BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 5, 1926.

I.-GENERAL; PLANT; MACHINERY.

Adsorption of gases [ammonia, sulphur dioxide, chlorine] by wood charcoal at pressures exceeding 1 atm. F. A. HENGLEIN and M. GRZENKOVSKI (Z. angew. Chem., 1925, 38, 1186-1188). -The apparatus comprises essentially two glass limbs, one of which contains the charcoal and is evacuated and sealed, whilst the other is graduated and contains the liquefied gas. When a protruding neck of the charcoal chamber is broken, connexion is established between the limbs, and some of the liquefied gas volatilises and is adsorbed by the charcoal, the quantity adsorbed being calculated from the loss in volume of the liquefied gas. With ammonia and at a temperature of 30°, the charcoal used reached saturation at 8.19 atm., and then contained 34% of its weight of gas. With sulphur dioxide the same charcoal showed no saturation point, and at 25° and 3.35 atm. had taken up 62% of its weight of gas. The results with chlorine were less regular, and some chemical action of the gas on the charcoal is assumed. At 15° and 4 atm. the charcoal took up about 70% of its weight of chlorine within a few minutes. W. T. K. BRAUNHOLTZ.

Application of rotating tubes in crystallisation. and evaporation. H. BARKHOLT (Chem.-Ztg., 1926, 50, 45-46).—The advantages of the apparatus described (cf. G.P. 333,304 and 351,947; B., 1921, 422 A; 1922, 737 A) lie in their continuous operation, low maintenance and installation costs with respect to the yield, immunity from corrosion and consequent purity of the product, and wide range of applicability. Since the evaporator works on the same principle as the crystalliser, the two operations may be performed in one tube, the evaporating portion of which is heated by flue-gases or other heating agent. E. S. KREIS.

Laboratory kiln for obtaining high temperatures. F. H. NORTON (J. Amer. Ceram. Soc., 1925, 8, 826—828).—A small cubical chamber has a tuyère (through which gases of combustion enter from a Premix blower) placed near the crown, so that the flames pass over the charge and down to flues in the bottom, from which the gases pass out through four stacks, one at each corner of the kiln. The efficiency of the furnace is due to careful design of the tuyère, the combustion space, the baffles, and the flues, and not to preheating or to heavy insulation. A temperature of 1400° is reached in one hr. and 1800° in 4 hrs. The actual flame temperatures are estimated to be almost as high as the maximum theoretical values for the natural gas used in the experiments. F. SALT.

Technical dispersoid analysis. F. V. von HAHN (Kolloid-Z., 1925, 37, 377—379).—A discussion of the measurement of the degree of dispersion by methods specially applicable to the control of industrial processes, namely methods depending mainly on filtration, ultrafiltration, microscopy, and sedimentation. N. H. HARTSHORNE.

Plasticity. A. DE WAELE (Kolloid-Z., 1925, 38, 27).-The extrusion of an heterogeneous system through a capillary orifice under pressure comprises a combination of two régimes, i.e., that of the shear of the continuous phase resulting in a velocity gradient within it, and mere extrusion of accompanying unshearable disperse phase not susceptible to a velocity gradient. By deriving the empirically-obtained proximate equation for the "shear" of a heterogeneous system through a capillary from these principles, ψ in the equation $P/V^{\psi} = \text{constant}$, is shown to denote the volume proportion of shearable, truly viscous phase. Many, if not all heterogeneous systems show in addition, evidence of a static elasticity or yield value (f), the actual resultant of which is, however, variable in magnitude with the stress applied, thus: yield value at any moment $(F)=fe^{-\text{stress}}$, where $e=\log$ base. This static yield value is recoverable on rest according to the relationship: $F=fe^{-\eta/t}$. The complete equation showing the discontinuity in capillary shear owing to loss of yield value with stress then is:

$$\pi g R^4 (P - f e^{-r R/24}) / 8 V^{\varphi} l = \eta$$

The mechanism of this deflocculation on shear and re-flocculation with subsequent rest is suggested as being de-orientation and re-orientation respectively of molecules of the viscous phase at the boundary surfaces of the unshearable phase. A. DE WAELE.

Corrosion and erosion of steam turbine blading. E. HONEGGER (Brown Boveri Rev., 1924, 11, 263—268; Chem. Abstr., 1925, 19, 3471).— Corrosion and erosion usually assist one another, the latter exposing fresh surfaces to the action of the former. The incidence of both factors on the lowand high-pressure parts of steam turbine blading during operation has been examined, and the resistance of "stainless" steels has been studied. It appears that below a critical steam speed for any metal no erosion can take place. A. A. ELDRIDGE.

Action of ammoniacal water on feed pipes and boiler plates. M. TILGNER (Chem.-Ztg., 1926, 50, 48).—Water containing ammonia to the extent of 8—15 mg. per litre has been observed to produce heavy corrosion. E. S. KREIS.

D

Evaporation of mineral oils and its bearing on the use of oil-sprinkled air-filters. W. ALLNER (Z. angew. Chem., 1926, **39**, 16—20).—The harmful consequences of the presence of oil in the supposedly purified atmosphere of electrical powerhouses fitted with air filters of the Delbag-Viscin type (consisting of cells filled with oil-coated rings which retain dirt etc.) have been wrongly attributed to the use of the oil in the filter. Experiments are described showing that under the working conditions the volatilisation of oil is negligible, whilst formation of oil mist or mechanical entrainment of droplets is impossible. S. S. WOOLF.

See also A., 1926, 118, Theory of atmolysis (SAMESHIMA). 119, Influence of thin surface films on evaporation of water (RIDEAL). 122, Determination of size of colloidal particles (BURTON and REID); Diffusion analysis (AUER-BACH); Measurement of particle size (KUHN). 124, Theory of peptisation (SEN). 142, Viscosimetry (STAUF).

Method of measuring porosity. ROBINSON.— See VIII.

PATENTS.

Manufacture of large [or single] crystals. GEN. ELECTRIC Co., Assees. of PATENT TREUHAND GES. FÜR ELEKTRISCHE GLÜHLAMPEN M.B.H. (E.P. 234,449, 2.3.25. Conv., 26.5.24).—The material to be converted into a single crystal is melted in a tube of which the largest diameter corresponds to the required thickness of the crystal and which is tapered to a smaller diameter and finally brought to a point at one end. The tube and its contents are then slowly cooled progressively along its length, starting at the small end. The material in the thin stem of the tube first solidifies to a single crystal before the melt in the main portion of the tube has solidified. As the tube is progressively cooled the crystal grows at the expense of the solidifying B. M. VENABLES. material.

Pulverising and drying of materials. O. Y. IMRAY. From INTERNAT. COMBUSTION ENGINEER-ING CORP. (E.P. 244,575, 28.11.24 and 21.4.25).-A pulverising mill of the type having a reservoir of material which is kept in agitation just before entry or re-entry into the actual grinding chamber (e.g., the Raymond pulveriser), is operated with the bulk of the air travelling continuously in a circuit, but to effect drying as well as pulverising, a portion of the air saturated with moisture is withdrawn from the return conduit after leaving the collector for pulverised material and replaced by fresh air, which may with advantage be heated, and preferably enters the same return conduit at a point close to that of withdrawal. To prevent waste or dust in the withdrawn air it may be used to transport the finished material to the points of use, e.g., to the furnaces in the case of powdered fuel.

B. M. VENABLES.

Refrigeration. B. H. COFFEY (U.S.P. 1,565,795, 15.12.25. Appl., 9.8.20).—The pressure on a liquid

refrigerant is gradually reduced to vaporise a portion without substantial heat exchange, and a further portion is vaporised at low pressure and temperature to produce the refrigeration. The pressure on the resulting mixture of liquid and vapour is then gradually increased, thus condensing part of the vapour without substantial heat exchange, and the remaining vapour is condensed at the higher pressure and a higher temperature. H. HOLMES.

Filter. W. P. COTTRELL (U.S.P. 1,565,988, 15.12.25. Appl., 28.10.24).—A compressible filtering medium is packed between spaced walls of impermeable material provided with perforations for passage of fluid. The perforations are arranged out of register to compel the fluid to follow a lengthy path between the walls, and means are provided for adjusting the pressure of the walls on the filtering medium. H. HOLMES.

Evaporator. M. J. KERMER (U.S.P. 1,566,539, 22.12.25. Appl., 15.8.23).—A "deck" or nest of tubes, 10, is situated within an evaporating chamber,



and a steam chamber, 13, surrounds the tubes. A trough, 14, is formed inside the steam chamber below the level of the tubes and water is forcibly projected through the trough, which is completely open to the steam. B. M. VENABLES.

Preventing loss by evaporation from storage tanks. R. E. WILSON, Assr. to STANDARD OIL CO. (U.S.P. 1,566,944, 22.12.25. Appl., 30.1.24).—A storage tank for volatile liquids is vented through a mass of silica gel, the temperature of which is regulated to be at least as high while the air is being drawn into the tank as the temperature prevailing during expiration. B. M. VENABLES.

Continuous drying kiln and method of drying ware. T. LARSSON, ASST. to NORTON CO. (U.S.P. 1,567,023, 22.12.25. Appl., 30.10.20).—A continuous drying kiln is provided with a series of heaters arranged along its length, each being thermostatically and separately controlled. Fans are provided to circulate the air continuously across the kiln, and the air is also circulated longitudinally of the kiln, the return passage for this circulation being external to the actual drying chamber. The moisture content of the air is maintained constant by withdrawing a portion of the moist air and admitting a corresponding quantity of fresh air to the return passage. The amounts of the forward or longitudinal circulation and of air withdrawn for re-conditioning are also thermostatically controlled.

B. M. VENABLES.

Treatment of gases at high temperatures. M. FOURMENT (F.P. 593,298, 22.12.24).—Gases are passed through a chamber constructed of electricalconducting material, which is brought to a high, uniform temperature by means of a high-frequency induction current. L. A. COLES.

Production of highly efficient catalysts. K. WOLF (G.P. 418,724, 30.7.21).—Insoluble, porous material is impregnated with solutions containing salts of catalytic metals and colloidal substances, and, after drying at low but slowly rising temperature, is heated at dull redness in the presence of reducing or oxidising agents, according to whether the metals or their oxides are required.

L. A. COLES.

[Platinum-ruthenium] catalyst. GES. FÜR NAUTISCHE INSTRUMENTE G.M.b.H., and O. MAR-TIENSSEN (G.P. 418,868, 11.10.24).—Alloys of platinum and ruthenium are used instead of platinum as catalysts. L. A. Coles.

Ammonia-absorption refrigerator. C. SENS-SENBRENNER (U.S.P. 1,568,476, 5.1.26. Appl., 16.12.24).—See E.P. 236,101; B., 1925, 656.

Refrigerating system. W. H. CARRIER, Assr. to CARRIER ENGINEERING CORP. (U.S.P. 1,569,214, 12.1.26. Appl., 2.9.22).—See E.P. 230,936; B., 1925, 385.

Method of heating material at successively different temperatures. A. McD. DUCKHAM and J. S. MORGAN, ASSIS. to THERMAL INDUSTRIAL AND CHEMICAL (T.I.C.) RESEARCH CO., LTD. (U.S.P. 1,568,886, 5.1.26. Appl., 9.8.21).—See E.P. 174,690; B., 1922, 205 A.

Heat exchangers. C. Roszak (E.P. 235,149, 7.4.25. Conv., 5.6.24).

Rotary drying or torrefying apparatus. P. SCRIVE (E.P. 238,871, 7.8.25. Conv., 20.8.24).

Doll-head bearings for drying cylinders. S. C. BULLEN (E.P. 245,548, 31,10.24).

Rotary dryer for whinstone, limestone, sand, clinker, and like material. R. H. and F. KIRKUP and J. THOMPSON (E.P. 245,561, 1.8.25).

Apparatus for separating dust from air. J. B. TAYLOR (E.P. 245,243, 10.11.24). Preventing and dissolving scale in steam boilers. P. SCHENITZA (E.P. 245,365, 10.7.25).

Furnaces. VICKERS AND INTERNAT. COMBUSTION ENGINEERING, LTD., and F. H. ROSENCRANTS (E.P. 245,496, 6.10.24).

Washing apparatus for granular material. H. J. GREAVES (E.P. 245,630, 24,2.25).

Combined evaporator and feed heating systems. BABCOCK AND WILCOX, LTD., and A. SPYER (E.P. 245,664, 16.6.25).

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

Conversion of ligneous plant substances into coal. III. Formation of sugars as an intermediate phase of the production of coal. C. G. SCHWALBE and R. SCHEPP (Ber., 1925, 58, 2500-2502; cf. B., 1924, 240) .- Humic acids are generally regarded as the precursors of coal and are possibly produced from sugars formed by hydrolysis. Experimentally it is shown that wood cellulose yields sugars when heated with 2% of oxalic acid at 170°. Dilute sucrose solutions when heated with oxalic acid at 130° afford humic acids which are transformed by concentrated solutions of magnesium chloride at 180° into substances resembling but not identical with lignite or coal. The production of the latter appears to depend on the simultaneous presence of lignin. When, therefore, spruce wood is heated successively with oxalic acid and magnesium chloride solutions a product is obtained which appears to have properties intermediate between those of lignite and coal. The carbon content increases with increasing duration of the treatment with magnesium chloride. H. WREN.

Heat of adsorption of gases by coal and charcoal. A. G. R. WHITEHOUSE (J.S.C.I., 1926, 45, 13-20 T).-The heat of adsorption of carbon dioxide, sulphur dioxide, methane, oxygen, nitrogen, and carbon monoxide by charcoal and different types of coal at 0° was measured with a Bunsen ice-calorimeter. The values obtained for the heat evolved per c.c. of gas adsorbed are as follows :---Carbon dioxide 0.346-0.255 cal., sulphur dioxide 0.627-0.359, methane 0.245-0.199, oxygen 0.19-0.17, nitrogen 0.22-0.17, and carbon monoxide 0.198-0.188 cal. A simple exponential equation connecting the total heat evolved per gram of adsorbent with the total volume of gas adsorbed per gram of adsorbent is found to hold over the range covered by the experiments. The gases studied are adsorbed more slowly by coal than by charcoal, the difference being most pronounced in the cases of methane and nitrogen.

Refractories for gas retorts, with special reference to silica. W. EMERY (Trans. Ceram. Soc., 1924-5, 24, 361-402; cf. B., 1924, 674; also Robinson, p.158)—An examination of the actual results with silica retorts in practice shows that in general p 2

the throughput and rate of carbonisation are increased for horizontal retorts, this being chiefly due to the greater diffusivity of silica compared with fireclay at temperatures above 1000°, giving higher thermal conductivity. Silica retorts retain their rigidity and shape much better at higher temperatures than fireclay, the type of retort and setting, especially the position of the cross walls, exerting a considerable influence. The tendency to form less scurf on silica has also a favourable effect. Silica is more resistant to abrasion and to corrosion by salty coals or by ash in the producer gas. The high thermal expansion of silica can be dealt with by special design of the settings, and spalling troubles have been reduced by more careful attention to the manufacture of the silica. Experience to date seems to show that the advantages of the silica retort outweigh the disadvantages of higher capital outlay and the necessity for careful handling, and special precautions during the setting of the retorts. B. W. CLARKE.

Sodium chloride solution as a confining liquid for gas analysis. F. G. HOFFMANN (Z. angew. Chem., 1926, 39, 23—24).—The solubility of carbon dioxide in dilute hydrochloric acid and in dilute sulphuric acid is almost as great as in pure water. The solubility in aqueous sodium chloride solution is not altered by addition of sulphuric acid, and the solubility of sodium chloride in dilute hydrochloric acid solutions is higher than in aqueous solutions of the same concentration. Thus the use of dilute acids or dilute acid solutions of sodium chloride as confining liquids offers no advantage over that of aqueous solutions. A 22% solution of chemically pure sodium chloride is recommended as the best confining liquid. In this solution carbon dioxide is less soluble than in one of commercial salt of the same concentration in the proportion 14:15.

E. H. SHARPLES.

Detection and determination of traces of carbon disulphide in small gas volumes. W. J. HUFF (J. Amer. Chem. Soc., 1926, 48, 81-87).-The method of Harding and Doran (B., 1907, 1190) has been modified as follows :- A volume of gas, e.g., coal gas or carburetted water-gas, containing at least 1 mg. of carbon disulphide is freed from carbon dioxide and moisture, and passed into a saturated solution of potassium hydroxide in absolute alcohol. The solution is then made acid with glacial acetic acid, excess of standard 0.01N-copper sulphate solution added, and the mixture left overnight. The copper xanthate, precipitation of which is aided by the presence of excess cupric ion, low acidity, and keeping the volume of solution as small as possible, is filtered off, and the excess of copper determined iodometrically. A sensitive qualitative method of detection is based on the same process. R. CUTHILL.

Determination of water in mineral oils. S. REINER (Elektrotechn. Z., 1925, 46, 1447—1448; Chem. Zentr., 1925, II., 2334).—For the determination of small quantities of water in mineral oils the oil is heated at 120° for 3 hrs. in a flask while a current of

compressed air is passed through. The air is previously dried by passing it through a tower filled with calcium chloride, then through two U-tubes packed with phosphorus pentoxide, and the moisture removed from the oil is collected in two small Utubes containing phosphorus pentoxide, protected from the atmosphere at the far end by a washing flask containing strong sulphuric acid.

A. R. POWELL.

See also A., Feb., 1926, 116, Heat of combustion of salicylic acid (BERNER); Heats of combustion of standard substances (SWIENTOSLAWSKI).

Evaporation of mineral oils. AllNER.-See I.

Use of silica refractories. ROBINSON.-See VIII.

PATENTS.

Apparatus for drying or preheating coal or like material. S. R. ILLINGWORTH, and ILLING-WORTH CARBONIZATION Co. (E.P. 244,505, 9.9.24).— The coal or other material to be dried or preheated passes through vertical metal retorts, built up of tubes, or of plates which may be of H-section. These are enclosed in a retort setting of metal or refractory material, and can be heated by radiant heat from flue-tubes through which circulate hot gases from other processes, or from gas burners placed in the flue-tube itself. Each flue-tube forms a separate, easily manipulated unit. The retorts are stepped to provide vents for the escape of steam or gases into the space between the retorts and flue-tubes; or these gases may be taken away through perforated tubes passing down inside the retorts. Dampers are provided whereby, if required, the flue-gases may be caused to flow from any flue-tube to the interior of the retort setting and thence to the chimney. Scrapers, which can be reciprocated in the retorts, are provided for dealing with coal of high moisture content. The retorts are fed from bunkers having outlets of smaller size than the retorts, and agitating arms which may be moved to and fro in the outlets. The discharge of material from the retorts is controlled by reciprocating plates. Coal can be dried in this apparatus without coming into contact with the heating gases, whereby oxidation is reduced to a A. B. MANNING. minimum.

Binder [for fuel briquettes]. S. W. CARPENTER and G. N. WHITE (E.P. 244,517, 16.9.24; cf. Goodwin and White, B., 1926, 34).—Soft vegetable tissue, e.g., straw, waste of potato, beet, or other crops, hemp waste, etc., is subjected to aerobic fermentation so that the whole of the non-gaseous products are retained in the mass, which is boiled with an alkali to form an agglutinous material. The product is used either alone, or dried and mixed with water, as a binder in the manufacture of fuel briquettes. The amount of binder required decreases with increasing briquetting pressure and with increased fineness of grinding of the fuel; from 4 to 8% is sufficient when using a pressure of 1—3 tons per sq. in. B. W. CLARKE. Electrical [blast-furnace] gas purification. LODGE-COTTRELL, LTD. From METALLBANK U. METALLURGISCHE GES. (E.P. 244,372, 29.8.25. Addn. to 177,117 and 238,480; B., 1923, 1A; 1925, 790).— The gas after being pre-cleaned electrically and cooled by washing with water, is delivered directly, without preheating, into the apparatus for precipitating the fine dust particles electrically. H. HOLMES.

Treatment of gas liquor. Recovery of phenols from ammoniacal liquor. L. R. W. HEFFNER (U.S.P. 1,566,795-6, 22.12.25. Appl., 16.12.24).—(A) Phenol is recovered from gas liquor by subjecting the liquor to a temperature of 98° or higher, in the presence of sufficient ammonia to drive off the phenol as phenoxide. (B) The liquor is distilled for the recovery of ammonia, the temperature of the liquor and of the vapours at the outlet of the still being kept at about 98° or higher, in order to drive off ammonium phenoxide with the ammonia. A. B. MANNING.

Recovering benzene from gases. É. E. M FRÉCHOU (F.P. 594,531, 15.5.24).—Oils containing benzene are distilled in a vacuum and the vapours condensed at different temperatures. A. GEAKE.

Recovering by-products of coal distillation. É. M. E. FRÉCHOU (F.P. 594,012, 3.5.24).—Activated charcoal is used for absorption under pressure. A. GEAKE.

Oil-shale retort. J. T. POPE (U.S.P. 1,564,271, 8.12.25. Appl., 24.10.22. Renewed 29.6.25).— The retort consists of a sheet metal casing mounted above a furnace with interposed firebrick slabs to prevent direct heating. An inclined table, capable of small longitudinal movement, is suspended in the metal casing and conveys crushed shale from one end of the retort to the other, entry and exit of the shale being controlled by roller gates. Superheated steam is distributed along the surface of the shale and a vapour offtake is provided. T. A. SMITH.

