the formation of coke from lump coal (such as occurs in vertical-retort practice) it is argued that the opposing forces, swelling power and cohesion, determine the strength of the resultant coke.

Behaviour of carbonised fuels in the open firegrate. (Mrs.) M. F. BLIGH and H. J. HODSMAN (J.S.C.I., 1927, 46, 92-99 r).—The radiant efficiency was measured for several solid fuels burning in an open domestic grate adapted to consume carbonised fuels. The importance of heat-insulation of the fire-back was noted. The cokes usually radiated more of their heat of combustion than the coal. The size of coke had little effect on the efficiency, but large pieces were more difficult to ignite. The radiant efficiency of a lowtemperature coke (9.5% volatile matter) was greater than that of the coal, but less than that of a gas coke (vertical retort). The highest figure (29%) was obtained with a special coke of very low ash content prepared under high-temperature conditions. A by-product coke was difficult to ignite, and it burned only so long as a deep column of fuel was maintained. It is considered

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that if a high radiant efficiency is sought a low content of ash and of volatile matter should be aimed at. Impregnation of the gas coke with sodium carbonate increased its rate of combustion, but lowered the thermal output by radiation.

Heat of combustion of the carbon in coke, and reactivity of coke. M. W. TRAVERS (J.S.C.I., 1927, 46,128—130 T).—The results of calorific value determinations of samples of coke produced under known conditions are analysed. It is shown that the derived heat of formation of carbon dioxide is generally lower for high-temperature coke than for low-temperature coke. It is suggested that a practical limit to the value of this constant for coke produced in modern plant will be in the neighbourhood of 95,000 C.H.U. per lb.-mol. or 14,250 B.Th.U. per lb. of carbon. So long as the material is porous the surface energy will be considerable, and the value for massive graphite, 94,275, will not be reached.

Method of ignition in calorimetric determinations of calorific value. O. KÜNLE (Brennstoff-Chem., 1927, 8, 107).—The powdered substance is enclosed in a wrapper made of cigarette paper, weighing about 0.05 g., and having a calorific value of about 155 kg.-cal. The packet is wound round the centre and at each end with the ignition wire, and is suspended between the ignition terminals in the calorimeter. In the case of substances which are difficult to ignite, a wrapper of waxed paper may be preferable (cf. Steuer; B., 1927, 161). W. T. K. BRAUNHOLTZ.

Determination of nitrogen in fuels. G. LAMBRIS (Brennstoff-Chem., 1927, 8, 108; cf. B., 1927, 289).— A correction of a printer's error in the original article. W. T. K. BRAUNHOLTZ.

Thermal study of the manufacture of water-gas. A. PARKER (J.S.C.I., 1927, 46, 72-76 T).-The principal thermal changes involved in the manufacture of blue water-gas by the intermittent process are discussed. A thermal account constructed from data procured during an investigation of the process is given. The total heat supplied to the generator in the form of coke, steam, and air is accounted for in eight items: (1) Potential heat value of the water-gas made; (2) sensible heat of water-gas; (3) total heat of undecomposed steam; (4) potential heat of blow gas; (5) sensible heat of blow gas; (6) potential heat of ashes, clinker, and dust; (7) sensible heat of ashes, clinker, and dust; (8) a difference item including losses by radiation etc. A suggested scheme for separate thermal analyses of the air-blow periods and steaming periods is discussed, and a new scheme, which does not require assumptions regarding the nature of coke carbon, is proposed. Examples of separate thermal accounts are given.

Study of flame movement. O. C. DE C. ELLIS (Safety in Mines Res. Brd., Paper No. 32, 1927, 35 pp.). —The methods of measuring flame speeds are discussed. An improved photographic method working on the cinematographic principle is described. A series of apertures on the periphery of a rapidly revolving disc passes before the lens of a plate camera. A similar, auxiliary disc revolves before this in the reverse direction, so that the gradual obscuring of the lens ends at its centre and not at its rim. A radial knife device, which can be released at will, is attached to the first disc. When in use, the two discs are revolved at a constant known speed, and the flashes from the mercury break of an electrically-controlled tuning fork are registered on the revolving drum of an auxiliary camera (which thus serves both as a chronograph and to record the speed of propagation of flame). When the radial knife is released, (i) the primary of an inductively-coupled circuit is ruptured and immediately re-made, thus causing a spark which is registered on the revolving drum; (ii) just afterwards, a second, similar circuit is broken, the resulting spark igniting the gas mixture; and (iii) the first circuit is again broken when the wheel has completed half a revolution, causing another flash to be registered on the drum. Thus the drum record of sparks and of the time scale gives the necessary information for calculating the time interval between ignition and the first exposure on the plate, and the intervals between successive exposures. A series of photographs on the same plate can be taken at accurately-timed intervals during the course of an explosion, or a series of explosions can be carried out under identical conditions and a number of photographs taken at chosen time intervals on separate plates. Both methods of application are illustrated by photographs of the flames in mixtures of air or oxygen with carbon S. K. TWEEDY. monoxide.

Estonian shale oil. I. Isolation and properties of phenols. P. N. KOGERMAN (J.S.C.I., 1927, 46, 138-143 r).-Estonian oil shale, "kukersite," on distillation at 500-600° affords about 20% of its weight of crude oil, d¹⁵ 1.007, viscosity (Engler) 5.5°, moisture 0.9%, containing 72.1% of neutral substances, chiefly hydrocarbons, 22.4% of phenols, 4.0% of carboxylic acids, and 0.2% of bases. Investigation of the fraction of b.p. 230-270°/760 mm. (the lowest containing phenols in quantity) indicates a tendency for the phenolic content to decrease on redistillation. This fraction, which forms 8% of the crude oil, contains $17 \cdot 2\%$ of phenols, 2.5% of acids, and less than 1% of bases. The neutral oil, yellow, has d²⁰ 0.857 (d¹⁷ 0.847 on redistillation in vacuo at 60 mm.), and when treated with liquid sulphur dioxide at -20° affords $32\cdot8\%$ of refined oil, d^{17} 0.7995, $n_{\rm D}^{20.5}$ 1.4471, 35% of which is absorbed by sulphuric acid; the extract $(61 \cdot 2\%)$, has d^{17} $0 \cdot 8663$, $n_{\rm D}^{20.5}$ 1.4792, and 70% is absorbed by sulphuric acid. The phenolic fraction contains o-, m-, and p-cresols, 1:4:5-,1:2:4-, and 1:3:4-xylenols, and the presence of mesitol or propylphenol is indicated. Pyrocatechol is present in the aqueous fraction obtained on distillation of the oil. The phenols of kukersite thus resemble those of low-temperature coal tar (Weindel; B., 1925, 795), but, in addition, phenolic ethers are present. Guaiacol has been detected qualitatively. With strong sodium hydr-oxide solution the fraction, b.p. 230-270°, is completely resinified. For the extraction of phenols from the crude oil, the oil is mixed with an equal volume of benzene, and the concentration of the sodium hydroxide solution should not exceed 15%. The quantity of oil dissolved by the latter reagent increases both with the quantity and concentration of the sodium hydroxide solution. Variations were not so great on extracting the fraction of b.p. 230-270°. For the determination of the phenols, the water content is determined by distillation with xylene and also the matter insoluble in ether; a second sample of equal weight is distilled up to 160° , the remaining oil dissolved in a measured volume of ether, and extracted three times with 10% sodium hydroxide solution. The extract is washed with an equal volume of ether, and the washings are added to the oil. The ether is evaporated *in vacuo*. The flask containing the oil is warmed for 10 min. and weighed, the water being determined by xylene distillation. R. BRIGHTMAN.

Determination of paraffin wax in crude wax. L. M. HENDERSON and S. W. FERRIS (Ind. Eng. Chem., 1927, 19, 262-264).-Various methods for the analysis of oil-wax mixtures are reviewed. A modification of the method of Wyant and Marsh (B., 1926, 37), using nitrobenzene as a solvent, is proposed. The determination is carried out in a Kjeldahl flask with shortened neck, to the bottom of which a portion of the bottom of a test tube is fused. A surrounding beaker provides an air bath for hot or chilled air. The flask is fitted with a thermometer, a tube for the withdrawal of the dissolved oil, and a suction tube for vapours. 20 c.c. of nitrobenzene are added to a 10 g. sample in the flask, air is slowly bubbled through, and the temperature raised to 70°, and then lowered to 32° for 5 min. The wax solidifies and floats on the surface of the solvent. The oil solution is withdrawn, and a second 20 c.c. of solvent are added, and the temperature is raised to 135° and later to 150°. Nitrobenzene vapours are removed by suction, when the m.p. of the wax may be determined, and the yield of wax weighed. The yields closely agree with those of sweating ovens. H. MOORE.

Specific gravity of paraffin wax. F. J. MORRIS and L. R. ADKINS (Ind. Eng. Chem., 1927, 19, 301-302). -The minute bubbles in commercial wax contain air, which, however, is not held in solution when wax is melted at atmospheric pressure. A modified form of the Nicholson hydrometer made of glass was used to determine sp. gr. The pan in the liquid consisted of two crystallising dishes one inside the other, the inner one, inverted, having two V notches in the edge to allow of the flow of water. Samples of wax were kept under 30 mm. pressure for 24 hrs. at 60-65°, and cooled in vacuo at room temperature. Samples were weighed on the instrument in air and in water at 15.5°. The results differed greatly from those taken at 54.5° and reduced to 15.5° by the petroleum conversion tables. To determine the expansion coefficient, sp. gr. were taken at every 5.5° from 15.5° to 43°, and at every 2.3° from 43° to 54.5°. The results, given in graph form, show the break in the curve where most of the hydrocarbons of the sample melt, and also flat spots of unknown H. MOORE. significance.

Deterioration of mineral oils. I. Mechanism of oxidation and action of negative catalysts as determined by a dynamic method. R. T. HASLAM and P. K. FROLICH (Ind. Eng. Chem., 1927, 19, 292—296). —Tests were made on a highly refined Parke-Davis oil. Dry oxygen was bubbled at 10 litres per hr. through 75 g. samples in large test tubes, the temperature being kept at 130° or 140°. The resulting acidity was titrated as oleic, and the viscosity taken by Lang's method. The action

of catalysts was expressed by the hours by which oxidation was accelerated or retarded. The addition of 0.01'g. of a negative catalyst gave a delay in hours as follows for the substances tested : β -naphthylamine, 3; ethyl- α naphthylamine, 3; methyl-a-naphthylamine, 4.5; anaphthylamine, 4.5; p-aminophenol, 5; diphenylamine, 5.5; phenyl-a-naphthylamine, 11, and diphenylhydrazine, 18. The oxidation curve after the delay has the same form as with the pure oil. Experiments on the loss of power of the catalyst were made, using heat alone, oxygen, and nitrogen. p-Aminophenol disappears by evaporation; diphenylamine is mainly destroyed by heat, whilst diphenylhydrazine is partly evaporated and partly destroyed by oxidation. Preheating with nitrogen accelerated oxidation with oxygen. The rate of oxidation was the same with dry as with damp oxygen. Oxidation curves tend to attain a maximum at about 28 hrs., and rise only slightly up to 40 hrs. The viscosity curves show similarity with the oxidation curves, but the viscosity increases more rapidly, particularly after the oxidation curve has reached its maximum. Only a small proportion of the oxygen in oxidised oils is present as acids, the rest being probably as products of condensation or polymerisation. Aldehydes are not stable in the heated and oxidising oil. The process of oxidation appears to follow the series :---hydrocarbons, alcohols, aldehydes, ketones, naphthenic and fatty acids, condensation and polymerisation products. H. MOORE.

Evaluation of turbine oils. T. H. ROGERS and C. E. MILLER (Ind. Eng. Chem., 1927, 19, 308-312).-Deterioration of turbine oils in service is due to oxidation, with the formation of asphaltic material insoluble in oil, and of free acids soluble in oil, which form insoluble soaps in contact with iron or copper. Both the soaps and the asphaltenes are emulsifying agents of the water-in-oil type. Turbine deposits consist mainly of heavy-metal soaps. A proposed stability test consists in subjecting 500 c.c. of oil to oxygen (2 or 3 bubbles per sec.) at 100°. Acidity and Herschell demulsibility tests were taken at intervals of 48 hrs. The test is made in a flask supported in a water bath, and fitted with a thermometer and tube for oxygen supply. Acidities were determined by titration, using 95% alcohol and phenolphthalein as indicator. The standard tests were made with a spiral of iron wire present. The acidity curves do not always conform with the demulsibility curves. Confirmatory tests were run on 1000-kw. turbines over a period of some 7 months; 1 hr. of stability test was roughly equal to 9 hrs.' running. The stability tests were confirmed by the behaviour of the oil in the dry turbine. H. MOORE.

Prediction of flash point of blends of lubricating oils. E. W. THIELE (Ind. Eng. Chem., 1927, 19, 259— 262).—An approximate method of calculation is based on the principle that the antilogarithm of one-hundredth part of the flash point is an additive property of the oil (on a volume basis), the flash point of an ordinary mixture of lubricating oils being calculated from the values of its constituents. Tables are given for mixtures of two oils. Assuming that the flash point is the temperature at which the vapour pressure of an oil reached about 10 mm. of mercury, the author demonstrates 356

CL. II.-FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

mathematically the correctness of the method, which does not, however, apply to oils of low b.p., *e.g.*, kerosene. H. Moore.

Acids in automobile crank-cases. A. F. MESTON (Ind. Eng. Chem., 1927, 19, 312-315).-The oil and vapours in a crank-case are always acid, containing naphthenic and other acids. Running trials were made on cars using gasoline and Pennsylvanian lubricating oil, to ascertain the rate of increase of chamber acidity; with a 6-cylinder car the acidity value attained a maximum at 0.35 mg. of potassium hydroxide per g. of oil, and the dilution a maximum of 19% after running 400 miles, and in other tests the maximum acidity was 0.50 mg. High acidity does not always accompany high dilution, but the acidity is reduced by driving off the diluent. Naphthenic acids were found in the used oil, but not in the condensed water from the crank-case, which contained calcium, magnesium, and iron, and was very corrosive to steel. The acidity values were determined by titration with phenolphthalein as indicator, and the dilution values by the Sligh test in a modified form. H. MOORE.

Determination of phenol. WILLIAMS.—See III.

Ammonium sulphate. WEINDEL.-See VII.

Amyl acetate from gasoline. Koch and BURRELL.—See XX.

PATENTS.

Plant for washing coal and other minerals by means of liquid streams. A. FRANCE (E.P. 265,077, 18.8.26).—In washing coal with streams of water flowing in launders, the heaviest fractions are made to move with the lowest possible velocity relative to that of the cleaned portions by subdividing the launder by adjustable partitions running parallel to its edges, such partitions being completely submerged in the moving stream. S. PEXTON.

Apparatus for making artificial fuel. R. HAMP (E.P. 264,309, 14.5.26).—Wood refuse is transferred by means of rollers from a hopper to a cylinder in which moves a reciprocating piston. The outlet of the cylinder being of restricted section compresses the fuel in its passage from the cylinder. During the transference of fuel to the cylinder prior to compression, suction is applied to the cylinder through small holes in its side. This ensures complete filling of the cylinder at each stage. S. PEXTON.

Briquetting bituminous coal. S. R. WAGEL, Assr. to LEHIGH COAL & NAVIGATION CO. (U.S.P. 1,623,764, 5.4.27. Appl., 16.6.24. Renewed 31.7.26). —A mixture of bituminous coal with clay and a binder of sulphite liquor, clay, and asphalt is passed through regions of increasing temperature with alternate cooling.

C. O. HARVEY.

Preparation of a bituminous composition. L. KIRSCHBRAUN (U.S.P. 1,620,899 and 1,620,900, 15.3.27. Appl., [A], 23.4.21; [B], 25.4.24).—(A) Bentonite is of value as a dispersing agent in the preparation of aqueous bituminous emulsions of the type described in U.S.P. 1,517,075 (B., 1925, 65). (B) Asphalt is emulsified with from 10 to 40% of its weight of bentonite, and dried. The product is infusible, and of value as a waterproofing and roofing material. T. S. WHEELER. Manufacture of agglomerated adsorbent carbon. Soc. DE RECHERCHES ET D'EXPLOITATIONS PÉTROLIFÈRES (E.P. 261,692, 8.12.25. Addn. to E.P. 244,461; B., 1927, 133).—The mechanical strength of the absorbent carbon is improved by lightly agglomerating the powder with a low proportion of organic binder (preferably with admixture of a dehydrating salt such as ferric chloride), drying the shapes moulded from the resulting pasty mess, coating them superficially with the binder (with or without a small proportion of powdered carbon), calcining at 700° in a luted refractory pot, and washing if necessary. C. O. HARVEY.

Means for gas separation. H. DUMARS and W. S. BOWEN, ASSTS. to BOWEN-DUMARS POWER CORP. (U.S.P. 1,622,134, 22.3.27. Appl., 28.11.23).—An apparatus for separating from a gas mixture by refrigeration substances which are, respectively, solid and liquid at the same temperature consists of jacketed condensing and conveying tanks containing baffles on which the solid condensate collects, and a brine tank for receiving liquid condensate. C. O. HARVEY.

Distillation of carbonaceous substances. T. M. DAVIDSON, and PATENT RETORTS, LTD. (E.P. 263,933, 5.10.25).—A carbonising kiln, built in the form of a box, and open at the top, is filled with low-grade fuel. The fuel is ignited at the top and partly burnt by the admission of air through ports in the side. Within the charge are distributed perforated tubes, each of which communicates through a cock to an exhausting fan. The air admission and the removal of distillation products are regulated so that the distillation zone moves slowly down the kiln. S. PEXTON.

Distillation of carbonaceous materials. B. McCORMICK (E.P. 265,046, 7.5.26).—Carbonaceous material is distilled in a continuous vertical retort in a stream of superheated steam. The steam flows countercurrent to the material, which is supported on a rotating helical partition while undergoing treatment, and the primary oils, conserved by the presence of steam, are removed immediately into zones of lower temperature. S. PEXTON.

Conversion of mineral oils and the like of high boiling point into aliphatic hydrocarbons of low boiling point. F. W. TODT and P. SUMPF (E.P. 267,364, 2.6.26).—Oils particularly suitable for engine fuels, and containing a minimum quantity of unsaturated hydrocarbons and no trace of aromatic compounds, are obtained in yields of up to 80% by a cracking process which involves passing vaporised heavy oils through a cylindrical retort lined with chamotte, charcoal, or the like, charged with porous material, e.g., pumice stone, coke, etc., and maintained at a temperature suitable for successful cracking. The operation is carried out in stages, each stage being adapted (by virtue of temperature or duration of treatment) to the cracking of that fraction of the oil or cracking residue with which the retort is fed. C. O. HARVEY.

Conversion of heavy or complex hydrocarbon oils into lighter oils. J. F. DONNELLY (E.P. 243,339, 10.11.25. Conv., 21.11.24).—Hydrocarbon oil is cracked in a pipe coil under sufficient pressure to prevent vaporisation, and is injected (after admixture with cooler oil to prevent the formation of solid products) into an expansion chamber connected with a reflux condenser from which all but the heaviest fractions may be drawn off for admixture with the heated oil, the vapours passing on to another condenser. C. O. HARVEY.

Conversion of higher-boiling hydrocarbons into lower-boiling petroleum hydrocarbons. C. B. BUERGER, Assr. to GULF REFINING Co. (U.S.P. 1,623,025, 29.3.27. Appl., 22.7.25).—Oil, cracked by distilling with aluminium chloride, is freed from hydrochloric acid and other foreign matter by condensing the vapours with water brought into contact with them. C. O. HARVEY.

Production of lower-boiling distillates from higher-boiling petroleum hydrocarbons. A. J. H. HADDAN. From GULF REFINING Co. (E.P. 267,386, 24.7.26).—50—75% of an oil is cracked to produce gasoline and kerosene containing less sulphur and more saturated hydrocarbons than normal gas oil by distilling continuously with 1—3% of aluminium chloride, and removing the vapours as they are produced, preferably until at least 75% of the still charge has passed over.

C. O. HARVEY.

Conversion of heavy into light hydrocarbons. C. AB-DER-HALDEN (F.P. 610,448, 8.5.25).—The heavy hydrocarbon mixed with steam is passed through molten metal, the water being thereby decomposed into its elements at a temperature at which the hydrocarbon vapour is cracked. A. B. MANNING.

Treatment of hydrocarbon oil. H. M. LASHER, Assr. to KANSAS CITY GASOLINE Co. (U.S.P. 1,622,453, 29.3.27. Appl., 16.11.20).—The oil is heated in one chamber and cracked by spraying over a fixed catalyst situated above the bottom of a second chamber so as to provide a quiet zone below the catalyst where uncracked oil and carbon may collect, the former being returned to the heating chamber. The lighter cracked products are withdrawn from above the catalyst.

C. O. HARVEY.

Cracking oils and tars. Soc. LUXEMBOURGEOISE DES HYDROCARBURES, and F. BRIMEYER (E.P. 267,776, 20.7.26).—Oils are vaporised and cracked under their own pressure by continuous passage through a heated chamber, and, after condensation, the more volatile fractions are re-evaporated by subjection to the heat of the cracked vapours. After passage through a superheater, the vapours enter a hydrogenating tower maintained at a temperature not above 300°, mix with a stream of hydrogen sulphide (prepared by the action of hydrogen on a metal sulphide), and pass through a metallic mass which liberates nascent hydrogen and thus effects hydrogenation. C. O. HARVEY.

Manufacture of benzol and like aromatic hydrocarbons. I. W. HENRY (E.P. 267,359, 28.5.26).— Aromatic hydrocarbons are produced from carbonaceous materials (e.g., powdered bituminous coal with or without admixture with limestone) by heating in a retort and subjection to a high-frequency oscillating electro-magnetic field in the presence of hydrogen. The carbon particles suspended in the gas become ionised and react, yielding enriched hydrocarbon gas.

