

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 6, 1928.

I.—GENERAL; PLANT; MACHINERY.

Calibration liquids for viscosimeters. D. KRÜGER (Z. angew. Chem., 1928, 41, 375).—Castor oil and glycerin are not recommended for calibrating capillary viscosimeters as they are liable to changes, the former under the influence of air and light and the latter on account of its hygroscopic nature. Solutions of sucrose in water, up to 60%, give the best results, and the absolute viscosities, in centipoise units, for such solutions at different temperatures are tabulated.

R. H. GRIFFITH.

Crushing and grinding. GROSS and ZIMMERLEY.—See VII. **Drying furnace.** ORTLEPP. **Boiler plate steels.** POMP. **Alloys in motor construction.** STEUDEL.—See X.

PATENTS.

Heating kiln. B. M. JOHNSON, Assr. to CARBORUNDUM Co. (U.S.P. 1,669,563, 15.5.28. Appl., 24.8.25).—The furnace consists of a heating compartment separated from a combustion chamber by baffles of higher thermal conductivity at the base than at the top.

H. ROYAL-DAWSON.

Grading machine. J. D. GRABILL (U.S.P. 1,661,657, 6.3.28. Appl., 10.9.27).—The openings of the endless sizing chain of a grading machine are closed by means of a flexible endless cover member which moves with the sizing chain, and is fixed thereto. F. G. CLARKE.

Screening of wet material. J. CREDO, Assr. to LOUISVILLE DRYING MACHINERY Co., Inc. (U.S.P. 1,669,973, 15.5.28. Appl., 4.12.26).—Wet material is fed by gravitation on to a screen and maintained in an approximately uniform layer while passing over it. Material passing through part of the screen is collected and returned to the remaining part. L. A. COLES.

Liquid and gas contact apparatus. F. H. WAGNER, Assr. to BARTLETT HAYWARD Co. (U.S.P. 1,669,638 and 1,669,795, 15.5.28. Appl., [A] 7.2.25, [B] 17.6.25).—(A) The apparatus consists of a casing containing a number of superposed pans, each provided with a device for raising the liquid therein and spraying it in an outward direction. The gas passes upwardly around the pans through perforations in horizontal, annular baffle plates placed above the pans, at least in the lower portion of the apparatus. In (B) the perforations are provided with devices which prevent the liquid from coalescing on the under surface of the container.

F. G. CLARKE.

Catalysts for gas reactions. DEUTS. GASLÜHLICHT-AUER-GES.M.B.H. (B.P. 263,758, 6.11.26. Ger., 29.12.25).—The exhaustion by water, carbon dioxide, sulphur dioxide, etc. of catalysts for gas reactions is

indicated by adding 2–3% of a substance which under the influence of the particular catalyst poison causes disintegration of the active mass. W. G. CAREY.

Preparation of foams for fire extinction. BRIT. DYESTUFFS CORP., LTD., and E. CHAPMAN (B.P. 289,630, 25.4.27).—Solutions of the following substances are used to assist foam formation in fire-extinguishing appliances:—sulphonates of aldehyde-naphthalene or aldehyde-phenol condensation products, alkylated naphthalenesulphonic acids, sulphonates of sulphonatable mineral oil fractions of b.p. above 200°, sulphonated alkylated mineral oil fractions, lignosulphonic acid, naphthenic acids, taurocholic acid, and sulphonated higher fatty acids. These substances are used preferably in the form of their neutral salts. C. O. HARVEY.

Hardness testing device. D. C. DAVIS (U.S.P. 1,661,718, 6.3.28. Appl., 2.2.27).—The device comprises a plunger slidably mounted in a hollow operating member which in turn moves in one direction within a casing. A compressible measuring element of known value in one end of the operating member engages one end of the plunger, at the other end of which is a test member in contact with the sample. A stop device arrests the travel of the operating member when the latter applies a predetermined compression to the measuring element. F. G. CLARKE.