Obtaining oil from oil sands. T. W. PRITCHARD (Can. P. 244,540, 27.2.24).—The sand is heated in a retort to a temperature between 340° and 430°, the oil vapours and gases are passed through a condenser, and a part of the uncondensed gas is led back through the retort in order to prevent the temperature rising to a height at which cracking of the oil occurs. A. B. MANNING.

Obtaining oils from oil shales. JURA OELSCHIEFER-WERKE A.-G. (G.P. 415,847, 20.8.20).— The shale is distilled by the direct passage through it of the hot gases from the combustion of the shale residue. The distillation of the shale and the combustion of the residue take place in separate chambers, one above the other, the material passing through the chambers in succession. A. B. MANNING.

Purification of liquid fuels, in particular those containing alcohol. Soc. DE RECHERCHES ET DE PERFECTIONNEMENTS IND. (F.P. 589,712, 16.1.24).— The liquid fuel is distilled in the presence of less than 1% of a purifying agent (metal, oxide, hydroxide, carbonate, alkyloxide, or carbide of an alkali or an alkaline-earth, or a mixture of these). A. B. MANNING.

Motor fuel. J. D. RIEDEL A.-G. (G.P. 416,838, 16.8.21. Addn. to 414,245; cf. E.P. 202,805, B., 1923, 1012 A).—To the mixture of hydrogenated naphthalenes and low-boiling aliphatic hydrocarbons a further addition of a small quantity of ether or of an alcohol of higher boiling point is made so that a homogeneous mixture resistant to cold is produced. This prevents carbon formation in the cylinder when too rich a mixture is used, and at the same time gives an increase of power. A. B. MANNING.

Catalyst and adsorbent for the production of hydrocarbons. LE PÉTROLE SYNTHETIQUE (F.P. 589,831, 28.11.24. Conv., 20.9.24).—A catalyst and adsorbent for the preparation of hydrocarbons similar to natural petroleum products consists of a mixture of charcoal and nickel, cobalt, iron, manganese, copper, boron, calcium, sodium, titanium, uranium, or vanadium, and a solid hydrocarbon.

A. B. MANNING.

Conversion of heavy [mineral] oils into lighter products. M. A. H. DE DAMPIERRE (F.P. 590,616, 19.12.24. Conv., 20.12.23).—The heated oil is treated with hydrogen under pressure and the evolved vapours are passed over a large surface of catalyst. A. R. POWELL.

Wax sweating and crystallising apparatus. BURMAH OLL CO., H. L. ALLAN, and J. MOORE (E.P. 243,447, 29.8.24. Addn. to 208,195; B., 1924, 165).—In an apparatus of the type described in the chief patent, consisting of a number of superposed wax compartments separated from one another by heating and cooling cells, the charging and discharging pipes are located in a space between the sides of the compartments and the container; separate inter-cell charging and discharging pipes are dispensed with. Solid metal fins project downwards from the cells into the wax beneath to increase the heating or cooling effect. T. A. SMITH.

Manufacturing lubricating oils. J. W. WEIR, Assr. to J. C. BLACK (U.S.P. 1,564,501, 8.12.25. Appl., 21.3.22).—Lubricating oil stock is treated with sulphuric acid and the greater portion of the sludge removed by settling. Finely comminuted solid absorbent material is then added and the mixture heated until sulphur dioxide is evolved. The absorbent material and absorbed matters are then separated from the stock. T. A. SMITH.

Preparation of stable lubricating and insulating oils. F. FRANK (G.P. 417,835, 20.2.23).— Refined lubricating and insulating oils before or after use may be improved by treatment with methyl alcohol, which extracts the compounds which are readily decomposed. A. R. POWELL.

Dehydration process [for oil emulsions]. H. C. Eddy, Assr. to Petroleum Rectifying Co. of California (U.S.P. 1,565,992, 15.12.25. Appl., 8.9.24).—An emulsion containing suspended solids is subjected to an electric field to agglomerate and precipitate water and solids, the precipitate is removed, and the residue is filtered to agglomerate the remaining water. H. HOLMES.

Centrifugal dehydrator [for oil emulsions]. C. W. GIRVIN, ASST. to PETROLEUM RECTIFYING CO. OF CALIFORNIA (U.S.P. 1,565,997, 15.12.25. Appl., 3.4.23).—The emulsion is subjected to an electric field to coalesce the particles of water, and the oil is separated centrifugally. H. HOLMES.

Distillation of carbonaceous material. R. M. CATLIN, ASST. to CATLIN SHALE PRODUCTS CO. (Reissue 16,252, 12.1.26, of U.S.P. 1,509,667, 23.9.24. Appl., 18.7.25).—See B., 1924, 933.

Refractory diaphrams for use in surfacecombustion apparatus. F. J. Cox (E.P. 245,182, 2.9.24).

Combustion apparatus. B. VALJEAN (E.P. 245,497, 6.10.24).

Pulverising and drying materials (E.P. 244,575). —See I.

Combustion of ammonia (G.P. 418,622).— See VII.

Manufacture of hydrogen (E.P. 228,153).— See VII.

Activating decolorising char (U.S.P. 1,565,911). —See XVII.

III.—TAR AND TAR PRODUCTS.

Light oils from low-temperature tars. A. KLEIN (Brennstoff-Chem., 1926, 7, 3—7).—A detailed analysis of the light oils from tar obtained from Saar coals by the Thyssen low-temperature (550°) carbonisation process shows these oils to consist of saturated and unsaturated paraffinoid hydrocarbons with only a very small proportion of aromatic hydrocarbons. B. W. CLARKE.

Reduction of cresols by the Bergius process. F. FISCHER and H. TROPSCH (Brennstoff-Chem., 1926, 7, 2—3).—The reduction of o-, m-, and p-cresol by hydrogen under pressure proceeds very slowly, with the formation of phenol, toluene, and methane and its homologues, showing that a reduction to hydrocarbons as well as the splitting off of the methyl group has taken place. The speed of the reaction is so slow, however, that it is impossible for the phenols formed in the primary decomposition of coal to be appreciably acted upon by hydrogen during the Bergius process. B. W. CLARKE.

Reduction of cresols. G. STADNIKOV, N. GAVRILOV, and A. WINOGRADOV (Brennstoff-Chem., 1926, 7, 7-9; cf. A., 1926, 60).—Cresol passed over finely-divided metallic iron (supported on asbestos) at 500°, is converted almost entirely into toluene. The first effect of the iron is to decompose the cresol, with the deposition of carbon; the carbon thus

produced, activated by the iron, then reduces the remaining cresol (92.5% of the whole) to toluene, *p*-cresol being more readily reduced than its isomerides. B. W. CLARKE.

PATENTS.

Tar bitumen emulsions. J. A. MONTGOMERIE (E.P. 244,561, 28.10.24. Addition to 226,032; cf. B., 1925, 91).—A small percentage of soap is added to a bituminous emulsion prepared as described in the chief patent, and a small proportion of melted coal tar or pitch or blast-furnace tar or pitch is incorporated. B. W. CLARKE.

Distilling crude tar oils. G. L'RIOKHUHN (G.P., 417,974, 25.1.25).—The crude oil mixture from the tar still is led into an oil still, separated from the tar still by a fireproof, heat-insulating material.

A. GEAKE.

Separation of the acid fractions, in particular phenols, from mixtures with neutral oils. GELSENKIRCHENER BERGWERKS-A.-G., ABTEIL. SCHALKE, and H. HOCK (G.P. 417,971, 15.8.22).— The oil mixture is treated with a 5—10% solution of sodium carbonate under pressure at about 250°. The phenols dissolve, and the solution of phenoxides is separated from the remaining oils. On cooling the solution the phenols separate, and the carbonate solution can be used for a further extraction. Thus, after a third extraction of a tar oil containing 40%of phenols with 10% sodium carbonate solution at 250° , 83% of the phenols had been removed, only the very feebly acid homologues remaining.

A. B. MANNING.

Recovering phenols from ammoniacal liquor (U.S.P. 1,566,795-6).—See II.

Recovering benzol from gases (F.P. 594,531).---See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Fastness of pigments to light. WAGNER.— See XIII.

PATENTS.

Manufacture of condensation products [dyestuffs] of the anthraquinone series. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 222,125, 16.9.24. Conv., 18.9.23. Addn. to 205,502, B., 1924, 627).—Vat dyestuffs are prepared by condensation of derivatives of benzanthrone substituted in the 1'-position and aromatic acid chlorides by the process described in the chief patent. Yellow dyestuffs are obtained from 1'-methylbenzanthrone or 2-chlorobenzanthrone and benzoyl chloride; the former dyestuff crystallises from nitrobenzene in microscopic spears, m.p. above 300°, and the latter has m.p. 197°. A. J. HALL.

Manufacture of vat dyestuffs from benzanthrone. BADISCHE ANILIN U. SODA-FABR. (F.P. 589,323, 31.10.24. Conv., 10.1.24).—Benzanthrone or 2:2'-benzanthronyl is heated with an alkali alkyloxide or an alcoholic solution of a caustic alkali. For example, a mixture of dibenzanthrone and iso dibenzanthrone is obtained by heating benzanthrone at 170° with methyl alcohol and caustic potash. The mixture may be purified by precipitation from sulphuric acid and the constituents separated by taking advantage of the difference in the solubilities of the alkali salts of their leuco-compounds (obtained by reduction with alkaline solutions of sodium hyposulphite); the alkali salt of the leucocompound of *iso* dibenzanthrone is insoluble. Methyl alcohol may be replaced by *iso* propyl or butyl alcohol. A. J. HALL.

[Manufacture of] vat dyestuffs. KALLE U. Co. A.-G. (F.P. 591,535, 12.1.25).—Vat dyestuffs are prepared by the action of compounds containing reactive hydrogen atoms on chloro-, bromo-, and nitroketoperylene dyestuffs. For example, greenish-blue and reddish-blue dyestuffs are obtained by treating chlorodibenzanthrone and chloro*iso*dibenzanthrone respectively, with phenol in the presence of an alkali. Blue, blue, and greyish-black vat dyestuffs are prepared by heating chlorodibenzanthrone, dichlorodibenzanthrone, and nitrodibenzanthrone, respectively, with *p*-toluidine in the presence of sodium carbonate.

A. J HALL.

[Manufacture of] sulphur dyestuffs fast to chlorine. KALLE U. Co. A.-G. (F.P. 588,874, 14.11.24).—Blue to black sulphur dyestuffs fast to chlorine are prepared by heating aromatic dihydroxycompounds, aminocarbazole, and alkali polysulphides together under pressure. The aromatic dihydroxycompounds, *e.g.*, quinol, may be replaced by compounds, *e.g.*, benzoquinone, which are converted into dihydroxy-compounds when treated with polysulphides, and aminocarbazole may be replaced by nitro-, nitroso-, or nitrosonitro-carbazole.

A. J. HALL.

[Manufacture of] diphenylmethane dyestuffs. G. L. HUGEL (F.P. 589,745, 9.2.24).-Diphenylmethane dyestuffs are condensed with phenylmethylpyrazolone, β -naphthol, or nitromethane in aqueous solution and the leuco-products subsequently oxidised. For example, a dyestuff capable of yielding fluorescent shades fast to light on silk and cellulose acetate silk is obtained by condensing Thiopyronine with phenylmethylpyrazolone in an aqueous solution of caustic soda. Under similar conditions goldenyellow (with green fluorescence) and red fluorescent dyestuffs for silk and cellulose acetate silk are obtained from Acridine Orange and phenylmethylpyrazolone and from Thiopyronine and nitromethane respectively. A. J. HALL.

[Manufacture of] greenish-blue dyestuffs [lakes]. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (F.P. 590,464, 13.12.24. Conv., 24.12.23 and 19.5.24).—A greenish-blue lake is obtained by the action of ferric chloride on a solution containing sodium carbonate, sodium acetate, and 8-chloro-1:2-naphthylenediamine-5-sulphonic acid (obtained by reduction of the dyestuff obtained by coupling 8-chloro-1-naphthylamine-5-sulphonic acid with diazobenzene chloride) and precipitating the resulting dyestuff by the addition of barium chloride, aluminium sulphate, and barium sulphate. A. J. HALL.

[Manufacture of a] monoazo dyestuff. CHEM. FABR. ROHNER A.-G. PRATTELN (Swiss P. 109,706, 10.5.24).—A bluish-scarlet pigment is produced directly on textile fibres or in substance by coupling diazotised 2-benzamido-*p*-toluidine with 2:3-hydroxynaphthoic acid anilide (Naphthol AS). It forms lakes very fast to light. A. J. HALL.

[Manufacture of] dyestuffs from peat. J. KOZAK, M. F. WEINBERGER, AND M. PROKOPCZUK (Austr. P. 99,926, 30.1.24).—Humic acids precipitated by the addition of acids to the product obtained by digesting peat with a dilute solution of sodium carbonate containing caustic soda or potash, are heated at 200° with alkali sulphides or polysulphides in the presence of the salts of heavy metals such as copper, zinc, and iron. The resulting dyestuff may be purified and its colour strengthened by the introduction of nitro-groups. A. J. HALL.

[Naphthalene] chlorination apparatus. S. BROWN, Assr. to HALOWAX CORP. (U.S.P. 1,564,044, 1.12.25. Appl., 13.1.21).—Molten naphthalene is treated with chlorine under reduced pressure to form monochloronaphthalene, and the hydrochloric acid formed is sucked through water and absorbed. Chlorination and absorption proceed satisfactorily under the reduced pressure and there is no risk of leakage from the apparatus. T. S. WHEELER.

Production of anthraquinone derivatives containing nitrogen [isoxazolones]. BADISCHE ANILIN- U. SODA-FABR., Assees. of H. NERESHEIMER 16.8.23).—Anthraquinoneisox-(G.P. _418,270, azolones, which are of use in the manufacture of dyestuffs, are produced by oxidising anthraquinoneisoxazoles not substituted in the isoxazole ring, by treatment in solution or in suspension in acids, with halogens or substances liberating them. For example, chlorine is passed into a suspension of anthraquinoneisoxazole in 62% sulphuric acid at 50-60°, until a filtered test portion dissolves completely in ammonia, yielding a yellowish-green solution. The product, of formula, $C_{15}H_7O_4N$, is separated by dilution with water and filtration; it has m.p. $214-215^{\circ}$ (crystallised from benzene), and is reduced by alkaline sodium hyposulphite to 1-aminoanthraquinone-2-carboxylic acid. 5-Nitroanthraquinoneisoxazolone is obtained in a similar manner from the corresponding isoxazole derivative. L. A. COLES.

Preparation of primary aromatic amines. FINOW-G.m.b.H., and H. MÜLLER (G.P. 418,497, 29.3.23).—Aromatic nitro-compounds are reduced to the corresponding amines by treatment with nascent ferrous hydroxide, carbonate, or acetate. For example, an intimate mixture of nitrobenzene with anhydrous sodium carbonate or with flaked sodium hydroxide, is added, with stirring, to ferrous sulphate solution at 30°; stirring is continued for a short time, and aniline is separated by steam distillation. o-Nitrobenzaldehyde is reduced quantitatively to o-aminobenzaldehyde by similar treatment at the ordinary temperature. In cases where excess of alkali is not harmful, the reduction can be effected by adding a mixture of the nitrocompound with ferrous sulphate to sodium carbonate solution. L. A. COLES.

Manufacture of azo-dyestuffs. O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (E.P. 245,287, 24.1.25).—See U.S.P. 1,546,328; B., 1925, 751.

Condensation products [dyestuffs] of the anthraquinone series. Soc. of CHEM. IN BASLE, Assees. of P. SCHETELIG (U.S.P. 1,568,627, 5.1.26. Appl., 4.5.25).—See E.P. 234,086; B., 1925, 624.

Lakes (F.P. 582,506).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Sulphur content of wool. S. R. TROTMAN and H. S. BELL (J.S.C.I., 1926, 45, 10-12 T).-Published results for the sulphur content of wool vary from 0.5 to 5%. Whether keratin be either a single protein or a mixture of proteins one would expect it to have a definite and not a variable composition. Variation in results may be due partly to faulty methods of analysis. The authors have examined many published processes, compared with that of Carius, and recommend the following modification of the Benedict-Denis method (J. Biol. Chem., 1909, 363; 1910, 401). About 0.2 g. of the wool is digested with pure sodium hydroxide solution made from metallic sodium, till it has just dissolved. A drop of bromine is added and, after a few minutes, the mixture is neutralised with nitric acid, 10 c.c. of the Benedict-Denis reagent are added, and the determination of the sulphuric acid is completed in the usual manner. Different purified wools, of known origin, were analysed by this process. The percentage of sulphur showed but little variation, 3.42% being the maximum, whilst the mean of 40 experiments gave 3.22%. It is contended that the sulphur content of wool is not variable as supposed hitherto, but practically constant.

Proteins of wool. S. R. and E. R. TROTMAN and R. W. SUTTON (J.S.C.I., 1926, 45, 20-24 T) .--The authors have repeated the work of Raikov, Allwörden, and other investigators. They could not obtain any confirmation of Raikov's statement that wool evolves sulphur dioxide when treated with phosphoric acid. Neither Allwörden's "elasticum" nor Justin-Müller's "gelatin" could be isolated. No volatile sulphur compounds were obtained from the solution of wool in sodium hydroxide. When wool is digested with hydrochloric acid, part of it dissolves, leaving a comparatively insoluble residue which dissolves readily in ammonia. From the hydrochloric acid solution and the undissolved residue two different proteins (termed A and B) were obtained, with sulphur contents of 3.67% and 1.49% respectively. After removing protein A from the hydrochloric acid solution, a third was obtained by saturating the filtrate with magnesium sulphate. As the result of experiments made with cold acids and alkalis on wool, it is suggested that protein A is present chiefly in the epithelium, whilst the cortex consists principally of protein B. Protein A is more readily attacked by oxidising agents and chlorine than protein B, and these facts are in accordance with practical experience in the chlorination of wool. On the other hand, protein B being more readily soluble in alkalis is possibly connected with damage by alkalis.

Chemical analysis of cotton. Waxes of cottons of different origin and their characteristics. L. V. LECOMBER and M. E. PROBERT (J. Text. Inst., 1925, 16, T 338—344).—The quantities of the carbon tetrachloride extracts (fat and wax) and of the chloroform extracts (fat, wax, and resin), and their analytical characteristics have been determined for a range of cottons. The table summarises the most useful data :—

		Sapon-		
	Fat and	Acid	ification	Unsap. Iodine
Cotton	wax	value	value	matter value
a second second second	%			%
American (12				
samples)	0.435	25	66	. 64 22
Egyptian (7				
samples)	0.39	27	78	54 23
Sea Island (2				
samples)	0.51	21.5	61	$. 61.5 \dots 23$
S. American	1			
(5 samples)	0.29 - 0.54	30	71	. 60 25
Australian (1		-		
sample)	0.42	29	58	. 68 19
Mean of these				
27 samples	and the second	26	70	. 61 23
Indian (6 sam-	0.04	0.0	101	15 00
pies)	0.34	38	121	. 45 32

Native Indian cottons can obviously be readily distinguished from other growths by the properties of their wax extracts. J. C. WITHERS.

Chemical analysis of cotton. Identification of the fatty ingredients in sized goods. L. V. LECOMBER and M. E. PROBERT (J. Text. Inst., 1925, 16, T 345-351).-The analytical characteristics of cotton wax (cf. preceding abstract) are sufficiently divergent from those of tallow, Japan wax, spermaceti, castor oil, paraffin wax, and similar "softeners" commonly used in compounding sizes to make it possible to identify these substances in the carbon tetrachloride extracts from sized goods. In the absence of the original unsized cotton, the average values for its particular type may be used for reference. Examples are quoted to prove the utility of the method for cloths containing the above softeners singly or in certain pairs. The chief sources of error are possible adulteration of the softener presumed to have been used, and possible oxidation of unsaturated fats or oils on exposure of the cloth to the air. J. C. WITHERS.

Common faults in cotton goods. F. SUMMERS (J. Text. Inst., 1925, 16, T 323-337).—The value of microscope technique in elucidating the causes of typical faults is shown. Of chemical interest is a chapter dealing with mercerisation, which shows how the degree of penetration by the alkali and the efficiency of the tension employed may be judged from cross-sections of the material.

J. C. WITHERS.

Action of ammonia on cotton cellulose. G. BERNARDY (Z. angew. Chem., 1925, 38, 1195-1197).-The action of 22% aqueous ammonia on cellulose does not lead to additive compounds, to adsorption, to entry of an amino- or imino-group into the molecule, to hydrolysis, or to oxidation. It is analogous to, though feebler than, that of other alkalis, and leads simply to the dissolution of alkalisoluble celluloses. When the mixture is heated under pressure, black, amorphous, humin-like substances are also formed, and under prolonged heating at 200° and 40 atm. the cellulose breaks down into a brown powder. The nitrogen content of the cellulose is only very slightly increased by the action of ammonia. The cleansing action of 22% ammonia on raw cotton cellulose is less than that of 1% sodium hydroxide at 100°. Whereas ammonia in this concentration does not cause any swelling of cellulose, permanent swelling is produced by liquid ammonia at -33° to -35° , though unaccompanied by any decomposition. W. T. K. BRAUNHOLTZ.