C. O. HARVEY.

Manufacture of [anti-knocking] liquid fuels. I. G. FARBENIND. A.-G. (E.P. 252,019, 10.5.26. Conv., 8.5.25. Addn. to E.P. 226,731; B., 1925, 163).—The antiknocking effect of iron carbonyl is enhanced and the quantity necessary reduced by adding to benzine containing more than 0.2 g./gal. of the carbonyl a non-metallic organic compound (other than an alcohol) containing oxygen or nitrogen (ketones, aniline, nitrobenzene, etc.), with or without the addition of an organic halogen compound (e.g., halogenides of aliphatic or aromatic hydrocarbons). C. O. HARVEY.

Motor fuels. DEUTSCHE PETROLEUM-A.-G., and E. H. RIESENFELD (G.P. 436,945, 25.2.25).—A motor fuel is made by mixing alcohol and benzine, and adding chlorinated rape oil. This addition ensures uniform combustion in the motor, and homogeneous mixing of the alcohol and benzine. A. B. MANNING.

Purification of benzene, benzine, and similar motor fuels. BENZOL-VERBAND G.M.B.H. (G.P. 436,944, 2.2.26).—The oils are freed from sulphur by being brought into contact with amalgams of the heavy metals, in the form of amalgamated metal shavings, wire, or netting, at the ordinary or at higher temperatures. A. B. MANNING.

Obtaining light oils by washing gases. I. G. FARBENIND. A.-G., Assees. of A.-G. FÜR ANILIN-FABR. (E.P. 254,287, 1.6.26. Conv., 25.6.25).—Light oils are removed from coal and natural gas by washing with the oil dissolved from mineral oils by extracting them with liquid sulphur dioxide (Edeleanu process), which takes up 6% of its weight of benzene as compared with 3% taken up by tar or petroleum oil, and which shows no tendency to thicken. C. O. HARVEY.

Refining oils. H. O. PARKER, ASST. to MATHIESON ALKALI WORKS (U.S.P. 1,622,879, 29.3.27. Appl., 22.1.25). —The oil is passed through aqueous hypochlorite solution containing excess of caustic alkali, and chlorine is introduced into the solution near the point of entry of the oil, which thereby comes in contact first with that portion of the solution containing the highest concentration of hypochlorite. C. O. HARVEY.

Method of treating petroleum. R. CROSS (U.S.P. 1,623,018, 29.3.27. Appl., 26.4.24).—A minor flow of oil is treated with chlorine, submitted to the action of light, and combined with the major flow.

C. O. HARVEY.

Separation of hydrocarbon fractions. D. PVZEL, Assr. to SIMPLEX REFINING Co. (U.S.P. 1,622,737, 29.3.27. Appl., 23.8.23).—The partial vapour pressure of a portion of a mixture of hydrocarbons is reduced by injecting vapour having a different vapour tension (heat being supplied from the remainder of the mixture which is maintained under its initial vapour tension), and the mixture of vapour is removed. C. O. HARVEY.

Removal by centrifuging from fluid hydrocarbons of the solid hydrocarbons precipitating at low temperatures. BERGEDORFER EISENWERK A.-G. (E.P. 267,038, 26.10.26. Conv., 16.8.26).—In a process for the centrifugal separation of paraffins frozen out from lubricating oils etc. the difference in sp. gr. necessary for successful separation is imparted to the mixture of oil and paraffins by adding a heavy solvent (e.g., trichloroethylene, carbon tetrachloride, etc.) for the liquid hydrocarbons instead of adding gasoline, as is common practice. Only relatively small proportions of these heavy solvents are required, and the paraffins travel to the centre of the bowl, where they are removed along with some of the fluid hydrocarbons, the viscosity being kept sufficiently low, if necessary by the addition of a low-viscosity hydrocarbon. C. O. HARVEY.

Condensation of hydrocarbons. E. W. ISOM and J. E. BELL, Assrs. to SINCLAIR REFINING CO. (U.S.P. 1,623,790, 5.4.27. Appl., 10.9.23).—The hot vapours are injected into a large volume of cooled liquid condensate, circulation being maintained by the incoming vapours. C. O. HARVEY.

[Liquid] fuels. J. F. P. DE LA RIBOISIÈRE (E.P. 244,797, 17.12.25. Conv., 17.12.24).—See U.S.P. 1,558,967; B., 1926, 6.

Removal of water from peat and the like. O. Söderlund, T. Boberg, and N. Testrup, Assrs. to Techno-Chemical Laboratories, Ltd. (U.S.P. 1,624,769, 12.4.27. Appl., 30.9.24. Conv., 7.11.23).—See E.P. 228,628; B., 1925, 274.

Coal drying. O. W. RANDOLPH (U.S.P. 1,623,553, 5.4.27. Appl., 24.12.23).—See E.P. 226,785; B., 1925, 976.

Recovery of sulphur compounds of ammonia in the form of salts from gas mixtures containing sulphuretted hydrogen and ammonia. F. SIEMENS A.-G., and H. BÄHR (E.P. 268,024, 23.12.25).—See F.P. 609,931; B., 1927, 252.

Production of a porous mass for storing explosive gases. G. DALEN, Assr. to AMERICAN GAS-ACCUMULATOR CO. (U.S.P. 1,623,169, 5.4.27. Appl., 14.8.22. Conv., 16.8.21).—See E.P. 203,255; B., 1923, 1060 A.

[Discharging device for] washing and separating apparatus for coal and other minerals. E. Delcu-VELLERIE (E.P. 265,112, 16.11.26).

[Burner for] combustion of pulverised fuel in furnaces. L. GROTE (E.P. 264,563, 15.10.25).

Coal distillation (E.P. 266,402).—See I.

Pulverising mill (E.P. 267,426).—See I.

Asphaltic road material (U.S.P. 1,620,813).—See IX.

Bituminous mixtures (E.P. 267,317).-See IX.

Ionising retort (E.P. 267,358).-See XI.

Production of changes in dielectric carbon compounds (U.S.P. 1,621,143).—See XI.

III.—TAR AND TAR PRODUCTS.

Manufacture of pure naphthalene. A. VON SKOPNIK (Chem.-Ztg., 1927, 51, 211-212).—A suitable tar-oil fraction may contain 40% of naphthalene, but it is desirable to re-distil this in order to obtain the naphthalene with a suitable crystalline structure. The re-distilled oil is subjected to slow crystallisation in vessels of 13 cub. m. capacity, the oil drawn off, and the crystals drained on

stages beneath the crystallisers. They then pass through a breaker and are elevated, one third to a melting pan and two thirds to a mixing vessel where the melted portion is reincorporated with the rest. The whole is passed into the press at 50-60°, and the pressure, at first low, is gradually raised to 300 atm. The pressed naphthalene (m.p. $79 \cdot 0 - 79 \cdot 1^{\circ}$) is broken and passed into melting pans. The melted naphthalene is washed with 1% of sulphuric acid $(d \ 1.70)$ to remove moisture and then with 3% of acid (d 1.84). A sample of the washed naphthalene after distillation should give no red coloration with sulphuric acid. After water and alkali washes, the product should have m.p. 79.3-79.4°. It is subjected to vacuum distillation with indirect steam in a still having a high column, in presence of a little caustic soda (d 1.14). The temperature at the top of the column should be 135-150°, and that of the condensers 105-120°, the latter being maintained with the aid of steam. Tests of the distillate are taken, and when the initial impurities are drawn off the m.p. rises sharply to $79 \cdot 9^{\circ}$. The pure liquid naphthalene is run into iron crystallising pans in a closed chamber, the cocks being controlled from outside on account of the naphthalene vapour. The aqueous fore-runnings and the resinous still residue are returned to the process. The refined product should give no coloration on melting with an equal volume of sulphuric acid or after remaining 2 hrs. on a watchglass over concentrated nitric acid. If the pressed naphthalene is washed with monohydrate (100% H2SO4) the final distillation may be omitted. The product is, however, not quite so good, and the working losses are greater. C. IRWIN.

Pitch softening points. R. G. W. EADIE (J.S.C.I., 1927, 46, 109—111 T).—Softening points by the Kraemer and Sarnow, ring and ball, and $\frac{1}{2}$ inch cube-in-air methods are given for three grades of vertical-retort tar pitch, along with values for specific gravity, volatile matter, and free carbon content. A simple apparatus for determining softening points is described which gives values corresponding closely with those of the Kraemer and Sarnow and ring and ball methods.

Rapid determination of phenol in ammonia liquor and other solutions. R. D. WILLIAMS (Ind. Eng. Chem., 1927, 19, 530-531).-In the determination of phenols by their quantitative conversion into bromophenols, special care must be exercised to free the sample from impurities likely to react with bromine. Precise experimental details are given of a method involving the removal of cyanide as cyanate by adding a few drops of ammonium polysulphide, the expulsion of ammonia by boiling with an excess of sodium hydroxide, and the removal of sulphide by oxidation with hydrogen or sodium peroxide, or preferably by precipitation as lead sulphide by means of lead oxide or carbonate. The method may be used with equal facility for determining phenols in ammoniacal liquor, ammonia still waste, benzene containing phenols, and sodium phenoxide solutions equivalent to a wide range of sodium hydroxide and phenol concentration. E. HOLMES.

Estonian shale oil. KOGERMAN.-See II.

Preparation of 2-hydrindone from coal tar. WALTERS.—See IV.

PATENTS.

Recovery of phenols from ammoniacal liquor or technical effluents. ZECHE M. STINNES, Assees. of F. ULRICH (G.P. 436,522, 14.3.25. Conv., 3.2.26) .--Phenols are recovered from ammoniacal or other liquors by extraction with a mixture of benzene or its homologues with the bases from a low-temperature or cokeoven tar. Tar oils from which the phenols have been removed, but which still contain the bases, may be used for the purpose, or the bases themselves may be used alone. Benzene containing 20% of pyridine or quinoline, or preferably such of their homologues as are only slightly soluble in water, extracts phenol quantitatively from an ammoniacal liquor, from which benzene alone removes only 65%. The efficiency of the extraction is due to the chemical action of the bases; the benzene acts merely as a diluent, and may be replaced by benzine or other neutral oil. A. B. MANNING.

Manufacture of benzol etc. (E.P. 267,359).— See II.

Purification of benzene (G.P. 436,944).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Analysis of mixtures of the isomeric toluidines. H. H. Evers and N. Strafford (J.S.C.I., 1927, 46, 114-117 T).—The existing methods for the determination of p-toluidine in o-toluidine are reviewed and shown to be of limited accuracy. The determination of *m*-toluidine is effected by titration with standard bromate (Callan and Henderson, ibid., 1922, 41, 161 T). The meta-isomeride absorbs 3 atoms of bromine per molecule, whereas each of the remaining isomerides absorbs only 2 atoms ; hence the proportion of meta can be calculated. p-Toluidine is determined from the setting point of the sample to which pure p-toluidine has been added in known amount; from the data given, the proportion of this isomeride can readily be calculated. In such mixtures replacement of a portion of the ortho- by meta-toluidine has no effect on the setting point obtained, hence the method is specific for determination of the para-isomeride.

Volumetric analysis of Malachite Green with titanous sulphate and titanous chloride solutions. H. WILKINSON and A. G. TYLER (J. Soc. Dyers Col., 1927, 43, 116).—Setoglaucine, Malachite Green base, oxalate, and double zinc chloride are decolorised gradually by titration with dilute titanous sulphate solution in 4N-sulphuric acid in presence of Rochelle salt. With titanous chloride they are decolorised after the addition of a few c.c. of the reagent, but the colour returns in a few moments, disappearing and returning with each addition of the solution until the end-point is reached, when the colour no longer reappears (cf. Knecht and Hibbert, "New Reduction Methods in Volumetric Analysis," 2nd ed., p. 107). This latter behaviour is shown by Fast Green J.J.O. (sulphate) with both titanous chloride and sulphate. Acid Green G is decolorised gradually with both reagents. E. H. SHARPLES.

Preparation of 2-hydrindone from coal tar. L. S. WALTERS (J.S.C.I., 1927, 46, 150-152 r).—The following method is recommended for the preparation of 2-hydrindone from the "middle" oil fraction of Woodall-Duckham vertical-retort tar. The fraction 175-185°

is re-distilled and, after washing with 10% sodium hydroxide solution and 10% sulphuric acid, the bromine value of the dry fraction, 176-182°, is determined. 2 g. of the oil are dissolved in 10 c.c. of carbon tetrachloride in a stoppered flask and kept in the dark for 1 hr. after adding 25 c.c. of 10% bromine in carbon tetrachloride. Excess of 10% potassium iodide solution is added and the iodine titrated with 0.1N-sodium thiosulphate, a blank titration being performed on the bromine solution. Three fourths of the total bromine absorption is calculated to indene (1 mol. bromine = 1 mol. indene), and the indene fraction is then heated at 150-160° with the amount of sodium calculated from this figure (at 110-120° if sodamide is employed), 2% of aniline being added as catalyst. The oil is decanted from the sodium-indene, the latter is crushed, added to 10 times its weight of ice-water, and distilled in steam. The oil is separated from the distillate, washed with 1% sulphuric acid, and converted into 2-bromo-3hydroxyhydrindene by mixing with 10-15% of kieselguhr, emulsifying with 10 vols. of water, and adding slight excess of 10% bromine solution in 10% potassium bromide (cf. Read and Hurst, J.C.S., 1922, 121, 2550). The bromo-hydroxy-compound is refluxed for $\frac{1}{2}$ 1 hr. with 50% alcoholic potassium hydroxide (1 mol.), the alcohol distilled, and the residue refluxed 10-15 min. with excess of 20% sulphuric acid. On rapid steam distillation of this mixture, 2-hydrindone, m.p. 57-58°, is obtained. The bromo-hydroxy-compound cannot be prepared direct from the crude indene fraction.

R. BRIGHTMAN.

Determination of carbon dioxide in carbonates [in dyestuffs]. T. CALLAN (Analyst, 1927, 52, 222; cf. B., 1927, 42).—Hepburn's modification of Van Slyke's method of determining carbon dioxide in carbonates has been found useful with dyestuffs. The dry dyestuff is first wetted with 1—2 c.c. of alcohol to prevent frothing, and the reaction was found to be complete in 6 hrs. D. G. HEWER.

PATENTS.

Preparation of azo dyes. I. G. FARBENIND. A.-G., Assees. of H. EICHWEDE (G.P. 432,426, 11.4.24).-Diazotised amines or aminoazo compounds are coupled with monoazo dyes (or the corresponding ψ -azimines) in which the second component contains at least one hydroxyl, in addition to the auxochromic group, capable of further coupling; sulphonic and carboxylic groups are excluded from all components. The dye 1:7-amino $naphthol \rightarrow resorcinol is coupled with p-nitrodiazobenzene$ (2 mols.); the product, reduced and tetrazotised, gives a deep black when coupled on the fibre with 2: 3-hydroxynaphthoic anilide. The dye from 2:7aminonaphthol diazotised and coupled acid with 2:7-aminonaphthol, is developed on the fibre with diazotised methyl 4-nitro-2-aminobenzoate for a fast C. HOLLINS. yellow-brown.

Diazo preparations. I. G. FARBENIND. A.-G., Assees. of K. SCHNITZSPAHN (G.P. 430,621, 13.2.25).—A mixture of the mineral acid salt of a diazotisable amine, with or without excess of mineral acid or of acid salt, a diluent, and a dry nitrite, remains stable and easily wetted even after long keeping at 60—65°. C. HOLLINS. 360

CL. V.-FIBRES; TEXTILES; CELLULOSE; PAPER.

Manufacture of vat dyes and intermediates. O. Y. IMRAY. From Soc. CHEM. IND. IN BASLE (E.P. 267,177, 12.10.25). — Chloro-2-methylphenylthioglycollic acid, m.p. 104°, prepared from 6-chloro-o-toluidine, is converted by means of phosphorus trichloride and aluminium chloride into 6-chloro-3-hydroxy-7-methylthionaphthene [6-chloro-7-methylthioindoxyl], m.p. 102-103°, volatile in steam, which condenses with p-nitrosodimethylaniline to form the p-dimethylaminoanil, m.p. 154° 256-257°, of 6-chloro-7-methylthiosatin, m.p. (obtained by hydrolysis of the anil with sulphuric acid). The thioindoxyl is oxidised by air to the thioindigo (carmine red on cotton from a greenish-yellow vat), or may be condensed with acenaphthaquinone (for orange), isatin a-anil (violet), 5:7-dibromoisatin (red-brown), α-naphthathioisatin (bordeaux-red), bromo-β-naphthathioisatin (from 5-bromo-β-naphthylamine, brown), or with the *p*-dimethylaminoanils of 6-chloro-4-methylthioisatin (bluish-red), 6-ethoxythioisatin (bluish-scarlet), thioisatin (red), 6-chloroisatin (red), and 4-chloro- $\beta\beta$ naphthathioisatin (violet). C. HOLLINS.

Manufacture of diamino- and aminohydroxydiaryl sulphones from sulphinosalicylic acids and quinoneimines. W. CARPMAEL. From I. G. FARB-ENIND. A.-G. (E.P. 267,366, 7.6.26).-Intermediates for indamines, indophenols, and chromable azo dyes are prepared by condensing an o-hydroxycarboxysulphinic acid with a quinone mono- or di-imine, or with a p-diamine or p-aminophenol and an oxidant. The diamines are readily acylated in the amino-group remote from the sulphone grouping. 5-Sulphinosalicylic acid is condensed with p-benzoquinonedi-imine (or with a mixture of p-phenylenediamine hydrochloride and ferric chloride) to give 2:5-diamino-4'-hydroxy-3'-carboxydiphenyl sulphone in 85-90% yield. 2:5-Diaminoanisole or -phenetole or p-aminodimethylaniline may be used in place of the p-phenylenediamine, and 5-sulphinoo-hydroxytoluic acid in place of the salicylic derivative. 5-Sulphinosalicylic acid is also condensed with a mixture of ferric chloride and p-aminophenol, giving 2-amino-5:4'-dihydroxy-3'-carboxydiphenyl sulphone; the 4:5'dimethyl derivative is similarly prepared from 5-sulphinoo-hydroxytoluic acid and 5-amino-o-cresol. The monoamines may be diazotised and coupled with resorcinol for yellow dyes, or with R-acid for red dyes.

C. HOLLINS.

Manufacture of condensation products of the anthraquinone series. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 252,029, 11.5.26. Conv., 11.5.25). — 3-Aroylbenzanthrones, when fused with alkali, give good yields of compounds of the dibenzpyrenequinone type. Benznaphthapyrenequinone, obtained in this way from $3-\alpha$ naphthoylbenzanthrone, m.p. $244-245^{\circ}$ (the condensation product from benzanthrone-3-carboxylic chloride and naphthalene), dyes cotton brownish-orange from a red vat. C. HOLLINS.

Manufacture of [indigoid] intermediates and dyes of the anthraquinone and anthracene series. Soc. CHEM. IND. IN BASLE (E.P. 249,489, 19.2.26. Conv., 18.3.25. Addn. to E.P. 210,413; B., 1925, 66).—1(2)-Halogenoanthraquinone-2(1)-carboxylic acids are treated

with sodium hydrogen sulphide to give the mercaptan, which is reduced to the mercaptoanthracenecarboxylic acid, and then condensed with halogenoacetic acid to give the thioglycollic-carboxylic acid, which on cyclisation with acetic anhydride and sodium acetate gives 1:2or 2 : 1-anthrathioindoxyl. Further, 1-carboxy-2anthraquinonylthioglycollic acid, prepared from 2halogenoanthraquinone-1-carboxylic acid and thioglycollic acid, or from 2-mercaptoanthraquinone-1-carboxylic acid and halogenoacetic acid, is cyclised to anthraquinone-2:1-thioindoxyl, or is reduced to 1-carboxyanthryl-2-thioglycollic acid. The thioglycollic acids and thioindoxyls so prepared are condensed with isatins, thioisatins, acenaphthaquinone, β -naphthaquinone, etc., or are oxidised to symmetrical thioindigos. The dyes may finally be halogenated. The following compounds are described : 1-mercaptoanthraquinone-2-carboxylic acid, the 2:1-isomeride, and the corresponding anthracene derivatives; 2-carboxy-1-anthrylthioglycollic acid, the 1:2-isomeride, and the corresponding thioindoxyls (the 1:2-thioindoxyl has m.p. 200°); 1-carboxyanthra-quinonyl-2-thioglycollic acid and the corresponding thioindoxyl; vat dyes by oxidation of 2:1-anthrathioindoxyl (yellowish-bordeaux on cotton) and the 1:2isomeride (yellowish-brown), by oxidation of 1-carboxy-2-anthraquinonylthioglycollic acid (brownish-grey), by condensation of 2-carboxy-1-anthrylthioglycollic acid with isatin- α -anil (greyish-violet), and by condensation of 1-carboxy - 2 - anthraquinonylthioglycollic acid with acenaphthaquinone (brownish-orange). Eleven other condensations for vat dyes are tabulated, the colours on cotton being brown or bordeaux, except in the case of the product from 1:2-anthrathioindoxyl and the p-dimethylaminoanil of 2:3-anthrathioisatin, which gives yellowish-green dyeings. C. HOLLINS.

Manufacture of tetrahalogenated 4:4'-dimethylthioindigotins. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 254,340, 28.6.26. Conv., 27.6.25).—Bright reddishviolet vat dyes, fast to boiling, are obtained by chlorination or bromination of 6:6'-dichloro- (or bromo-)4:4'dimethylthioindigotin, preferably in chlorosulphonic acid or nitrobenzene in the presence of a little iodine. The halogen enters the 5:5'-positions. C. HOLLINS.

Manufacture of hexasubstituted thioindigotins. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 254,743, 2.7.26. Conv., 2.7.25. Addn. to E.P. 254,340; preceding abstract).— The method of the prior patent is applied to the production of 5:5'-dichloro- and 5:5'-dibromo-derivatives of 4:4':6:6'-tetrahalogeno-, 4:4':6:6'-tetramethyl-, and 4:4'-dihalogeno-6:6'-dimethyl-thioindigotins, which are violet vat dyes for cotton. C. HOLLINS.