Apparatus for standardising solutions. H. H. WERMINE, Assr. to BELDEN MANUF. Co. (U.S.P. 1,664,840, 3.4.28. Appl., 14.5.25. Renewed 23.2.28).—The rise and fall of a member submerged in a solution, due to changes in density of the latter, operate a valve device for supplying liquid to compensate for the changes in composition of the solution. In order to maintain the composition constant through large ranges of temperature, the submerged member has a relatively high temperature coefficient of expansion compared with that of a solid material. For this purpose a collapsible sealed capsule containing a liquid having the same coefficient of expansion as the solution may be used.

F. G. CLARKE.

Treatment of substances with heat. W. G. LAIRD, Assr. to HEAT TREATING Co. (Re-issue 16,971, 22.5.28, of U.S.P. 1,546,345, 14.7.25).—See B., 1925, 743.

Refrigerating systems or apparatus. CHICAGO PNEUMATIC TOOL Co., Asscs. of R. W. DAVENPORT (B.P. 270,655, 21.3.27. U.S., 5.5.26).

Absorption refrigerating apparatus. P. M. LE ROY (B.P. 273,315, 23.6.27. Fr., 23.6.26).

Clarification of liquids (U.S.P. 1,665,167).—See XVII.

II.—FUEL; GAS; TAR; MINERAL OILS.

[Determination of] total carbon in coal. A. R. CARR and A. M. RENTE (Ind. Eng. Chem., 1928, 20, 548—549).—About 0.3—0.6 g. of coal is exploded in a bomb calorimeter and the products are expanded into a rubber balloon contained in a large bottle filled with water. The displaced water is weighed and the temperature and pressure of the gas are recorded. The carbon dioxide content of the gas is determined by measuring the percentage absorption in caustic soda solution.

J. S. CARTER.

Determination of elementary oxidisable carbon in solid fossil fuels. I. BLUM (Bul. Soc. Romane Stiin., 1928, 30, 43—49).—In determining the amount of elementary oxidisable carbon in solid fossil fuels from the quantity of carbon dioxide produced by their combustion, it is necessary to take into account the carbon dioxide originally adsorbed or absorbed in the fuel, that formed by the decomposition of inorganic carbonates, and that liberated from organic compounds under the influence of heat. The total amount of this correction may be obtained by heating a sample of the fuel in a current of nitrogen, or in a Fischer-Schrader aluminium retort. Determinations of this kind have been made with some Rumanian lignites.

R. CUTHILL.

Determination of the reactivity of coke. D. J. W. KREULEN (Z. angew. Chem., 1928, 41, 498—501).—A current of air is passed at a constant speed over a weighed amount of the coke in a porcelain tube maintained at 700°, and the amount of carbon dioxide evolved in a definite period of time is determined. The boat containing the coke, which is in the cold part of the tube while the furnace is heating up, is pushed by means of a wire into the heated portion and 2 min. are allowed for it to reach 700°. Connexion is then made to the carbon dioxide absorption apparatus (a tower containing coke moistened with 30% potassium hydroxide solution), and absorption is allowed to proceed for 5 min. The increase in weight of the tower is taken as a measure of the reactivity.

W. J. POWELL.

Characteristics of colliery surface and mine waters. N. SIMPKIN (J.S.C.I., 1928, 47, 114—115 r).—Attention is drawn to the need for discrimination in the use of local colliery waters for boiler or other purposes. Drainings from pit heaps and certain seams may contain considerable quantities of metal sulphates and be acid in character, e.g., 617 grains per gallon of iron, aluminium, and manganese sulphates; others may be distinctly alkaline and may be utilised to some extent to neutralise acid waters; a third class may contain sodium chloride in deleterious quantity for such purposes as washing coal for coking. Usually considerable variation in composition occurs in the different sources of water even at the same colliery.