Lignin. I. Spruce lignin. A. FRIEDRICH and J. DIWALD (Monatsh., 1925, 46, 31-46).-Finelysieved spruce sawdust was freed from resin by Soxhlet extraction for 7 hrs. with alcohol-benzene (1:1), and from gum by subsequent extraction with cold, aqueous 5% sodium hydroxide for 36 hrs. After removal of the alkali and drying at 60-70°, the wood was triturated with an equal weight of hydrochloric acid (1 vol. of acid, d 1.17, to 1 vol. of water). After keeping for 48 hrs. the mixture was boiled for 8-10 hrs. with a tenfold proportion of 96% alcohol, filtered, the filtrate evaporated to one-third of its original volume, and diluted with 10 pts. of water, addition of a little mineral acid improving the filtering properties of the precipitate. This consists of primary lignin, C34H33O9(OMe)5, and forms, after several purifications by dissolving in alcohol and re-precipitation with water, a pale brown, amorphous, hygroscopic powder, soluble in alcohol, chloroform, acetone, pyridine, carbon di-sulphide, acetic acid, and ethyl acetate, but insoluble in ether and benzene (cf. Schmidt, A., 1923, i., 274). The yield is 8-10%, calculated on airdried wood. When dissolved in warm, 2N-sodium hydroxide, and re-precipitated with acid after 2 hrs., the primary lignin is partially demethylated with formation of the product C₃₄H₃₅O₁₁(OMe)₃, which is soluble in sodium carbonate solution. The methoxyl content of the primary lignin (20.9%) is also reduced (to 16.8%) by treatment with concentrated hydrochloric acid (d 1.21), the product being dark brown, insoluble in all media except alkalis, and apparently identical with the lignin described by Willstätter and Zechmeister (A., 1913, i, 955), obtained by the direct extraction of spruce wood with concentrated hydrochloric acid. Treatment of the primary lignin with bromine in acetic acid affords a product containing 4 atoms of bromine,

but its formation is accompanied by evolution of hydrobromic acid, and by considerable changes in the constitution of the lignin. Prolonged action of bromine affords successively an alcohol-soluble and an alcohol-insoluble product, each containing 5 bromine atoms, and finally an ether-soluble hexabromo-derivative. Oxidation with dilute alkaline hydrogen peroxide affords a pale yellow, amorphous, compound C₂₈H₃₃O₉(OMe)₃, which does not reduce Fehling's solution. Phenylhydrazine converts the primary lignin into a reddish-brown, amorphous product, C₃₀H₂₇O7N(OMe)3, whilst semicarbazide affords a yellowish brown, amorphous powder, the composition of which is also not in accordance with simple semicarbazone formation. Treatment with excess of methyl sulphate in alkaline solution affords the compound C₃₄H₃₂O₈(OMe)₆, yellow-brown, decomp. above 110°, insoluble in alkalis, whilst benzoylation affords the tribenzoate, $C_{55}H_{45}O_{12}(OMe)_5$, pale yellow. This capacity for attaching three benzoyl groups but only one methyl group is possibly to be ascribed to the hydration, in the former case, of an aldehyde group (cf. Powell and Whittaker, A., 1924, i, 375). The typical colour reactions with aniline and phloroglucinol are exhibited by the primary lignin and the methyl derivative. The product of the reaction with aniline corresponds to the addition of 1 mol. of aniline to 1 mol. of the primary lignin, and forms a yellow powder. The colour reaction with phloroglucinol only takes place in presence of hydrochloric acid, and disappears on dilution with water. When the reaction is carried out in alcoholic hydrochloric acid, evaporation of the solution affords a dark reddish-brown product. A bisulphite compound of the primary lignin is described.

F. G. WILLSON.

Lignin. W. KÜSTER and E. SCHNITZLER (Z. physiol. Chem., 1925, 149, 150-172).-Lignin is decomposed by fusion with β -naphthol to give at least two distinct products, one of which, merolignin, is a crystalline chemical individual. The mass is treated with chloroform, filtered, and precipitated by the addition of ether, when a substance is obtained which on drying in a vacuum gives analyses agreeing with the formula C18H13O3(OMe)3. It is soluble in aqueous potash but not in soda. It melts at 155° (decomp.). The filtrate from the ether precipitation is evaporated, cooled, and treated with light petroleum, when a second precipitate is obtained. This is filtered off quickly, dried, and washed with light petroleum. The dried yellow powder, m.p. 160—168° (decomp.), gives analyses which agree with $(C_{18}H_{14}O_3)_x$. The filtrate is evaporated to dryness, and crude merolignin extracted by digestion of the residue with cold alcohol. Better yields are obtained by extracting the β -naphthol melt direct with petroleum of b.p. 110-130° the solution is evaporated to dryness and treated with cold alcohol, when crude merolignin is extracted. This may be purified by recrystallisation from chloroform or benzene. It melts at 205-206 Analyses agree with the formula $C_{22}H_{14}O$.

H. D. KAY.

152

Use of acid hydrolysis in the determination of lignin. L. PALOHEIMO (Biochem. Z., 1925, 165, 463-464).-Replacement of 41% hydrochloric acid by 70% sulphuric acid gives, on hydrolysis, concordant results for the lignin residue, viz., for pine wood 30%, for hay and straw 20%. More accurate investigation shows that these figures are too high. If the time of hydrolysis is too short, carbohydrate material is precipitated along with the lignin, and if too long, humus is formed. By hydrolysing over increasing periods up to one hour, it was shown that the amount of residue was constant for a short time, but then slowly increased, due to adsorption by the lignin of secondary products. This increase could be retarded by use of large amounts of acid. For each 1 g. of material 200 c.c. of acid are used and the residue after half-an-hour is filtered off and submitted to a second hydrolysis for 1 hour. Very difficultly hydrolysable protein material remains, however, after hydrolysis for 24 hrs. A correction for this is applied by subtracting from the lignin figure the protein content as calculated from the total nitrogen of the residue. The accepted figures for pine wood (30%), for clover hay (25%), and for rye straw (20%) are re-duced by these methods to 25%, 7%, and 3%, respectively. The amount of lignin is not changed by heating the residue with dilute acid or alkali, nor when such treatment precedes acid hydrolysis. If, therefore, the material receives a preliminary treatment with dilute alkali, which removes most of the substances likely to interfere with the determination, a single acid hydrolysis is sufficient. Lignin of hay is not attacked in the alimentary canal of ruminants. P. W. CLUTTERBUCK.

Formation of sugars as intermediate phase of production of coal from ligneous plant substances. Schwalbe and Schepp.—See II.

PATENTS.

Fireproofing textile fibres and fabrics, and other porous or absorbent substances. T. J. I. CRAIG, and P. SPENCE AND SONS, LTD. (E.P. 244,503, 18.8.24).—The material is impregnated with sodium aluminate and squeezed. It is then immersed for $\frac{1}{4}$ — $\frac{1}{2}$ min. in a saturated solution of sodium bicarbonate at a temperature above 50°, but below that at which carbon dioxide is evolved. After squeezing off the excess solution, the material is immersed for $\frac{1}{4}$ — $\frac{1}{2}$ min. in a boiling solution of sodium carbonate or other salt, when the alumina is rapidly fixed on the material. The treatment with sodium bicarbonate may be partly or wholly replaced by exposure to gaseous carbon dioxide.

A. GEAKE.

Retting flax and similar vegetable fibres. BADISCHE ANILIN- U. SODA-FABR., Assees. of G. PFÜTZER and O. FLIEG (G.P. 419,730, 7.9.24. Addn. to 411,697; B., 1925, 626).—Formamide, or a mixture of formamide and carbamide, is added to the ret water; the time required for retting is thereby reduced. A. GEAKE. Washing artificial fibres wound upon bobbins. B. BORZYKOWSKI (E.P. 239,482 and 244,324, 9.4.25. Conv., [A] 2.9.24, [B] 13.12.24).—(A) The fibre is wound upon hollow, perforated bobbins, and the washing liquid drawn through it by suction applied to the interior. (B) Coagulating liquor adhering to the fibre is removed by suction before washing as described in (A). After washing, the washing liquid is similarly removed. A. GEAKE.

Manufacture of cellulose derivatives [acetate]. J. O. ZDANOWICH (E.P. 244,148, 15.8.24).—In the production of a solution of cellulose acetate, which can be directly converted into films, filaments, and the like, the acetylation mixture is first chlorinated (cf. E.P. 196,641; B., 1923, 650 A), and a substance containing 20—30% of acetyl obtained. The chlorine remaining in this solution is utilised by adding sulphur dioxide or trioxide, or an oxide of phosphorus. The chlorine compound thus formed acts as a condensing agent for the second stage of acetylation. Acetylation is completed by the addition of a very small quantity (0.01-0.1%) of a powerful condensing agent, such as sulphuric acid. A. GEAKE.

Production of plastic masses. R. GARKE, E. MEYER, and W. CLAASEN (E.P. 241,858, 28.4.25. Conv., 22.10.24).-A mixture of ar- and ac-tetrahydronaphthyl acetate, or either of these separately, is a solvent for rubber and the like, and also for cellulose esters. These may thus be combined to form solid plastic masses, varnishes, impregnating media for textiles, paper, wood, leather, and the like, and adhesives. A solution of rubber in tetrahydronaphthyl acetate may be added to nitrocellulose before spinning artificial filaments, and the filament then obtained has great resilience and is not affected by water. The addition of a small quantity, e.g., 10%, of tetrahydronaphthyl acetate to rubber hinders atmospheric oxidation and consequent deterioration. The ester is non-volatile at the ordinary temperature and remains in the goods made from it. A. GEAKE.

Manufacture of cellulose esters or ethers in a solvent. N. B. GRILLET, ASST. to SOC. CHIM. USINES DU RHÔNE (U.S.P. 1,566,398, 22.12.25. Appl., 22.11.24).—Cellulose is esterified, in the presence of a liquid solvent of the desired product, in a large, rotating, closed vessel.

A. GEAKE.

Prevention of corrosion in steel digesters [for wood pulping]. F. G. RAWLING; dedicated to the citizens of the United States (U.S.P. 1,566,118, 15.12.25. Appl., 21.3.25).—Corrosion of steel digesters for wood pulping is prevented by adding the sodium salt of a weak polybasic acid to the sodium sulphite solution used. Subsequently, during the cooking, aqueous sodium hydroxide is added. A. GEAKE.

Digesting pulp in rotating digesters. E. MORTERUD (U.S.P. 1,566,339, 22.12.25. Appl., 28.5.25).—A tube located in one of the trunnions of the digester is connected to an external heater, and internally alternately to two tubes leading to opposite ends of the digester. A. GEAKE.

Manufacture of cellulose. L. BRADLEY and E. P. McKEEFE (Can.P. 246,477, 12.4.24).—Finely divided wood is boiled in a solution of sodium hydroxide, sulphite, and thiosulphate. A. GEAKE.

Manufacture of paper pulp from vegetable fibres. B. S. SUMMERS (Can. P. 246,537, 16.2.24).— Fibre is boiled in a liquor containing 1-4% of phosphoric acid. A. GEAKE.

Manufacture of pulp. MASCHINENBAU-ANSTALT HUMBOLDT (G.P. 418,304, 3.6.23).—Fibrous material is first beaten, and while beating is continued solutions of caoutchouc, balata, gutta-percha, or drying, oxidised, or sulphonated oils are added. This procedure avoids the formation of lumps.

A. GEAKE.

Purifying cellulose. C. G. SCHWALBE (G.P. 418,976, 19.4.23).—Cellulose is boiled with bases and sulphites in the presence of substances soluble in such solutions, such as carbohydrates, especially sugars, and salts of organic acids. Boiling may be with or without pressure, and preferably with agitation. The above substances may be replaced by molasses or sulphite-cellulose waste liquor.

A. GEAKE.

Treatment of black liquor [from sodacellulose manufacture]. L. BRADLEY, Assee. of E. P. MCKEEFE (Can. P. 245,831, 1.12.21).—A solution of alkali aluminate is formed in the liquor, the alumina and organic matter are then precipitated together as an intimate mixture, the precipitate is distilled, and the mixture of carbon and alumina obtained is electrolysed in the molten state.

A. B. MANNING.

Recovering constituents from muds produced in purification of cellulosic alkali lyes with heavy metals. A.-G. F. ANILIN-FABR. (G.P. 419,665, 16.9.24).—Muds containing heavy metals, alkalis, and cellulose are boiled at the ordinary pressure until organic substances are dissolved, and the heavy metal is obtained in a recoverable form. Thus from cellulosic precipitates in viscose manufacture sodium hydroxide is recovered, and also copper as cuprous oxide or metal.

A. GEAKE.

Utilisation of sulphite-cellulose waste liquor. E. L. RINMAN (Swed. P. 57,863, 8.6.23).—Sulphitecellulose waste liquor or spent wash from the distillation of sulphite spirit is evaporated to dryness, and mixed with calcium sulphate and materials containing alumina, ferric oxide, and silica, in such proportion that on ignition sulphur dioxide is evolved and a residue is obtained suitable for the production of a cement. A. B. MANNING.

Preparation of alkali cellulose. E. VAN WEYEN-BERGH, Assr. to COURTAULDS, LTD. (U.S.P. 1,569,692, 12.1.26. Appl., 6.3.25).—See E.P. 237,685; B., 1925, 800. Grinding stones for grinding wood pulp. NORTON CO., Assees. of W. W. GREENWOOD (E.P. 238,176, 19.12.24. Conv., 6.8.24).

Coal briquettes (F.P. 594,080).-See II.

Improving the odour of methyl sulphide (G.P. 418,129).—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Leather dyeing. I. H. SALT (J. Soc. Leather Trades Chem., 1925, 9, 518-520).-Comparative dyeings on vegetable-tanned leather were made with different acid dyes without added acid or salts, and with addition of sulphuric acid, acetic acid, sodium chloride, sodium sulphate, and sodium sulphate and sulphuric acid respectively. Acid dyes gave as good a colour with acetic acid as with sulphuric acid and in some cases fuller and more even shades. Sodium chloride reduced the colour value of acid dyes on vegetable-tanned leathers. Many acid dyes gave full shades without the addition of acid. Sodium sulphate had a similar action to sodium chloride. Sodium sulphate and sulphuric acid gave slightly paler shades than sulphuric acid alone. D. WOODROFFE.

Leather dyeing. II. D. McCANDLISH and H. SALT (J. Soc. Leather Trades Chem., 1925, 9, 520-525).-Comparative dye tests on chrome-tanned and vegetable-tanned skivers with a range of acid, basic, and direct dyes have shown that unmordanted chrome leather has a much greater affinity for acid and direct dyes than vegetable-tanned leather, The hydroxyl group in the dyestuff is unimportant. The object of mordanting chrome leather is not to facilitate dyeing but to reduce the attraction between the leather and the dye, thus producing more even shades. A better feel is produced on the finished leather if it has been mordanted. The dyeing of chrome leather with basic dyes is unsatisfactory unless the leather has been mordanted. Dyes with a reddish shade tend to appear more reddish when used on chrome leather. D. WOODROFFE.

PATENTS.

Bleaching agent. Soc. ALSACIENNE DE PROD. CHIM. (F.P. 590,876, 22.2.24).—Aromatic sulphonamide derivatives halogenated in the side chain, such as *p*-toluenesulphochloramide, in solution or in admixture with soap, sodium carbonate, or sulphonated castor-oil, are used for cleaning and bleaching vegetable and animal fibres. L. A. COLES.

Preparation of a mordant [for cotton] in place of tannin. L. SELLET (F.P. 589,081, 17.1.24). —Hydrogenated aromatic hydrocarbons are treated with concentrated sulphuric acid, with or without a preliminary oxidation. For example, tetralin is oxidised with bromine water and phosphoric acid, the aqueous layer removed, and the oxidation completed by treatment with air in the presence of ferric chloride as catalyst. Any unchanged tetralin is distilled off and the resinous residue is rendered soluble in water by treatment with concentrated sulphuric acid. A similar mordant may be prepared by the direct treatment of tetralin with concentrated sulphuric acid, oxidation of the tetralin taking place simultaneously. A. R. POWELL.

Dyeing cellulose acetate products. L. B. HOLLIDAY AND CO., LTD., and A. YOUNG (E.P. 244,936, 24.1.25).—Cellulose acetate products are dyed by the substances obtained by condensing 1chloro-2: 4-dinitrobenzene-6-sulphonic acid or 1chloro-2: 6-dinitrobenzene-4-sulphonic acid or their salts with substances containing one or more aminoor one or more hydroxy-groups, *e.g.*, aniline.

A. GEAKE.

Dyeing cellulose acetate. TEINTURERIE DE LA RIZE (F.P. 590,738, 15.2.24).—Cellulose acetate products are dyed by means of direct cotton dyestuffs after treatment for from 15 min. to 2 hrs. at 50—70° in an aqueous solution containing 1 kg. of barium hydroxide and 1 kg. of barium chloride per 100 litres. A. J. HALL.

Easily soluble preparations of vat dyestuffs. FARBENFABR. VORM. F. BAYER U. Co. (F.P. 589,914, 2.12.24. Conv., 10.12.23, 29.2, and 6.6.24).-Preparations of vat dyestuffs which are soluble in cold or warm water are obtained by mixing unreduced vat dyestuffs with a caustic alkali, a hyposulphite, and a salt of a sulphonic acid capable of acting as a dispersing agent, e.g., sulphonic acids of unsaturated fatty acids, ligninsulphonic acid, sulphite-cellulose waste lye, or water-soluble carbohydrates such as glucose and dextrin; or by the addition of suitable quantities of a hyposulphite and a caustic alkali to an aqueous solution of an alkali salt of a sulphonic acid containing a vat dyestuff. For example, a mixture containing 2-(4'-chloroanilido)-1:4-naphthaquinone, Monopole soap (twice sulphonated castor oil), and caustic soda is dried, and sodium hyposulphite added ; or the mixture containing the dyestuff, soap, and caustic soda is warmed, filtered, and mixed with caustic soda and sodium hyposulphite; or the dyestuff is mixed with molasses, a caustic alkali, and sodium hyposulphite. A. J. HALL.

Process of dry-dyeing. I. LEVY and L. D. GIMBERG (F.P. 591,075, 16.8.24).—Designs are printed on fabric, paper, and the like by means of a viscous colour paste prepared by adding paraffin wax or a similar substance to a mixture obtained by the addition of an aqueous solution of an organic dyestuff, *e.g.*, Methyl Violet, and lactic acid to a 25—50% solution of a soap derived from stearic acid, the printed fabric being subsequently calendered. Lactic acid may be replaced by citric, tartaric, malic, or oxalic acid. A. J. HALL.

Increasing the fastness to rubbing of dyed materials. CHEM. FABR. A. SCHMITZ (F.P. 592,452, 6.1.25. CONV., 15.9.24).—Dyed animal or vegetable fibres, particularly cotton materials dyed with "ice" colours, are treated with a 1.5-2.0% aqueous solution of a viscous emulsion prepared by treating fats and oils, *e.g.*, castor and olive oils, with sulphuric

acid, converting the product into a soap, adding gelatin or glue or similar substance, and mixing the product with a chloro-derivative of a hydrocarbon, such as carbon tetrachloride. The emulsion does not form insoluble soaps when added to hard water.

A. J. HALL.

Preparation for finishing natural or artificial fabrics. G. L. BRUGÈRE (F.P. 592,205, 27.3.24).— A mixture of an aromatic amino- or hydroxy-carboxylic acid, formaldehyde, and ammonia is heated in a closed vessel to 90° or 150°. The product is a viscous mass which sets to a jelly when kept in the air. For use in the finishing of fabrics the substance is made into a colloidal suspension in an organic solvent. Treatment with this material increases the strength of the fabric. A. R. POWELL.

Monoazo dyestuff (Swiss P. 109,706).-See IV.

Soaps (F.P. 594,146).—See XII.

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

Factors governing lime-kiln capacity and fuel economy. V. J. AZBE (Tech. Papers, Nat, Lime Assoc., May, 1925, 37-58).-The regulation of the air supply is the most important factor in determining the efficiency and fuel economy of a lime kiln. A minimum temperature of 900° is necessary to convert limestone into lime, and a small percentage of excess air will be sufficient to lower the combustion temperature below this point. On the other hand, too small a supply of air will produce too high a temperature, giving overburnt lime, and there will be a risk of incomplete combustion. The fuel and air supply must be regulated to give a long, luminous flame, which is much more efficient for transferring heat to the limestone than a short, non-luminous and hotter flame. It is impossible to obtain a regular supply of heat with direct hand-fired kilns, considerable losses occurring at the drawing periods. Properly designed automatic gas producers will give good results; their operation is not improved by steaming, but is improved by passing carbon dioxide from the waste gases. Oil firing with mechanical burners is only economical where cheap oil fuel is available. A determination of the percentage of carbon dioxide and oxygen will be a guide as to the operation of the kiln, using charts developed by the author. The output of lime per cubic foot of kiln space is suggested as a basis for the comparison of kiln capacities. The life of the kiln depends on the type of brick used and the structural design of the kiln. It should be possible to obtain without difficulty 4 tons of lime per ton of fuel burnt, with a thermal efficiency of over 60%. B. W. CLARKE.

Comparative tests of methods for the quantitative determination of sodium sulphide. P. BUDNIKOV (Z. anal. Chem., 1925, 67, 241-248).— A series of analyses of pure and technical grades of sodium sulphide has been made by the following eight methods: gravimetric as barium sulphate,

volumetric after conversion into benzidine sulphate, direct and indirect iodometric titration, titration with permanganate of the amount of ferrous sulphate formed when the sulphide is heated with excess of ferric sulphate, precipitation as cadmium sulphide, direct titration with zinc sulphate, and titration with 0.1N-hydrochloric acid using phenolphthalein as indicator (Podreschetnikov, B., 1908, 21). The last-named method and indirect iodine titration gave the most satisfactory results, and the zinc sulphate method the least satisfactory. The advantage of Podreschetnikov's method is that free sodium hydroxide and sodium hydrogen sulphide may be determined at the same time. The pink colour of the indicator disappears when all the hydroxide and half the normal sulphide has been neutralised; addition of formaldehyde then converts all the sodium hydrogen sulphide into sodium hydroxide, which is titrated as before. From the figures so obtained the composition of the preparation is readily calculated. A. R. POWELL.