Manufacture of [azo] dyestuffs. F. STRAUB and H. SCHNEIDER, Assrs. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,624,637, 12.4.27. Appl., 6.3.25. Conv., 20.3.24).—See E.P. 231,149; B., 1925, 875.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER. Effect of alkalis on wool. II. Effects of sodium and potassium compounds and of temperature. H. C. CHAPIN (Proc. Amer. Assoc. Textile Chem. Colorists, 1927, 42—46).—In solutions of equimolecular concentration, sodium and potassium carbonates produce an equal weakening effect. The effect of sodium or potassium soap is small compared with that of the hot water in which it is dissolved, and much smaller than that of 0.1N-sodium or potassium carbonate solution. The weakening effect of alkali carbonate solution may be reduced by the simultaneous presence of a soap. A rise of 1° in scouring temperature near 50° has the same weakening effect as an increase of 25% in carbonate concentration near 0.2N. CHEMICAL ABSTRACTS.

Dehydration of sulphite spirit with quicklime. E. SCHLUMBERGER (Papier-Fabr., 1927, 25, 180-183).-A method depending upon the elevation of the critical separation temperatures of alcohol-petroleum mixtures by the addition of small quantities of water was used to investigate the dehydration of liquid and gaseous sulphite spirit by lime. A curve for the critical separation temperatures of a series of alcohol-petroleumwater mixtures of known water content was first obtained, and similar temperatures observed for sulphite spirit mixtures then gave directly from the curve the water content of the spirit. Impurities present in the latter, e.g., aldehyde etc., do not affect the measurement. With liquid spirit of 94-96% alcohol content, dehydration was carried out at different temperatures and. corresponding pressures in an autoclave, the time in hrs. for the change of a 94% into 98.5% spirit being taken as a measure of the dehydration velocity. A rise in temperature of 10° increases this velocity 2-3-fold, and it is of secondary importance whether the lime is in the form of powder or coarse lumps, or whether the mixture is stirred or not. It is suggested that the dehydration velocity in the heterogeneous system lime_{solid}-alcohol_{liquid} is dependent not upon diffusion (stirring, and the surface development of the lime), but upon a slowly proceeding chemical reactionapparently the decomposition of an alcohol hydrate. The lime is the more effective the less its water content, i.e., the better it is burnt, marble lime being somewhat less efficient than equally well burnt, but less dense, mortar lime. The most favourable proportion for the dehydration is 125% of the theoretical amount of lime necessary. As less than 1% of alcohol remains in the lime before the combined water passes over, the distillation yield is about 99%. For gaseous spirit, with lime heated at 100°, contact for 15 sec. suffices to dehydrate a spirit from 6% to 0.5% water content. In this case, however, the lime increases in volume about 5-fold through slaking; further, it disintegrates into a very fine powder which is carried over during the distillation, and requires several days to settle out. The advantage of this method consists in the possibility of a continuous process, and still more in the saving of the total heat of vaporisation of the alcohol, which for 1 kg. amounts to about 270 kg.-cal. The disadvantage lies in the increased space required for treatment in the gaseous state.

PATENTS.

B. P. RIDGE.

Preparation of plant fibres, particularly jute, resistant to washing. G. G. GUARNIERI (F.P. 611,289, 16.2.26).—Fibrous materials are immersed in a bath containing enzymes capable of rapidly attacking glutinous impurities, and are then bleached with an alkali hypochlorite, soured, and thoroughly washed.

A. J. HALL.

Production of fine, soft viscose fibres with good physical properties. I. G. FARBENIND A.-G., Assees. of H. LUMMERZHEIM, J. HUBER, and P. ECKERT (G.P. 436,792, 9.8.23).—Highly dispersed emulsoids, preferably of vegetable origin, are added to the liquor for dissolving the xanthate. L. A. Coles.

Preparation of aqueous emulsions of substances insoluble in water. CHEM. FABR. POTT & Co. (Swiss P. 115,701, 28.4.24. Conv., 9.7.23).—Aqueous emulsions of such substances as tetralin and *cyclo*hexanol which are useful agents in the washing and scouring of animal and vegetable raw textile materials, are prepared by means of small quantities of at least one emulsifying agent, *e.g.*, water-soluble sulphonic acids (or their salts) of substituted aromatic hydrocarbons (especially naphthalene derivatives), such as the sodium salt of propylnaphthalenesulphonic acid. A. J. HALL,

Production of patterned weaving effects. HEBER-LEIN & Co. A.-G. (E.P. 262,477, 6.12.26. Conv., 5.12.25).—Fabric is woven from both ordinary and nitrated vegetable fibres, treated locally with an alkali, and subsequently heated or steamed, the nitrated fibres being thus removed from the treated parts. Alternatively, the material is printed with a resist or a denitrating agent, or printed with a resist, denitrated, and the resist removed before being passed through an alkaline solution. Finally the whole fabric is made insensitive to subsequent alkaline treatment by being denitrated.

B. P. RIDGE.

Mercerising vegetable fibres. I. G. FARBENIND. A.-G., Assees. of L. LÖCHNER (G.P. 433,733, 16.9.24).— The tenacity of the fibres is increased 40—50%, and they are not turned yellow, by treating them, before or after spinning, at a low temperature, with a hypochlorite mercerising bath of $d \ 1.162$. L. A. COLES.

Manufacture of hollow artificial textile fibres. W.J.TENNANT. From J. ROUSSET (E.P. 267,187,11.11.25. Cf. E.P. 189,973; B., 1923, 91 A).—The usual cellulose solutions (e.g., cellulose acetate in a mixture of acetone and alcohol) are spun into a cell containing air at a temperature substantially higher than the b.p. of the solvent used, a comparatively high rate of flow of this air being maintained in order to produce an active superficial evaporation from the filaments as they emerge from the nozzle. B. P. RIDGE.

Manufacture of a non-hygroscopic pulverulent product from sulphite-cellulose waste liquor. I. G. FARBENIND. A.-G., Assees. of A.-G. FÜR ANILIN-FABR. (E.P. 250,956, 15.4.26. Conv., 15.4.25).—The total solid content of the liquor is obtained in a dry non-hygroscopic form either by evaporating the liquor at a temperature above 100° (e.g., on a drying drum at 140°) or by first heating it at a temperature above 100° and then evaporating it. Maximum stability of the product in air is obtained by subsequently heating it for a short time at a high temperature (e.g., at 180°).

B. P. RIDGE.

Manufacture of cellulose. H. WALKER (E.P.267,226, 12.12.25 and 19.7.26).—Cellulose pulp is obtained from

the leaves and stems of unripe potato plants (or other solanaceæ) by alternate freezing and thawing alone, or by such treatment followed by open or pressure boiling with water or sodium hydroxide solution.

B. P. RIDGE.

[Manufacture of] artificial silk from nitrocellulose. J. DELPECH (Addn. No. 30,982, 6.10.25, to F.P. 561,614).—Pyroxylin is dehydrated before conversion into spinning solutions. A. J. HALL.

Bleaching cellulose acetate. R. BAYBUTT and E. S. FARROW, JUN., Assrs. to EASTMAN KODAK Co. (U.S.P. 1,623,519, 5.4.27. Appl., 28.10.25).—Cellulose acetate is bleached by bringing ozone into active contact with it. W. G. CAREY.

Production of artificial leather. R. CLAVEL (F.P. 611,401, 30.5.25).—Cellulose esters are treated with aromatic hydroxy-compounds and with metal compounds. *E.g.*, cellulose acetate is successively treated with 1% tannin solution at 70—80°, washed, treated with 3% ammoniacal bismuth nitrate solution for 1 hr., the temperature being gradually raised to 65° , steeped in ferric chloride solution, washed with dilute hydrochloric acid, dyed, dried, calendered, and printed. Zinc, silver, and iron salts can be used instead of bismuth nitrate. L. A. COLES.

Production of waterproof materials from textiles, paper, and the like, by impregnation with metal chlorides. A VOHL & Co., A.-G. (G.P. 433,983, 8.8.25. Addn. to G.P. 377,659; B., 1923, 971 A).—The necessity for heating the material on rollers to effect partial hydrolysis of the cellulose to amyloids, as described in the earlier patent, is avoided by using a bath of $d 1 \cdot 616$ — $2 \cdot 244$ maintained at 60— 90° . L. A. COLES.

Production of films not sensitive to electrical action. PATHÉ CINÉMA (ANCIENS ÉTABL. PATHÉ FRÈRES) (F.P. 611,136—7, 26.5.25).—Nitrocellulose films are coated on the one side with the photographic emulsion, and on the other with a mixture of (A) cellulose nitroacetate, or (B) cellulose benzoate, with a plasticising agent. L. A. COLES.

Altering the viscosity of materials containing cellulose. H. P. BASSETT (U.S.P. 1,620,938, 15.3.27. Appl., 19.12.23).—Cellulose fibre is heated with 0.05— 1% sulphuric acid at 20—100°, washed, treated with 0.1-2.5% sodium hydroxide solution, and washed again. The product when dissolved in the usual solvents for cellulose gives solutions of high or low viscosity, according as the weaker acid and the lower temperature, or the stronger acid and the higher temperature, have been employed. T. S. WHEELER.

Waterproofing textile and other materials. C. J. MORETON (U.S.P. 1,625,672, 19.4.27. Appl., 22.1.26. Conv., 24.12.25).—See E.P. 262,605; B., 1927, 165.

Manufacture of a cellulose product having the appearance of wool. A. Pellerin (E.P. 263,727, 12.2.26. Conv., 30.12.25).

Apparatus for boiling or otherwise treating cork and like materials. W. BRIDGES (E.P. 267,652, 11.1.26).

Means for obtaining fibrous materials from the stalks of hemp and like plants. F. B. DEHN. From FABRICORD INC. TRUSTEE (E.P. 267,763, 31.5.26).

[Spinning head] for spinning artificial silk. A. E. O'DELL. From BERLIN-KARLSRUHER INDUSTRIE-WERKE, A.-G. (E.P. 267,727, 12.4.26).

[Paper-]pulp forming machine. C. J. KEENAN, E. P. KENNEDY, and G. H. KIRSCH, ASSIS. to ALASKA PULP & PAPER Co. (U.S.P. 1,621,671, 22.3.27. Appl., 9.2.26).

Treatment of glass (E.P. 267,428).-See VIII.

VI.-BLEACHING; DYEING; PRINTING; FINISHING.

Progress in the application of vat dyes. F. M. Rowe and C. P. BEAN (J. Soc. Dyers Col., 1927, 43, 99-105).- A review of the application of vat dyes and an investigation of the properties and application under varying conditions of development on cotton and wool of Soledon Jade Green, Soledon Brilliant Purple RR, and Soledon Yellow G. In dyeing cotton and wool from a Glauber's salt bath more level dyeings are obtained, particularly with wool, by entering the material into the cold bath instead of a warm bath as recommended by the manufacturers. Soledon Yellow G gave the best results when the material, previously boiled out in 2% sodium carbonate solution and rinsed lightly, was entered into the cold dye-bath, which was heated quickly to 50°, then during $\frac{1}{2}$ hr. at 70°, and kept at that temperature for $\frac{1}{2}$ hr. The presence of the trace of alkali prevented any decomposition in the dye-bath, but diminished the rate of exhaustion. The behaviour of cotton and wool dyed with these dyes on development with mineral acids, nitrous acid, acid persulphate, acid ferric chloride, and acid dichromate is described.

E. H. SHARPLES.

Cloth dyeing for rubber-proofing. H. L. HOCKNEY and C. W. BANCROFT (J. Soc. Dyers Col., 1927, 43, 105-110, and Trans. Inst. Rubber Ind., 1927, 2, 435-445).-The precautions necessary in the dyeing of cloth for rubber-proofing are discussed. The pronounced tendering effect of traces of mineral acidity in materials to be hot cured and of oxalic acid in the steam cure is emphasised. The presence of minute amounts of metals in the cloth accelerates the degradation by oxidation of the vulcanised rubber, the most common and deleterious being copper. Rubber cured by the cold process is most susceptible to traces of copper, and it is considered that cloth containing 0.01% or more of copper is useless for proofing, and thus those dyeing processes directly employing copper salts and dyeing or dye-making plant containing copper fittings are to be avoided. Manganese is not as dangerous as copper, but it is not advisable to use the permanganate process for bleaching. The presence of grease or oil, especially in presence of a trace of copper, has a very deleterious effect on the physical properties of the rubber, and they should not be present in amounts more than 2%. The effect of the curing processes on the dyes is discussed, and it is considered that it is almost imperative to submit sample dyeings to the actual conditions of proofing. E. H. SHARPLES.

Applications of vat colours in printing. L. SMITH (J. Soc. Dyers Col., 1927, 43, 110-114).—A

description of the methods employed and the practical difficulties encountered in printing with vat dyes.

E. H. SHARPLES.

PATENTS.

Production of fast-coloured discharges on fast dyeings. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 246,183, 19.1.26. Conv., 19.1.25).—Two vat dyes are used, one of which is not fast to formaldehydesulphoxylate in the presence of Leucotrope W, the other being fast to these reagents. The material is dyed with the dischargeable vat dye and then printed with a paste containing the fast vat dye, together with Leucotrope W, formaldehydesulphoxylate, anthraquinone, etc. Suitable pairs of dyes are Indanthrene Orange RRT printed on Indanthrene Olive R; Indanthrene Blue 3G and Indanthrene Yellow G on Indanthrene Red-violet RH; Indanthrene Violet BN on Indanthrene Brown G; Indanthrene Brilliant Blue R on Helindone Fast Scarlet B.

C. HOLLINS.

Dyeing and printing cellulose esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 267,695, 25.2.26).—Sulphamic acids or sulphamates, especially the N-sulphonic acids derived from aminoanthraquinones, dye cellulose esters; the dyeings may further be developed with diazo compounds, or may be diazotised and coupled with suitable components. Examples are the sulphamic acids derived from 1:4-diaminoanthraquinone (reddish-violet), 1:5-diaminoanthrarufin (reddish-blue), 1:4-aminomethoxyanthraquinone (bluish-red), 4-aminobenzeneazo- β -naphthol (reddish-orange), 2:4-dinitro-4'-aminodiphenylamine (golden-yellow).

C. HOLLINS.

Protection of animal fibres in mordant dyeing. I. G. FARBENIND. A.-G., Assees. of K. DAIMLER, G. BALLE, and F. JUST (G.P. 435,899, 21.12.23).—Resinous sulphonic acids obtained from condensation products of aromatic or hydroaromatic hydrocarbons with sulphur chloride, alkyl, aryl, or aralkyl chlorides, alcohols, etc. are added to the mordanting and dyeing baths. An example is sulphonated benzylnaphthalene resin. C. HOLLINS.

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

Manufacture of sulphuric acid. The Tenteleff P. P. BUDNIKOV (Chem.-Ztg., 1927, 51, process. 209-210, 230-232).-The plant and process differ in various details from the Badische process. The burnergas after dust deposition is passed downwards through a lead cooling tower surrounded by an iron water jacket with cross connexions passing through the tower. The gas leaves at the bottom at 90-120°, and some dilute and impure sulphuric acid is condensed and drawn off. Coke or quartz filters are now not used ; the gas is passed through a washer in the shape of a heavy lead bell with water jacket, in which the wash water is changed once daily. It then enters the washing tower, which consists of five superimposed lead cylinders fitted with perforated bells. Contact is further promoted by perforated plates in each section, and the water is changed either intermittently or continuously. In some cases alkali is added to the water in both types of washer to assist

in removing chlorine. The gas is then dried in four coke towers of lead, the last three of which are fed with sulphuric acid of increasing concentration. The moisture content of the dried gas, determined by means of phosphorus pentoxide, should not exceed 0.01%. The dried gas enters a cigar-shaped vessel which serves as a reservoir for the compressors and also for the deposition of entrained acid spray. After passing the compressors the gases are freed from oil in two lead-lined iron vessels charged, respectively, with coke and asbestos, and are preheated at 220-240°. The converter consists of a wide upper part containing the principal contact mass of platinised asbestos, and a narrower lower part having 25 perforated plates with further contact masses between them. Conversion averages 96%. Three absorbers in series are used for an output of 10 tons of sulphur trioxide per 24 hrs. These are enamelled cast-iron vessels with four inlet pipes, fed continuously with acid and cooled with water. The last absorber produces monohydrate. The exit gases contain 0.01% SO3. Alternative forms of some of the apparatus are described and dimensions given. C. IRWIN.

Active forms of silicic acid (silica gel) and their adsorptive powers. O. RUFF and P. MAUTNER (Z. angew. Chem., 1927, 40, 428-434).-Largely a critical review of the literature. It is suggested that the adsorptive power of a solid depends on three effects : (a) an unspecific, purely surface effect, analogous to the adsorption of gases, (b) a specific adsorption of substances of molecular dispersity, (c) adsorption of colloids, which for very small particles is specific, but for large particles is a kind of filter action. Silica gel adsorbs appreciable quantities of acetic acid from aqueous solution, and also smaller quantities of aniline. In respect of the adsorption of acetic acid from solution in petrol, kieselguhr dried at 350° is superior to ordinary ground quartz or powdered vitrified quartz, but is not so good as silica gel. R. CUTHILL.

Causes of discoloration of ammonium sulphate produced at coking plants. A. WEINDEL (Brennstoff-Chem., 1927, 8, 104-107).-The grey colour of " neutral" ammonium sulphate is primarily due to iron sulphide, and to a less extent to sulphides of lead and copper. Arsenic sulphide, being yellow, may be disregarded in this respect. The discolouring effect of tarry substances present in the crude ammonia liquor is negligible. Sulphuric acid made by the contact process is preferable to chamber acid, as it contains less metallic impurities, and the traces of nitrogen oxides in chamber acid increase its corrosive action. Acid sulphate may be neutralised either before or after centrifuging, or in the centrifuge itself. In no case is complete neutralisation attained, the "neutral" salt containing both free acid and free alkali. Neutralisation with ammonia liquor in the centrifuge leads to an undue amount of copper in the mother-liquor, due to corrosion of the copper basket and gauze. The two methods recommended are: neutralisation of the hot salt and mother-liquor, before centrifuging, with gaseous ammonia free from hydrogen sulphide, or the treatment of the centrifuged acid salt with ammonia gas or solution free from hydrogen sulphide. It is for many reasons advantageous to remove the tarry matter from the crude ammonia liquor before

it reaches the still, and this may be done by filtering it through a crushed hydrophobic substance, such as coal dust. W. T. K. BRAUNHOLTZ.

Volumetric determination of magnesium in magnesium chloride solutions. J. E. W. RHODES (J.S.C.I., 1927, 46, 159–160 T).—2.5 c.c. of a solution containing 30—35% of magnesium chloride, or an equivalent amount of a weaker solution, are run into a weighed 100 c.c. flask. The flask is again weighed and filled to the mark with 0.25N-baryta solution, shaken, and kept for 4 hrs. 25 c.c. of the supernatant liquor are then pipetted off and titrated with 0.1Nhydrochloric acid in presence of phenolphthalein. The first disappearance of colour is the correct end-point. The percentage of magnesium chloride found is usually within 0.15 of that found by gravimetric analysis.

Stability of titanous sulphate solutions in air. H. WILKINSON and A. G. TYLER (J. Soc. Dyers Col., 1927, 43, 114-115).-A titanous sulphate solution having a fair degree of stability is prepared as follows. 1500 c.c. of 4N-sulphuric acid are run into a 2-litre stock bottle, and zinc amalgam is distributed evenly over the bottom. A few pieces of granulated zinc are added to remove the dissolved oxygen, and a layer of solvent naphtha is run over the surface of the liquid. 30 c.c. of commercial titanous sulphate solution are made up to 200 c.c. with 4N-sulphuric acid in a conical flask, and a quantity of zinc amalgam is added. Carbon dioxide is passed through the liquid, which is boiled until a clear violet solution is obtained. It is then cooled and decanted into the stock bottle. With simple standardisation with potassium permanganate from time to time such a solution is very convenient for technical purposes, although for accurate work the solution must be kept under an atmosphere of hydrogen, as recommended by Knecht and Hibbert. E. H. SHARPLES.

Ionic exchange of zeolitic silicates with hydrolysable salts. I. Experiments with permutite. H. KAPPEN and F. RUNG (Z. Pflanz. Düng., 1927, A8, 345-373).-The effect upon the exchange acidity shown by a pure sodium and calcium permutite of treatment with various salts has been studied in detail. The results of experiments with ferric chloride, chromic sulphate, and aluminium chloride indicate that it is impossible to obtain a direct exchange of ferric, chromic, and aluminium ions with the bases of the permutite. It is considered that the physical condition of the permutite accounts for this. On the other hand, treatment with ferrous, cupric, and zinc salts gives permutites in which exchangeable ferrous, cupric, and zinc ions are present. The formation of aluminium permutite by treatment with acids was also studied. The discussion of the bearing of these results on problems of soil acidity is deferred until the results of experiments with natural crystalline silicates are available.

C. T. GIMINGHAM.

Carbon dioxide in carbonates. CALLAN,—See IV. Toxic action of selenium and tellurium compounds. Stover and HOPKINS,—See XVI.

PATENTS.

Manufacture of nitric acid. C. TONIOLO (E.P. 267,721, 27.3.26).—Instead of absorbing oxides of

nitrogen from the combustion of ammonia or other source in refrigerated dilute acid circulating in towers in counter-current to the gases (cf. E.P. 121,635; B., 1919, 104), the acid and the gases both enter the towers through the top, the hot gases thereby undergoing sudden cooling by the acid, which is cooled to near zero before entry. The gases instead of the acid may be cooled to considerably below zero, the acid circulating in the absorption towers at a higher temperature, but not so high as to cause dissociation. The refrigeration of the gases may be brought about in towers through which strongly cooled nitric acid circulates in the same direction as the gases. W. G. CAREY.

Production of hydrochloric acid and magnesia. VEREIN FÜR CHEM. & MET. PROD. (G.P. 436,241, 11.3.25). —Magnesium chloride or oxychloride is treated with steam in a revolving tube furnace heated both internally and externally. L. A. Coles.