C. A. KING.

Effect on the gas-making value of gas oil of its previous use for removal of naphthalene from coal gas. H. H. THOMAS (Gas J., 1928, 182, 538—540).—When coal gas is treated in a rotary washer with gas oil to remove naphthalene, the spent oil is usually consumed on the carburetted water-gas plant. The properties of the oil are affected both by the naphthalene

which it dissolves and by lower-boiling hydrocarbons; using 15.3 gals. of oil per million cub. ft. of gas, the sp. gr. was changed from 0.8709 to 0.8804, the flash point was lowered from 76° to atmospheric temperature, and 10% of the oil was distilled at 150° in comparison with 221° for the original sample. Gas-making tests were carried out at different temperatures in a modified form of Ross and Leather apparatus; a factor expressing the value of the oil was obtained by multiplying the volume of gas per c.c. of oil by its content of hydrocarbons, and it was evident that the value of the oil was lowered by use in the washer. Further tests were performed on the plant, using in one case new oil, and in the other a mixture of 4 pts. of new oil with 1 pt. of spent oil; by assuming a constant calorific value for blue-gas and a constant ratio of oil-gas to water-gas, the yield of therms per gallon of oil was found to be 1.0787 and 1.0218, respectively. Interpretation of these figures shows that the therm yield per gallon of spent oil is 0.7942.

R. H. GRIFFITH.

Composition of the light fractions of tar from the Kashperovka shales. G. L. STADNIKOV and A. E. VEJCMAN (J. Russ. Phys. Chem. Soc., 1927, 59, 859—866).—The light neutral oils from tar obtained by the low-temperature carbonisation of Kashperovka (Volga) shales was investigated, with a view of identifying the sulphur compounds contained in them. The shale, containing 13.4% of moisture, 46.25% of ash, and 3.8% of sulphur, was carbonised in a Fischer rotating retort, and yielded 11.1% by wt. of tar without (15% with) a current of carbon dioxide passed slowly through the mass. On straight distillation 11% by vol. came over below 140° and 36% below 200°. On removal of acid sulphur compounds with 3% formaldehyde solution in the presence of sulphonic acids, bases with 10% sulphuric acid, and phenols with 10% alkali, and distillation, the distillate between 105° and 220° was divided into six fractions, in which the sulphur content was determined and the thiophen derivatives were separated and identified by conversion into ketones by a modified Friedel-Crafts method with acetyl chloride, lead chloride being used as catalyst, owing to the prevalence of secondary reactions with aluminium chloride. The fraction 140—160° (13.1%) gave a mixture of ketones, which yielded two isomeric semicarbazones, m.p. 192—193° and 199—200°, differing markedly in crystalline form, formed probably from isomeric dimethylthiophen methyl ketones or ethylthiophen methyl ketones, and two isomeric semicarbazones, m.p. 177° and 166°, belonging to isomeric propylthiophen methyl ketones. The four *p*-nitrophenylhydrazones were also prepared, m.p. 155°, 158°, 135—136°, and 127—128°, and these confirmed the identity of the ketones.

M. ZVEGINZOV.

Continuous treatment of kerosene distillate. L. SELSKI (Azerbaijansk Neftyanoye Khoz., 1927, No. 12, 53—58).—The time of treatment is diminished by the use of an apparatus providing for atomisation of the oil, so that a large surface is exposed to the reagent and the mixing is thorough. CHEMICAL ABSTRACTS.

Determination of asphaltum in oils containing paraffin. J. MZOUREK (Petroleum, 1928, 24, 403—

404).—Asphaltum in lubricating oil is determined by precipitation with benzine or with a mixture of alcohol and ether, but if paraffin is present it will be thrown out simultaneously, and has to be removed by subsequent extraction. If, however, the oil is treated with 40 times its volume of benzine and refluxed for 2–3 hrs. this difficulty is overcome, and, after cooling, the asphaltum appears as a residue. R. H. GRIFFITH.