Determination of nitrate. K. KÜRSCHNER and K. SOHARRER (Chem.-Ztg., 1925, 49, 1077—1078).— Reduction of nitrates to ammonia by means of finely divided copper and sulphuric acid yields low results owing to imperfect reduction, but accurate results may be obtained if an iron-copper couple is used. 0.5 g. of the nitrate is shaken in a Kjeldahl flask with 3.5 g. of iron wire, 0.5 g. of cupric oxide, and 30 c.c. of 1 : 2 sulphuric acid. After 1 hr., the solution is boiled for 30 min., cooled, treated with sodium hydroxide, and distilled, the ammonia being collected in standard acid. A. R. POWELL.

See also A., Feb., 1926, 112, X-Ray examination of some ammonia catalysts (WYCKOFF and CRITTENDEN). 113, Hydroxides of aluminium and iron (BöHM); Incandescence of metal oxides (Böhm). 119, Adsorption of barium chloride by colloidal hydrated manganese dioxide (CHLOPIN and BALANDIN). 126, System water and nitrates and sulphates of ammonium and potassium (OSAKA and INOUYE); Separation of manganese and zinc with sodium sulphide (RUFF and HIRSOH). 127, Decomposition of metal sulphates by heat (MARCHAL). 131, Chemical reactions in powdered mixtures of two kinds of crystals (TAMMANN). 134, Catalysis of detonating gas at high temperatures by metals inactive at ordinary temperature (REMY and GÖNNINGEN). 136, Decomposition of alkaline-earth phosphates by alkali carbonates (RAQUET); Reduction of heavy metal sulphides by barium oxide (BILTZ and VON MÜHLENDAHL). 138, Hypochlorous acid and alkali hypochlorites (von DIENES). 139, Preparation of fluorine (LEBEAU and DAMIENS) ; Determination of hydrochloric acid by means of potassium iodate (KOLTHOFF); Micro-titration of iodide (KOLTHOFF); Acidimetric determination of mercuric chloride (Rupp and MAISS).

Adsorption of ammonia, sulphur dioxide, and chlorine by wood charcoal. HENGLEIN and GRZENKOVSKI.—See I. Determination of carbon disulphide. HUFF. —See II.

PATENTS.

Sulphur burner. H. HOWARD, Assr. to GRAS-SELLI CHEMICAL Co. (U.S.P. 1,566,538, 22.12.25. Appl., 26.12.24).—A receptacle, such as a rotary cylinder, for burning the sulphur is provided with an air inlet and gas outlet and is enclosed in a chamber to which air under pressure is supplied, the air passing partly within and partly without the burner and the combined streams being exhausted through an outlet in the outer chamber adjacent to the outlet in the burner. B. M. VENABLES.

Manufacture of pure hydrochloric acid. SALZWERK HEILBRONN A.-G., T. LICHTENBERGER, and K. FLOR (G.P. 418,389, 7.2.25).—The acid is produced by treating solutions of alkaline-earth sulphates in fused alkaline-earth chlorides with steam. L. A. COLES.

Manufacture of pure, concentrated nitric acid. BADISCHE ANILIN- U. SODA-FABR., Assees. of C. BECK and O. BALZ (G.P. 418,428, 16.12.24).—Nitric acid containing impurities of higher boiling point is purified by scrubbing it in the form of vapour with boiling concentrated nitric acid. L. A. COLES.

Manufacture of ammonia. C. DEGUIDE (F.P. 591,019, 3.3.24).-Barium cyanide is produced by treating mixtures of barium carbonate, coal, and barium silicate at 1200-1300°, with a countercurrent of nitrogen in a rotating tubular retort surrounded by a heating jacket in which carbon monoxide generated during the reaction is burned. The retort is constructed of material of low permeability, such as carborundum, and is maintained under a higher pressure than that prevailing in the outer jacket, to prevent passage of carbon monoxide into the retort. The barium cyanide is decomposed into barium carbonate, ammonia, carbon monoxide, and hydrogen, by treatment at 300-500° with superheated steam in a similar furnace, the carbon monoxide and hydrogen being burnt to supply the necessary heat. L. A. COLES.

Catalysts for the synthesis of ammonia. G. CARRARA (F.P. 591,384. 5.1.25).—Catalytic material for the synthesis of ammonia is worked up into the form of network or is deposited as powder upon network carriers. The network may be rolled up into cylinders. L. A. COLES.

Production of stable bicarbonate of ammonium. J. Y. JOHNSON. From BADISCHE ANHLIN- U. SODA-FABR. (E.P. 244,645 21.5.25).— Pure stable ammonium bicarbonate is produced by crystallising it from initially hot solutions and continuously passing a current of carbon dioxide through the solution during the cooling and crystallisation. The salt is separated from the liquor by centrifuging, and dried in air at 80—90°, the mother liquor being used again in the process. The product is improved by adding to the solution, prior to crystallisation, up to about 1% of substances which influence, and preferably increase, its surface tension; e.g., benzene, petrol, carbon tetrachloride, iron carbonyl, tar oils, ammonium sulphide, or sugar may be added to increase, or naphthalenesulphonic acids substituted in the nucleus by one or more propyl, *iso*propyl, butyl, or *iso*butyl radicals may be added to decrease its surface tension. The product can be stored, e.g., for use as a fertiliser, without any material loss. L. A. COLES.

Combustion of ammonia with oxygen. W. SIEBERT and E. UNGER (G.P. 418,622, 21.10.24).— In apparatus for burning ammonia with oxygen for the generation of heat and light, the gases, in suitable proportions and under suitable pressure, are passed through separate inlets into a mixing chamber which may or may not be provided with nozzles. The mixture is burnt at the mouth of a combustion tube filled or nearly filled with rods or with capillary tubes, which may contain concentrie rods constructed of the same or of similar material.

L. A. Coles.

Separating zirconium and hafnium halogenides. N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 221,802, 8.9.24. Conv., 15.9.23; cf. E.P. 219,327; B., 1925, 499).—Zirconium and hafnium halides are separated by fractional distillation after the addition of a halide of an element of the fifth or sixth group. For example, 1 pt. of phosphorus pentachloride is added to 2 pts. of a mixture of zirconium and hafnium chlorides. When fused, compounds are formed between the phosphorus and metal chlorides. Between 230° and 360° a fraction rich in hafnium distils over, and above 360° a fraction rich in zirconium. Further separation is effected by repeating the fractionation, and the final products are converted into oxides by treatment with a base. A. GEAKE.

Treatment of [precipitation of tin from] solutions containing alkali oxysalt of tin [and arsenic]. H. HARRIS (E.P. 244,526, 18.9.24).— Solutions containing alkali oxy-salts of tin and arsenic, obtained, *e.g.*, in refining lead, are treated with calcium hydroxide after previous addition of carbonates, *e.g.*, sodium carbonate. Tin compounds are precipitated whilst arsenic remains in solution (cf. E.P. 213,638; B., 1924, 522). L. A. COLES.

Making [lead] arsenates. T. A. MITCHELL and K. TOABE (U.S.P. 1,564,093, 1.12.25. Appl., 21.7.22).—Metallic lead and arsenic trioxide are added to a concentrated solution of arsenic acid containing nitric acid. Lead arsenate is precipitated, the concentration of the arsenic acid remaining constant. At intervals the precipitate is removed and the arsenic acid solution concentrated and used again.

T. S. WHEELER.

Production of aluminium chloride. L. BURGESS, ASST. to M. BARNETT (U.S.P. 1,566,269, 22.12.25. Appl., 27.7.21).—Aluminium chloride is produced by treating material containing aluminium silicide with hydrogen chloride. L. A. COLES. Manufacture of nickel salts. W. N. KOHLINS (Can. P. 244,129, 6.6.23).—Solutions of commercial nickel in sulphuric acid are treated with oxidising agents to convert ferrous salts into ferric salts, and, after addition of nickel carbonate, are filtered and concentrated to crystallisation. L. A. COLES.

Neutralising titanic acid obtained by hydrolytic precipitation from titanic sulphate. FABR. DE PROD. CHIM. DE THANN ET DE MULHOUSE (F.P. 590,743, 15.2.24).—Freshly precipitated titanic acid is treated with aluminium hydroxide or zinc hydroxide, or with sodium aluminate or zincate, whereby any sulphuric acid present is converted into soluble sulphates, which are removed by washing. Small quantities of aluminium hydroxide or zinc hydroxide are retained by the titanic acid, and neutralise residual traces of acid not removed during the washing. L. A. COLES.

Manufacture of barium sulphide. C. DEGUIDE (F.P. 592,805, 4.4.24).—Barium sulphide is produced by heating barium sulphate in the presence of a reducing gas, the gas being burnt to provide the necessary heat, without the flame coming in contact with the barium sulphate. L. A. COLES.

Rendering common salt suitable for table and industrial use. M. KRÜGER and S. R. UNKEL (F.P. 593,279, 12.8.24. Conv., 2.8.24).—Crude sodium chloride is purified by repeated washing with a counter-current of water or more or less saturated mother-liquor. L. A. COLES.

Manufacture of metal [e.g., copper] sulphate solutions. F. KLEINMANN (G.P. 418,723, 24.6.23). -Copper and other metal sulphates are produced by treating the finely-divided metal in a towershaped apparatus, with a stream of sulphuric acid containing oxides of nitrogen, the reaction products being continuously oxidised by a counter-current of air and steam, which is injected into the apparatus at such a temperature that the reaction velocity increases, or at least does not diminish, until the sulphate solution flows out of the apparatus. After separation of the sulphate crystals, the motherliquor is mixed with fresh sulphuric acid and used again. The gas outlet of the apparatus can be throttled so as to produce sufficient pressure to force the copper sulphate solution through a filter in the lower part of the apparatus. L. A. COLES.

Treatment [purification] of zinc sulphate solutions. ARTIEBOLAGET OSKARSHAMNS KOPPAR-VERK (Swed. P. 57,951, 19.1.22).—Zinc sulphate solutions for use in the production of electrolytic zinc are treated with precipitated copper, copper sulphate, and a reducing agent or cuprous oxide, whereby chlorides are precipitated as cuprous chloride. L. A. COLES.

Apparatus for effecting recovery of heat and water vapour in the catalytic manufacture of hydrogen by the action of water vapour upon carbon monoxide. G. L. E. PATART (E.P. 228,153,

16.1.25. Conv., 21.1.24).—Carbon monoxide or gas containing it is preheated and charged with water vapour by passage upwards through the lower of two sections of a tower, in which it is scrubbed by a descending stream of hot water, and on leaving the section at the top, steam is injected into the gas to increase its moisture content, and the mixture is passed into a catalyst chamber for the production of hydrogen. On leaving the chamber, the gases pass upwards through the upper section of the tower, in which they are cooled and deprived of the greater part of their moisture content by scrubbing with cold water. Water is circulated continuously through both sections of the tower, which are provided with bubbling plates or other means for effecting intimate contact between the gas and the liquid, the hot water collecting at the bottom of the upper section, passing through a siphon tube to a spraying device at the top of the lower section, and the cold water collecting in a constant-level reservoir below the lower section being pumped to the top of the upper section. L. A. COLES.

Production of pure nitrogen from combustion gases. N. CARO and A. R. FRANK (G.P. 418,495, 14.2.23).—Catalysts for use in the production of pure nitrogen from combustion gases contain copper, cobalt, chromium, iron, uranium, and other heavy and noble metals, and their oxides, in varying proportions, deposited upon non-reacting porous carriers. Carbon dioxide is subsequently removed from the nitrogen by absorption. L. A. COLES.

Apparatus for production of hypochlorites and chlorates. M. WILDERMAN (U.S.P. 1,568,787, 5.1.26. Appl., 13.4.21).—See E.P. 183,671; B., 1922, 812 A.

Treating sulphide ores and concentrates (U.S.P. 1,566,379).—See X.

Electrolysis of alkali chlorides (U.S.P. 1,565,943).—See XI.

VIII.—GLASS; CERAMICS.

Analysis of recent measurements of the viscosity of glasses. II. G. S. FULCHER (J. Amer. Ceram. Soc., 1925, 8, 789-794).-The method of analysis described in a previous paper in relation to three-component glasses (cf. B., 1925, 590) has been applied to two series of four-component glasses, namely, soda-lime-magnesia-silicate glasses and soda-lime-alumina-silicate glasses. The first of these was formed by the molecular substitution of MgO for CaO in the glass 6SiO₂, 1.15 Na₂O, 0.84 CaO, and the second by substituting Al_2O_3 for CaO in the glass $6SiO_2$, $1\cdot11Na_2O_3$ for CaO in the glass $6SiO_2$, $1\cdot11Na_2O$, $0\cdot95CaO$. In the first series, the aggregation temperature, T_a , falls to a sharp minimum when the ratio MgO: CaO is 1.13, corresponding to the composition 6SiO₂, 1.15Na₂O, 0.445MgO, 0.395 CaO. In the second series, a similar sharp minimum is observed for the composition 6SiO2, 1.11Na2O, 0.81CaO, $0.14A1_2O_3$. F. SALT.

Calcining as an aid to grinding [flint]. W. M. MYERS (J. Amer. Ceram. Soc., 1925, 8, 829-842).-Comparative grinding tests were conducted on calcined and on uncalcined flint, care being taken to ensure uniformity in the grinding conditions; e.g., all material from the jaw crusher was passed through a meshed feed to the pebble mill. A similar series of tests was carried out on calcined flint, part of which had been quenched in cold water, and the remainder allowed to cool slowly in air. The advantage of calcining was most marked in material ground for 6 hrs. in the pebble mill. The improvement however, scarcely warrants the additional cost of calcining. The use of more suitable crushing equip-ment will, in the future, probably tend to eliminate the calcining operation. F. SALT.

changes Constitutional occurring in clays on heating. RESEARCH STAFF OF GEN. ELECTRIC Co. (J. F. HYSLOP) (Trans. Ceram. Soc., 1924-5, 24, 402-406).—The X-ray diffraction patterns of various clays, e.g., china clay. silicious clays, ball clay, show that mullite, not sillimanite, is formed at temperatures above 1050°, as well as transformed quartz or cristobalite. The temperature at which complete stability is reached varies with the nature of the clay. Kaolinite breaks down at 600° to form a silicate, presumably Al₂O₂,SiO₂, stable to 900°; above this mullite is formed. B. W. CLARKE.

Thermal expansion of refractories. F. H. NORTON (J. Amer. Ceram. Soc., 1925, 8, 799-815).-A number of materials were tested for thermal expansion or contraction up to 1700°. The tests were conducted on specimens 9 in. long and 1 in. square in a gas-fired furnace with a neutral atmosphere. The length of the test-pieces in the furnace was measured directly with filar telescopes. The materials tested included silica, kaolin, fireclay, silicon carbide, zircon, zirconia, mullite, magnesite, chrome, spinel, lime (calcined between 1850° and 1900°), fused alumina, and insulating bricks. In a series of tables, these materials are arranged, in order of merit, in accordance with their m.p., mean coefficient of expansion, maximum coefficient of expansion between 300° and 700° (regarded as giving an indication of resistance to spalling), and temperature at which irreversible contraction begins.

F. SALT.

Fractional fusion of refractories. R. HUSTIN (Chim. et Ind., 1925, 14, 691-692).—Bricks which had done service in a reheating furnace, expecially those in the hearth, were found, on examination after 8 days, to have undergone partial fusion at the exposed surface. The fused, surface portion proved, on analysis, to be much richer in alumina and fluxes than the unaltered portion of the brick. This change is shown to be due to a reaction between the fluxes introduced by the coal and the free silica in the brick, resulting in the formation of fusible silicates. A protective coating, rich in alumina, is thus formed on the surface. This is gradually destroyed and a new surface becomes exposed, whereupon the process is repeated. The fusion process is thus assumed to take place in intermittent stages and not continuously. F. SALT.

Use of silica refractories. P. B. ROBINSON (J.S.C.I., 1926, 45, 29-33r).-The constitution and thermal properties of silica bricks of different specific gravities are described, and illustrated by photomicrographs and diagrams correlated with expansion curves, and methods of overcoming expansion troubles are outlined. Silica brick has a higher thermal conductivity and greater refractoriness under load than fireclay; this allows of a greater throughput in carbonisation plants. By suitable selection and treatment of bricks, spalling troubles and the abrasion of the brick due to hot dusty gases can be reduced. Bricks should be stored in a dry place. Jointing material must be of refractoriness comparable with that of the brick and give sound adhesive joints at all temperatures.

Laboratory load furnace [for testing refractories]. P. D. HELSER (J. Amer. Ceram. Soc., 1925, 8, 822—825).—An Ajax–Northrup type of high-frequency induction furnace is so constructed that a load can be applied directly to the test-piece, which is held between two anvils and supported on a scale platform. The resistance material may be either graphite or molybdenum. Change of volume in the specimen can be followed throughout the test by means of an Ames dial. F. SALT.

Porcelain for high-tension insulators. K. H. REICHAU (Trans. Ceram. Soc., 1924-5, 24, 279-301). -Porcelain for high-tension insulators must be a dense, homogeneous body, with high electrical resistance, and considerable mechanical strength. It must be able to withstand rapid temperature changes and be stable under varying atmospheric conditions. Correct proportioning and thorough mixing of the quartz, china clay, and felspar are essential to produce a homogeneous porcelain which will not suffer distortion on firing; the size of the silica grains is most important. A fired cylindrical test-piece, about 6-8 in. in diameter and the same height, should show a dense structure on fracture, and a thin section should show a felted network of sillimanite crystals with very little free silica. The extent of the formation of sillimanite can be estimated by the degree of resistance to hydrofluoric acid. The electrical and mechanical tests of the porcelain must be varied according to the work the insulator is to do; in many cases dynamic tests are more useful than static tests. B. W. CLARKE.

Metal porosimeter for determining the pore volume of highly vitrified ware. L. NAVIAS (J. Amer. Ceram. Soc., 1925, 8, 816—821).—A porosimeter of the McLeod gauge type is constructed of steel to meet the requirements of routine work, for which the glass apparatus is too delicate. The receptacle is in two parts, the lower being made of ordinary steel, and the upper, or cap, of 20% chrome steel, to which the glass capillary can be welded. The results of porosity determinations vary considerably according to the fluid medium used, whether air, water, etc. The term "pore volume" is suggested to distinguish the results of the air method from those obtained by the absorption method. F. SALT.

Method for measuring porosity [of ceramic materials etc.]. P. B. ROBINSON (J.S.C.I., 1926, 45, 33-34 T).—The material is coated with an impervious layer of plasticene, and after weighing in air and in water the bulk density is calculated. From this and the powder specific gravity of the material, the porosity can be calculated.

Testing the cross-bending strength of enamels. R. R. DANIELSON and W. C. LINDEMANN (J. Amer. Ceram. Soc., 1925, 8, 795—798).—An apparatus for measuring the bending strength of enamelled steel sheets consists of stationary supports and a movable roller fulcrum, which is raised vertically in applying the load, by an eccentric arrangement operated by a ratchet wheel. F. SALT.

Laboratory high-temperature kiln. NORTON. --See I.

Refractories for gas retorts. EMERY.—See II. Sintered glass crucibles. ELSDON.—See XVII.

PATENTS.

Utilisation of domestic and industrial refuse [for making glass]. A. GROTE (E.P. 244,358, 16.7.25).—The silicious material separated from town refuse by screening is sintered and is charged into a smelting furnace together with the clinker obtained by the combustion of the coarse screenings. The charge is treated with a mixture in suitable proportions of substances containing silica and saline constituents, e.g., quartz, sand, firebrick, potash, soda, Glauber's salt, in quantity such that a product suitable for glass-making is obtained directly by fusion. A softening agent, such as red lead, and colouring agents, such as cobalt, cupric oxide, iron, uranium oxide, chromium oxide, cuprous oxide, or bone meal, may be added to the charge. H. HOLMES.

Manufacture of abrasives. C. GERGONNE (G.P. 417,888, 4.10.24).—Slags containing alumina, e.g., from aluminothermic or thermoelectric processes, are fused, preferably in an arc furnace, so that the size of the crystal grains is increased. Reducing agents are added during the fusion process, so that on cooling and grinding, reduced iron can be removed magnetically, and other impurities by screening, hand sorting, etc. B. W. CLARKE.

Manufacture of sheet glass. E. C. R. MARKS. From E. DANNER (E.P. 245,362, 30.6.25).

Cleaning chalk-flint stones. W. NOAKE (E.P. 245,613, 30.1.25).

IX.—BUILDING MATERIALS.

Wood preservation. C. SCHANTZ (Brennstoff-Chem., 1926, 7, 1-2).—The preservative effect of creosote on timber is due to the poisonous nature of phenol and the cresols, whereby insects and fungoid growths are destroyed, and to the water-repelling property of the tar oils, whereby atmospheric attack is resisted and decay prevented. In general, it is only the sap wood which is affected, and in order to obtain the maximum effect with the minimum consumption of creosote, it is only necessary to impregnate completely the cells of the sap wood. It is shown how this optimum impregnation may be calculated for pine wood from the specific gravity of the wood itself and of the actual woody tissues ; further the life of the preserved timber only increases asymptotically after a certain degree of impregnation, and consideration of this fact enables considerable economy in creosote to be effected.