Evolution of hydrocyanic acid [for fumigation]. H. LEHRECKE, ASST. to ROESSLER & HASSLACHER CHEMICAL CO. (U.S.P. 1,620,365, 8.3.27. Appl., 20,10.25. Conv., 6.11.24).—Anhydrous zinc chloride absorbs 3 mols. of anhydrous hydrogen cyanide, which is liberated on exposure of the product to the action of moisture. For fumigation it is of advantage to add an excess of zinc chloride or other substance with a large heat of hydration. T. S. WHEELER.

Production of colloidal silicic acid solutions. M. PRÄTORIUS and K. WOLF (F.P. 612,486, 8,3.26. Conv., 18.3.25).—Sodium silicate solution is electrolysed in a cell containing at least three diaphragms constructed of non-reacting material, such as wool, asbestos, or alumina, between the two poles. L. A. COLES.

Recovery of titanic acid, iron, and magnesia from titaniferous ores. F. E. BACHMAN (U.S.P. 1,618,795, 22.2.27. Appl., 18.3.21).- A mineral containing titanium, e.g., ilmenite, intimately mixed with carbon and sodium carbonate is heated in a rotary kiln, and the clinker formed is introduced with carbon into a blast furnace, when metallic iron and a slag are obtained. The slag is heated with water under pressure to dissolve the sodium compounds present, and leave hydrated titanic oxide and magnesium compounds, which are heated with dilute sulphuric acid under pressure at 150°. The residue from this treatment is dissolved in sulphuric acid, treated with titanous chloride to reduce the trace of iron present, and reprecipitated by heating under pressure. Finally, the sulphate present is removed with barium hydroxide, leaving titanic acid. T. S. WHEELER.

Apparatus for the catalytic combustion of mixtures of ammonia and oxygen. I. W. CEDER-BERG (E.P. 244,134, 8.12.25. Cf. E.P. 236,145; B., 1925, 713).—The walls of the apparatus are two hexagonal chrome-nickel steel plates connected to form a gas-tight joint and having jackets for cooling water. The intermediate space is extremely narrow, especially at the centre, so that a gas layer is formed sufficiently thin to prevent explosions, and this space contains a catalyst of platinum gauze or sheet. W. G. CAREY.

Synthesis of ammonia. Soc. CHIM. DE LA GRANDE PAROISSE, AZOTE & PROD. CHIM. (F.P. 611,417, 2.6.25).- The supply of the mixed gases to each unit in apparatus comprising a number of catalyst units in parallel is controlled by regulating the pressure before or behind each unit, or by the provision of a pump having a cylinder for supplying each unit. L. A. COLES.

Manufacture of sodium peroxide. ROESSLER & HASSLACHER CHEMICAL CO. (E.P. 265,124, 7.6.26. Conv., 29.1.26. Cf. E.P. 264,724; B., 1927, 218).—Molten sodium, highly diluted with powdered sodium monoxide, is oxidised to sodium monoxide in a dry atmosphere poorer than air in oxygen preferably below 200° and not above 250°, and the resulting monoxide is oxidised to sodium peroxide in an atmosphere richer than air in oxygen at 200—350°. W. G. CAREY.

Recovery of sulphur [from charcoal]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 267,246, 17.12.25).—Sulphur is recovered from masses containing it, such as active charcoal from the catalytic oxidation of hydrogen sulphide, by passing superheated steam through them in a downward direction at a speed of at least 1 m./sec., and collecting the molten sulphur. The steam is washed with water to free it from sulphur vapour or mist, and is superheated and used again.

W. G. CAREY.

Eliminating water from liquid sulphur dioxide. ALLGEM. GES. FÜR CHEM. IND. M.B.H. (E.P. 261,732, 6.11.26. Conv., 19.11.25).—Water taken up by liquid sulphur dioxide in the refining of hydrocarbons or oils and fats is eliminated by compression and cooling, and at the first stage of the condensation the liquid, which contains all the water, is tapped from a preliminary condenser, the sulphur dioxide therein being re-introduced to the system after distillation to separate the water. W. G. CAREY.

Manufacture of nitric acid. C. TONIOLO (U.S.P. 1,623,606, 5.4.27. Appl., 9.9.26).—See E.P. 267,721; preceding.

Production of concentrated solutions of alkali cyanides. DEUTSCHE GOLD- U. SILBER-SCHEIDEAN-STALT, VORM. ROESSLER (E.P. 245,152, 23.12.25. Conv., 23.12.24).—See U.S.P. 1,615,208; B., 1927, 218.

Manufacture of siliceous alkaline-earth compounds. A. J. H. HADDAN. From CELITE Co. (E.P. 268,011, 18.12.25).—See U.S.P. 1,574,363; B., 1926, 487.

Converting hafnium and zirconium phosphates. J. H. DE BOER, Assr. to N. V. PHILIPS' GLOEILAMPEN-FABR. (U.S.P. 1,624,162, 12.4.27. Appl., 19.6.25. Conv., 13.8.24).—See E.P. 238,543; B., 1925, 881.

Sulphur compounds of ammonia from gas (E.P. 268,024).—See II.

Utilisation of phosphate residues (E.P. 252,367).— See IX.

Zirconium and hafnium (U.S.P. 1,618,960).— See X.

VIII.-GLASS; CERAMICS.

U.S. Government master specification for plastic fire-clay refractories. (U.S. Bur. Standards, Circ. No. 297, Oct. 20, 1926, 5 pp.).—The fire clay, specially adapted for the construction of small furnaces, must contain not more than 65% of total silica calculated on

the dry weight, and the softening point shall not be less than 1680°; the water content as delivered shall not exceed 15% calculated on the plastic weight, nor the total linear dry-and-burned shrinkage 4% of its plastic length. B. W. CLARKE.

U.S. Government master specification for fire clay. (U.S. Bur. Standards, Circ. No. 298, Oct. 20, 1926, 6 pp.).—Fire clay is divided into two classes, fine and commercial, according to its fineness. Of the former, not more than 4% is retained on a U.S. standard sieve No. 20, and of the latter, 10%. It must have a softening point not more than 60° lower than that of the brick with which it is to be used. B. W. CLARKE.

U.S. Government master specification for fireclay brick. (U.S. Bur. Standards, Circ. No. 299, Oct. 20, 1926, 7 pp.).-Fire-clay brick is divided into the following classes : SH 75, for severe conditions of boiler practice, with a softening point of not less than 1680°; H 75, for general boiler practice; and H 57, for use where resistance to spalling is unimportant, with ability to withstand 15, 12, and 5 quenchings, respectively, without failure. Other classes are :-- M 73, for use at moderate temperatures, softening point not less than 1640°; also H 25 and M 7, for silicious brick with deformation under load at high temperatures of less than 3% and 4% respectively. The resistance to spalling, slagging, or high temperatures varies for each class, and is determined in a "simulated service test," in which the brick is built into one wall of an experimental oilfired furnace of special design, for comparison with standard brick, observation being taken by radiation pyrometers of the inner- and outer-face temperatures of the furnace and a spalling test conducted at the end of the run. B. W. CLARKE.

PATENTS.

Ray-filter glass. I. FRANK (U.S.P. 1,615,448, 25.1.27. Appl., 6.7.25).—A glass which is practically colourless in commercial thickness and gives normal transmission in the visible region, but which absorbs the greater portion of the ultra-violet and infra-red rays, is obtained by addition of suitable proportions of ferric oxide and manganese dioxide to the batch. For lenses approximately 1.5 mm. thick, a proportion of 0.00256 pt. by wt. of the batch of the mixture of oxides in the ratio of manganese dioxide to ferric oxide of 3:2 is used. A. COUSEN.

Readily fusible glasses and enamels. GENERAL ELECTRIC CO., Assees. of PATENT-TREUHAND-GES. FÜR ELEKTRISCHE GLÜHLAMPEN M.B.H. (E.P. 267,815, 25.9.26. Conv., 11.5.26).—Glasses with a low coefficient of expansion and without yellow coloration, for coating glass articles by fusion, have the following limiting composition, 60-75% of lead oxide, 10-25% of zinc oxide, and 15-30% of boric oxide, the most suitable formula being 65% of lead oxide, 15% of zinc oxide, and 20% of boric oxide. They may be made opaque by adding up to 2% of arsenic and up to 2% of an ammonium compound. W. G. CAREY.

Treatment of glass plates and the like to prevent moisture affecting their transparency. O. DEMARET (E.P. 267,428, 1.11.26. Conv., 27.4.26).—A transparent sheet or film of coagulated or reverted cellulose, previously treated with 50% caustic potash solution at 30° , washed, and dried, is secured by transparent glue to the surface of the glass. B. W. CLARKE.

Moulding silica. BRITISH THOMSON-HOUSTON CO., LTD., Assees. of L. B. MILLER (E.P. 262,110, 23.11.26. Conv., 24.11.25).—Silica is rendered plastic by heating at 1700°, and a moulding member of carbonaceous material at approximately the same temperature is introduced into the mass. To prevent chemical action between the silica and the mould these are removed and cooled rapidly in the air, thereby facilitating the removal of the mould from the solidified silica.

B. W. CLARKE.

Treatment of silica articles. BRITISH THOMSON-HOUSTON CO., LTD., Assees. of E. HERZOG (E.P. 264,863, 21.1.27. Conv., 21.1.26).—Articles of vitreous silica, having a rough surface which is to be glazed, are mounted so that they can be rotated and heated in an enclosed space at about 1000°, which is well below the softening temperature, but sufficiently high to prevent cracking during the subsequent process. The articles are then subjected to a fusing temperature from the flames of suitably arranged burners, the region of fusing heat being continuously changed by rotating the article and traversing the burners until the whole surface is glazed. B. W. CLARKE.

Kiln for glass melting and like purposes. G. E. CRAWFORD, ASST. to AMERICAN DRESSLER TUNNEL KILNS INC. (U.S.P. 1,623,779, 5.4.27. Appl., 19.10.23).— A tunnel kiln has an elongated kiln chamber and a movable hearth separated from a portion of the kiln structure by a clearance space at the side of the hearth. A conveyor beneath removes material passing downwards through the clearance space. W. G. CAREY.

Manufacture of raw plate glass. M. BICHEROUX (E.P. 263,847, 29.12.26. Conv., 30.12.25. Addn. to E.P. 182,551).

Stretching glass. Soc. Anon. des Manuf. des Glaces et Prod. Chim. de St. Gobain, Chauny, et Cirey (E.P. 257,590, 12.8.26. Conv., 27.8.25.)

[Fritted-glass] filter (U.S.P. 1,620,815).—See I.

IX.—BUILDING MATERIALS.

Aqueous vapour pressure of magnesium oxychloride cement, and the state of the water contained in the latter. T. MAEDA (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1926, 5, 133-140).-The aqueous vapour pressure isotherms of two magnesium oxychloride cements are constructed for 25°, with a view to the elucidation of the state of the water contained in them. This vapour pressure is due, in one specimen of cement containing 33% of water, mainly to the water of crystallisation of the double compound 3MgO,MgCl, 12H,O, whilst in the other, containing 55% of water, it is ascribable chiefly to magnesium chloride solution held by capillarity within the pores of the cement. Cements in which the water is preponderatingly of the former type are brittle, and sweating of the surface of the cement in a moist atmosphere is more marked, this being due either to the lower vapour pressure of its water, or to its more compact surface preventing drops of water from soaking into the interior of the mass.

R. TRUSZKOWSKI.

Hardening of magnesium oxychloride cement, and the function of free water, water of crystallisation, and adsorbed water. T. MAEDA (Sci. Papers . Inst. Phys. Chem. Res., Tokyo, 1926, 141-154).-Measurements of the hardness of magnesium oxychloride cements, using a modified Vicat apparatus, indicate that hardening is a continuation of the setting process, and is due to the further formation of the double salt 3MgO, MgCl₂, 12H₂O. Elimination of water and the absorption of carbon dioxide play only a secondary rôle in this process. A high proportion of solid constituents without sufficient adsorbed water leads to the formation of brittle cements, as does also the use of old specimens of magnesium oxide. Free water is necessary for the plasticity of cement, and water of crystallisation and adsorbed water influence the setting and hardening processes, the latter form of water also serving to reduce the brittleness of the cement. Soft cements are characterised by an unduly high free-water content after hardening. R. TRUSZKOWSKI.

Influence of humidity changes upon the composition of building materials. J. W. MCBAIN and J. FERGUSON (J. Physical Chem., 1927, 31, 564-590).-Data on the sorption of water vapour under varying conditions of humidity have been obtained using the sorption balance of McBain and Bakr (A., 1926, 493) in the case of the following materials : powdered clear quartz crystal, fibres of fused silica, kieselguhr, Filter cel ("treated" diatomaceous earth), Millstone grit (Idle Quarry and Darley Dale), Kentish rag, Portland stone, Mona's Park stone, dolomitic limestone (Gold Coast), chalk (Setch, Norfolk), Stoneycombe stone, Buxton limestone, Meldon granite, and the following bricks :- red rubber, red wire-cut, blue wire-cut, blue Stafford, Fletton, Stock and sand-lime. Quartz, granite, the pure limestones, and highly-burned brick show practically no sorption from an atmosphere saturated with water vapour. Portland stone and Monk's Park stone show initially a slight absorption of water vapour which remains unchanged over a wide range of humidity. The building stones absorb 1% or less, varying with humidity changes, and the amorphous silica, such as kieselguhr, sorbs up to 10% of water at high humidity, the amount varying largely with conditions. The sorption and swelling of sandstone is due to the contained amorphous building material. The hysteresis shown by kieselguhr, Filter cel, and Gatton stone has also been studied. Absorption of methylene-blue and magenta by building materials parallels the absorption of water vapour. Heating at 850° destroys the colloidal matter present in sandstones, which then lose the power of sorbing water vapour or dyes. The inter-grain cement of sandstone sorbs the vapour of water, methyl and ethyl alcohols, chloroform, benzene, and octane according to the size of the molecules concerned. The heats of sorption have been calculated for certain cases. The results of the sorption experiments agree with the conception of micellar linkage advanced in connexion with other work (cf. A., 1926, 351), and on

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this basis is advanced the definition that a stone preservative is a substance which in the presence of water is preferentially sorbed by the mineral gel, and remains permanently in combination with it. L. S. THEOBALD.

PATENTS.

Apparatus for agitating slurry etc. J. S. FASTING, ASST. to F. L. SMIDTH & Co. (U.S.P. 1,622,280, 29.3.27. Appl., 5.9.24).—A carrier supported on the walls of a slurry tank, along which it is free to move, is provided with a shaft carrying a series of sprocket wheels which can be rotated, and from which chain loops are suspended within the tank. Weighted air pipes, connected to a supply of compressed air, are supported pivotally from the carrier. B. W. CLARKE.

Manufacture of hydraulic cement and products produced thereby. H. S. SPACKMAN (E.P. 244,756, 10.12.25. Conv., 17.12.24).—A charge, consisting of bauxite and limestone in such proportions that the acidic components are approximately equal to the basic components, is heated in a rotary cement kiln in an oxidising atmosphere, producing a clinker which is ground to form a hydraulic cement containing 11% of iron oxide, together with alumina, lime, and silica.

B. W. CLARKE. Manufacture of cement. L. KERN (U.S.P.1,623,876, 5.4.27. Appl., 23.12.25).—A mixture of natural calcareous and siliceous material, after calcination at a temperature too low to cause sintering, is pulverised, slaked with sufficient water to give a mass capable of being moulded, and, after moulding, is hardened under pressure at steam heat and burnt at 400—600°.

W. G. CAREY.

Production of a porous cementitious article. A. T. SCHENCK (U.S.P. 1,622,396, 29.3.27. Appl., 4.1.26).—Cement in granulated form is mixed with hydrated lime, aluminium, and warm water, and during the liberation of gas the mixture is moulded and rotated during the setting to form substantially uniform voids in the mass. B. W. CLARKE.

Utilising and producing cement from the residue obtained in the reduction of molten crude phosphates. I. G. FARBENIND. A.-G., Assees. of M. PLATSCH (E.P. 252,367, 17.5.26. Conv., 23.5.25).—Cement is obtained by fusing a crude phosphate in an electric furnace with sand and carbon or coke, and, after the phosphorus vapours are expelled, adding calculated amounts of alumina (bauxite or clay) until a homogeneous cement mixture is produced, which is tapped off and ground. W. G. CAREY.

Production of gypsum product. L. M. PARK-HURST, ASST. to FEDERAL GYPSUM PRODUCTS CO. (U.S.P. 1,620,915, 15.3.27. Appl., 6.5.22).—A mortar for making gypsum products is prepared by mixing calcined gypsum with water and carbon dioxide. W. G. CAREY.

Composition capable of being moulded. MICHELIN ET CIE. (E.P. 263,138, 13.12.26. Conv., 19.12.25).—A mixture of 15 pts. of sodium silicate solution and 100 pts. of ground lava is moulded under pressure, dried in air, stoved at 40—50°, and finally baked at 800—900°. The product is resistant to shocks and cold.

B. W. CLARKE.

Bituminous mixtures for use in road construction and the like. UNIVERSAL RUBBER PAVIORS (MANCHESTER 1923), LTD., and A. E. BROWN (E.P. 267,317, 12.9.25).— Residual products such as guttapercha pitch or balata pitch are added to bituminous mixtures other than those described in E.P. 246,186 (cf. B., 1926, 276).

B. W. CLARKE.

Preservation of wood. L. P. CURTIN, ASST. to WESTERN UNION TELEGRAPH Co. (U.S.P. 1,620,152, 8.3.27. Appl., 24.6.25).—Wood is impregnated with an aqueous solution containing copper sulphate, sodium hydrogen sulphite, sodium carbonate, sodium chloride, arsenious oxide, and sodium fluoride. On exposure to air cupric hydrogen orthoarsenite is deposited on the wood. T. S. WHEELER.

Apparatus for impregnating wood. K. BUBLA (E.P. 267,579, 18.11.25).⁻

Manufacture of mastic sheet material. L. MELLERSH-JACKSON. From F. R. MULLER INC. (E.P. 267,414, 1.10.26).

Bituminous composition (U.S.P. 1,620,900).— See II.

Coating wood etc. (E.P. 252,364).—See XIII.

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

Influence of atmosphere and temperature on scaling of steel. I. Scaling by air, water vapour, and carbon dioxide. C. B. MARSON and J. W. COBB. II. Prevention of scaling by hydrogen and carbon monoxide. H. T. ANGUS and J. W. COBB (J.S.C.I., 1927, 46, 61—72 т).—The production of a scale of oxide on iron or steel in re-heating operations may occur even in an atmosphere containing no free oxygen. Laboratory experiments have been made to ascertain the conditions which determine scale production on a boiler-plate steel (0.2% C) at 900° and 1000°. The weight of scale was measured when samples identical in form and size were exposed to different atmospheres for 3 hrs. At 1000° the weights of scale produced in g./cm.² were : in dry air 0.0635, in 20% steam : 80% nitrogen 0.0166, and in 20% carbon dioxide: 80% nitrogen 0.0024. When both carbon dioxide and steam were present the results were intermediate. Adding carbon monoxide to carbon dioxide retarded but did not stop scaling altogether until 58% of carbon monoxide was present. Scaling by steam was arrested only when 50% of hydrogen was present. The addition of hydrogen to carbon dioxide increased the rate of scaling owing to the formation of steam by way of the "water-gas reaction," the maximum scaling occurring with an initial mixture of 26% H_2 : 74% CO₂. Further addition of hydrogen reduced the scaling, which was completely arrested by 57% of added hydrogen. The addition of carbon monoxide to steam reduced the rate of scaling, but did not stop it until 80% of carbon monoxide was present. It required the addition of 30% of methane to stop the scaling of an atmosphere containing 10% of carbon dioxide and 10% of steam. The scale exercised a protective action against decarburisation of the metal which nearly always occurred. The experiments indicate

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that fuels rich in hydrogen will tend to cause rapid scale formation which cannot be stopped by reducing gases unless these are present in large proportions.

Influence of cold work and annealing on magnetic properties of thin wires of invar, iron, and steel. C. BIÉLER-BUTTICAZ (Arch. Sci. Phys. Nat., 1927, 9, 32-51).-In a previous paper (*ibid.*, 1925, 7, 19) it was shown that for invar the internal friction increases suddenly at 340° in hard-drawn wire, and at 250° in annealed wire. These temperatures correspond to the maxima in the torsion moduli. It is now shown that no corresponding displacement occurs in the magnetic transformation point, although the form of the curve is altered by cold work. The magnetic susceptibility falls most rapidly between 200° and 250° both for cold-drawn and annealed wires. The magnetisation curve shows an anomaly at about 150°, which is attributed to the effect of cold work, and can be removed by annealing at 800°. A similar effect was observed in piano wire and in pure iron. A much smaller anomaly, attributed to the nickel transformation point, was observed at 340°. C. J. SMITHELLS.

Effect of prolonged annealing on the phosphide eutectic [in cast iron]. H. PINSL (Stahl u. Eisen, 1927, 47, 537-540).-After annealing at 980° for 48 hrs. a cast-iron rod containing 1.95% C, 2.46% Si, 0.30% Mn, 1.18% P, and 0.026% S, the microstructure of the cross-section showed the presence of three distinct zones, of which the central and outer consisted of practically pure ferrite saturated with phosphorus (0.8-1%), and containing coarse inclusions of graphite. Between these two zones was a ring of phosphide eutectic in globular masses surrounded by pearlite; the phosphorus content of this ring was as high as 1.98% in the middle, and decreased regularly towards the centre and edges of the bar. A sample of the metal taken from near the tuyères after a week's running of a furnace melting an iron containing 3.30% C, 2.20% Si, 0.40% Mn, 1.30% P, and 0.08% S, had the following composition :--0.03% C, 0.005% Si, 2.10% P, 0.12% S, 0.052% Cu, and Mn a trace, showing that the action of the hot air blast is to remove the carbon, silicon, and manganese, in preference to the phosphorus and sulphur. As this metal had been allowed to cool down very slowly with the furnace, its microstructure was entirely free of phosphide eutectic, and consisted of the saturated solid solution of phosphorus in iron containing inclusions of long needles of iron phosphide segregated along the grain boundaries.