B. W. CLARKE.

Determination of the quality of a concrete. R. STUMPER (Bull. Soc. chim. Belg., 1925, 34, 296— 303).—A method is given for calculating the sand : cement ratio in a concrete from determinations of the silica, lime, and loss on ignition of the concrete, cement, and sand respectively. A simpler method requires the silica or lime values only of the three components, the solution being partly graphic. The first method is shown experimentally to be the more exact. A. COUSEN.

Factors governing lime-kiln capacity and fuel economy. Azbe.—See VII.

PATENTS.

Manufacture of fused cement. Soc. DES CIMENTS FRANÇAIS, and BUREAU D'ORGANISATION ECONOMIQUE, Assees. of J. BIED (E.P. 225,858, 4.12.24. Conv., 4.12.23).—The raw materials are subjected to dehydration and decarbonation, at a temperature insufficient to cause partial fusion, and then conveyed to a separate fusion furnace. Partial fusion and blockage in the conduit pipe is prevented by providing a separate passage through which the hot products of combustion (at 1400—1500°) used to heat the fusion furnace are conveyed.

B. W. CLARKE.

Making cement and mortar. O. SIMON (E.P. 244,603, 22.1.25).—Fine meal (ground to pass a mesh of 10,000 per sq. cm.) of stone, cement, slate, slag, etc. is added to cement for waterproofing purposes. In addition, oils, fats, albumins, bitumen, etc. may be used for this purpose; the rate of setting of the product may be regulated by the addition of gypsum meal or an alkali lye, or chlorides of alkali or alkaline-earth metals. Bituminous mixtures are improved by the addition of acid. The density of the product is increased by passing an electric current through the mass while setting.

B. W. CLARKE.

Concrete material. K. P. BILLNER (E.P. 244,671, 22.7.25).—Cement to which powdered zinc or aluminium has been added in the proportion of 0.03-0.2%, is mixed to a grout with water and lime, with or without the addition of fine aggregate. Screened coarse aggregate is added to the grout mix-

ture, forming a porous or expanding concrete material owing to the bubbles of gas evolved from the metal. B. W. CLARKE.

Manufacturing cement. C. LEONARDT (U.S.P. 1,563,755, 1.12.25. Appl., 19.11.23).—Limestone is treated with hydrochloric acid, asphalt or heavy oil is added, and the mixture incorporated with cement, which it renders waterproof and plastic.

T. S. WHEELER.

Tar bitumen emulsions (E.P. 244,561).—See III.

Utilising sulphite-cellulose waste liquor (Swed. P. 57,863).—See V.

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

Thermal treatment of electrolytic iron in a vacuum and recovery of gases. R. HUGUES (Rev. Mét., 1925, 22, 764-775).-A volume of gas equal to two and a half times the volume of iron is obtained by heating electrolytic iron in vacuo for 2 hrs. at 1500°. At 730° the volume of gas liberated is one and a half times the volume of metal. The gas contains CO₂ 6%, CO 34%, H₂ 49–51%, hydro-carbons 4–2%, other gases 7%, and O₂ nil. The curves showing evolution of gas as a function of the temperature indicate the break up of a definite quantity of hydrogen-iron complex for each temperature. The magnetic properties appear to be directly related to the liberation of gas, the point of minimum remanent magnetism corresponding to the maximum quantity of gas evolved. The evolution of gas causes at 100° a progressive variation in the mechanical properties which is not marked by a change in the microstructure. This variation commences before any crystalline change and yields a curve similar to that for evolution of gas as a function of the temperature for the same period of heating. A change in the crystal structure commences at 620° and also a further variation in the mechanical properties. There is a sharp variation in the curve between 730° and 910° due to the combined effects of structural change and evolution of gas on the mechanical properties. The hydrogen-iron complex, which is hard and brittle, is believed to be a compound in solution in iron. M. COOK.

Analysis of precious metal doublé, solderfilled wire, etc. A. SAUERLAND (Chem.-Ztg., 1925, 49, 1078—1080).—As the gold layer in goldsilver or gold-base metal doublé rarely contains less than 40% Au and is therefore not attacked by nitric acid, the silver or base metal layer may be removed from 1 g. of the doublé by treatment with 25 c.c. of 1 : 4 nitric acid, with the addition of tartaric acid if the base metal contains tin or antimony. The gold layer is washed, dried at 120—150°, weighed, and assayed for gold and silver by cupellation. The nitric acid solution is analysed as usual for base metals and silver. Doublé of standard silver and base metal alloy (brass, tombak, or nickel-silver) may be separated by anodic solution of the base

metal layer in 1:5 sulphuric acid, using a platinum cathode, or by treating the doublé with very dilute aqua regia. The silver coating of solder-filled silver wire may be removed by dipping the wire for a few seconds in a mixture of 5 c.c. of strong sulphuric acid and 0.5 c.c. of strong nitric acid. A second layer of nickel-silver may be removed by means of dilute aqua regia, leaving a thin core of solder which is analysed as usual. The gold layer of platinum-gold doublé can be removed only by anodic solution in 5% potassium cyanide solution, in which platinum is insoluble, but the method is very slow. It is preferable therefore to dissolve the whole sample in aqua regia and determine the gold, silver, platinum, and palladium. The silver chloride is reduced by heating in a current of coal gas, and after weighing, the silver is dissolved in nitric acid, any platinum residue being dissolved in aqua regia and added to the main solution. Gold is removed with sodium hydrogen sulphite, the filtrate is oxidised with chlorine, the platinum and palladium are precipitated together by addition of ammonium chloride and alcohol, and the copper is determined by precipitation with thiosulphate. The sum of the platinum and palladium gives the weight of the platinum layer; that of the gold layer is found by difference. If the gold layer consists of "white gold," palladium and platinum may form part of this alloy, in which case the palladium and platinum in the gold layer are separately determined in some thin filings scraped from the lower surface of the doublé and allowance is made for the amount so found in calculating the weights of the two layers. A. R. POWELL.

Separation of the platinum metals. L. WÖHLER and L. METZ (Z. anorg. Chem., 1925, 149, 297-323).-By distillation with acid permanganate or with chromic and sulphuric acids, osmium is separated from all metals, except ruthenium, as the volatile tetroxide. Tellurium remains in the residue as the non-volatile telluric acid. Separation from ruthenium is effected by heating at 275° in a current of nitrous gases. Oxidation to the volatile osmium tetroxide takes place more readily than in oxygen, because in the latter, gas oxidation is hindered by the formation of the non-volatile dioxide. Tellurium is not volatile below 500°, or ruthenium below 600°. Volatilisation of osmium is hindered by sulphur and sulphides, and a higher temperature is then requisite ; separation is still quantitative. Osmium is determined in the nitrous distillate by reducing with Devarda's alloy and distilling with permanganate or chromic acid. In nitrous gases tellurium forms the monoxide as an unstable intermediate product. When heated in nitric oxide to 520°, osmium forms the dioxide, OsO2. Rhodium and iridium are separated by chloridising with excess of sodium chloride at 650°, when the double salts Na2IrCl6 and Na3RhCl6 are obtained. These are separated by extracting with a mixture of equal volumes of acetone and ether, in which the iridium salt is soluble. Rhodium is also separated from iridium and ruthenium by fusing with more than 20 times its weight of bismuth. A rhodium-bismuth alloy is formed which is

completely dissolved by hot 50% nitric acid. When less bismuth is used a new crystalline compound RhBi₂, is formed. Ruthenium is separated from iridium and rhodium by fusing with sodium hydroxide at 550°. The greater part of the ruthenium is converted into sodium ruthenate, and the remainder into ruthenium dioxide, which is soluble in nitric acid. Iridium forms the dioxide and rhodium the sesquioxide, both of which are insoluble in nitric acid. Iridium is partially dissolved by molten potassium hydroxide and, to a greater extent, by potassium carbonate, on account of its higher m.p. Ruthenium is also separated from rhodium and iridium as the volatile tetroxide by distillation with sodium hydroxide solution and chlorine. If it is alloyed with iridium it is necessary first to convert it into the soluble ruthenate by repeated fusion, preferably with potassium hydroxide and potassium nitrate. The platinum metals can therefore be separated as follows. Osmium is volatilised in nitrous gases, and platinum and palladium extracted from the residue with aqua regia. From the solution platinum is precipitated with ammonium chloride. Rhodium is removed from the undissolved residue by fusion with bismuth, and the iridium and ruthenium are separated by fusion with sodium hydroxide or by distillation with alkali chloride. The order of removal of rhodium and ruthenium may be reversed.

A. GEAKE.

System aluminium-zinc. O. TIEDEMANN (Z. Metallk., 1926, 18, 18-21).-After ageing for 6 weeks at the ordinary temperature alloys of zinc and aluminium containing 0-20% Zn, which have been annealed at temperatures between 250 and 500° for several hours, characteristic changes take place in the ductility and tensile strength so that the field of the aluminium-zinc solid solution may be divided into four parts according to the behaviour of the alloys during the above treatment. Field I. is bounded by curved lines joining the points (560°-0% Zn) and $(275^{\circ}-6\%$ Zn) and the latter point with $(0^{\circ}-3\%$ Zn), field II. by the first line, the horizontal at 275°, and a second curved line joining the points (560°-0% Zn) and (275°-17% Zn), field III. by the last-named line, the solidus, and the boundary between the α solid solution and the heterogeneous region, and field IV. by the continuation of this boundary below 275°, the 275° horizontal, and the lower curved line of field I. Alloys containing less than 3% Zn in field I. consist entirely of β solid solution, which remains unchanged on ageing, whilst only a small increase takes place in the hardness and ductility. Alloys containing 3-6% Zn are in this field above 250° and in field III. below this temperature, so that, on ageing them after quenching, the β solution slowly breaks down into a "new crystal form" and the tensile strength is increased whilst the ductility shows a slight increase (quenched from above 350°) or decrease (quenched from below 350°). Alloys in field II. contain α and β solid solutions in equilibrium but, on cooling below 275°, field IV. is entered and the alloys then consist of β solid solution and the "new crystal form";

consequently alloys quenched from within this field and then aged show a marked increase in tensile strength but a great decrease in ductility. Alloys quenched from field III. (a solid solution) show similar alterations in tensile strength and ductility on ageing to those in field I. containing 3-6% Zn. On ageing alloys containing more than 8% Zn quenched from temperatures below 275° in field IV. a considerable increase in the ductility and a marked decrease in the tensile strength take place owing to decomposition into β solid solution and free zinc. A. R. POWELL.

Annealing, quenching, and reheating of some industrial nickel-brasses. F. M. OSTROGA (Rev. Mét., 1925, 22, 776—786).—The three alloys investi-gated, containing Cu 50.42%, Ni 6.25%; Cu 44.70%, Ni 10.34%; and Cu 39.65%, Ni 14.75%, consist at ordinary temperatures of α and β . Differential dilatometric curves (Chévenard) do not yield any evidence of an abrupt change below the α - β inversion point, but indicate a progressive transformation similar to that found in ordinary brasses. On cooling, two points are indicated, the position of the higher one being little affected by the cooling rate, while the lower one is depressed to 100° by cooling in air. The martensitic structure has not been obtained in quenched alloys. Heating to 200° has no appreciable effect on the hardness; heating to and heating above 550° results in a rapid fall in hardness. Quenching in oil or water from high temperatures renders the alloys excessively brittle. Maintaining the alloys at high temperatures is distinctly harmful, since the crystals grow rapidly and produce a coarse structure and brittleness. The allovs are correctly hardened by water-quenching from 600° or oil-quenching from 775° and reheating to 550°. The reheating to 550° results in a greater hardness than oil-quenching from 775° without further treatment. М. Соок.

Copper containing bismuth. W. STAHL (Metall u. Erz, 1925, 22, 421–422; Chem. Zentr., 1925, II., 2324–2325).—Copper containing 0.02% Bi as metal is red-short whereas that containing 0.05% is appreciably cold-short. If the bismuth is converted into antimonate or arsenate 0.06% Bi does not render the metal red-short. In order to overcome the deleterious effect of small amounts of bismuth in copper, the metal is melted with the requisite amount of copper-arsenic alloy, allowed to oxidise, and finally poled until the oxygen content is reduced to 0.08%. After this treatment the metal has all the mechanical properties of tough-pitch copper.

A. R. POWELL.

Viscosity of alloys at high temperature. J. COURNOT and K. SASAGAWA (Rev. Mét., 1925, 22, 753-763; cf. B., 1925, 994).—An apparatus is described for determining the rate of flow and the viscosity limit of metals, and details are given of a special compensating device to eliminate errors due to the diminution of cross-section of the test-piece

with elongation. In the work of previous investigators this source of error has not been taken into consideration. The test-pieces were 1 sq. mm. in section and 100 mm. long and were heated in an electric furnace in an atmosphere of nitrogen. The amount of elongation produced by a given load at definite temperatures has been plotted against time for several carbon and alloy steels and the viscosity limits are deduced from these curves. The time of application of the load was $1\frac{1}{2}$ hr. For a soft carbon steel the limit of viscosity is 19.7 tons/in.² at 350° and falls to 2.5 tons/in.² at 600°. The limit of a nickel-chromium alloy falls from 26.7 tons/in.² at 550° to 1.9 at 850° and that of silicon-chrome steel from 38.1 at 500° to 0.6 at 800°. The increase in the rate of flow due to small additional load is more pronounced as the temperature is raised and for special steels at 800° is less than it is for ordinary steels at 600°. The importance of the viscosity limit in fixing factors of safety is emphasised. M. COOK.

Recrystallisation [of metals]. H. HANEMANN (Z. Metallk., 1926, 18, 16-17).-A further discussion of the results of earlier work (cf. B., 1925, 674, 925), in which it is shown that the harder a metal is at the temperature of rolling the greater is the amount of work required to obtain a predetermined grain size after annealing. If, however, the metal is subjected to too severe a rolling the grain size after recrystallisation will not be appreciably reduced and the desired improvement in the mechanical properties of the metal will not be produced. On the other hand, too gentle rolling may result in a coarse grain structure owing to the amount of work expended being below the minimum necessary to produce recrystallisation. The latent energy induced in the metal by rolling is proportional to the degree of rolling and therefore inversely proportional to the grain size after recrystallisation. The fact that recrystallisation of a worked metal commences along the slip planes and that the cause of hardening is also to be found in the slip planes indicates that there is a definite relation between the grain size of a metal after recrystallisation and the hardness induced by deformation.

A. R. POWELL.

Present position of the electric furnace for the melting of non-ferrous metals. M. TAMA (Z. Metallk., 1926, 18, 7—14).—A review of the present position of electric furnaces, especially induction furnaces of the Ajax–Wyatt and Northrup type, for use in the melting of non-ferrous metals and alloys. in which are briefly discussed the effects of the electrical resistance, volatilisation point, specific heat, and viscosity of brass on the design of the furnace and its efficiency and on the life of the refractories. A. R. POWELL.

Tensile properties of single iron crystals and influence of crystal size on the tensile properties of iron. C. A. EDWARDS and L. B. PFEIL (J. Iron and Steel Inst., 1925, 112, 79-110).—See B., 1925, 806. Orientation of crystals produced by heating strained iron. C. F. ELAM (J. Iron and Steel Inst., 1925, 112, 111—112).—See B., 1925, 806.

Dilatation of cast iron during repeated heating and cooling. J. H. ANDREW and R. HIGGINS (J. Iron and Steel Inst., 1925, 112, 167-189).— See B., 1925, 806.

Magnetic and electrical properties of cast iron. J. H. PARTRIDGE (J. Iron and Steel Inst., 1925, 112, 191-224).—See B., 1925, 805.

Direct process for manufacture of steel. H. FLODIN (J. Iron and Steel Inst., 1925, 112, 9-25). -See B., 1925, 807.

Effect of temperature on the behaviour of iron and steel in the notched-bar impact test. R. H. GREAVES and J. A. JONES (J. Iron and Steel Inst., 1925, 112, 123—165).—See B., 1925, 807.

See also A., 1926, 111, X-Ray analysis (GÜNTHER and STRAUSKE). 112, X-Ray examination of a-iron plastically strained (ONO); X-Ray examination of mechanism of crystal rearrangement and cause of strain hardening (ONO); Allotropy of chromium (BRADLEY and OLLARD); X-Ray examination of gold-copper and palladiumcopper mixed crystals (JOHANSSON and LINDE); Density of tungsten (DAVEY); Arrangement of micro-crystals in rolled platinum plate (TANAKA). 117, Allotropy of zinc (STOCKDALE); Vapour pressures of metals (RODEBUSH and DIXON). 119, Alloys of titanium and aluminium MANCHOT and LEBER). 131, Catalytic phenomena when zinc is dissolved in acids (CENTNERSZWER and STRAUMANIS). 133, Passivity, catalytic action, etc. (RUSSELL). 135, Electrolytic deposition of nickel (SAXON). 138, Solubility of ruthenium in hypochlorite solutions (Howe and MERCER); Behaviour of carbon monoxide towards salts of palladium and platinum (MANCHOT). 140, Determination of copper (Azzalin); Differential reaction of cerium (FERNANDES); Electrometric determination of iron with bromate (COLLENBERG and SANDVED). 141, Benzoylmethylglyoxime as precipitant of palladous salts (HANUS, JILEK, and LUKAS); Separation of iridium from rhodium and platinum, and separation of platinum and rhodium (WADA and NAKAZONO).

Rust-protective power of paints. HERRMANN. --See XIII.

PATENTS.

Treating [iron] ores. W. R. VAN SLYKE (U.S.P. 1,565,689, 15.12.25. Appl., 7.3.21).—The ore is sintered to obtain a porous clinker which is then smelted with a non-porous fuel.

A. R. POWELL.

Purification of cast iron. J. G. PLATON (F.P. 591,053, 27.12.24).—Air is introduced under pressure into the molten iron, through openings in the wall of

the furnace or through refractory ducts which pass through the wall and terminate below the surface of the metal. Additional heat is not required, since the removal of sulphur and silicon which takes place is an exothermic reaction. The purified iron is drawn off, leaving the impurities behind as a slag, at the end of the process. B. W. CLARKE.

Decarbonisation of metals, especially iron and steel. Soc. DES ACIÉRIES ET FORGES DE FIRMINY (F.P. 591,981, 13.3.24).—The metal is decarbonised by hydrogen saturated with steam at 40°, in vessels from which the air has been removed. The hydrogen is saturated by passing through warm water, then through a fine spray of water, and is finally heated to 100°. The carbon monoxide and dioxide and methane produced may be removed from the gas mixture, or a corresponding amount of gas allowed to escape into the air. The carbon content of the iron is reduced to less than 0.01 %. B. WiCLARKE.

Manufacture of deoxidised iron or steel. W. TAFEL (G.P. 399,906, 19.6.23 and 409,347, 3.6.24).—Slag from weld iron or a slag of similar composition is spread on the ingot mould in a solid state before the metal is poured, and the molten metal mixed well therewith. The product is suitable for hot working and is stable at high temperatures. The method described prevents melting of the cast iron plates at the bottom of the mould.

B. W. CLARKE.

Iron blast furnace process. HALBERGERHÜTTE G.M.B.H. (G.P. 404,700, 16.11.23).—To ensure smoother running of a blast furnace, especially when a high temperature is required, oxygen and fuel in the ratio necessary for the formation of carbon monoxide are simultaneously blown into the furnace. A. R. POWELL.

Desulphurisation of iron and steel. W. KROLL (G.P. 418,074, 12.3.22).—The molten metal is treated with a mixture of alkalis and strong reducing agents, such as calcium carbide, aluminium, magnesium, copper, and silicon, so that the material of the walls of the bath is not attacked by the alkali. The sulphur content of an iron may thus be reduced from 0.12% to 0.06%. B. W. CLARKE.

Increasing the amount and phosphoric acid content of slag from the Thomas process. H. HILBERT (G.P. 418,102, 20.2.25).—The limestone or dolomite added in the converter process is replaced by calcium phosphate, forming a slag containing the phosphate in a readily soluble form, with no harmful effects on the properties of the steel. B. W. CLARKE.

Refining the grain of steel. F. KRUPP A.-G., Assees. of H. SCHOTTKY (G.P. 418,124, 6.11.23).— Steel is heated to above the upper transformation temperature (Ac2), quenched rapidly in a bath of liquid, and subjected to a second heating to just above the upper transformation temperature, followed by a moderately rapid cooling. The product posresses exceptional strength and toughness.

B. W. CLARKE.

Electrolytic iron. SIEMENS U. HALSKE A.-G., Assees. of H. GERDIEN (G.P. 418,139, 23.6.23).— Cast iron anodes with a carbon content of more than 3% are used, so that after dissolution of the iron, a skeleton of carbon still remains. The voltage should be less than 1 volt and the temperature at the bottom of the vessel should be as low as possible.

B. W. CLARKE.

Coating ferrous metals to make them rust-proof. C. F. HENDRICK (E.P. 244,523, 18.9.24).—Ferrous metals are subjected to the heat of an intensely hot flame whereby superficial fusion is produced, and simultaneously a protective nonferrous metal or alloy, of lower melting point than iron or steel, is fed in a finely divided state at a rate such that it becomes diffused in molten form on or in the fused ferrous surface. The protective metal is introduced either directly on to the ferrous surface or, in powdered form, is driven on by the gases of the flame-producing mechanism or by a separate air stream. Oxidation during the process may be prevented by the use of a deoxidising flux.

L. M. CLARK.

Electrodepositing nickel, cobalt, or nickel alloys. E. POTTER. From MADSENELL CORP. (E.P. 244,166-7, 11.9.24).—(A) Soft, highly malleable and ductile nickel deposits may be obtained from a bath containing 240 g. of nickel sulphate crystals, 20 g. of nickel chloride, and 40 g. of boric acid per litre of water if the bath is first neutralised with sodium hydroxide or nickel hydroxide and treated with chlorine to oxidise and precipitate the iron and copper impurities. The bath is filtered before use and 4 c.c. of 3% hydrogen peroxide per litre are added every 24 hrs. The anode consists of nickel or a nickel alloy containing more than 0.1% of oxygen and is obtained by adding nickel peroxide to, or passing air through, molten nickel until a black deposit forms on the surface of the metal. (B) Hard, but malleable and ductile nickel containing more or less iron is deposited from a bath containing 300 g. of nickel sulphate crystals, about 30 g. of nickel fluoride or nickel hydrogen fluoride, and a small amount of quinol per litre. The anode consists of nickel or a nickel alloy free from chromium and silicon but containing more than 0.5% C; it is made by stirring molten nickel at 1500° in contact with carbon for 30 min., cooling nearly to the m.p., and casting quickly. A. R. POWELL.

Electroplating. J. G. SWAIN, ASST. to FIRESTONE STEEL PRODUCTS CO. (U.S.P. 1,565,683, 15.12.25. Appl., 12.11.23).—The article to be electroplated is treated in an electrolyte charged simultaneously from anodes of zinc and mercury. A. R. POWELL.

Producing electrolytic copper. F. L. ANTISELL (U.S.P. 1,566,265, 22.12.25. Appl., 11.1.22).— Solutions containing copper are electrolysed in cycles, the current being passed through the solution for a predetermined fraction of each cycle. L. A. Coles.

Electroplating method. R. J. SHOEMAKER (U.S.P. 1,566,984, 22.12.25. Appl., 1.10.23).—In electroplating iron articles the surface is covered with a permanently adherent film of mercury and a metal capable of forming an amalgam with mercury, and then plated. M. Cook.

Electrolytic deposition of chromium. G. LE BRIS (F.P. 590,777, 1.12.24).—Chromium is deposited electrolytically from a bath containing chromic salts together with salts or acids having an oxidising action, using lead anodes, a current of 4—6 volts and 7—8 amp. per sq. dm., and a temperature of 35—40°. L. A. COLES.

Welding. C. H. HUMPHRIES, ASST. to METALS PROTECTION CORP. (U.S.P. 1,563,748, 1.12.25. Appl., 31.8.25).—Iron or steel surfaces to be welded are treated with a solution of chromic acid and then allowed to dry before welding. Sputtering of the metal is thus prevented. T. S. WHEELER.

Recovering tin from ores. J. R. STACK, Assr. to AMER. SMELTING AND REFINING CO. (U.S.P. 1,566,352, 22.12.25. Appl., 23.5.19).—Ores, concentrates, and by-products containing compounds of tin, iron, and other elements are crushed, heated in a closed receptacle to a temperature above the reducing point of tin and below the reducing point of iron, and subjected to the action of a gas which will reduce tin oxides. Tin so produced is removed, and the residue treated for the recovery of other metals.

L. M. CLARK.

Treating sulphide ores and concentrates. M. DE KEYSER (U.S.P. 1,566,379, 22.12.25. Appl., 21.7.22).—The material is roasted to produce sulphur dioxide which is then mixed with ozone and passed in contact with a catalyst. M. COOK.

Producing an alloy of copper. C. KAZEMER (U.S.P. 1,566,406, 22.12.25. Appl., 11.6.25).—Copper is melted with glass and made into sheets which are reduced in thickness by pressure and re-melted with a mixture of silver and glass. This melt is formed into a sheet which is reduced in thickness and into a workable form. M. COOK.

Method of electrolytically producing aluminium. J. B. RAILSBACK (U.S.P. 1,566,694, 22.12.25. Appl., 20.10.24).—The bath contains compounds of aluminium and a metal the oxide of which is not reducible by aluminium. M. Cook.

Treating ores. C. A. MCCOURT (U.S.P. 1,566,755' 22.12.25. Appl., 12.1.24).—In a continuous process for recovering the lighter metal values from slimes, an amalgamating solution is caused to flow through an electrolytic cell and streams of this solution are then diverted on to the slimes at separate points. The amalgamated lighter values are separated from the solution and the spent liquor is returned to the electrolytic cell to prepare it for use again in the process. L. M. CLARK.

Apparatus for manufacturing metal dust. M. H. NEWELL, Assr. to ALLOYS CO. (U.S.P. 1,566,913, 22.12.25. Appl., 29.8.16).—In an apparatus for manufacturing metal dust by condensing vaporised CL. XI.-ELECTROTECHNICS. CL. XII.-FATS; OILS; WAXES.

metal, a condensing chamber is provided with an aperture and means for retarding the inflow of air through the aperture, the chamber being otherwise L. M. CLARK. substantially airtight.

Heat treatment of grey iron castings. A. K. SCHAAP (E.P. 245,172, 1.7.24).—See U.S.P. 1,514,070; B., 1925, 44.

Deoxidation of ingot iron and steel. W. TAFEL (E.P. 245,486, 18.9.24).—See G.P. 399,906; preceding.

Treatment of oxidic raw materials. T. R. HAGLUND (U.S.P. 1,569,483, 12.1.26. Appl., 5.8.22). -See E.P. 232,549; B. 1925, 957.

Metal melting furnaces. L. HALL (E.P. 245,390, 24.12.24).

See also pages 145, Platinum-ruthenium catalyst (G.P. 418,868). 147, Blast-furnace gas purification (E.P. 244,372). 153, Treatment of black liquor (Can. P. 245,831). 156, Precipitating tin (E.P. 244,526); Purifying zinc sulphate solutions (Swed. P. 57,951). 158, Abrasives (G.P. 417,888). 164, Electro-thermal process (F.P. 592,045).

XI.—ELECTROTECHNICS.

High-frequency induction furnace. D. F. CAMPBELL (J. Iron and Steel Inst., 1925, 112, 69-77). -See B., 1925, 812.

See also A., 1926, 104, Action of visible light on electrodes (AUDUBERT). 115, Unipolarity pheno-mena in compressed powders (TREY). 130, Overvoltage (MEUNIER); Electrolysis of acid solution of copper sulphate (REDMAN). 134, Production of sulphuric acid by electrolysis (SAXON). 147, Electrolytic reduction of aldehydes (SHIMA).

Electric-furnace melting non-ferrous of metals. TAMA.-See X.

PATENTS.

Galvanic cell of the copper oxide type. G. W. HEISE, Assr. to NAT. CARBON Co. (U.S.P. 1,563,980, 1.12.25. Appl., 27.5.24).-In a galvanic cell with a copper oxide depolariser and caustic soda as electrolyte, the depolariser is suspended in the electrolyte in a container lined with cellulose. This prevents any dissolved copper passing out into the cell where it would damage the zinc electrode. T. S. WHEELER.

Electrolysis of alkali chlorides. H. KLOPSTOCK (U.S.P. 1,565,943, 15.12.25. Appl., 25.5.25).-In the use of the mercury process the mercury is protected by an air-excluding fluid throughout its transfer by a propelling device from the amalgamdecomposition cell to the amalgam-formation cell. H. HOLMES.

Impregnated electrode for furnace work. H. A. LAVENE, Assr. to ACHESON GRAPHITE CO. (U.S.P. 1,566,409, 22.12.25. Appl., 5.2.23).—An electrode having in its pores a refractory compound formed by the interaction of two soluble compounds is claimed. M. COOK.

Electric furnace. A. GRAUEL (U.S.P. 1,566,855, 22.12.25. Appl., 30.4.23).-The furnace consists of a casing enclosing a water-jacket and a central closed heating chamber, the bottom portion of which is lined with an electrical insulating material. A bed of loose carbon particles is arranged within the lined portion of the chamber and electrodes are inserted through, and insulated from, the waterjacket walls. The carbon particles form a heatradiating body directing heat rays on to the inner surface of the water-jacket. М. Соок.

Electro-thermal process. Norske Aktiesel-SKAB FOR ELEKTROKEM. IND. (F.P. 592,045, 23.1.25. Conv., 8.2.24).-Electrodes having a higher electrical resistance than carbon, constructed, e.g., of mixtures of carbon, material of high resistance, and binding material, are used in heating electric furnaces. The material to be heated is stacked around the electrodes, which may be provided with metal ribs to facilitate passage of the current. The electrodes may be constructed of the reacting material itself, e.g., of mixtures of carbon, iron or zinc ores, and binding material, in which case they may be up to 2 m. in diameter. L. A. COLES.

Ozoniser. P. J. ARENA (F.P. 592,644, 8.11.24).-The apparatus generates a high-tension current, producing sparks which ozonise a current of air passing through the apparatus. L. A. COLES.

Apparatus for electrolysing fused salts of metals and recovering the metals and acid radicals. E. A. ASHCROFT (U.S.P. 1,569,606, 12.1.26. Appl., 6.2.24).—See E.P. 215,872; B., 1924, 602.

Diaphragms to be used in electric batteries. M. WILDERMAN (E.P. 245,252, 12.9.24).

See also pages 145, Treating gases at high temperatures (F.P. 593,298). 147, Electrical gas purification (E.P. 244,372); Insulating oils (G.P. 417,835). 147-8, Dehydrating emulsions (U.S.P. 1,565,992 and 1,565,997). 153, Treatment of black liquor (Can. P. 245,831). 158, Abrasives (G.P. 417,888). 159, Cement and mortar (E.P. 244,603). 170, Purifying sugar juice (F.P. 591,198).

XII.—FATS; OILS; WAXES.

Tung oil. W. NAGEL and J. GRÜSS (Z. angew. Chem., 1926, 39, 10-13).—The earlier theories of the gelatinisation process of tung oil are briefly indicated, attention being mainly given to the views of Mar-cusson, Grün, and Wolff. The last-named's insistence on the absence of molecular polymerisation has been criticised by various workers on the ground of the uncertainty attaching to molecular weight determinations, and the present authors use the iodine value as their criterion. When methyl α - and β -elæostearates were heated at different temperatures for various lengths of time in an atmosphere of carbon dioxide, a limiting value of 80-90 for the iodine value was obtained, further heating inevitably producing destructive distillation. The iodine value

of methyl elæostearate is 172.5, so that the observed value indicates the formation of a dimeride. Similar treatment of tung oil yielded a limiting iodine value of 110-120 for a product just about to gelatinise. In this connexion it was found that 10% ethereal hydrogen chloride was an effective reagent for liquefying without depolymerising, freshly gelatinised tung oil. The iodine value of elæostearin being 173.3, the fall in this value corresponds to polymerisation to the extent of 60% of the original oil. Typical formulæ for the dimeride are given, all of which are characterised by the presence of a tetramethylene ring. By parallel experiments with amyl and glycol esters of elæostearic acid it was shown that the solidification of the dimeride is dependent on the nature of the alkyl group, since methyl and ethyl esters remain liquid, the amyl ester partially solidifies, whilst the glycol and glyceryl esters completely solidify on polymerisation. This is in harmony with the authors' views and previous work on shellac (cf. B., 1923, 1234 A), whereas according to Wolff the methyl ester must differ fundamentally from the glyceryl ester, a view not borne out by these experiments. S. S. WOOLF.

Detection and determination of coconut oil and milk fat in cacao butter. J. KUHLMANN and J. GROSSFELD (Z. angew. Chem., 1926, 39, 24-25).-The method is similar to that used by Bertram, Bos, and Verhagen (Z. Deuts. Oel- u. Fett-Ind., 1924, 44, 445-447, 459-461) for the determination of coconut oil and milk fat in margarine, and furnishes two values : the "A" number, for the fatty acids the magnesium soaps of which are soluble and the silver soaps insoluble in water, and the "B" number for volatile fatty acids with soluble silver soaps. Both numbers are in c.c. of 0.1 N-acid (or 0.1 N-silver nitrate and 0.1N-sodium hydroxide) expressed on 6.4 g. of fat. An examination of many pure cacao fats shows that the "A" number does not rise above 0.2 and is generally about 0.1, whereas that of pure coconut oil is 27.7 and of milk fat 6.7, the "B" numbers being 0.3, 2.75, and 33.4 respectively. Both values for mutton tallow, mowrah fat, sesamé oil, linseed oil. and whale oil are below 1.16; for palm kernel oil the "A" value is 16.53-16.76 and the "B" value 1.80-1.92. A special advantage of the method is that the "A" value practically represents the coco-nut oil content, and the "B" value the milk fat content, an "A" number of 1 c.c. corresponding to 3.6% of coconut oil. Exactly 20 g. of the fat and 30 g. of glycerol are heated with 8 c.c. of 75% potassium hydroxide solution in a tared flask until saponification is complete. Warm water is added to make 409 g., the temperature adjusted to 80°, and 103 c.c. of magnesium sulphate solution (150 g. MgSO4,7H2O in 1 litre) at 80° are added with shaking, which is continued for 10 min. The liquid is cooled to 20°, kept for 5 min., and filtered. 200 c.c. of the filtrate are added to 20 g. of sodium nitrate and neutralised with 0.5N-sulphuric acid using phenolphthalein (1 drop), 25 c.c. of 0.2N-silver nitrate are added, and then water to 250 c.c. After shaking and keeping in water at 20° for 5 min.

the mixture is filtered and the excess of silver nitrate in 200 c.c. of filtrate determined by Volhard's method. The difference between this and the blank is the "A" number. 200 c.c. of the filtrate from the magnesium soaps are neutralised with 0.5N-sulphuric acid, made up to 250 c.c. with water, and 2 g. of powdered silver nitrate added with shaking at 20°. It is then filtered and 200 c.c. of the filtrate are acidified with 50 c.c. of dilute sulphuric acid (2.6%) and distilled from a 500-c.c. flask. 200 c.c. of the distillate are titrated with 0.1N-sodium hydroxide solution and the result minus the number of c.c. for the blank is the "B" number, E. H. SHARPLES.

Free thiocyanogen and its application in volumetric analysis. New criterion for fats and oils. H. P. KAUFMANN (Arch, Pharm., 1925, 263, 675-721).—The chemistry of thiocyanogen is exhaustively reviewed, with copious citation of the literature (cf. in particular, Bjerrum, "Die Rhodanide des Goldes und das freie Rhodan," Copenhagen, 1918; Söderbäck, A., 1920, i, 219); the pharmacological importance of thiocyanogen compounds is similarly discussed; and the following new observations and applications are described in detail. Free thiocyanogen, which can be prepared in various ways (cf. Söderbäck, loc. cit., Kaufmann and co-workers, A., 1925, i, 1252, etc.), displaces iodine from iodides, but is displaced from thiocyanates by bromine. It takes part in addition and substitution reactions like a halogen, being less active than bromine but more active than iodine (cf. Kaufmann and co-workers, A., 1923, i, 765; 1924, i, 209, 835, 840). On these facts new titration methods, applicable to fats and oils, and yielding results not otherwise obtainable, are based. For this purpose the thiocyanogen is obtained as a solution in an indifferent solvent (by interaction of lead thiocyanate and bromine). The stability of the solution depends on the nature of the solvent, as this has an influence on the rate at which polymerisation and decomposition take place. The nature of these changes is discussed at length (cf. Lecher and co-workers, Ber., 1923, 56, 1204). The strength of the solution decreases at first slowly, but later very rapidly. Thus, in pure carbon disulphide 7% disappears in 12 days, whereas in technical carbon disulphide over 90% is lost in the same time. Similar losses take place in chloroform, carbon tetrachloride, and ethylene dichloride, the last of these being itself attacked. Ether and methyl alcohol likewise undergo substitution, both very Solutions in acetic acid (absolutely rapidly. anhydrous) are, however, more stable, i.e., there is no loss in 3 days, and such solutions, in 0.05N or 0.1N strength, are well adapted for volumetric work. A series of "rhodanometric" determinations of the iodine values of fats and oils has accordingly been made, using 100-150% excess of thiocyanogen, and determining the excess by means of potassium iodide (cf. A., 1924, i, 840). The results are calculated as if iodine were used so as to be directly comparable with the usual iodine values. In the list following these figures are denoted by R and I respectively, the iodine values proper being

166

determined either by the Hanus method or by Kaufmann's bromometric method (cf. A., 1925, ii, 555). The period of interaction is indicated in each case. Castor oil, 6-12 hrs., R, 81.6; I, 82.6. Olive oil, 5 hours, R, 76.6; I, 80.8. Arachis oil (three samples), 12-15 hours, R, 78.5, 70, 68.1; I, 87.1, 89.3, 88. Almond oil (two samples), 14 hours, R, 83.8, 85.2; I, 97.59, 97.9. Rape oil, 5 hours, R, 77.4; I, 105.9. Sesamé oil, 7 hours, R, 75.5; I, 108.9. The differences between the two sets of figures in some cases are due to the selective action of the thiocyanogen (cf. Kaufmann and Wolff, A., 1924, i, 835). The following figures were obtained for purified fatty acids. Oleic acid, R, 90.6; I, 90.6. Elaidic acid, R, 81.5; I, 81.6. Erucic acid, R, 73.7; I, 74.0. Brassidic acid, R, 73.2; I, 73.8. These results show that compounds with one ethylenic linkage give the same result by either method and that stereoisomerism has no effect. Stearolic and behenolic acids are not affected by the reagent. Linoleic acid (as glyceryl ester), however, gave the numbers : R, 82.5; I, 169.1, i.e., the thiocyanogen attacks only one ethylenic linkage. From the results obtained the composition of various natural oils was calculated. Castor oil is thus found to contain 1.15% of linoleic triglyceride; olive oil has the composition 10.92% of glycerides of saturated acids, 84.2% of olein, 4.88% of linolein; in arachis oil (third sample) the proportions are $21\cdot2\%$, $55\cdot66\%$, and $23\cdot14\%$ respectively; and in almond oil (second sample) $1\cdot5\%$, $83\cdot73\%$, $14\cdot77\%$ respectively. Rape oil contains $33\cdot14\%$ of linolein, 10.4% of saturated glycerides, the remainder being those of erucic and rapinic acids. Sesamé oil consists of 38.84% of linolein, 48.61% of olein, and 12.55% of saturated compounds. These results compare favourably with those obtained by a laborious chemical separation, in the cases in which such has been carried out. W. A. SILVESTER.

Rapid method for determining reaction velocity in soap boiling. G. I. FINCH and A. KARIM (J.S.C.I., 1926, 45, 35—36 T).—A sample of the reaction mixture is diluted to a 2—3% solution, 25 c.c. of which are saturated with sodium chloride and then titrated against standard acid with phenolphthalein as indicator. Near the end-point some light petroleum is added to break down the grains and after shaking the titration is completed. The total alkali is obtained by titrating a separate portion, using methyl-orange as indicator. The results are corrected for carbonate, if any is present. The difference of the two readings gives the combined alkali, from which, and the saponification value of the original oil, can be calculated the degree of saponification.

See also A., Feb., 1926, 122, Viscosity of soap solutions (CLARKE). 123, System sodium stearate-water (Von BUYAGH). 134, Catalytic hydrogenation (SCHMIDT).

Waxes of cottons of different origin. LECOMBER and PROBERT.—See V.

Identifying fatty ingredients in sized goods. LECOMBER and PROBERT.—See V.

PATENTS.

Extraction of oil from oil-bearing substances. A. W. SIZER (E.P. 244,557, 25.10.24).—The material, with or without preliminary heating and crushing, is forced through perforated vessels, *e.g.*, by a worm, and the oil is thus continuously removed, after which the material is treated with volatile solvent, to extract the remaining oil. D. G. HEWER.

Production of soaps. R. VIDAL (F.P. 594,146, 26.2.25).—Oils, fats, and waxes are treated with alkali hydroxides in the presence of aldehydes, such as furfuraldehyde and methylfurfuraldehyde. The soaps thus obtained are specially suitable for brightening sulphur dyes. A. GEAKE.

Grease from slaughterhouse offal (U.S.P. 1,567,014).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Isomerism of Guignet's green, a phenomenon of particle size. L. WÖHLER and J. DIERKSEN (Z. angew. Chem., 1926, 39, 13-16).-Guignet's green is a hydrated oxide of chromium, produced in practice by fusion of potassium dichromate with boric acid, and the failure of attempts to convert the dull olive-green precipitated hydroxide into the brilliant pigment by heating with water under pressure, supported the view that the compound 2Cr₂O₃,6B₂O₃ is responsible for the brightness. It is now shown that the presence of boric acid is not essential to Guignet's green, for which a gel structure is suggested. Reduction in the amount of water in chromium hydroxide gels, with increase in particle size, is found to increase the brilliance of the product progressively. Boric acid, silicic acid, etc. are effective in producing the required flocculation. Chromium oxides of differing hydration and particle size were prepared both from the pure precipitated hydroxide and by various flocculation processes, and the vapour pressure-temperature curves for these are discussed; brilliant flocculated hydroxides show higher vapour pressure than dull non-flocculated hydroxides of identical water-content. X-Ray photographs reveal the absence of any lattice structure.