A. R. POWELL.

Oxygen in pig iron and cast iron. P. OBERHOFFER and E. PIVOVARSKY (Stahl u. Eisen, 1927, 47, 521—533). —Tables are given showing the variation in the composition, especially the oxygen content, of 20 pig irons from coke-fired furnaces, 14 pig irons from charcoal-fired furnaces, and 30 cast irons made up from many different types of raw material, full details of furnace dimensions, blast pressure and temperature, composition of feed to the furnace, and analyses of the slags produced being included. The mean hydrogen contents of the three varieties of iron examined were $13 \cdot 5$, $19 \cdot 5$, and $15 \cdot 2$ c.c./ 100 g., and the mean oxygen contents $0 \cdot 022\%$, $0 \cdot 023\%$, and $0 \cdot 038\%$ respectively. Practically the whole of the oxygen content in the second case was present as silica; in the other cases, however, there was no relation between the oxygen and silicon contents of the metal. The structure and properties of cast iron are seriously affected by the presence of more than 0.045% O. A high furnace temperature tends to reduce the proportion of included oxides in the castings, and this may be most satisfactorily obtained by increasing the quantity of air supplied; at the same time, this increases the rate of melting of metal, and consequently results in a greater output from the furnace. A. R. POWELL.

Specific resistivity of copper-refining electrolytes and method of calculation. S. SKOWRONSKI and E. A. REINOSO (Trans. Amer. Electrochem. Soc., 1927, 51, 113-131).-Sulphuric acid concentration is the main factor in obtaining electrolytes of low ohmic resistance, and the specific resistivity of sulphuric acid at different concentrations and at various temperatures has been determined. All resistivity measurements carried out at different temperatures are compared with measurements made at 55° (the normal operating temperature) as 100%, the comparison being called the "percentage resistivity." At constant temperature the specific resistivity of a solution of sulphuric acid containing 150 g./litre is taken as 100%, and any change in resistivity due to variation in the composition of the electrolyte is referred back to this standard figure. By using percentage resistivity data it is not only possible to calculate the resistivity from the chemical analysis of the electrolyte used, but also to make up any particular solution to any predetermined resistivity. The percentage resistivity of sulphuric acid decreases as the concentration increases, but is independent of the temperature within a wide range. Copper, nickel, and iron as sulphates increase the specific resistivity to a marked degree, whilst the effect of arsenic is slight; the increase is directly proportional to the amounts added. Glue has no effect on the resistivity of sulphuric acid solutions. Formulæ have been derived by means of which the resistivity of a tank-house electrolyte may be calculated from its chemical analysis.

M. E. NOTTAGE.

Properties of electrolytic copper sheets. Sheets deposited on rotating cathodes. S. SONODA (Trans. Amer. Electrochem. Soc., 1927, 51, 191-205). -Electrolytic sheets produced on rotating cathodes are inferior in their physical properties to standard sheets rolled from cast ingots. This inferiority is connected with the microstructure; in ordinary annealed sheets the fine crystals are irregularly oriented, but in electrolytic sheets the crystals are developed perpendicular to the surface; hence the tensile strength is low in the direction perpendicular to the surfaces of the crystals. The ductility and malleability of electrolytic sheets is also low, and the sheets are difficult to work. The drawing, stamping, and compressive properties of the sheets were measured by the Erichsen test. Measurements were also made of the tensile strength and hardness. It was found that by rolling and annealing the electrolytic sheets, both surfaces could be made smooth, and the mechanical properties and microstructure developed were very similar to those of ordinary copper sheets; annealing alone did not produce this effect. The direction of rolling had little effect, but heavy rolling produced marked physical changes. The best annealing temperature for the rolled sheets was $400-600^\circ$; annealing at temperatures higher than 800° gave rise to rapid grain growth and a decrease in the percentage elongation. Preliminary annealing before rolling is not necessary when the reduction is less than 50%. Electrolytic sheets rolled directly without annealing showed a tensile strength and percentage elongation greater than those for ordinary rolled copper sheets.

M. E. NOTTAGE.

Gaseous reduction of tin concentrates. C. G. FINK and C. L. MANTELL (Trans. Amer. Electrochem. Soc., 1927, 51, 175-189):-The low-temperature reduction of stannic oxide by purified hydrogen is complete at 250°, the action being made irreversible by using an excess of hydrogen which sweeps out the water vapour formed; with stannic oxide in excess the action ceases as soon as the concentration of water vapour has reached N%, where N varies inversely as the temperature. The reduction takes place in one step or two, according as the temperature employed is above or below the thermal decomposition point of the lower oxide. The velocity of reduction is greatest at those points first affected; it is also dependent upon the velocity of diffusion of the water vapour formed. The reduced metal forms a fine grey powder with very little sintering even when the reduction takes place above the m.p. of tin. The temperature-time of reduction curves are logarithmic, taking the form $T_{k}^{n} M = C$, where T_{k} is the absolute temperature, M, the time in min., and n and C are constants; extrapolation of the curve shows that the reaction would be instantaneous at 1000°. The rate of reduction of ferric oxide by hydrogen has also been studied, the shape of the curve being the same as for tin, but the constants smaller; hence it is inferred that the reduction of ferric oxide by hydrogen takes place more easily than that of stannic oxide. The nonappearance of metallic iron in the reduced masses is probably due to the re-oxidation of the finely-divided metal. Temperature-time of reduction curves for Bolivian concentrates are of the same form as those for stannic oxide. The process of gaseous reduction depends for its success upon the absence of all slagging phenomena until reduction is complete, as even incipient fusion prevents the intimate contact of ore and gas. 80% of the tin metal content may be liquated from the non-fluid ore in this manner, the remaining 20% being almost completely recovered by fusing the residues at 1000-1050°, when the gangue forms a liquid slag from which metallic tin settles out. The economics of the hydrogen reduction of tin ores is also discussed. M. E. NOTTAGE.

Diffusion law as applied to diffusion in solid solution. J. S. DUNN (J.S.C.I., 1927, 46, 109 T).—A criticism of Tammann and Bredemeier (cf. A., 1925, ii, 541), who claim that the law of growth of films of reaction products upon metals is correlated with the mechanism of conduction of electricity in these films. Diffusion of gold in lead and of zinc in copper which should vary logarithmically with time upon their theory is shown to follow a parabolic law.

PATENTS.

Manufacture of steel. F. N. SPELLER (U.S.P. 1,617,726, 15.2.27. Appl., 16.8.26).—In the manufacture of steel, the ladle is preheated by pouring a quantity of superheated steel into it, prior to the addition of the bulk of the steel, which can then be held sufficiently long in the ladle to permit of separation of the non-metallic inclusion. T. S. WHEELER.

Plant for treating molten iron. E. C. R. MARKS. FROM NEW PROCESS MULTI-CASTINGS CO. (E.P. 267,590, 15.12.25).—A blast furnace is used in conjunction with a number of electric furnaces mounted on a turn-table and arranged successively to receive molten metal from the blast furnace and to condition the metal as to composition and temperature. J. S. G. THOMAS.

Annealing and hardening steel wires and bands. F. KÖSTER (E.P. 254,278, 14.5.26. Conv., 23.6.25).— The wires etc., after passing through the annealing furnace, are drawn first through a pipe contained in a lead-bath and then through an oil-bath, air being excluded. M. E. NOTTAGE.

Heat treatment of iron or steel. AUTOMATIC AND ELECTRIC FURNACES, LTD., and L. W. WILD (E.P. 267,253, 30.12.25).—In an electric furnace heated by direct current, magnetisation of the charge may be prevented by connecting a neutralising winding on the outside of the furnace in series with the heating winding, whereby a magnetising force equal and opposite to that generated by the heating winding is produced. Thus, magnetising coils carrying alternating currents, and detecting coils can be added to the furnace, and the change in the magnetic state of the charge from ferroto para-magnetic can be detected. M. E. NOTTAGE.

Case-hardening of steel articles. RHEINISCHE METALLWAAREN- U. MASCHINENFABR. (E.P. 242,978 and 244,431, [A] 5.11.25, [B] 6.11.25. Conv., [A], 17.11.24, [B], 13.12.24.)—(A) After case-hardening by known methods, including annealing or subsequent heat-treatment, the article is subjected to a controlled heating agent, e.g., a molten bath of lead, for such time that the carburised layer only is raised to the hardening temperature. The depth in the carburised layer to which "glass hardness " is to be produced is determined by trial. (B) When "glass hardness" is desired over the whole surface of steel articles the time of heating is. maintained so that the heating to hardening temperature penetrates a short distance into the non-carburised core, whereby, on quenching, a normally hardened, uncarburised zone is formed between the carburised surface layer and the core. . C. A. KING.

Desulphurising fine iron pyrites. METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 254,691, 7.6.26. Conv., 4.7.25).—The pyrites is deprived of part of its sulphur in a hearth or drum roasting furnace, and the remaining sulphur is driven out by aspirating or blowing air through the material in a Dwight-Lloyd blast roasting furnace, agglomerating the burnt pyrites if desired. The cooling between the operations may be with complete or partial exclusion of oxygen, whereby iron sulphate is formed. W. G. CAREY.

Metallurgy of tin. E. A. C. SMITH, Assr. to GUGGEN-HEIM BROS. (U.S.P. 1,620,580, 8.3.27. Appl., 27.6.25).— A partially reduced tin ore is leached with a solution of stannic chloride, ferrous chloride, and either hydrochloric or sulphuric acid, and the metallic tin, which is dissolved with formation of stannous chloride, is recovered by electrolysis, with regeneration of the leaching solution. A portion of this solution is electrolytically oxidised in a cell fitted with a diaphragm to form ferric chloride, and this solution is used to leach the tailings from the first leach, from which it extracts stannous sulphide, antimony, and bismuth. The latter metals are precipitated by treatment with metallic tin, and the solution of stannic chloride and ferrous chloride remaining is returned to the first leaching vessel.

T. S. WHEELER. **Preparation of [low-grade lead] ores for flotation.** H. H. HENDRICKSON, Assr. to GRAND CENTRAL MINING Co. (U.S.P. 1,620,761, 15.3.27. Appl., 9.9.26).—Lowgrade lead ores are ground and mixed with sodium sulphide and water at about 70°. The product when submitted to flotation gives a 60% recovery of lead.

T. S. WHEELER.

Production of metallic [zinc] oxides. G. B. FILMER (U.S.P. 1,620,880, 15.3.27. Appl., 12.12.24).— Zinc is added to molten slag, which is heated in a furnace by passing an electric current through it. A current of air is mixed with the zinc vapour as it leaves the furnace. The slag removes any impurities in the zinc.

T. S. WHEELER.

Separation of zirconium and hafnium. D. COSTER and G. von Hevesy, Assrs. to N. V. PHILIPS' GLOEI-LAMPENFABR. (U.S.P. 1,618,960, 22.2.27. Appl., 13.3.24. Conv., 26.4.23).—A mineral containing zirconium and hafnium is fused with potassium hydrogen fluoride, and the mixture of potassium zirconium fluoride and potassium hafnium fluoride obtained is dissolved in a boiling aqueous solution containing 10% of hydrogen fluoride and 5% of potassium fluoride. The precipitate which separates on cooling contains less hafnium than the original material, and by repetition of the process a practically complete separation can be attained. To prepare metallic hafnium, a solution of the double fluoride is treated with an excess of a base, and the hydroxide precipitated is converted into the metal by any reaction suitable for the preparation of zirconium from its hydroxide. T. S. WHEELER.

Metallurgical furnace. F. A. FAHRENWALD (U.S.P. 1,623,469, 5.4.27. Appl., 12.10.25).—A number of one-piece hollow rolls are set horizontally and parallel in the furnace chamber, and have co-axial, solid shafts at each end projecting through the furnace walls. Provision is made for rotating all the rolls in the same direction, at the same speed. W. G. CAREY.

Heating metals by electricity. D. F. CAMPBELL (E.P. 267,704, 3.3.26).—Materials to be heated, *e.g.*, wires or bars, are drawn through perforations in a block of magnetic material magnetised by a coil energised by an alternating electric current, the block being heated at about its temperature of recalescence by effects due to hysteresis and eddy currents. J. S. G. THOMAS.

Electrodeposition of chromium. C. HAMBUECHEN, Assr. to ELECTRO METALLURGICAL CO. (Re-issue 16,598, 19.4.27, of U.S.P. 1,544,451, 30.6.25).—See B., 1925, 726. Refining brass and bronze secondary metals and their residues. T. LEWIN (E.P. 269,253, 15.1.26). —See U.S.P. 1,574,043; B., 1926, 369.

Extrusion of metals [e.g., magnesium propellers]. E. MITCHELL (E.P. 267,412, 23.9.26).

Method and apparatus for making metal wool from wire. A. K. CROAD. From BRILLS MANUF. Co., INC. (E.P. 268,244, 2.11.26).

Ore reduction (E.P. 266,402).—See I.

XI.-ELECTROTECHNICS.

PATENTS.

Electric furnace. A. D. KEENE, Assr. to WESTING-HOUSE ELECTRIC AND MANUF. Co. (U.S.P. 1,622,650—1, 29.3.27. Appl., [A, B] 24.4.25).—(A) A refractory resistorsupporting member has a portion projecting into the furnace, and is provided with a groove and locking means interfitting with the groove for holding it in the refractory wall of the furnace. (B) The resistor-supporting member is held in position in the furnace wall by means which are prevented from moving longitudinally. J. S. G. THOMAS.

Electric melting pot. F. SCHUSTER (U.S.P. 1,623,209, 5.4.27. Appl., 16.12.25).—An electric heating element is arranged between an inner vessel and an outer jacket, in close proximity to the former. A heat-insulating ring is arranged between the upper edge of the inner vessel and an annular top plate secured to the upper edge of the jacket. J. S. G. THOMAS.

Electric battery. E. R. GILL (E.P. 266,565, 1.5.26). —A wet-battery system comprises a series of intercommunicating cells, having an inlet at one end and an outlet at the other end of the series. An arrangement of automatic valves permits the forward movement of fluid under pneumatic pressure from the inlet end of the series through the intermediate cells to the outlet end, where it is returned to the inlet end. A filling chamber is connected with the inlet, and is provided with a gas escape pipe having an automatic safety valve for controlling the pressure in the chamber. J. S. G. THOMAS.

Manufacture of storage batteries. C. M. ANGELL, Assr. to VESTA BATTERY CORP. (U.S.P. 1,622,304, 29.3.27. Appl., 5.11.23).—The surfaces of freshly-pasted batteryplates are dried, and the plates burned into groups in which they assume their final structural relationship of positive and negative plates, groups of which are assembled to form a battery. J. S. G. THOMAS.

Incandescence filament. G. R. FONDA, Assr. to GENERAL ELECTRIC Co. (U.S.P. 1,623,784, 5.4.27. Appl., 1.5.23).—An incandescence filament consists of tungsten and fractional percentages of thorium and copper.

J. S. G. THOMAS.

Manufacture of articles from molten carbon. I. G. FARBENIND. A.-G., Assees. of A.-G. FÜR ANLIN-FABR. (E.P. 254,679, 18.2.26. Conv., 30.6.25).—Suitable relative movement is produced between a carbon electrode conveying sufficient current to fuse it, and a counter electrode, so that an article of desired shape plate, rod, tube, etc.—is built up on the latter electrode by deposition of molten carbon from the electrode undergoing fusion. J. S. G. THOMAS. CL. XII.-FATS; OILS WAXES. CL. XIII.-PAINTS; PIGMENTS; VARNISHES; RESINS.

Galvanic cell. H. D. NYBERG (U.S.P. 1,624,845 12.4.27. Appl., 24.5.22. Conv., 28.3.22).—See E.P, 195,580; B., 1923, 1184 A.

Production of physical and chemical changes in dielectric carbon compounds by means of ionised gases. H. E. R. VOGEL (U.S.P. 1,621,143, 15.3.27. Appl., 8.4.25. Conv., 26.3.20).—See E.P. 254,375; B., 1926, 757.

[Electrode-supporting sheets for] electric storage batteries or electric accumulators. Fuller's UNITED ELECTRIC WORKS, LTD., and S. C. WHAITE (E.P. 266,497, 31.12.25).

Electric cell. G. WEISSMANN (E.P. 262,049, 20.8.26. Conv., 30.11.25).

Metallising insulating sheets etc. (E.P. 263,728).---See X.

XII.—FATS; OILS; WAXES.

Drying of fatty oils. A. V. BLOM (Z. angew. Chem., 1927, 40, 146-147).-The behaviour of a thin film of a drying oil is dependent on the rate of formation therein of nuclei, consisting of molecular complexes. These have lower surface tension than the surrounding oil, and are consequently forced to the surface, where they pile together, their "solvate" sheaths being gradually compressed. Tackiness and yield value are the first evidence of this change, which ultimately leads to the formation of a surface skin. The "period of induction" is due to the slow rate of diffusion of these colloidal aggregates towards the surface. Chemical action does not begin until the concentration of micelles in the surface is sufficiently high to permit of molecular attraction. Solidification of the film then proceeds inwards in layers. The drying through of a film, i.e., the production of a minimum of unchanged oil, thus depends on the relationship between thickness of layer and the accession of nuclei-inducing agents. By conversion to "stand oil," the formation of nuclei is inhibited, whilst some highly dispersed pigments, e.g., French ochre, carbon blacks, etc., adsorb nuclei already formed. On the contrary, a tendency to assist nucleus formation constitutes the drying action of metallic soaps and certain pigments. In poppy seed and similar oil-films, the surface micelles are more strongly "solvated" than in linseed oil, and the film has too loose a structure for condensation reactions to occur, whilst tung oil, on the other hand, has more surface activity. Gelatinisation is not a normal stage in the drying of an oil film, but may occur when too many nuclei are formed, as in air blowing or admixture with stand oil. Feeble drying power is a consequence of this anomalous gelatinisation. S. S. WOOLF.

Chemistry of palm oil. A. RAYNER (J.S.C.I., 1927, 46, 160 T). W. BRASH (*ibid.*, 160 T).—Polemical.

Saturation of resin acids. MARGOSCHES and others. —See XIII.

Determination of fat in milk. Popp.—See XIX. Glycerol tables. BOSART and SNODDY.—See XX.

PATENT.

Treatment of oils (E.P. 267,174).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Arsenic in printing ink. T. H. BARRY (Analyst, 1927, 52, 217—221).—Suggested limits for arsenic in printing ink pigments which are to be used on confectionery wrappers are (1) pigments which may be used unconditionally, containing less than 1 pt. of arsenic in 50,000 of pigment; (2) those containing between 1 in 20,000 and 1 in 50,000, to be used in conjunction with those in class (1); and (3) those containing over 1 pt. in 20,000, to be rejected. The ink contains also considerable proportions of oil, and a maximum of less than 1 pt. of arsenic in 50,000 of ink is assured by the above limits, but this method of fixing limits is unsatisfactory, and one in terms of the weight of arsenic per unit area of wrapper or carton is suggested. D. G. HEWER.

Degree of saturation of the resin acids. II. B. M. MARGOSCHES, K. FUCHS, and W. RUZICZKA (Chem. Umschau, 1927, 34, 73-76).-In continuation of earlier researches (B., 1927, 228), it is pointed out that in the determination of iodine values by the rapid method (ibid.) the halogen acids produced correspond to 50% of the iodine consumed, whilst in the case of resins or resin acids the amount may rise to 80-90%. From the examination of numerous mixtures of oleic acid and various resins, it is demonstrated that an empirical value, the "Plussäure," which is the excess of halogen acids produced over 50%, bears a linear relation, within limits, to the amount of resin in the mixture. Hence, multiplication of this value by a factor, in this case 1.4, gives an approximate value for the amounts of resin in such mixtures. With mixtures containing more than 30% of resins the relation does not hold, the "Plussäure" value increasing less rapidly than the amount of resin. In practice the iodine value of a mixture is determined by the Margosches rapid method (*ibid.*), the excess of halogen back-titrated directly, then 20 c.c. of a 3% solution of potassium iodate are added and the halogen liberated is determined by a further titration. Although indirect and approximate only, the method is claimed as rapid, and of especial value in technical soap analysis. E. HOLMES.

PATENTS.

Manufacture of compositions containing colouring materials [pigments etc.]. W. H. WHATMOUGH (E.P. 267,207, 7 and 22.12.25).-Insoluble pigments or lakes are precipitated in the presence of dilute aqueous dispersions of oleaginous materials (e.g., drying oils, mineral oils, waxes, etc.), the latter separating from the aqueous phase and being carried down by the pigment. The composite masses so produced (the consistency of which is controllable) are characterised by ready dispersion, and are used in printing inks, paints, typewriter ribbons, etc. S. S. WOOLF.

Coating wood, iron, or other structures. W. BAUR (E.P. 252,364, 14.5.26. Conv., 20.5.25).— Stationary structures, particularly of iron or wood, are coated with a refractory varnish which is "burnt on" at 50—600° by means of a hot-air blast. A suitable coating is prepared by heating castor oil at 280—300° with 5—10% of metal oxide (lead, cobalt, zinc, etc.) until the mixture is clear. On cooling, 10—20 pts. of this wax-like material are incorporated with 80 pts. of an ester gum-linseed or tung oil varnish base and thinners. To 80 pts. of this medium are added 20 pts. of metal powder, *e.g.*, aluminium, brass, or lead. S. S. WOOLF.

Manufacture of lead compounds. R. Wilhelm, Assr. to Commonwealth White Lead & Paints Pro-Prietary Ltd. (U.S.P. 1,617,887, 15.2.27. Appl., 24.9.25. Conv., 30.3.25).—See E.P. 249,809; B., 1927, 259.

Manufacture of resins. C. O. TERWILLIGER, Assr. to F. VON BRIESCU (U.S.P. 1,624,082 and 1,624,638, 12.4.27. Appl., [A] 29.6.23, [B] 5.7.23. Renewed [A, B], 7.2.27).—See E.P. 218,277 and 218,638; B., 1925, 890, 999.