S. S. WOOLF.

Fastness of pigments to light and its determination in natural and artificial light. H. WAGNER (Z. angew. Chem., 1925, 38, 1191—1195).— In estimating the fastness of a dyestuff or pigment it is important to follow the course of its fading, and this may be done graphically by plotting time as abscissæ and the corresponding shade according to the Ostwald scale as ordinates. Curves of a similar nature are usually obtained by illuminating with sunlight or with a "Uviol" (mercury) lamp, but the latter is generally 5—30 times as intense in action. A better method is to plot the progress of illumination by means of Ostwald's triangular axes, omitting time. The nature of the binding medium may have a profound influence on the fastness of a pigment, and its refractive index should be as near as possible to that of the pigment. The least favourable medium for fastness to light is an aqueous glue wash, and the most favourable an oil or concentrated dextrin medium in which the medium constitutes an optical bridge and allows the light to strike deeper into the colour. This influence of binding medium on the behaviour of pigments on illumination is also best expressed graphically by means of triangular coordinates. W. T. K. BRAUNHOLTZ.

Heterochromatic photometry. C. SCHAEFER (Physikal. Z., 1925, 26, 908—913).—Earlier work on the measurement of the brightness of pigments (B., 1925, 179) is extended, confirmed, and placed on a more accurate basis. R. A. MORTON.

Testing the rust-protective power of paints. P. HERRMANN (Korrosion u. Metallschutz, 1925, 1, 80—84; Chem. Zentr., 1925, II., 2326).—The painted surface of the iron is covered with a layer of cotton wool saturated with a solution of an electrolyte and in contact with a calomel electrode, and the point at which the paint film has been perforated is ascertained by means of a galvanometer. With micaceous paints rust can sometimes be observed even when there is no deflection of the galvanometer.

A. R. POWELL.

Tung oil. NAGEL and GRÜSS.-See XII.

PATENTS.

Preparation of lakes from organic dyestuffs. L. D. GIMBERG and M. H. BAILLY (F.P. 582,506, 29.4.24).—An aqueous or alcoholic solution of an aniline salt is treated with an organic sulphonic acid, and a nitro- or aminoazo-compound is added to the mixture. After filtering off the organic acid the filtrate is treated with a solution of an aluminium salt and the precipitated lake is washed free from all soluble substances. It may then be mixed with pastes of aluminium hydroxide or silicic acid.

A. R. POWELL.

Lakes (F.P. 590,464).—See IV.

Monoazo dyestuff (Swiss P. 109,706).—See IV.

Plastic masses (E.P. 241,858).-See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Spraying [rubber] latex with protective colloids. W. C. DAVEY (J.S.C.I., 1925, 44, 515—517 T). —Experiments on the spraying of latex to powder form by means of protective colloids, using an experimental Krause machine, are described. Glue was found to be the most suitable substance. Compounded moulded articles were made from suitable powders by pressing and curing in plunger moulds. With vulcanised latex 3% of glue yielded a powdery product which could also be moulded to shape. The use of a sprayed glue-rubber powder mixture is suggested as a suitable means of incorporating glue in rubber mixings on the rolls, and results are shown indicating improved reinforcement. The uncured mixes tend to deteriorate during storage. When using glue it is advisable to have zinc oxide present in the mix in order to obtain the best results in regard to tensile strength.

Factors influencing plasticity of sole crêpe [rubber]. H. P. STEVENS (Bull. Rubber Growers' Assoc., 1925, 7, 716—722).—The spread of crêpe rubber soles is due to the plasticity induced by machining, which has been held to rupture the protein films surrounding the particles of the rubber hydrocarbon; the behaviour of rambong (*Ficus elastica*) and *Castilloa elastica* rubbers, the particles of which have no skin, makes this hypothesis doubtful. The method and results of plastometer tests are described. S. I. LEVY.

Alkali [rubber] reclaiming process. W. E. STAFFORD (India-rubber J., 1925, 71, 59).—Reclaiming rubber by heating vulcanised waste with sodium hydroxide solution under pressure removes free sulphur and depolymerises a portion of the vulcanised material, reducing the coefficient of vulcanisation in this portion and rendering it soluble in chloroform. The resin content is not increased. S. I. LEVY.

PATENTS

Drying [rubber] latex. P. C. RUSHEN. From GENERAL RUBBER Co. (E.P. 244,619, 7.3.25).— Latex is fed, at a rate controlled by an automatic float device, into a shallow trough, in which rotates a cylindrical metal or other perforated screen. Heated air or flue-gases, forced through a narrow slit within the cylinder, detach the latex from the perforations and carry the material thus divided into a drying chamber; the exit gases pass through a baffle tower. S. I. LEVY.

Incorporation of rubber in non-aqueous materials. J. M. WEISS (U.S.P. 1,563,410, 1.12.25. Appl., 18.12.23).—Rubber latex is mixed with a high-boiling non-aqueous solvent, such as paraffin wax, at 120°. The water present evaporates and an intimate mixture of rubber and the non-aqueous solvent is obtained. T. S. WHEELER.

Rubber latex compositions. [Distempers mixed with rubber latex.] T. M. RIGBY (E.P. 245,540, 21.10.24).

Plastic masses (E.P. 241,858).—See V.

XV.-LEATHER; GLUE.

Preparation of hide powder for [tannin] analysis. L. MEUNIER, P. CHAMBARD, and A. JAMET (J. Soc. Leather Trades Chem., 1925, 9, 510—512; cf. B., 1925, 603).—Powders prepared from the same species of animal hide (*e.g.*, calf hide, fresh or salted) give approximately the same results. Pelt fleshings should not be used. The best material is the belly portion of ox-hide, which should be limed with lime only and after removing the flesh and grain surfaces by splitting, delimed with ammonium chloride or acetic acid, well washed, minced to a fine pulp, and left for 6 hrs. in a saturated solution of carbon dioxide, to reduce the $p_{\rm H}$ to 4-4.5. The pulp is then drained, dehydrated with acetone, dried at 30°, and subdivided with a rasp or grater. The product contains about 0.1% of ash and has $p_{\rm H}$ 5.5. D. WOODROFFE.

Report of the International Commission on the analysis of chrome leathers and chrome liquors. R. F. INNES (J. Soc. Leather Trades Chem., 1925, 9, 508-509; cf. B., 1924, 25) .- It is proposed to modify the peroxide method of chromium determination by extracting the fused peroxide-leather ash mixture with cold water containing 5 c.c. of saturated potassium permanganate solution and then filtering. Leather should be dried before extracting the grease with petroleum spirit. A further extraction with alcohol will give oxidised oils, and extraction with benzene will give the asphalt. The Woodroffe and Green method (B., 1922, 641 A) of determining alkali metals is recommended as provisional. Spent chrome liquors should be filtered after boiling with sodium peroxide, to remove in-soluble calcium peroxide. The excess of sulphurous acid in spent thiosulphate liquors can be determined by titrating 25 c.c. of the filtered liquor with 0.1Npotassium dichromate To determine chromium in spent thiosulphate liquors, after determining the acidity the precipitated chromium hydroxide is filtered off, washed, dissolved in acid, and treated with sodium peroxide as usual. D. WOODROFFE.

Structure of gelatin gels. KRAEMER.—See A., Feb., 1926, 124.

Leather dyeing. I. SALT. II. MCCANDLISH and SALT.—See VI.

PATENTS.

Process for rapid tanning. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of F. MERKEL (G.P. 417,865, 13.1.22. Addn. to 414,867; cf. B., 1925, 858).-The chromium salts used in the process of the chief patent are replaced completely or partly by soluble aluminium salts of inorganic acids, principally aluminium chloride. Hydrolysis of the aluminium salts is diminished by concentrating the bath as far as possible. Neutral as well as basic aluminium chloride can be used. The tanning bath is of such concentration that the Al₂O₃ content is about the same as in the solid crystalline alum, or even higher concentrations may be used. A more rapid rate of tanning is thereby attained, and the hydrolysis and proteolysis, which occur in more dilute solutions, and adversely affect the tensile strength of the leather, are much decreased; further, crystallisation of aluminium salts on the skin, which occurs frequently when alum is used, is avoided. In this way soluble aluminium salts can be used for tanning heavy and medium leathers as well as for glace and skin tanning. In many cases a combination with other tanning agents, e.g., chromium salts, is advantageous.

A. B. MANNING.

Preparation of tanning agents. CHEM. FABR. WORMS A.-G. (G.P. 417,972, 11.7.20).—Two mols. of acetaldehyde are allowed to react with one mol. of

a phenol in the presence of a normal sulphite, or on one mol. of an alkali phenoxide in the presence of the corresponding quantity of bisulphite, and acid or a metal salt is added to the product until it is neutral to Congo-red. The product, either before or after being acidified, may be mixed with natural tanning agents, or with glucose. As an example, commercial xylenol is heated with a solution of sodium sulphite and two mols. of acetaldehyde for several hours under pressure. The reaction product is acidified, the sulphurous acid boiled off, the residue neutralised with alkali, and treated with chromium sulphate. The dry product is only partially soluble in water, with separation of basic salts; the filtrate is acid to litmus and neutral to Congo-red, and precipitates gelatin from solution. On being heated it chars without melting. The soluble sodium salts of the condensation products are completely insoluble in benzol, chloroform, ether, and alcohol, and couple with aromatic diazo-compounds, so that skins treated with them can subsequently be A. B. MANNING. dyed.

XVI.—AGRICULTURE.

Steam and chemical soil disinfection with special reference to potato wart. N. R HUNT, F. G. O'DONNELL, and R. P. MARSHALL (J. Agric. Res., 1925, 31, 301-364).-Potato wart can be eliminated from soil by sterilisation with steam under pressure or by chemical treatment. Fourteen of the twenty-two chemicals used produced wart-free plots during the season when treated and the two following seasons, but sodium fluoride inhibited growth of the plant completely and sulphur almost completely in all three seasons. The cost of treatment per acre and the water requirements are stated. The temperature changes occurring in soil heated under pressure with steam and the degree of penetration in various soils of various chemicals have been studied. A table showing the growth obtained in the seasons following treatment is given. H. J. CHANNON.

Colloidal silica and the efficiency of phosphates [as plant nutrients]. P. L. GILE and J. G. SMITH (J. Agric. Res., 1925, 31, 247-260).—The addition of colloidal silica greatly increased the yield of millet in sand cultures when phosphorus was supplied as rock phosphate and slightly increased it when the source of phosphorus was superphosphate. On the other hand, the effect of a mixed gel containing iron, alumina, and silica, when rock phosphate was given, was to reduce the yield below that obtained with no phosphate. The amount of phosphoric acid taken up by the plants was proportional to the yield; and, in the presence of colloidal silica, an increased solubility of rock phosphate in the nutrient solution used was demonstrated. The silica, therefore, appears to act by bringing a greater quantity of phosphoric acid into solution and hence increasing the availability. The unfavourable action of the mixed gel was probably due to the production of soluble iron and aluminium by base exchange, with consequent diminution of soluble phosphate. The experiments are a preliminary to a study of the effect of the

colloidal material of soils on the efficiency of phosphates as plant nutrients. C. T. GIMINGHAM.

Reciprocal action of ground phosphorite and soils not having an acid reaction. A. N. LEBEDIANTZEV (Ann. sci. agron., 1924, 41, 320-329; Chem. Abstr., 1925, 19, 3340) .- The biological activity of soils is of great importance in modifying the availability of phosphorite. Usually phosphorite was more active when added immediately before planting than when added to fallowed and subsequently cropped soil. Phosphorite is more sensitive to desiccation than basic slag or superphosphate, and it is consequently believed that the greater part of the phosphate of phosphorite passes into organic combination. The phosphate of phosphorite is thus believed to be inherently much more mobile under soil conditions than that of superphosphate or basic slag. A. A. ELDRIDGE.

Influence of aluminium, manganese, and iron salts on the growth of sugar cane, and their relation to the infertility of acid Island soils. W. T. McGEORGE (Exp. Sta. Hawaiian Sugar Planters' Assoc. Bull., 1925, [49], 95 pp.; Chem. Abstr., 1925, 19, 3558).—Under the conditions described, aluminium salts exert a direct toxic action on sugar cane, whilst manganese salts have no effect. The resistance of the plant to aluminium toxicity is increased by increasing its phosphate or potash reserve. Phosphate applications are effective only over a short period, and heavy applications of potash are usually required. Lime gave no immediate response, but there was a greater residual stimulation of plant growth. A. A. ELDRIDGE.

Determination of nitrate. KÜRSCHNER and SCHARRER.—See VII.

PATENTS.

Stable ammonium bicarbonate (E.P. 244,645).—See VII.

Reducing offal to tankage (U.S.P. 1,567,014).— See XIX.

Colloidal metal solutions (G.P. 417,933).— See XX.

XVII.—SUGARS; STARCHES; GUMS.

Hydrogen-ion concentration as a basis of [sugar] refinery alkalinity control. A. A. BLOWSKI and A. L. HOLVEN (Ind. Eng. Chem., 1925, 17, 1263—1266).—Colour charts, consisting of dyed celluloid sheets representing the colours obtained with methyl-red, bromothymol-blue, and phenol-phthalein between $p_{\pi} 4.5$ and 10, are used, these greatly simplifying the "spot test" method of operating. In the refining process the p_{π} is kept at approx. 7.0 by neutralising the acidity in the entering products and also that developed later in the refining operations. This control has resulted in more uniformly neutral conditions with the use of only half the amount of lime formerly added, thereby reducing the molasses production arising from the formation of melassigenic calcium salts. J. P. OGILVIE.

Influence of the filter-cloth on scum press work [in sugar refining]. J. HRUDA (Z. Zuckerind. Czechoslov., 1925, 50, 73-80, 81-87, 89-93, 113-118, 121-128).—Varying results in respect of rate of filtration, volume of sweetening-off water, clarity of filtrate, cleanliness of surface, durability, etc., were obtained in a series of factory tests made with carbonatation juices, using different kinds of cloth for the dressing of the filter-presses. Particularly good results were obtained with the marks "1105" (Herold) and PZZI (Slechta und Sohn).

J. P. OGILVIE.

Solubility of sucrose in impure solutions. J. ROBART (Bull. Assoc. Chim. Sucr., 1925, 43, 128—132).—Working with beet molasses containing very melassigenic non-sugars, results were obtained showing that the solubility of sucrose is not affected by the presence of such substances. J. P. OGILVIE.

Jena sintered glass crucibles [in sugar determinations]. G. D. ELSDON (Analyst, 1926, 51, 30–31).—The crucibles of the size (1bG3/<7) are useful for weighing cuprous oxide in sugar determinations but must be selected by actual experience owing to individual differences. Efficient filtration should be obtainable at a trickle rate. Hot alkalis used in determinations by means of Fehling's solution cause a loss in weight of the crucible of the order 3 mg. D. G. HEWER.

Nitrogenous substances of beet molasses. E. PARISI (Annali Chim. Appl., 1925, 15, 555-568). -The possible uses of beet molasses are discussed and the following results given for the various forms in which nitrogen exists in Italian molasses : total, 2.11; protein, 0.11; ammoniacal, 0.02; amide, 0.04; nitric, 0.10; basic, 0.57; total monoaminoacids, 0.95; aminic, 0.45%, calculated on the weight of molasses. The nitrogen precipitated by Neuberg's reagent (mercuric acetate, sodium carbonate, and alcohol) amounts to 1.19%, composed of : aminic, 0.40; precipitated by normal lead acetate, 0.04; purine bases, trace; hexone bases, 0.16; total monoamino-acids (by difference), 0.95. The nitrogen not precipitated by Neuberg's reagent is 1.02%, comprising : basic, 0.40; aminic, traces; slightly soluble in alcohol, 0.45; soluble in alcohol, 0.57. T. H. POPE.

Preparation of *l*-arabinose from mesquite gum. E. ANDERSON and L. SANDS (Ind. Eng. Chem., 1925, 17, 1257—1258).—Gum from the mesquite tree (*Prosopis juliflora*) is treated with 6 times its weight of 4% sulphuric acid, for 3 hrs. at 80°, the acid removed as barium sulphate, the filtrate concentrated, salts are precipitated by alcohol, and the alcoholic liquor is concentrated and allowed to crystallise. A yield of *l*-arabinose amounting to 27—36% of the gum can thus be obtained. J. P. OGILVIE.

Behaviour of starch components towards iodine, and their protective colloid action. SAMEC and KLEMEN.—See A., 1926, 22. 170

Hexahexosan and trihexosan. CASTAN and PICTET.—See A., 1926, 52.

Dihexosan and tetrahexosan. PICTET and SALZMANN.—See A., 1926, 52.

Sulphur-containing sugar. SUZUKI and MORI. --See A., 1926, 96.

Influence of aluminium, manganese, and iron salts on growth of sugar cane. McGeorge.— See XVI.

PATENTS.

Activating decolorising char for use in refining sugar. C. B. DAVIS (U.S.P. 1,565,911, 15.12.25. Appl., 10.10.23).—A carbon-forming distillate of coal, non-volatile at normal temperatures, is incorporated with the bone char, which is then passed, out of contact with air, through a kiln at a red heat below 1000°. The product is cooled out of contact with air, washed with hot water, dried, and again passed through a kiln, out of contact with air, prior to use. H. HOLMES.

Purification of sugar juice by chlorination. TOKYO IMPERIAL INDUSTRIAL LABORATORY (F.P. 591,198, 24.12.24).—Sugar juice is treated with hypochlorous acid at 55°, or chlorine gas is led into the juice, and the free chlorine is afterwards removed by distillation *in vacuo* or neutralised by the addition of an alkali. Alternatively, free chlorine may be generated in the sugar juice by adding sodium chloride and subjecting the solution to electrolysis.

A. J. HALL.

XVIII.—FERMENTATION INDUSTRIES.

Changes in the degree of dispersion of the proteins of barley during malting, mashing, and fermentation. W. WINDISCH, P. KOLBACH, and E. WENTZELL (Woch Brau., 1925, 42, 287-288, 295-297, 303-306, 313-317, 323-325, 333-335).-At various stages in the malting of a barley and the production of beer from the same malt, the products were investigated to ascertain the proportions of soluble nitrogenous matter capable of passing through a series of ultra-filters having pores of approximately the following sizes : 500, 120-150, 20-30, 18, and 15 $\mu\mu$. It is concluded that during malting the finer particles increase in amount more than the coarser ones. Kilning reduces the amounts of both kinds. Boiling of the wort with hops increases the quantity of proteins which are only just soluble, at the expense of those of medium colloidal dimensions, but equilibrium between the particles of different sizes is restored during fermentation. The hops exert a temporary stabilising influence on the very fine protein particles. In a wort acidified until it has the normal reaction of beer, and then stored, great changes take place in the sizes of the protein particles, and a large amount of coagulation occurs. Unacidified wort undergoes similar changes, but not to the same extent. It is impossible to determine correctly the amount of nitrogenous matter assimilated by yeast during fermentation, owing to coagulation of proteins which takes place at the same time. When stored unfermented wort is boiled, there occurs an increase in the amount of nitrogenous matters having degrees of dispersion within the range of an ultra-filter, but when fermented wort is boiled such changes as occur concern chiefly the particles too large to be within this range. J. H. LANE.

Diastatic power of malt and barley. H. L. HIND, H. THREADGOLD, and C. W. B. ARNOLD (J. Inst. Brew., 1926, 32, 26-32).-Owing to want of control of the reaction of the starch solution, preparations of soluble starch from different sources are liable to give discordant results when used for the determination of the diastatic power of malt or barley. Addition of a buffer mixture ensures a constant reaction and avoids the necessity of relying on washing or neutralisation to a given indicator. The chance of false results through accidental and unnoticed contamination of starch or distilled water is eliminated. In the case of malts concordant results are secured by using Sörensen's acetate buffer in quantity sufficient to give p_{π} 4.6. The variation in the diastatic activity of the enzymes of barley as determined by unbuffered starches is greater than is the case with malt. Concordant results for barley can only be obtained if, in addition to buffering to a constant $p_{\rm ff}$, every care is taken to use a perfectly soluble starch. It is proposed to modify the standard method for determination of diastatic power adopted by the Malt Analysis Committee of the Institute of Brewing by the use of 20 c.c. of the acetate buffer solution in each litre of starch solution and, after completion of the conversion, replacing the usual addition of 10 c. of 0.1N-sodium hydroxide by 20 c.c. to stop the diastatic action. C. RANKEN.

Determination of the colour of malts. V. BERMANN (Woch. Brau., 1925, 42, 317—318).— Ostwald's doctrine of colour is explained at greater length than in the previous paper by the author and Laufer (B., 1925, 820). J. H. LANE.

Evaluation of hops by chemical and biological tests. J. S. FORD and A. TAIT (J. Inst. Brew., 1926, 32, 19-25; cf. B., 1924, 568).-The preservative values of hops as obtained by a biological and by a chemical method are compared. The chemical values, $(\% \alpha + \beta/5) \times 10$, were obtained by multiplying by 10 the amount of preservative substance estimated according to the empirical chemical method previously published by the In the bacteriological test varying authors. quantities of hopped wort were added to a sterile cold water malt extract of d 1.010 and $p_{\rm H}$ 5.5, and after re-sterilisation, inoculated with a vigorous 24-hrs. culture of "Bact. X." After being kept at 30° for 24 hrs., the point of suppression of the growth of the bacterium was noted. The authors consider that, using "Bact. X.," the conditions cannot be standardised so as to obtain satisfactory uniformity of development of this organism. Owing to the biological tests being made at different times with consecutive lots of the samples, a difficulty arose in expressing the whole series on a common numerical basis. As the agreement between chemical and biological results was fairly satisfactory, the chemical values were used as a standard for reducing the various biological values to a common basis.

C. RANKEN.

Titration of the bitter substances of hops. L. HEINTZ (Woch Brau., 1925, 42, 325).-In Seibriger's method for determining the bitter substance of hops (B., 1913, 442; 1914, 37) greater precision in the titration can be attained by adopting the method described by Lüers for the titration of coloured liquids (B., 1914, 761). Small square bottles of colourless glass may be used. 50 c.c. of the filtrate to be tested are run into each of two bottles. To the left-hand one 5 c.c. of phenolphthalein solution (in 96% alcohol) are added 1 c.c. at a time, and then 5 c.c. of 96% alcohol 0.5 c.c. at a time to clear the emulsion formed at first. Occasionally, 1 or 2 c.c. more alcohol may be required. Then 10 c.c. of alcohol are added to the liquid in the right-hand bottle. Two other bottles are placed behind, the left-hand one containing water and the right-hand one a phenolphthalein solution having the standard red colour. The titration is then carried out, equal volumes of alkali being added to the contents of each of the two front bottles, until on looking through the left-hand pair the colour appears the same as on looking through the righthand pair. J. H. LANE.

PATENTS.

Improving molasses as a nutrient medium for cultivation of aroma-producing bacteria. P. NITSCHE (Austr. P. 100,447, 26.7.24).—Molasses is heated with tannic acid or similar substances and then filtered, whereby basic substances, *e.g.*, betaine, and vegetable proteins injurious to aromaproducing bacteria are precipitated and removed.

A. J. HALL.

Utilising sulphite-cellulose waste liquor (Swed. P. 57,863).—See V.

XIX.—FOODS.

Filtration method of measuring sediment in milk. A. R. TANKARD (Analyst, 1926, 51, 31).— Although filtration methods of measuring sediment in milk are useful as a rough sorting test and for giving ocular demonstration of the presence of impurities, none can be regarded as quantitative, and results by such methods cannot be made the basis of a prosecution under the Sale of Food and Drugs Act. D. G. HEWER.

Composition of pectin. Determination of galacturonic acid in pectin. W. H. DORE (J. Amer. Chem. Soc., 1926, 48, 232-236).—A method for the determination of galacturonic acid in pectin has been developed, based on measurement of the carbon dioxide evolved, when this acid is distilled with hydrochloric acid, according to the equation

 $CHO.(CHOH)_4.CO_2H \rightarrow C_5H_4O_2$ (furfuraldehyde) +CO₂+3H₂O (cf. Nanji, Paton, and Ling, J.S.C.I., 1925, 44, 253 T). The galacturonic anhydride 1925, 44, 253 r). The galacturonic anhydride contents of various materials, evaluated by this method, were as follows: "dearabanised" beet pectin (Ehrlich's "crude pectin" extracted with 70% alcohol), 39.66—44.48%; impure "cytopectic acid" from beet pulp (cf. Clayson, Norris, and Schryver, A., 1922, i, 206), 41.10—44.82%; pectic acid from beet pulp ("crude pectin" demethylated with solution hydroxide, acidified, and precipitated with solution 54.20%; and hydrolysed beet pectin with alcohol), 54.30%; and hydrolysed beet pectin (autoclaved with 1% sulphuric acid at 120°, neutralised with barium carbonate, and precipitated with alcohol), 59.20-61.26%. Commercial pectin gave irregular results, apparently due to the production of carbon dioxide by secondary reactions. Attempts to utilise the production of furfuraldehyde as a method for the determination of galacturonic acid by leaching out the "araban" portion of the pectin with 70% alcohol (cf. Ehrlich, Schwalbe's "Chemische Untersnehmen effective, Schwalbe's Chemische Untersuchung pflanzlicher Rohstoffe," 1920, 94) indicated that two extractions with 70% alcohol reduced the soluble furfuraldehyde-yielding matter to a small and constant amount, but no method of calculating the galacturonic acid content from the

of calculating the galacturonic acid content from the furfuraldehyde yield of the alcohol-soluble portion could be devised. It is concluded that the ring complex containing the galacturonic acid (cf. Nanji, Paton, and Ling, *loc. cit.*) has, associated with it, furfuraldehyde-yielding substances extraneous to the ring, in varying proportions. F. G. WILLSON.

Chemical composition of the separative septa of the lemon. A. FICHERA (Annali Chim. Appl., 1925, 15, 568—572).—These septa are composed of complex polysaccharides, the external ones of cellulose, galactosans, and glucosans, and the internal ones of cellulose and glucosans. T. H. POPE.

Determination of lignin. PALOHEIMO.-See V.

Coconut oil and milk fat in cacao butter. KUHLMANN and GROSSFELD.—See XII.

PATENTS.

Apparatus for reducing the offal in slaughter houses to grease and tankage. H. P. TAYLOR, Assr. to P. B. BROKAMP (U.S.P. 1,567,014, 22.12.25. Appl., 5.8.22).—The offal is cooked in a tank having a screened opening in the side near the top for the entrance of steam, and a conduit from the part behind the screened opening leading to a receiving tank. so that the melted grease and part of the finelydivided protein may be floated off. The grease is forced through an outlet at the top of the receiving tank by introducing water below, and the protein is withdrawn from the bottom to an evaporating tank, steam-jacketed at the bottom, and the concentrated protein returned to the first tank. D. G. HEWER.

Manufacture of fertilisers and stock foods. A. WOOSNAM. From J. J. BERRIGAN (E.P. 245,285, 23.1.25).—See U.S.P. 1,524,233 ; B., 1925, 260. 172

CL. XX.-ORGANIC PRODUCTS ; MEDICINAL SUBSTANCES ; ESSENTIAL OILS.

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

Determination of quinine. L. DAVID (Pharm. Ztg., 1926, 71, 26-28).-Quinine in cinchona bark is thus determined : to exactly 17 g. of 1% hydrochloric acid in a rubber-stoppered flask are added 1.7 g. of powdered bark, and the liquid is warmed for half-an-hour on the water-bath with frequent shaking. After keeping for half-an-hour with occasional shaking, the mixture is cooled, weighed, and made up to the original weight with 1% hydrochloric acid. It is then filtered, and the filter-paper squeezed out. To 10 g. of the filtrate are added, with shaking, 10 drops of 0.1N- sodium hydroxide and 10 c.c. of 20% disodium hydrogen phosphate, and the liquid is kept for half-an-hour, with stirring. The precipitate is filtered off, flask and precipitate are washed four times with 2% disodium hydrogen phosphate, the last traces of precipitate being wiped from the beaker with filterpaper, which is added to the precipitate. The latter is treated with 4 c.c. of 20% sodium hydroxide, shaken, 6 g. of sodium chloride are added, and the mixture is extracted twice with 15 c.c. of ether, shaking for 20 min. each time. After half-an-hour, 15 c.c. of the ethereal extract are evaporated, the residue is twice treated with 3 c.c. of ether and evaporated, and the residue dissoved in 20 c.c. of neutral alcohol and titrated with 0.1N- hydrochloric acid in the presence of lacmoid, using a micro-burette. B. FULLMAN.

Determination of santonin in santonin pastilles. R. EDER and W. SCHNEITER (Schweiz. Apoth.-Ztg., 1925, 63, 557—560; Chem. Zentr., 1926, I., 188—189).—Four pastilles are finely ground with seasand and shaken with chloroform (50 g.) for 5 min. After the addition of 20 drops of water, the vigorous shaking is continued for 0.5 min., after which 45 g. of extract are filtered into a tared flask; chloroform is removed by distillation and the flask with contents is dried at 100° for 1 hr. The residue should amount to 0.085—0.095 g., corresponding with a santonin content for one pastille of 0.024— 0.026 g. The santonin can be identified by its m.p. (170°). L. M. CLARK.

Determination of alcohol and ethyl chloride in chloroform. C. NEWCOMB (Analyst, 1926, 51, 19—30).—In addition to the B.P. tests for anæsthetic chloroform the density should be determined before and after washing for 20 min. The chloroform is then refluxed for 2 hrs., during which time a volume of 10% caustic potash equal to that of the chloroform is continuously added. The chloroform is subsequently separated, washed for 20 min., filtered, and the density again determined. The difference between the first two densities plus 0.0005 for the water dissolved, less 0.0002 if from 0.05 to 0.5% of ethyl chloride is present, gives a measure of the "water-soluble substances reckoned as alcohol." The difference between the second and third densities gives a measure of the ethyl chloride, and the density of the residual chloroform should be within 0.0002 of 1.4747 at 27°. If more than 0.03% of ethyl carbonate or 0.3% of carbon

tetrachloride are present the last density will indicate the fact (unless they should be present in equivalent amounts). The washing of the chloroform may be conveniently carried out in a separating funnel to which the wash water is supplied through a tube extending downwards to about $\frac{3}{4}$ in. above the stopcock, and drawn out to a fine point; the water is drawn through by means of a vacuum pump and the outlet tube can be adjusted to different levels. About 10-20 c.c. of water are run through per minute. The most convenient method for determining the density was found to be by means of a Westphal balance. A preliminary calibration is essential, and a series of at least six determinations of densities at different temperatures is made, the results are plotted, and the best line of the right slope for the known change in density with temperature is drawn through them. D. G. HEWER.

Free thiocyanogen. KAUFMANN.-See XII.

Materials containing tartaric acid. GLASER.— See XVIII.

PATENTS.

Manufacture of CC-disubstituted compounds of barbituric acid and 4-dimethylamino-1phenyl-2:3-dimethyl-5-pyrazolone. P. PFEIFFER (E.P. 244,035, 3.4.25. Conv., 3.12.24. Addn. to 231,512; B., 1925, 942).—The methods previously applied to the preparation of compounds, in molecular proportions, of diethylbarbituric acid and 4-dimethylamino-1-phenyl-2:3-dimethyl-5-pyrazolone are now applied to *iso*propylpropenylbarbituric acid (yielding a *compound*, m.p. 95—97°) and phenylethylbarbituric acid (*compound*, m.p. 129—131°). B. FULLMAN.

Medicinal preparation. J. K. LILLY, Assr. to E. LILLY AND CO. (U.S.P. 1,562,459, 24.11.25. Appl., 5.5.25).—A mixture of equal parts by weight of phenylcinchoninic acid and its hydriodide has superior analgesic properties to the hydriodide alone and does not produce iodism. T. S. WHEELER.

Morphine solution. Analgesic agent. C. W. HOOPER, ASST. to H. A. METZ LABORATORIES, INC. (U.S.P. 1,563,086-7, 24.11.25. Appl., [A] 8.10.24, [B] 22.1.25).—(A) A solution of 0.5 g. of morphine, 2.5 g. of novocaine, about 50 g. of magnesium sulphate, and about 0.1 g. of sodium bisulphite in 100 c.c. of water is stable on storage. (B) A solution of about 50 g. of magnesium sulphate and 2.5 g. of novocaine in 100 c.c. of water has analgesic properties and is stable. T. S. WHEELER.

Determining the physiological value of remedial and medical substances [antirachitics]. T. F. ZUCKER, ASST. to UNIVERSITY PATENTS, INC. (U.S.P. 1,563,134, 24.11.25. Appl., 26.11.23).— The antirachitic strength of a substance can be determined by measuring its capacity for promoting the secretion of hydrochloric acid in the stomach. The measure of this quantity is obtained by feeding an animal for a standard time on a standard alkaline diet calculated to produce rickets, and then determining the $p_{\rm H}$ value of the fæces. A known quantity of the antirachitic substance under test is then administered and the $p_{\rm H}$ value of the fæces again determined. T. S. WHEELER.

Styptics. C. R. DOWNS (U.S.P. 1,563,346, 1.12.25. Appl., 12.8.24).—A mixture of equal parts of phthalyl peroxide containing 40% of free phthalic acid and tale is a valuable styptic. T. S. WHEELER.

Manufacturing a perfume smelling like amber. A. CORTI, ASST. to CHEMICAL WORKS FLORA (U.S.P. 1,565,796, 15.12.25. Appl., 26.1.23).—The use of nitrodibromobutyl-*m*-cresol methyl ether, m.p. 100°, having a smell of musk and a true smell of amber, is claimed. H. HOLMES.

Preparation of dibromobarbituric acid. PARKE DAVIS AND Co., Assees. of A. M. CLOVER (Can. P. 245,806, 9.6.24).—Bromine is added to a saturated solution of barbituric acid at a temperature below 40°, dibromobarbituric acid is separated by filtration, and the mother liquor is saturated with barbituric acid and used again in the process. L. A. COLES.

Preparation of alcohols from ethers. E. MERCK, Assee. of W. KRAUSS (G.P. 417,926, 8.3.24. Addn. to 407,487).—Alcohols are produced by treating the benzyl ether of the desired alcohol with hydrogen in the presence of noble metals as catalysts at the ordinary temperature, as described in the chief patent (cf. B., 1925, 474). For example, an alcoholic solution of amyl benzyl ether containing palladiumised charcoal, is shaken with hydrogen at 15° until 1 mol. is absorbed. After removing the catalyst, the product is diluted with water and extracted with ether, toluene and amyl alcohol being recovered from the extract by distillation. L. A, COLES.

Conversion of methyl sulphide into carbon tetrachloride and other chlorine compounds. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of A. HALLSTEIN (G.P. 417,970, 1.2.24. Addn. to 416,603; B., 1926, 28).—Methyl sulphide practically free from mercaptans (v.i.) is converted into methyl chloride, sulphur chloride, and carbon tetrachloride, by saturating it with chlorine at the ordinary temperature and subsequently passing chlorine into it at 100—120°, preferably in the presence of iodine as catalyst. L. A. COLES.

Improving the odour of methyl sulphide. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of A. HALLSTEIN (G.P. 418,129, 18.5.24).— Evil-smelling impurities present in methyl sulphide obtained as a by-product in the manufacture of sulphate-cellulose, are removed by treating the material with metal oxides or hydroxides in solution or in suspension, *e.g.*, with 40% potassium or sodium hydroxide, or with powdered lime, or with aqueous or gaseous ammonia. L. A. COLES.

Preparation of protocatechuic acid and pyrocatechol. ZELLSTOFF-FABR. WALDHOF, and P. ZICKMANN (G.P. 418,219, 31.7.23).—In the production of protocatechuic acid and pyrocatechol by treating lignin with fused alkali hydroxides, the addition of reducing agents which liberate nascent hydrogen, e.g., zinc dust or aluminium powder, prevents oxidation of the protocatechuic acid to oxalic acid. The protocatechuic acid can be converted completely into pyrocatechol by splitting off carbon dioxide. For example, zinc dust is added to a melt containing lignin, potassium hydroxide, and water, at 250°; after cooling the product, dissolving it in water, and acidifying the solution with sulphuric acid, protocatechuic acid is separated by extraction with a solvent not miscible with water, and evaporation of the solvent. L. A. COLES.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Visible decomposition of silver halide grains by light. A. P. H. TRIVELLI and S. E. SHEPPARD (J. Physical Chem., 1925, 29, 1568-1581).-The photochemical darkening of specially prepared silver bromide crystals is definitely oriented in the crystals in a manner dependent on their growth and structure (cf. B., 1923, 908 A). In the case of silver bromide grains from photographic emulsions the decomposition is largely irregular. Statistical measurements showed that in a given emulsion, the sensitivity of the grains as indicated by visible decomposition increases with increasing size. Visible sensitivity is relatively independent of sensitivity nuclei in the grains, a fact which explains the lack of parallelism between developable sensitivity and visible sensitivity of different emulsions. A tentative theory based on a hypothesis due to Weigert (A., 1918, ii, 50) is proposed. It is suggested that photochemical decomposition is oriented in the crystals according to gradients of ionic deformation or perturbation of electron orbits following certain directions of growth in the crystals. Since foreign inclusions, such as sensitivity nuclei and also reduced silver, will introduce deformation in the surrounding silver bromide, the visible decomposition becomes autocatalytically oriented. This explains the contrast in behaviour between specially prepared silver bromide crystals and emulsion grains, and also the effect of size. Considerations of diffraction and internal reflection of light in the crystals are inadequate to explain the distribution of visible blackening. Oxidising agents have a relatively small effect on visible sensitivity, compared with their effect on W. CLARK. developable sensitivity.

See A., 1926, 109, Photographic spectrophotometry (DORGELO). 113, X-Ray analysis of mixed crystals of silver halides (WILSEY).

PATENTS.

Screens for colour photography. H. KELLER (E.P. 244,644, 16.5.25).—Screens are prepared by spraying a gelatin or other colloid layer with materials which dye the colloid only when it is unhardened, and which themselves harden the colloid. The colloid layer is sprayed successively with the required dye solutions, for example, of dyes of the Pina-type, or of most aniline dyes, the hardening agent being formalin, chrome alum, or some similar substance, until all the uncoloured spots are covered. An alternative method of dyeing is to sprinkle mixtures in powdered form of the dyes and hardener on the moistened colloid surface. The layer is finally washed to remove the dyes or solutions deposited on portions of the colloid which have been already dyed and hardened by a previous spraying.

W. CLARK.

Sensitising kinematograph films for positive copies by means of solutions of dichromate. A. WEINGARTEN (U.S.P. 1,564,161, 1.12.25. Appl., 12.1.25).—Films are sensitised by bathing the colloid in a solution of potassium dichromate, potassium ferrocyanide, and potassium bromide.

W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Stability of explosives as an additive property. M. TONEGUTTI (Annali Chim. Appl., 1925, 15, 542—547).—Measurements made by the Taliani method (B., 1921, 562 A) show that the stabilities of ballistites of different composition correspond with those calculated from the proportions of the component nitroglycerin and collodion cotton (cf. Lenze and Pleus, B., 1920, 281 A, 468 A; De Bruin, 1921, 903 A; Brunswig, "Explosivestoffe," 1923, 172). Analogous results are furnished by powders of the cordite type. T. H. POPE.

Reaction of nitroglycerin powders to the Angeli test in relation to the stability to heat. M. TONEGUTTI (Annali Chim. Appl., 1925, 15, 548-554).-The results obtained on applying Angeli's acidity test (B., 1918, 608 A) to acid and neutral nitroglycerin powders show that with ordinary ballistites there exists a close relationship between the acidity and the stability towards heat shown by the Taliani test, but not by the Abel test. With powders of the cordite type, on the other hand, the acid reaction corresponds with a low Abel stability but not with the results of the other stability tests. With such powders only the Angeli and Abel tests furnish results sufficiently definite to indicate the state of preservation of the explosive. Other stability tests, especially the silvered vessel test, serve moderately well to determine the various degrees of stability exhibited by cordites of different types in relation to their different chemical compositions, more particularly to their content of nitroglycerin and vaseline and to the presence or absence of T. H. POPE. stabiliser.

XXIII.—SANITATION; WATER PURIFICATION.

Sensitivity of the *o*-tolidine and starch-iodide tests for free chlorine [in water]. A. M. BUSWELL and C. S. BORUFF (J. Amer. Water Works Assoc., 1925, 14, 384—405).—Using varying quantities of carefully standardised chlorine water and distilled water chlorinated but free from excess chlorine for dilution purposes, colours were obtained with *o*tolidine solution which checked Ellms and Hauser's dichromate standards (cf. B., 1913, 1125) over the range 0.02-0.2 p.p.m. of chlorine. In the o-tolidine method iron when present as the chloride or nitrate above 1 p.p.m. gives a yellow solution and interferes with quantitative determinations of excess chlorine ; nitrites in low concentration do not interfere because they are oxidised by the chlorine added and the compounds formed have no effect on the o-tolidine reagent. Water more basic than $p_{\rm H}$ 8 should be treated with acid to bring it within the range $p_{\rm H} 3-8$; sensitivity is impaired at low temperatures. The starch-iodide test is affected by small amounts of iron and nitrites in low concentration; manganese also interferes; the hydrogen-ion concentration is an important factor, the most sensitive concentration being that given by 2 c.c. of concentrated hydrochloric acid in 50 c.c. of water. The starch-iodide test is more sensitive at low temperatures. At ordinary temperatures the two tests are equally sensitive and each gives a positive test for 0.005 p.p.m. of excess chlorine; neither is interfered with by the presence of oxidised organic compounds in solution. The authors recommend the o-tolidine test for excess chlorine in water, using Ellms and Hauser's standards in half portions in order that 50-c.c. Nessler tubes may be employed. Where the temperature is low the starch-iodide test is proposed, as the use of the o-tolidine method with the regular standards would lead to over-dosing, with consequent W. T. LOCKETT. odours and tastes.

Determination of the chlorine ion in water. H. W. VAN URK (Pharm. Weekblad, 1925, 62, 1338—1343 also Z. anal. Chem., 1925, 67, 281— 288).—The Dutch Codex specifies titration with silver nitrate in presence of magnesium oxide, using potassium chromate as indicator. The concentration of chromate should be between 0.07×10^{-2} and $1.5 \times 10^{-2} M$. Only if the water is alkaline towards tropeolin-O should it be acidified before addition of magnesium oxide; the latter is not the most suitable substance, and the use of sodium hydrogen carbonate is recommended. The author's conclusions are criticised in an added note by N. Schoorl. S. I. LEVY.

Micro-determination of oxygen content of water. RISCH.—See A., Feb., 1926, 140.

PATENTS.

Treatment of natural base-exchange zeolitelike materials. E. B. HIGGINS (E.P. 244,506, 10.9.24).—Clay-like impurities are rapidly separated from a base-exchange material for water-softening, *e.g.*, glauconite, by subjecting it to a peptising and hydraulic grading process in one or more columns of upwardly-flowing liquid, *e.g.*, a faintly alkaline solution (0.5—1 lb. of caustic soda per ton) or a solution of common salt. The speed of the column is regulated so that the finer solid particles flow upward out of the apparatus, and the coarser ones settle to the bottom, and the speed of the liquid increases in succeeding columns. D. G. HEWER.

Utilising refuse (E.P. 244,358).-See VIII.