Production of fibres etc. (F.P. 612,879).—See V. Cellulose composition (U.S.P. 1,620,977).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Coagulation phenomena in Hevea latex. IV. Latex in the second liquid zone. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Comm. Netherlands Rubber Testing Stat., Buitenzorg, 1927, No. 6, 503-565. Arch. Rubbercultuur, 1927, 10, [11]; cf. B., 1925, 17, 891).-Latex to which an excess of acid has been added attains a second zone of stability in which the sign of the electric charge on the globules has changed from negative to positive. The coalase in such latex is killed, but the latex gradually "creams" and the top layers ultimately coagulate. The latex coagula are paler in colour than the earlier ones, and contain a lower proportion of nitrogen. The rubber is always low in viscosity and very plastic, but has a tendency to develop tackiness, especially if exposed to light; in extreme cases it finally becomes hard and resinous. D. F. Twiss.

Ball rubber (rubber prepared after the Brazilian method). O. DE VRIES and W. SPOON (Comm. Netherlands Rubber Testing Stat., Buitenzorg, 1927, No. 8, 1-65; Arch. Rubbercultuur, 1927, 10, [1]).—The Brazilian method for the preparation of rubber from latex has been adopted for a time in Java on a large experimental scale; the method, however, showed no especial advantages, and the normal procedure for the production of plantation rubber has now displaced it. In its properties generally ball rubber appeared to be intermediate between ordinary plantation rubber and and hard fine Parà rubber. D. F. Twiss.

Direct determination of hydrocarbon in raw rubber, gutta-percha, and related substances. A. R. KEMP (Ind. Eng., Chem., 1927, 19, 531-533).-The hydrocarbon of rubber or of gutta-percha can be determined by a modification of the Wijs method. A 0.1 g. sample, from which the resins have been removed by any suitable method, is swollen overnight in 75 c.c. of purified carbon disulphide; 25 c.c. of 0.2N-Wijs solution are added with agitation so as to obtain a clear red solution. The flask stoppers are wetted with a drop of potassium iodide to prevent escape of iodine, and the flasks are kept in ice water for 2 hrs.; 25 c.c. of 15% potassium iodide solution and 50 c.c. of distilled water are then introduced, and the iodine is titrated with 0.1N-sodium thiosulphate. The theoretical iodine value for C_5H_8 with one double linking is 372.8. It is unnecessary to exclude diffused light, and a large excess

of the Wijs reagent does not affect the results. The results for samples of extracted rubber, gutta-percha, and balata correspond closely with the theoretical values, any discrepancy being in accord with the known protein, dirt, and humus contents. Milling for 2 hrs. in air or melting by prolonged heating in carbon dioxide at 280° causes no appreciable change in the chemical unsaturation of rubber. D. F. TWISS.

Cloth dyeing for rubber proofing. HOCKNEY and BANCROFT.—See VI.

PATENTS.

Preparing rubber compositions. G. W. ACHESON (U.S.P. 1,623,517, 5.4.27. Appl., 21.4.23).—Rubber latex is mixed with aqueous suspensions of deflocculated fillers and sulphur; coagulation of the rubber and flocculation of the filler are effected simultaneously with formation of a precipitate containing the ingredients in intimate admixture. D. F. TWISS.

Removal of liquid polymerides from synthetic rubber. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 267,808, 3.9.26).—The rubber-like masses obtained in the polymerisation of butadiene are accompanied by liquid polymerisation products. These are removed by applying active charcoal to the sheeted product, heating if desired at 40—50° for several hours under reduced pressure, and then removing the charcoal mechanically or by washing with water. D. F. TWISS.

Process and apparatus for devulcanising rubber. E. E. ROYER (F.P. 611,405, 30.5.25).—Old rubber is heated with a liquid such as mineral oil in an autoclave, from the bottom of which the resulting solution is forced into a still. The mineral oil which is recovered by distillation is returned to the autoclave, whilst the residual "reclaimed" rubber is removed from the still at the end of each distillation process. D. F. TWISS.

Preservation of latex. A. E. JURY and O. H. SMITH, Assrs. to GENERAL RUBBER CO. (U.S.P. 1,619,938, 8.3.27. Appl., 8.6.26).—Latex is treated with an organic antiseptic and an alkaline phosphate.

T. S. WHEELER.

Apparatus for vulcanising under fluid pressure. R. D. FRITZ, ASST. to B. F. GOODRICH CO. (U.S.P. 1,622,438, 29.3.27. Appl., 12.1.26).

Coating materials with rubber (E.P. 261,700).— See XI.

XV.-LEATHER; GLUE.

Fluorescence test for synthetic and natural O. GERNGROSS and G. SANDOR (Collegium, tannins. 1927, 12-20).-Solutions of a number of synthetic tannins (1:1000) show violet or bright blue fluorescence in the light of a quartz lamp. Some synthetic tannins (e.g., Maxyntan) show no fluorescence. 1:1000 solutions of vegetable tanning materials do not give characteristic fluorescence except oakwood extract, which shows green, donga reddish, pine bark and malet bluish-violet, and quebracho and tizerah yellowish-green. Acidification destroys the fluorescence, alkali changes the donga fluorescence to orange, and that of pine bark and malet to grass-green, and destroys that of quebracho and tizerah. Fibres treated with quebracho, tizerah, and mimosa show a yellow fluorescence, and to a smaller extent when treated with other vegetable tannins. Pine bark, malet bark, larch bark, hemlock and chestnut extracts all give a violet fluorescence on cellulose fibres treated with them. The treated fibre soon loses its fluorescing property unless a small amount of sublimate is added to the solution. The violet fluorescence is probably caused by some substance akin to fisetin.

D. WOODROFFE.

Adsorption of the violet fluorescing substances from pine bark extract and the yellow fluorescing substances from quebracho wood by different adsorbents. Relation between adsorption and chemical constitution. O. GERNGROSS, G. SANDOR, and K. TSOU (Collegium, 1927, 21-24).-1:500 solutions of pine bark and quebracho extracts, respectively, were treated with different acetyl- and nitro-cellulose fibres. Different nitrocelluloses absorbed the fluorescing material from quebracho solutions, but not from pine bark. Cellulose diacetate, "cellit" powder, and acetatesilk alone absorbed the fluorescing agent in pine bark solutions. Hydrocellulose, starch, agar-agar, wool, silk, and casein absorbed the fluorescent agent from both solutions. The irreversible adsorption diminished with increasing $p_{\rm H}$ value. D. WOODROFFE.

Detection of quebracho tannin and similar tannins in extract mixtures and in leather. POLLAK and W. SPRINGER (Collegium, 1927, 46-52).-1 g. of solid or dried extract is mixed into 10 g. of caustic soda solution (d $1 \cdot 32$) in a silver crucible, slowly heated to 260° with stirring until the dried mass re-melts, poured into hot water, neutralised with sulphuric acid (1:1), made up to 500 c.c., and 100 c.c. are shaken out into three lots of 50 c.c. of ethyl acetate, the ester extracts dehydrated over anhydrous sodium sulphate, the ester distilled off, and the residue weighed, and calculated per 100 pts. of dried extract. A similar determination is made on the precipitate obtained in the formaldehydehydrochloric acid test, using 100 c.c. of tannin solution. It is proposed to use such figures for detecting and identifying different tannins. Tabulated results are given, together with the fluorescence of solutions of the different extracts, of the residue from the ester extracts of their fused mass, and of the fused mass of the formaldehyde precipitate, also the colour of the ester after shaking with the solution for the fluorescence reaction. These tests provide a means of distinguishing the sub-group quebracho, tizerah, urunday, and mimosa bark from the larger group of pyrocatechol tans, even in mixtures and when strongly sulphited. For leathers, the formaldehyde precipitate from the water-soluble matter can be used, or the leather can be fused with caustic soda and treated as above. D. WOODROFFE.

PATENTS.

Manufacture of condensation products containing sulphur [tanning agents]. I. G. FARBININD. A.-G., Assees. of A.-G. FÜR ANILIN-FABR. (E.P. 252,694, 27.4.26. Conv., 28.5.25. Addn. to E.P. 218,316; B., 1925, 181). —Products having properties similar to those previously described, except that they are only slightly hygroscopic, are prepared by the interaction of hydroxyarylcarboxylic acid sulphochlorides, such as salicylic acid sulphochloride, with esters derived from hydroxyarylcompounds, such as 2-chlorophenyl carbonate, resorcinyl diacetate, or acetylsalicylic acid. L. A. Colles.

Process of tanning. J. K. TULLIS (U.S.P. 1,621,612, 22.3.27. Appl., 3.10.23).—Hides are partially tanned in weak tanning liquors, dried till bone dry, and then drummed with half their weight of strong tanning liquor to complete the tannage. D. WOODROFFE.

Leather dressing and tanning material. F. C. ATKINSON (U.S.P. 1,622,127, 22.3.27. Appl., 16.8.19). —Corn cobs are digested under pressure at 140—200° for 2—3 hrs. D. WOODROFFE.

Manufacture of glues, gelatins, and the like from plagiostomaceous materials. A. EHRENREICH (E.P. 267,041, 4.11.26. Conv., 12.7.26).—Skins, pectoral and other fins, damaged skins and all gelatinous waste of plagiostomi are washed in water, or limed for 2 weeks, washed and bleached with sulphurous acid, then treated with water in a dissolving vat provided with a perforated partition, composed of a grid of wooden laths or galvanised wire cloth, and a heating coil. The liquid is heated until it froths or bubbles (70-80°), the heating discontinued and re-started alternately for 2 hrs. or until the liquid has reached the desired consistency, the impurities are removed, the viscous liquid is transferred to a receptacle which is placed in a refrigerator, or mixed with 30 g. of boric acid per 100 litres of liquor, and allowed to set in hermetically sealed flat boxes to obtain sheets of gelatin or poured into a vat, cooled to 40°, then $2\frac{1}{2}$ kg. of hydrochloric acid or 28% acetic acid are added per 100 litres of liquor, and finally oil of sassafras to produce a liquid glue. D. WOODROFFE.

Artificial leather (F.P. 611,401).—See V.

Treatment of yeasts (E.P. 243,373).—See XVIII.

XVI.—AGRICULTURE.

Value of "dissolved " waste hide as a manure. G. JORET and E. RADET (Ann. Falsif., 1927, 20, 133-148).—In the solution process, which consists in boiling the hide with sulphuric acid and adding calcium phosphate to increase the consistency and partially neutralise the acid, a variable proportion of nitrogen is rendered soluble in water, depending on the amounts of acid and phosphate used. In all cases there is an insoluble residue, which is useless for agricultural purposes. When mixed with a highly calcareous earth, the mean value of the nitrifiable nitrogen may amount to two thirds, but with a non-calcareous earth to only one half of that for the same amount of desiccated blood. Free acid and tannin tend to hinder nitrification. The Street index is of no value in evaluating these products, but the determination of the insoluble residue, its nitrogen content, as well as the intensity of the tannin reaction, are useful indications as to their commercial value as F. R. ENNOS. manures.

Nitrification of ammonium sulphate in soil. H. J. HARPER and B. BOATMAN (J. Amer. Soc. Agron., 1926, 18, 876-888).—Air-drying soils does not appear to affect the nitrification of ammonium sulphate. A higher percentage of nitrate-nitrogen was produced when smaller amounts of ammonium sulphate were used. Phosphorus and potassium fertilisation did not increase the nitrifying power of soils better than ground limestone. CHEMICAL ABSTRACTS.

Biological activities in fertiliser composts. A. J. ENGLEHORN (Proc. Iowa Acad. Sci., 1925, 32, 85—94).— The presence of sulphur in a fertiliser compost reduces, and that of orthoclase or raw rock phosphate increases, the number of bacteria present; the presence of sulphur stimulates, and that of orthoclase depresses, the growth of fungi. CHEMICAL ABSTRACTS.

Determination of the fertiliser requirements of soils by the Neubauer seedling method. S. GERICKE (Z. angew. Chem., 1927, 40, 366-368).-100 rye seedlings were grown for 18 days in 100 g. of the soil to be tested in glass bowls. The young plants were then washed free from soil and their potash and phosphorus contents determined. If these fall below certain values, originally given as 8 mg. P₂O₅ and 24 mg. K₂O, the soil is considered to be deficient in the fertiliser concerned. The method is not applicable to nitrogen. Various critical investigations of this method are discussed, and it is concluded (1) that the period of growth should be 14-15 days, in which time a maximum absorption of the fertiliser is obtained; (2) the soil should be diluted with sand and a blank test performed to compensate for interchanges between the plant and the sand, of minerals originally present in the seed; (3) the amount of water added, temperature, and light intensity all have a slight effect on the results, and should be standardised. The amount of fertiliser found in the grown seedlings which is considered to represent a satisfactory soil also varies with the species of plant grown. In spite of these limitations, the method gives results much more closely in accordance with field trials than are the indications of soil analysis by chemical methods, and is of great value. C. IRWIN.

Fungicidal and bactericidal action of selenium and tellurium compounds. N. M. STOVER and B. S. HOPKINS (Ind. Eng. Chem., 1927, 19, 510-513).-Sodium selenite and tellurite, potassium selenite and tellurite, and tellurium hydrogen tartrate in 0.05-0.0005N-solutions have practically no fungicidal action on the fungus causing chestnut blight, Endothia parasitica, even after exposure for a week, whilst selenious acid, lithium and thallous selenites are definitely toxic. Apple blotch, Benturia inæqualis, is not controlled by 0.1-0.04N-selenious acid even after repeated application. Selenious acid and thallous selenite are quite bactericidal to pear blight, B. amylovorus, according to laboratory tests, but these results have not been substantiated in the field. Selenious acid injected into pear and chestnut trees enters the circulatory system, but appears to be too injurious to the tree to be usefully employed against parasitic fungi. On the other hand, it is sufficiently toxic to be used as a herbicide against dandelion, Canada thistle, and burdock. E. HOLMES.

Ionic exchange of zeolitic silicates. KAPPEN and RUNG.—See VII.

Fumigation tests with chloropicrin. Hoyt and ELLENBERGER.—See XIX.

PATENTS.

[Seed] disinfecting composition. Non-hygroscopic seed disinfectants. M. ENGELMANN and A. R. ALBRIGHT, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,618,369 and 1,618,370, 22.2.27. Appl., [A], 11.1.23; [B], 8.6.23).—(A) An insoluble, organic mercury compound, e.g., o-acetoxymercuriphenol (10-30%), is intimately mixed with a dry, pulverised, alkaline-earth oxide or hydroxide (90—70%). (B) A mercuriphenol is mixed with an alkaline carbonate and an alkalineearth oxide or hydroxide. T. S. WHEELER.

Production of [seed] disinfectants. M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,618,371—2, 22.2.27. Appl., [A], 19.2.24 ; [B], 25.7.25). —(A) Finely-divided calcium carbonate or other insoluble basic substance (1000 pts.), copper sulphate or other soluble copper salt (500 pts.), and water (6000 pts.) are agitated together, and the product is filtered off, dried, and ground. (B) Mercuric acetate (50 pts.), kaolin (500 pts.), sodium o-nitrophenoxide or similar compound (23 pts.), and water (1500 pts.) are heated at 80° for 4 hrs., when the corresponding mercuriphenol compound is obtained, dispersed on the kaolin ; its value as a seed disinfectant is thus increased. T. S. WHEELER.

Lactonitrile fumigant. L. J. CHRISTMANN, ASSR. to AMERICAN CYANAMID Co. (U.S.P. 1,620,074, 8.3.27. Appl., 3.8.25).—An insecticide comprises a mixture of a stable organic cyanogen compound, *e.g.*, lactonitrile, (50 pts.), a non-alkaline saponaceous adhesive material, *e.g.*, saponin, (25 pts.), and water (50 pts.). It is further diluted with water before spraying. T. S. WHEELER.

Preparation of a [sulphur] fungicide. J. H. REEDY and W. S. BROCK (U.S.P. 1,620,229, 8.3.27. Appl., 3.10.24).—Calcium polysulphide solution is treated with zinc sulphate, and the mixture of calcium sulphate, zinc sulphide, and finely-divided sulphur obtained is employed as a fungicide. T. S. WHEELER.

Dehydrating vegetable substances etc. (E.P. 267,203).—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Wort boiling. L. R. SKINNER (J. Inst. Brew., 1927, 33, 219-222).-The crystalline structure of copper, which is universally chosen as the container in which to boil wort, explains its exceptional heat-conductive power. Heat radiation is an ionic emission, and negatively charged ions pass from the source of heat—the furnace to the copper. In the cases where the copper has been "gassed" by the reducing action of the flue gases on the oxygen contained in the copper, the cohesion between adjacent crystals is reduced, and a quicker passage is offered to the ions of emission, the rate and velocity of impingement of which may have a direct bearing on the ultimate cooked flavour of the wort. The cooking value of steam-heated coppers is inferior to that of fire-heated coppers, since the energy is third-hand, and there is not sufficient difference of temperature between the steam and wort to get the true cooking effect and complete sterilisation. With pressure steam-heated coppers, owing to the higher temperatures, sterilisation is better, the concentration is greater, and the rate of evaporation is increased with an improved cooking effect. Steam, and even super-steam heated pressure coppers do not compare favourably with fired coppers, which ensure a better quality of heat and a higher heating temperature. Coal and oil give the sun's energy secondhand, whereas energy transmitted by steam is thirdhand, owing to the coal or oil having to be burned to generate the steam. C. RANKEN.

Production of *n*-butyl alcohol and acetone from Jerusalem artichokes. A. C. THAYSEN and B. M. GREEN (J. Inst. Brew., 1927, 33, 236—240).—The tubers of Jerusalem artichokes (*Helianthus tuberosus*) cannot be converted into *n*-butyl alcohol and acetone by *Bac. butylicus*, Weizmann, in the same way as raw materials containing starch, owing to the bulk of the carbohydrates in the tubers not being directly fermentable by the organism. A yield of 12 gals. of "oil," or more, per ton of fresh tubers can, however, be obtained if the original carbohydrates are hydrolysed and the mashes diluted to such an extent that the reducing sugars are prevented from exerting their inhibitory action on the micro-organisms or their enzymes. C. RANKEN.

Difficulties of the sulphuric acid treatment [of distillery mashes]. WENDEL (Z. Spiritusind., 1927, 50, 96-97).-If lactic acid is replaced by sulphuric acid in the treatment of distillery mashes, owing to the greater strength of the latter acid, measurement of the resulting acidity to litmus by titration is insufficient, and must be amplified by determinations of the hydrogen-ion concentration. The efficiency of the treatment depends not only on the strength of the acid, but also on the concentration of the mash and the kind of raw material. Mashes made with maize require a lower degree of acidification than those made with potatoes or rye, whilst, in addition, greater amounts of acid can be safely added to thick mashes. If ammonium sulphate is used as a nitrogenous pabulum for the yeast, regard must be had to the increase of acidity from this cause during fermentation. Satisfactory results are obtained with sulphuric acid with mash concentrations varying from 15-18° Balling, and with $p_{\rm H}$ 5.0 (approx.). For the chief mash, the optimal acidity to litmus is given as $0.15-0.20^{\circ}$, and as 0.3° or slightly higher for the subsidiary mash. C. RANKEN.

PATENTS.

Treatment of yeasts by autolysis. M. KAHN, E. LE BRETON, and G. SCHAEFFER (E.P. 243,373, 19.11.25. Conv., 19.11.24).—Food products and products for tanning and tawing are obtained by autolysing distillery yeast, rhizopus, and amylomyces. This is effected in a medium of 5—20% by wt. of sodium chloride, for a time varying from a few hours to 6 days, the temperature being maintained between 40° and that at which the ferments are destroyed. The autolysis may be accelerated by adding suddenly after a few hours' action 5-10% of sodium chloride to the mass. (Cf. E.P. 225,228; B., 1925, 605.) A. RAYNER.

Manufacture of beverages. H. MACKESON (E.P. 267,236, 14.12.25).—The juice or must of fruits, particularly apples, pears, or grapes, is mixed with malt extract, fermented, and filtered. It is then further mixed with a priming solution consisting of glucose syrup or a concentrated fruit juice, with the addition of malt extract, again filtered, and pasteurised.

F. R. ENNOS.

Production of lactic acid from sugar-containing raw materials by means of lactic acid bacteria. A. POLLAK (U.S.P. 1,624,674, 12.4.27. Appl., 11.9.24. Conv., 29.9.23).—See E.P. 222,514; B., 1926, 73.

Manufacture of butyric acid and other aliphatic acids. L. LE FRANC, Assr. to LEFRANC & CIE. (U.S.P. 1,625,732, 19.4.27. Appl., 18.8.22. Conv., 26.9.21).--See E.P. 186,572; B., 1923, 674 A.

XIX.—FOODS.

Determination of fat in milk by the Neusal method. M. POPP (Chem.-Ztg., 1927, 51, 270).— Sufficient solution for 10,000 determinations is made by dissolving 10.2 kg. of a mixture of sodium salicylate and citrate in 40 litres of water. After remaining overnight a slightly insufficient quantity of butyl alcohol (9800—9850 c.c.) is added, and determinations of fat with this solution on a series of milks are compared with those made by the sulphuric acid method. The amount of the alcohol is then adjusted so that the results obtained by the two methods are in agreement. For the fat determination, 4 c.c. of the above solution are mixed with 9.2 c.c. of milk in a butyrometer, heated at 65° , centrifuged for 5 min., and the fat read at 45° .

F. R. ENNOS.

Immersion refractometer and its value in milk analysis. G. D. ELSDON and J. R. STUBBS (Analyst, 1927, 52, 193-214).-Results from comprehensive tests with the immersion refractometer as a means of detecting added water in milk show that claims made in this connexion have been much exaggerated. Refraction is found to be proportional to the solids-not-fat, and in no case did a milk having a refraction over 37.1 have less than 8.5% of solids-not-fat. Also since refraction rises as acidity increases, up to a point, a watered milk may on keeping give a figure identical with that of the genuine milk. It is difficult to apply a correction factor for such increase due to acidity, but generally, on an average, an increase of 1.0 in the acidity (c c. of 0.1N-sodium hydroxide required to neutralise 10 c.c. of milk) accompanies an increase of 0.2 in the refraction. D. G. HEWER.

Basic viscosity of ice-cream mixes. A. LEIGHTON and O. E. WILLIAMS (J. Physical Chem., 1927, 31, 596-600).—Ice-cream mixes show a "basic" viscosity, *i.e.*, a viscosity independent of the changes in mechanical structure which occur on being kept. At constant temperature the basic viscosity changes with the concentration of water in accordance with the Arrhenius equation, $\log \eta = \theta c$. L. S. THEOBALD.

Formaldehyde in fish. D. B. DILL and P. B. CLARK (Analyst, 1927, 52, 222; cf. B., 1926, 339).— Formaldehyde obtained from the fish examined by the authors is regarded as not due to oxidation of trimethylamine (cf. Tankard and Bagnall; B., 1927, 25) since little or no formaldehyde was present at canning time, but developed with age in the absence of oxygen, and tests were made on distillates obtained by steam distilling the samples mixed with a 2% solution of phosphoric acid (c.c./g.), the volume being kept constant by immersing the flask in a heated salt bath.

D. G. HEWER.

Quality of the protein of whale meat products. W. L. DAVIES (J.S.C.I., 1927, 46, 99-100 T).-Attention is drawn to the high content of non-protein nitrogen in feeding stuffs manufactured from animal by-products and refuse. This non-protein nitrogen, which is watersoluble, has been examined in the case of whale meat flakes and whale meat meal, comparing the composition of the aqueous extracts in each case with the composition of the pure tissue protein. With respect to diamino-acid content, the aqueous extracts compare favourably in composition with pure tissue protein, and also with any complete protein, and, although the total nitrogen allotted to diamino-acid is slightly lower for the extract, this is counterbalanced by a higher amount of nitrogen allotted to monoamino-acid nitrogen in the extract. Such extracts are slightly inferior in composition to similar extracts of various fish meals. The flake had roughly a fifth, and the meal two fifths, of its nitrogen as non-protein water-soluble nitrogen. The flakes kept better under ordinary conditions of storage than the meal.

Changes during storage in the composition of mangels. J. P. DREW and G. T. PYNE (J. Dep. Lands Agric., Ireland, 1926, 26, 9—14).—The composition of fresh Red and Yellow Intermediate varieties, respectively, was: dry matter 9.43, 11.21; sucrose 4.55, 6.48; reducing sugars 0.16, 0.22; crude protein 0.89, 1.14; true protein 0.39, 0.55; oil 0.03, 0.03; fibre 0.65, 0.76; ash 1.13, 1.13%. On storage for 5 months the percentages of oil, fibre, ash, and true protein were scarcely changed; both varieties lost 28% of the dry matter and 26—40% of the sucrose. The crude protein decreased 0.17—0.35%; the amounts of nitrate nitrogen decreased from 0.041 to 0.025, and 0.045 to 0.026%, respectively. CHEMICAL ABSTRACTS.

Examination of foodstuffs for preservatives. A. C. CHAPMAN (Analyst, 1927, 52, 215—217).—From certain carbohydrate materials such as caramel an acid, or mixture of acids, may be obtained which closely simulates benzoic acid. It gives a violet coloration with ferric chloride, apparently due to a phenolic substance present in smaller proportion than the acid. The latter reacts with bromine, but gives no precipitate. Formaldehyde, or some substance very closely resembling it, is produced on heating sugar products, and formal-dehyde reactions in such cases do not necessarily indicate addition of that substance. Boron occurs naturally in agar-agar and other seaweeds. D. G. HEWER.

Fumigation tests with chloropicrin [on foodstuffs etc.]. L. F. HOYT and E. P. ELLENBERGER (Ind. Eng. Chem., 1927, 19, 461—464).—A large variety of food products, fumigated on the commercial scale with nitrochloroform for a week, at concentrations of 0.8—1.33 lb./1000 cub. ft. of storage space, appear to be undamaged in any way. Germination tests on corn, oats, sunflower, wheat, and buckwheat show that exposure to chloropicrin at the lower concentration for one week had no deleterious effect on the germination of the seed, but rather the reverse effect on the last two. Chloropicrin is recommended as a fumigant on the grounds of the protective warning afforded by its lachrymatory power in high dilutions, and because of its highly poisonous effect on such insects as the Indian meal moth, Mediterranean flour moth, and the more resistant confused flour beetle. E. HOLMES.

PATENTS.

Manufacture of bread. J. MATTI (E.P. 266,967, 25.6.26).—100 pts. of flour, $66 \cdot 5$ pts. of water, 2 pts. of salt, and 1 pt. of yeast are mixed at 38—40°, $0 \cdot 1$ pt. of an edible neutral fatty substance, *e.g.*, refined paraffin oil, is added, the whole mechanically kneaded for 12 min., and fermented for 40 min. After the moulding and rising in the pan, which occupy 15—30 min., the mixture is baked for 1 hr. F. R. ENNOS.

Manufacture of bread. H. A. KOHMAN (E.P. 244,489, 14.12.25. Conv., 12.12.24).—The usual ingredients of the dough are mixed with a dough-maturing agent, such as potassium persulphate, bromate, iodate, or periodate, in a quantity greatly in excess of that permissible when the dough is kept for the ordinary fermentation period. After mechanically mixing the batch for 10—20 min., it is divided, proofed, and baked in the usual way. F. R. ENNOS.

Milling products [flour]. H. C. J. H. GELISSEN, Assr. to NOYADEL PROCESS CORP. (U.S.P. 1,620,458, 8.3.27. Appl., 3.5.26. Conv., 11.3.25. Cf. E.P. 102,967; B., 1918, 71 A).—Flour is treated with a current of air at 20° in presence of a small quantity (0.003%) of an aldehyde, *e.g.*, acetaldehyde or benzaldehyde, and an acid anhydride, *e.g.*, acetic or succinic anhydride, together with a catalyst, *e.g.*, cobalt acetate or a similar salt of vanadium, chromium, etc. The peroxide produced bleaches the flour. T. S. WHEELER.

Butter and cream treating process. R. K. COONEY, ASST. to CAMPBELL-COONEY PATENTS CO. (U.S.P. 1,599,649, 14.9.26. Appl., 30.12.24. Renewed 24.2.26).— Highly acid cream is agitated with a small quantity of slaked lime at 38°, pasteurised, and centrifuged to remove solid matter. The keeping properties of the cream, and of the butter prepared from it, are improved. T. S. WHEELER.

Pickling of fruit etc. J. W. BECKMAN, Assr. to G. C. ROEDING (U.S.P. 1,621,188, 15.3.27. Appl., 8.12.25).—The fruit, such as olives, is treated with a 2% solution of caustic soda for 4—6 hrs. to open the pores, and then with 1.5% hydrochloric acid and brine solution for 2—4 days. It is then immersed in a 1% solution of sodium lactate and lactic acid, the time required to remove the free hydrochloric acid and give the fruit the desired acidity being approximately one week.

F. R. Ennos.

Preserving fruit juices and other liquids. W. MATZKA (E.P. 267,058, 5.9.25).—The liquid passes slowly through a vessel where it is subjected to gentle electrolysis between a gold cathode and an aluminium anode. The temperature of the liquid, which is heated by passing steam or hot water through the electrodes, is regulated between 30° and 55° , depending on its albumin content and acidity, and is substantially below the normal pasteurisation temperature, so that the natural aroma is retained. F. R. Ennos.

Dehydrating vegetable substances or products of organic character. B. J. OWEN (E.P. 267,203, 7.12.25).—The drying agent, consisting of heated air, is supplied to the mass of material under treatment (e.g., grain or root-crops) at ranges of temperature, pressure, and volume such as have been found to regulate the consolidation of the mass satisfactorily, and also to promote the naturally occurring physical and chemical reactions, so that the rate of dehydration is increased to a maximum, and the effects of the exothermic reactions occurring are utilised to the best advantage.

F. R. ENNOS.

Preparation of vitamin-rich products. H. Iscovesco and A. B. ADAMS (E.P. 267,410, 17.9.26).—Cod liver residues are treated with a solution of caustic soda (d 1.33) and kept at about 18° for 2 months or longer. The saponified mass, after drying *in vacuo* below 50°, is extracted with acetone; the solvent is then evaporated, and the vitamin-rich product dissolved in liquid fats or glycerin before addition to foodstuffs.

F. R. Ennos.

Production of fish-oil powder. W. E. MILLER, Assr. to SILMO CHEMICAL Co. (U.S.P. 1,622,390, 29.3.27. Appl., 5.3.26).—Fish oil is added slowly with agitation to an equal weight of fermented milk in an airtight vessel, and the mixture kept for 48 hrs. at 10°. An amount of water equal to that of the oil is added and the whole emulsified. After a further 48 hrs. at 10°, the solid matter is separated from the liquid at 20°, and subsequently powdered. S. S. WOOLF.

Cream-treating process. R. K. COONEY, Assr. to 'CAMPBELL-COONEY PATENTS CO. (Re-issue 16,576, 22.3.27, of U.S.P. 1,599,650).—See B., 1927, 26.

Butter and cream treating process. R. K. COONEY, ASST. to CAMPBELL-COONEY PATENTS CO. (Reissue 16,562, 1.3.27, of U.S.P. 1,599,649).—See above.

Treatment of yeasts (E.P. 243,373).-See XVIII.

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

Manufacture of amyl acetate from natural gasoline. G. T. KOCH and G. A. BURRELL (Ind. Eng. Chem., 1927, 19, 442-445).-A process for the conversion of a 25-40° fraction of gasoline from natural gas, essentially pentane, into amyl acetate is described, the main feature being an ingenious device for the preliminary chlorination of the hydrocarbon in the liquid phase, in the presence of an activated carbon or other porous catalyst. A 90-110° cut of the chlorinated product is then esterified with sodium acetate in an oil-heated autoclave at 205-230° and under a pressure of 200-250 lb., the time of reaction being 8 hrs. The fraction 120-175° of the resulting ester consists of 99-100% amyl acetate (d^{15.5} 0.88) having no moisture, a light yellow colour, and containing 0.196% of free acetic acid. A method of determining the amyl acetate in each fraction is based on the solubility of the ester, and the relative insolubility

of the chlorohydrocarbons, saturated ring compounds of the polymethylene series, and paraffin hydrocarbons in sulphuric acid ($d \ 1.84$). Cost figures are included derived from the running of a semi-commercial plant. E. Holmes.

Distillation of formaldehyde solution. A. ZIM-MERLI (Ind. Eng. Chem., 1927, 19, 524—525).—An apparatus is described in which it was found possible to concentrate solutions containing (a) $15 \cdot 6\%$ of formaldehyde and $3 \cdot 4\%$ of methyl alcohol, and (b) $17 \cdot 7\%$ of formaldehyde in water, to a point at which the distillate contained 56% of formaldehyde, corresponding to 90% of methylene glycol. This distillate deposits paraformaldehyde immediately on cooling. (Cf. Blair and Taylor, B. 1926, 339.) E. HOLMES.

Humic acids. II. Action of thionyl chloride, of bromine, and of chlorine dioxide on some humic acid samples. W. FUCHS and H. LEOPOLD (Brennstoff-Chem., 1927, 8, 101—103; cf. B., 1927, 315).—Merck's humic acid and that prepared from quinol behave practically identically when exhaustively treated with thionyl chloride, bromine, and chlorine dioxide, whilst humic acid prepared from starch behaves quite differently. In none of the specimens was any trace of carbohydrates found. Humic acids are feebly reactive substances of high mol. wt., resembling phenols in some respects, but differing from them in their comparatively low content of phenolic hydroxyl groups. W. T. K. BRAUNHOLTZ.

Properties of diethylene glycol. W. H. RINKEN-BACH (Ind. Eng. Chem., 1927, 19, 474-476).-A study of the chemical and physical properties of a carefully purified specimen of diethylene glycol has been made, the chief findings being as follows. The bluish crude material loses its colour on purification, and gives an odourless compound with a slightly sweet, burning taste. Densities are given at temperatures between 0° and 35° , that at $15^{\circ}/15^{\circ}$ being $1 \cdot 1212$, whilst the liquid has f.p. $-10.45^{\circ} \pm 0.05$, and b.p. (by extrapolation) $244.5^{\circ}/760$ mm. Calorimetric determinations of the heat of combustion of diethylene glycol gave the values 566.11 kg.-cal./g.-mol. at constant volume, and 566.69 at constant pressure, these values being roughly double those for the corresponding ethylene glycol. The heat of dilution was found to be positive, the compound probably forming a molecular complex with water, having a positive heat of dissolution. It is freely miscible with water, methyl alcohol, ethyl alcohol, ethylene glycol, glacial acetic acid, acetone, furfuraldehyde, pyridine, glycol diacetate, chloroform, nitrobenzene, and aniline, but immiscible with ether, benzene, toluene, carbon disulphide, and carbon tetrachloride, all at ordinary temperatures. Diethylene glycol absorbs more than its own weight of water in 9 days when kept in a closed space over water, but undergoes no hydrolysis. Numerous tables are given, including those for variation of viscosity and refractive index with temperature.

E. HOLMES.

Glycerol tables. L. W. BOSART and A. O. SNODDY (Ind. Eng. Chem., 1927, 19, 506-510).—New and comprehensive tables are given for the sp. gr. and percentage of glycerol, both apparent and true (*i.e.*, reduced to vacuum), in mixtures of glycerol and water,

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based on very careful determinations of sp. gr. of mixtures at $15^{\circ}/15^{\circ}$, $15 \cdot 5^{\circ}/15 \cdot 5^{\circ}$, $20^{\circ}/20^{\circ}$, and $25^{\circ}/25^{\circ}$ for every $10^{\circ}/_{0}$, and for 95 and $97 \cdot 5^{\circ}/_{0}$ glycerol. The results, calculated to the fifth decimal place, are believed to be more accurate than any hitherto published, agreeing best with those of Nicol. The apparent values now given for $100^{\circ}/_{0}$ glycerol are $1 \cdot 26557$ at $15^{\circ}/15^{\circ}$, and $1 \cdot 26362$ at $20^{\circ}/20^{\circ}$, the corresponding true values being $1 \cdot 26526$ and $1 \cdot 26331$. Tables are also given comparing the values given by various workers, and showing the rate of expansion of mixtures between the temperatures $15-20^{\circ}$, $15-25^{\circ}$, and $20-25^{\circ}$. An important correction is noted in a calculation occurring in a paper by Grün and Wirth (A., 1919, ii, 202). E. HOLMES.

Moderately dilute sulphuric acid as a reagent for the examination of drugs. H. TEUFER (Pharm. Zentr., 1927, 68, 225—226).—Colour reactions of some vegetable drugs with 70% and 80% sulphuric acid are compared, and the relationship between the colours produced by various barks with acids of the above strengths and the degree of lignification of the cell walls of the barks is discussed. E. H. SHARPLES.

Occurrence, detection, and determination of ethyl chloride in perfumes. T. SUDENDORF and O. PENNDORF (Pharm. Zentr., 1927, 68, 226-228).-A few grams of the ice-cold perfume are added to about 100 c.c. of ice-cold water containing slightly more than the calculated quantity of potassium hydroxide necessary to hydrolyse the ethyl chloride, and the mixture is shaken vigorously at room temperature for at least 5 hrs. About 10 g. of powdered wood charcoal are then added, and, after shaking for 10 min., the liquid is filtered. The filtrate is distilled and the first 5-10 c.c. of distillate are examined for alcohol by the Lieben-Serulla iodoform method or, in doubtful cases, by the Hofmann carbylamine reaction. For the detection of the halogens, an alcoholic solution of slightly more than the calculated amount of potassium hydroxide is added to a few grams of the perfume, both liquids being ice-cold. The mixture is shaken at room temperature, and, if halogens are present, small crystals are deposited on the sides of the vessel. The liquid is decanted, the crystals are dissolved in water, and the solution is examined for halogens by the usual methods. The dangers occasioned by the indiscriminate use of perfumes containing large amounts of ethyl chloride are emphasised. E. H. SHARPLES.

Genus Mentha. XI. Oil of Mentha Canadensis, L. H. A. BRAUN (J. Amer. Pharm. Assoc., 1926, 15, 337— 338).—The greenish-yellow oil (1·16%) had d^{24} 0·8974, α_D + 32·4°; ester 8·06, total alcohols 15·7, free alcohols 7·9, pulegone (R₂SO₃) 20·00, pulegone (RHSO₃) 18%. Phenols were absent. Fractionation did not indicate pulegone; the fraction b.p. 140—175° yielded two semicarbazones having m.p. 137° and 180°, respectively.

CHEMICAL ABSTRACTS.

Determination of phenol. WILLIAMS .- See II.

Dehydration of sulphite spirit. Schlumberger.— See V.

Medicinal creosote. BOBROV.-See XIII.

n-Butyl alcohol and acetone. THAYSEN AND GREEN.—See XVIII.

PATENTS.

Method of conducting chemical reactions. [Oxidation of acetaldehyde.] C. O. Young and C. J. HERRLY, Assrs. to CARBIDE AND CARBON CHEMICALS CORP. (U.S.P. 1,620,180, 8.3.27. Appl., 20.6.23).-Acetaldehyde is passed in counter-current to a stream of air through a series of vessels at a gradually increasing temperature. The product containing 90% of acetic acid is withdrawn from the last vessel, and the spent air carrying aldehyde vapours is passed from the first vessel through one of two absorbers containing silica gel or other adsorbent at a low temperature. When the adsorbent is saturated it is heated, and the fresh air passing into the apparatus is led through it to recover the aldehyde, the spent air being discharged through the second absorper. T. S. WHEELER.

Manufacture of oxygenated products from hydrocarbons or oxidisable derivatives of hydrocarbons. W. A. CASPARI (E.P. 263,201, 11.11.25)—A colloidal solution or suspension of an oxide or oxides of the metals of the fifth or higher group in the hydrocarbon to be treated is atomised with air into a heating chamber at 200—500°. The metallic catalyst is recovered from the condensed product, either unchanged or in the form of the lower oxide. Thus benzaldehyde and benzoic acid may be produced by spraying a colloidal solution of vanadium oxide in toluene with eight times its vapour volume of air, into a chamber at 200—300°. S. PEXTON.

Preparation of alkyl hydrogen sulphates. TEXAS Co., Assees. of R. DE M. TAVEAU (Can. P. 258,227, 12.6.24), —Gases containing olefines are led through a series of absorption vessels containing sulphuric acid in increasing concentration. In the first two vessels, kept at 30°, butylene and propylene, respectively, are absorbed, whilst ethylene is absorbed in the last vessel at 80— 120°. C. HOLLINS.

Production of chlorocarbonates [chloroformates]. J. A. S. HAMMOND, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,618,824, 22.2.27. Appl., 14.7.24).— Gaseous carbonyl chloride (95.5 pts.) is passed into absolute alcohol (58 pts.) at 20°, and the product is treated with water and carbon tetrachloride to separate ethyl chloroformate, which is produced in 90% yield.

T. S. WHEELER.

Preparation of a reactive chloride from pinene or turpentine. F. Höhn (G.P. 426,865, 4.4.25).-Purified turpentine oil or pinene is added in a current of hydrogen chloride, with or without light petroleum, to hydrochloric acid which has been cooled in ice and salt and saturated with hydrogen chloride, giving a very reactive solid chloride, C10H17Cl, together with about 30% of an oil from which further quantities of the solid chloride are obtainable by strong cooling. The solid chloride differs from the chloride prepared at temperatures above 15° in that it readily loses hydrogen chloride when warmed, forming camphene. The oil, after separation of the solid, still yields a little camphene when heated. The saturated hydrochloric acid may be used for further C. HOLLINS. batches.

Preparation of Bz-halogenated derivatives of oxindole-3-acetic acid. CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING), Assees. of W. SCHOELLER and K. SCHMIDT (G.P. 436,518, 11.3.25. Addn. to G.P. 431,510; B., 1926, 870).—Halogenated isatins are condensed with malonic acid, and the unsaturated acids so formed are reduced. Thus, 5-iodoisatin is melted with malonic acid at 130—140° until no more carbon dioxide is evolved, the mass is dissolved in very dilute sodium hydroxide and acidified with dilute acetic acid. The precipitate is filtered off and reduced in sodium hydroxide solution with aluminium amalgam to give 5-iodooxindole-3-acetic acid. Dichloro-, dibromo-, and di-iodooxindole-3-acetic acids are similarly prepared.

C. HOLLINS.

Manufacture of acetals. CONSORTIUM FÜR ELEK-TROCHEM. IND. G.M.B.H. (E.P. 257,622, 27.8.26. Conv., 27.8.25).—The reaction between acetylene and alcohols in the presence of a mercury salt is so conducted that the acetal is removed from the sphere of action as it is formed, *e.g.*, by means of an immiscible solvent, or by distillation, or by "salting out" with potassium acetate, etc. For the preparation of diethylacetal, acetylene is passed rapidly into a suspension of finely-divided mercuric acetate in alcohol containing 10% of sulphuric acid at 75°. Acetal and ethyl alcohol are carried over by the excess of acetylene, and fresh alcohol is added continuously to replace that distilled. C. HOLLINS.

Preparation of iodinated pyridine derivatives. DEUTSCHE GOLD- U. SILBER-SCHEIDEANSTALT, VORM. ROESSLER (F.P. 605,226, 20.6.25. Conv., 30.6.24. Cf. E.P. 251,578; B., 1926, 608).—The following are additional. 5-Iodo-2-hydroxypyridine, m.p. 191—192°, is precipitated by carbon dioxide from a solution of its sodium salt. 5-Iodo-2-aminopyridine, m.p. 129°, from the action of aqueous potassium iodide on aqueous 2-amino-5-diazopyridine, is isolated by making the solution strongly alkaline and distilling in steam. 3-Diazo-2-hydroxypyridine similarly gives 3-amino-2-hydroxypyridine (sodium salt described).

C. HOLLINS.

Manufacture of quinoline derivatives. W. CARP-MAEL. From FARBENFABR. VORM. F. BAYER & Co. (E.P. 267,457, 7.9.25).—8-Nitro-6-methoxyquinoline, m.p. 159—160°, and 8-nitro-6-ethoxyquinoline, obtained by Skraup's reaction from 2-nitro-p-anisidine and 2-nitro-pphenetidine, respectively, are reduced by stannous chloride and hydrochloric acid to the corresponding amines, m.p. 41° and 60°, and b.p. 137—138°/0·5—1 mm. and 144—145°/1 mm., respectively. The amines are antipyretic, and have also a specific destroying action on blood parasites. C. HOLLINS.

Manufacture of pharmaceutical products. W. CARPMAEL. From FARBENFABR. VORM F. BAYER & Co. (E.P. 267,169, 7.9.25).—Strongly basic compounds of the di- and tri-arylmethane, quinoline, phenazine, oxazine, thiazine, acridine, and xanthene series, usually β -dialkylaminoethylamino-derivatives and of pharmaceutical interest, are prepared by a variety of methods from amines of these series. β -Diethylaminoethylaniline, b.p. 121—123°/5 mm., is obtained from aniline and β -diethylaminoethyl chloride, and is converted into a thiazine dye by oxidation together with p-aminodimethylaniline and thiosulphate. The corresponding

methyl-(B-diethylaminoethyl)aniline, b.p. 124-126°/5 mm., from methylaniline, gives a similar thiazine dye. β - Phenylmethylamino - δ - dimethylaminoisopentane, NPhMe · CHMe · CHMe · CH₂ · NMe₂, b.p. 133-135°/6 mm., prepared from methylaniline and the appropriate aminoalkyl chloride, gives a thiazine when its p-nitrosocompound is reduced and the resulting amine is oxidised with dimethylaniline and thiosulphate. A homologue of malachite-green is obtained by condensing methyl-(β-diethylaminoethyl)aniline with benzaldehyde and oxidising the product. Michler's hydrol is condensed with N-(B-diethylaminoethyl)acetanilide, b.p. 134°/3 mm., to give a leuco-base, m.p. 120-125° (decomp.), which is oxidised to a dye base, m.p. 135-138°. o-Toluic acid is condensed with methyl- $(\beta$ -diethylaminoethyl)aniline. From 8-aminoquinoline there are obtained by condensation with β -diethylaminoethyl chloride and with β-chloro-δ-dimethylaminoisopentane, respectively, 8-(B-diethylaminoethyl)aminoquinoline, b.p. 180-182°/3-5 mm., and β-8 $quinolinylmethylamino-\delta$ -dimethylaminoisopentane, b.p. $175 - 183^{\circ}/7$ mm. Similarly 8-amino-6-methoxyquinoline (cf. E.P. 267,457; preceding abstract) may be condensed with β -chloro- ϵ -diethylamino-n-pentane hydrochloride, m.p. 93° (prepared by reduction of the methyl y-dimethylaminopropyl ketone, b.p. 83-85°, obtained by hydrolysis of ethyl B-dimethylaminoethylacetoacetate, b.p. 115-120°/5 mm., and treatment of the resulting secondary alcohol, b.p. 97°/15 mm., with thionyl chloride), to form β -(6-methoxy-8-quinolinyl)amino- ϵ -diethylamino-n-pentane, b.p. 189–190°/2 mm., or with γ-diethylamino-β-hydroxypropyl chloride hydrochloride (from epichlorohydrin and diethylamine hydrochloride) to give 8-(γ-diethylamino-β-hydroxypropylamino)-6-methoxyquinoline, b.p. 225—227°/2 mm. B-(6-Ethoxy-8-quinolinylamino)-δ-dimethylaminoisopentane, b.p. 204°/mm., is similarly prepared. 4-Chloro-6-methoxy-2-methylquinoline condenses with N-methyl-NN'-diethylethylenediamine to give 8-(methyl-3-diethylaminoethylamino)-6-methoxy-2-methylquinoline, b.p. 179-180°/ mm. δ -Dimethylamino - Δ^{α} - isoamylene (G.P. 0.5 254,529) is oxidised to the corresponding oxide, b.p. 44-46°/17 mm., which condenses with 8-amino-6methoxyquinoline to form 8-(δ-dimethylamino-β-hydroxyisoamylamino)-6-methoxyquinoline, b.p. 200-202° 1.5 mm. β -(6-Methoxy-8-quinolinylamino)- ϵ -dimethylamino-n-pentane has b.p. 196-198°/3 mm. A phenazine dye is obtained from m-tolylencdiamine and the pnitroso-compound of methyl-3-diethylaminoethylaniline. m-(Methyl-β-diethylaminoethyl)aminophenol, b.p. 156.5 $-157 \cdot 5^{\circ}/1$ mm., condenses with *p*-nitrosodimethylaniline to form an oxazine dye, isolated as double zinc From m-ethylaminophenol and B-diethylchloride. aminoethyl chloride is obtained m-(ethyl-3-diethylaminoethylamino)phenol, b.p. 180-182°/4 mm.; this condenses with phthalic anhydride to give a rhodamine, which may be esterified. m-Amino-B-diethylaminoethylaniline, b.p. 158.5-159.5°/1 mm., prepared from m-nitroaniline. is converted by the glycerol and oxalic acid method into an acridine dye. C. HOLLINS.

Preparation of derivatives of 6-alkoxy-4-methylquinoline. CHEM. FABR.AUF AKTIEN (VORM. E.SCHERING), Assees. of C. ZÖLLNER (G.P. 436,516, 25.12.24).—αMonophthalones of 6-alkoxy-2: 4-dimethylquinolines are oxidised with chromic acid, permanganate, etc., and the resulting 6-alkoxy-4-methylquinoline-2-carboxylic acids are decarboxylated. 6-Methoxy-4-methylquinoline-2carboxylic acid, m.p. 220° (decomp.), prepared from 6methoxy-4-methyl-2-phthalylmethylquinoline, gives 6methoxy-4-methyl-2-phthalylmethylquinoline, gives 6methoxy-4-methylquinoline, b.p. 298°. The corresponding 6-ethoxy-acid, m.p. 215-216° (decomp.), and base, m.p. 86°, are similarly obtained. C. HOLLINS.

Preparation of vanillin.A.-G. FÜR ANILIN-FABR.(Addn. No. 30,919, 7.9.25, to F.P. 583,856.Conv., 30.10.24.Cf. E.P. 219,676; B., 1925, 26).-4-Hydroxy-3-methoxyphenyltrichloromethylcarbinol, $CCl_3 \cdot CH(OH) \cdot C_6H_3(OH) \cdot OMe$, is first oxidised andthen hydrolysed, or oxidised and hydrolysed in thesame operation.The oxidant must be such as will notattack the aryl nucleus.C. HOLLINS.

Ethyl-n-butylbarbituric acid derivatives. E. H. VOLWILER, ASST. to ABBOTT LABORATORIES (U.S.P. 1,621,094, 15.3.27. Appl., 18.7.23).—5-Ethyl-5-nbutylbarbituric acid is heated with diethylamine to give diethylammonium hydrogen 5-ethyl-5-n-butylbarbiturate, m.p. about 60°, which is of value as a non-toxic hypnotic. Di-n-butylammonium hydrogen 5-ethyl-5-n-butylbarbiturate, m.p. 70°, has similar properties. T. S. WHEELER.

Preparation of arsenical [arsphenamine] solutions. O. Lowy, Assr. to STENECK TRUST Co. (U.S.P. 1,621,121, 15.3.27. Appl., 20.12.20. Cf. U.S.P. 1,559,899; B., 1926, 108).—Diaminodihydroxyarsenobenzene dihydrochloride is treated in solution with $4 \cdot 3$ —8 g.-mols. of sodium hydroxide, and kept out of contact with air for at least 4 hrs. to permit of complete formation of the disodium salt of the arsenobenzene derivative. *E.g.*, it is sealed in an ampoule in an atmosphere of nitrogen until required for use. Solutions so prepared are non-toxic. T. S. WHEELER.

Concentration of dilute acetic acid. H. SUIDA (U.S.P. 1,624,810-1, 12.4.27. Appl., 18.6.24. Conv., 26.6.23).—See E.P. 218,271-2; B., 1925, 899.

Manufacture of ester condensation products. R. M. CARTER, Assr. to U.S. INDUSTRIAL ALCOHOL CO. (Re-issue 16, 591, 12.4.27, of U.S.P. 1,472,324, 30.10.23).— See B., 1924, 77.

Manufacture of menthol. K. SCHÖLLKOPF, ASST. to RHEINISCHE KAMPFER-FABR. G.M.B.H. (U.S.P. 1,625,771, 19.4.27. Appl., 28.11.22. Conv., 23.11.21).— See E.P. 189,450; B., 1923, 743 A.

Manufacture of halogen substituted oxindole-3-acetic acids and homologues thereof. W. CARPMAEL. From CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING) (E.P. 266,539, 27.2.26).—See G.P. 433,099; B., 1927, 286.

Manufacture of OO-diacyl derivatives of diphenolisatin. E. PREISWERK, ASST. to HOFFMANN-LA ROCHE CHEMICAL WORKS (U.S.P. 1,624,675, 12.4.27. Appl., 18.10.23. Conv., 27.11.22).—See E.P. 221,976; B., 1925, 26. Phenols from ammoniacal liquor (G.P. 436,522).— See III.

Butyric acid etc. (U.S.P. 1,625,732).-See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic sensitivity. T. S. PRICE (J.S.C.I., 1927, 46, 145-150 T).—A review of recent work.

Effects of dilution and stirring of a photographic developer. A. H. NIETZ and R. A. WHITAKER (J. Franklin Inst., 1927, 203, 509-535).—See B., 1927, 60.

Flame movement. ELLIS.—See II.

PATENTS.

Photographic film and base. S. E. SHEPPARD and S. S. SWEET, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,623,500, 5.4.27. Appl., 27.4.26).—The film consists of a sensitive layer, such as a gelatino-silver halide emulsion, a flexible colloidised supporting layer of a cellulose compound, and between them and firmly attached to both, anon- strippable elastic waterproof cushioning stratum, such as a composition containing rubber, preferably depolymerised, and mixed with a resin. W. CLARK.

Production of coloured photographs on paper or the like. E. A. LAGE (U.S.P. 1,623,123, 5.4.27. Appl., 25.7.21. Conv., 30.7.20).—See E.P. 188,692; B., 1923, 73 Δ.

Films non-sensitive to electrical action (F.P. 611,136-7).—See V.

XXII.—EXPLOSIVES; MATCHES.

Solubility of nitroglycerol in water. W. LEDBURY and C. W. FROST (J.S.C.I., 1926, 46, 120 T).—Nitroglycerol, present in aqueous solution, can be determined by a method which involves the reduction of the nitrogen of the ester to ammonia. Data for the solubility of nitroglycerol in water have been obtained over a temperature range of 15° to 80°, and show that the solubility is not a linear function of the temperature. The solubility at 15° is approximately 0.13 g. per 100 c.c., whilst that at 80° is 0.35 g. per 100 c.c. The increase in solubility with temperature is most marked at the higher temperatures. No appreciable hydrolysis of the ester occurred over the temperature range in question.

PATENTS.

Triazo-nitrate [azidonitrate] explosives. Explosive composition. F. H. BERGEIM, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,620,714—5, 15.3.27. Appl., [A], 18.6.26; [B], 9.9.26).—(A, B) β -Azidoethyl alcohol is nitrated with a mixed acid containing 60% of sulphuric and 40% of nitric acid, to give β -azidoethyl nitrate, which is more sensitive and more powerful than nitroglycerin, and can be employed as a substitute for it. T. S. WHEELER.

Primer for cartridges. O. C. HORNEY (U.S.P. 1,619,867, 8.3.27. Appl., 17.7.25).—The primer consists of Monel metal. T. S. WHEELER. Brisant explosive. B. J. FLÜRSCHEIM (U.S.P. 1,624,401, 12.4.27. Appl., 9.10.24. Conv., 26.10.23).— See E.P. 226,913; B., 1925, 268.

XXIII.—SANITATION; WATER PURIFICATION.

Chlorination reduces foaming in Imhoff tanks. C. COHEN (Eng. News-Rec., 1927, 98, 563-564).-At Lufkin, Texas, the addition of lime to the vents and sludge digestion chambers reduced "foaming" only to a slight extent. With a view to inhibition of the organisms which give rise to foaming and elimination of bad odours, chlorination of the raw sewage was tried. A reduction of the rate of foaming was soon noticeable, and became more so with continued application of chlorine at the rate of 6 p.p.m. After a few weeks prechlorination was discontinued, and lime treatment recommenced, but foaming recurred. Feeding the chlorine directly into the sludge-digestion chambers was then tried without success. On reverting to prechlorination of the sewage, satisfactory conditions were again established. Latterly, foaming has been controlled by the application of chlorine during the period 8.0 a.m. to 5.0 p.m. only, at the rate of 3 p.p.m. of chlorine calculated on the daily flow. The tank effluent contains no residual "free' chlorine, and is of better quality than formerly.

W. T. LOCKETT.

Super-chlorination of [water to remove] chlorophenol tastes. L. B. HARRISON (J. Amer. Water Works Assoc., 1927, 17, 336-340).-Chlorine was added in increasing amounts to water containing 0.075 p.p.m. of phenol, and the solutions were kept for 30 min. Chlorophenol tastes made their appearance with 0.125 p.p.m. of chlorine, were not noticeable with 0.5-0.75p.p.m., but again appeared with 0.8-0.95 p.p.m. With 1.0-2.0 p.p.m. no chlorophenol tastes were detected. 0.8 p.p.m. of chlorine was sufficient to destroy the chlorophenol tastes produced by wood distillation wastes, and $1\cdot 2$ p.p.m. those produced by gas wastes, using concentrations of wastes of 1:10,000. Experiments, in which taste was observed after dechlorination indicated that the minimum time required for the reaction between chlorine (1.2 p.p.m.) and phenolic waste is at least 2 hrs. With a low temperature a larger dose of chlorine is required and a longer period of reaction. (Cf. Howard and Thompson; B., 1927, 204.) W. T. LOCKETT.

Prevention of phenol taste with ammonia. J. W. MCAMIS (J. Amer. Water Works Assoc., 1927, 17, 341— 350).—In the water supply of Greeneville, Tenn., phenol-like tastes developed during the summer months, some considerable time after chlorination. The origin of the tastes could not be definitely established. The conditions obtaining precluded largely the possibility of the presence of industrial wastes and the contamination of the water with impure air. Algal growth was not responsible. Elimination of the taste has been successfully brought about at very low cost by treatment of the water with ammonia, along the lines of the Metropolitan Water Board's experiments, prior to chlorination.

W. T. LOCKETT.

Moderns methods of sewage disposal. W. BUTLER and J. H. COSTE (J.S.C.I., 1927, 46, 49-59 T).-The efficacy of sedimentation processes is limited by the slow rate of fall of fine particles and by the fact that much of the matter, which should be removed from sewage or altered chemically before a satisfactory effluent can be discharged, is in solution. Land treatment can yield satisfactory results under some conditions, but easily drained land is costly in this country. The intensive biological treatment of sewage can effect in a few hours a degree of purification which takes much longer on land, and it is in biological processes that the solution of the sewage problem is to be found. The activated sludge process has been studied, and a comparison made between the results obtained by air diffusion or blowing methods as used by Fowler and agitation processes which have been applied with success by Haworth, Bolton, and others. The authors consider that air diffusion is particularly suitable for re-aerating used activated sludgean essential part of the cycle-whilst mechanical agitation is better adapted for actual treatment. In both types of plant it was found that the quantity of sewage effectively treated was increased three- or four-fold if the greater part of the suspended matter was previously removed by sedimentation. Analyses of activated sludge are given, and experimental work is described which shows that, notwithstanding the great part played by air in the changes produced, an immediate purification, yielding in some cases a stable effluent, occurs when activated sludge and sewage or sedimented sewage are mixed. Experiments are also adduced showing the advantages of re-aeration of activated sludge and the small effect of variations in the depth of diffuser tanks.

Dissolved oxygen absorption test [of sewage]. E. A. COOPER and W. H. READ (J.S.C.I., 1927, 46, 154-156 T).-In experiments with the object of accelerating Winkler's dissolved oxygen absorption test, evidence of an increased oxygen absorption on the addition of potassium dihydrogen phosphate to the sewage has been obtained. Other alkali phosphates have little or no effect. Potassium dihydrogen phosphate causes an increase in the number of bacteria in the sewage, but the addition of cultures of sewage organisms has an inappreciable influence on dissolved oxygen absorption. Gelatin adsorbs a greater amount of phosphate from solutions of the foregoing potassium phosphate than from equimolecular solutions of other phosphates. The specific effect of the potassium salt on dissolved oxygen absorption is thus not merely due to its capacity of increasing the number of bacteria present, but is more likely to be due to an increased metabolism of the individual bacteria, owing to the more ready availability of the phosphate.

Chemical oxidation of the constituents of sewage. Action of hydrogen peroxide. E. A. COOPER and W. H. READ (J.S.C.I., 1927, 46, 156-157 T).-Hydrogen peroxide oxidises certain constituents of sewage, but there is no advantage in employing a hydrogen peroxide test instead of the Forschammer permanganate process in the analysis of sewage effluents. The catalytic action of ferrous sulphate on the thermal decomposition of hydrogen peroxide is retarded by the presence of sewage, and this has been found to be due to the ammonia and primary amines present. The presence of one part per 100,000 of hydrogen peroxide diminishes the dissolved oxygen absorption of the sewage effluents, owing apparently to an inhibitory effect on the bacteria.

Destruction of germs by ammonia and chlorine. W. OLSZEWSKI (Chem.-Ztg., 1927, 51, 269-270).—The results of the bacteriological and chemical examination of water in a swimming bath, after treatment with chlorine and ammonia, are described. The gases, which are used in the ratio of 2 pts. of chlorine to 1 pt. of ammonia by wt., probably owe their bactericidal effect to chloroamine. Compared with the use of chlorine alone, the method has the advantage that no odour of chlorine is perceptible, the water remains clearer, and a careful regulation of the amount to be added is not required. F. R. ENNOS.

Chemical action of p-quinones on proteins. E. A. COOPER and S. D. NICHOLAS (J.S.C.I., 1927, 46, 59-60 r).-It is confirmed that whilst p-benzoquinone is a very much more active germicide than toluquinone, yet there is little difference in their reactivity towards gelatin and other proteins. Charcoal and asbestos adsorb the two quinones equally, so that the foregoing results may be due to the disturbing effect of the adsorption. With cystine, alanine, and creatinine, the difference in the reactivity of the two quinones is also surprisingly small, suggesting that the anomalous results with proteins are not entirely caused by adsorption, but have also in part a structural explanation. On the other hand, benzoquinone reacts 4-5 times as rapidly with glycine and asparagine as toluquinone, thus giving results similar to those obtained in the bacteriological work. The observations suggest that simple water-soluble substances may be of as much fundamental importance in the life of the cell as the more complex colloids, upon which greater emphasis is usually placed.

Water analysis. D. BURTON and J. K. HASLAM J.S.C.I., 1927, 46, 111-114 T).-The methods for determining temporary, permanent, and magnesia hardness and oxygen in boiler feed water are critically examined so that sufficiently accurate results for control purposes can be obtained in softening water supplies to the usual limit of about 3° hardness. The suitability of various indicators for determining the temporary hardness is discussed. Pfeifer and Wartha's method is shown to give reliable results for the permanent hardness providing a Munktell No. 1 F filter paper is used and the water contains no alkali carbonate. High results are obtained for the magnesia hardness due to (1) carbonation of the lime-water, (2) removal of lime by the acid required to change the colour of the indicator in removing the temporary hardness, (3) precipitation of lime from the lime-water because its solubility decreases with rise in temperature.

sanitary analysis. S. L. NEAVE and A. M. BUSWELL (J. Amer. Water Works Assoc., 1927, 17, 388-395).-A discussion as to the value of the free and albuminoid ammonia and total organic nitrogen figures obtained in the routine analysis of domestic sewage. Analyses have been made both by routine methods and by determinations of the nitrogenous components of the samples using the methods of the physiological chemist. In the latter the actual ammonia was aspirated into standard acid, the carbamide then converted into ammonia by adding urease, and the resulting ammonia similarly aspirated into standard acid, the difference between total (Kjeldahl) nitrogen and the sum of the nitrogen contained in the ammonia and carbamide being taken as the protein fraction. The analyses indicated that "free ammonia" as obtained by distillation represents actual ammonia, a small amount of amine, and a part (5-20%) of the carbamide; and "albuminoid ammonia" represents an indefinite portion (25-55%) of the carbamide and a very small amount of the protein fraction. A revision of the accepted methods for determining the nitrogen of sewage, with a view either to correcting their defects or abandoning them in favour of more significant determinations, is recommended. W. T. LOCKETT.

Bactericidal action of selenium and tellurium compounds. Stover and HOPKINS.—See XVI.

PATENTS.

Production of base-exchange substances. Coch-RANE CORP., Assees. of S. DAHL-RODE (E.P. 245,092, 11.12.25. Conv., 23.12.24).—Ground furnace slag containing silicates of aluminium and alkaline-earth metal is treated with a hot strong solution of caustic alkali, heated until substantially all the water has evaporated, dried, and graded. Granules of the required size are then leached to remove excess alkali, and subsequently treated with a dilute mineral acid or dilute acetic acid to remove the remaining alkali and undesirable calcium compounds. Finally the product, preferably after washing, is treated with brine, washed, drained, and airdried. W. T. LOCKETT.

Apparatus for regenerating zeolites and the like. W. H. GREEN, ASST. to GENERAL ZEOLITE Co. (U.S.P. 1,620,886, 15.3.27. Appl., 17.7.22).—A tank of regenerating solution has a pipe communicating with the treating vessel below the pervious bed of treating material, and a return pipe from the treating vessel to drain the space above the reaction bed. Suitable discharge pipes and valves for the waste are fitted to the reaction vessel. W. G. CAREY.

Product for destroying mosquito larvæ. E. C. C. ROUBAUD and R. A. VEILLON (U.S.P. 1,625,627, 19.4.27. Appl., 21.11.23. Conv., 30.11.22).—See E.P. 207,802; B., 1924, 804.

Filters [for sewage etc.] (E.P. 266,857).-See I.

Hydrocyanic acid for fumigation (U.S.P. 1,620,365). —See VII.

Significance of nitrogen determinations in