Detection of paraffin in ceresin. D. HOLDE and K. H. SCHÜNEMANN (Z. angew. Chem., 1928, 41, 368–375).—Wax-like products from Polish and Caucasian ozokerite, consisting chiefly of isoparaffins, are refined with sulphuric acid and worked up as ceresin. Paraffin is frequently added to a ceresin, and this addition was usually detected by fractional precipitation with alcohol from chloroform solution, the fractions being examined in a refractometer; the refractive index of a ceresin is higher than that of a paraffin. A number of samples of ceresin, m.p. 60–87°, and of paraffins which may be mixed with them, m.p. 50–60°, have been examined, and it is found that the latter are less easily precipitated from solution and have much lower refractive indices except in the single case of a Rangoon paraffin. The sp. gr. of paraffins is 0.867–0.915, and of ceresins 0.912–0.943; molten paraffins are less viscous than ceresins. Treatment with chlorosulphonic or 30% fuming sulphuric acid provides still further distinction, as the extent of reaction is small with paraffins, but may involve up to 70% of a ceresin. Examination of mixtures of pure ceresin with known amounts of different paraffins showed that treatment with acids combined with precipitation from solution provided the most sensitive test for detection of added paraffin.

R. H. GRIFFITH.

Separation of crude-oil emulsions by chemical methods. T. KUCYŃSKI (Petroleum, 1928, 24, 398–403).—A water-in-oil emulsion may sometimes be broken by the addition of a third substance which is either soluble in the oil or else is added as an aqueous solution; of the first type, phenol and naphthenic acids are important. The action of the phenol is complex, as it increases the mutual solubility of the two liquids, lowers their surface tensions, and has a solvent action on asphaltums and similar substances which collect at the interface. The other group of emulsion-breaking substances consists of compounds such as soaps, and work in this connexion has led to a study of the electric charges present on the droplets of an emulsion. It was found that a natural emulsion from Boryslav-Tustanowice contained positively-charged water particles, unlike those of American oils described by other investigators. Examination of various artificial emulsions showed that when suspended in pure hydrocarbons a water droplet was always positively charged, but emulsions can exist in which the reverse conditions appear, and where different degrees of acidity or alkalinity obtain in the aqueous phase. The function of asphaltic substances in stabilising emulsions, the mutual effect of an acid and alkaline emulsion, and a number of technical methods of separation are described and the principles concerned in the various cases are discussed.

R. H. GRIFFITH.

Setting point of petroleum and residues containing paraffin. A. SACHANEN (Petroleum, 1928, 24, 654–655).—The setting point of a heavy oil depends on its previous thermal treatment, and may be altered very considerably by prolonged warming. Experiments with samples of petroleum products in small test tubes kept at any desired temperature for 1 hr. showed that a maximum setting point is developed by heating at between 40° and 60°, and an oil containing a greater percentage of paraffin will need heating at a higher temperature in order to attain its maximum. An oil or masut which contains only traces of resin or has been treated with sulphuric acid will show very slight alteration on heating; it appears, therefore, that the behaviour of an oil depends not only on the solid paraffins which are present, but also on the asphaltic substances.

R. H. GRIFFITH.

PATENTS.

Coking oven. H. H. KOPPERS (U.S.P. 1,669,168, 8.5.28. Appl., 22.12.25. Ger., 13.9.24).—The retorts are arranged in the form of a series of walls each containing a number of horizontal retorts placed one above the other, so that the heating chambers between each wall of retorts comprise vertical flues. The heating gases pass from the tops of the flues to two horizontal waste-heat collecting conduits, placed one above the other, which extend above all the flues. Below the latter are two regenerators which communicate with the bottoms of the flues. F. G. CLARKE.

Removal of graphite incrustations from ovens, particularly coke ovens. G. CAPIAU, M. GAUQUIER, and L. LAHAUT (B.P. 289,308, 10.8.27).—The incrustation is subjected at the normal temperature of the oven to a stream of high-pressure air carrying particles of a hard granular substance, e.g., slag, which cuts away the graphite very rapidly. A. R. POWELL.

Distillation and liquefaction of coal. F. BERGIUS (U.S.P. 1,669,439, 15.5.28. Appl., 24.11.26).—Coal is carbonised to give coke, tar, ammonia, a gaseous fraction poor in hydrogen and of high calorific value, and a gaseous fraction rich in hydrogen. The tar is mixed with a further supply of coal and is hydrogenated at an elevated temperature and under a high pressure, the gaseous fraction rich in hydrogen being utilised in this process, together with hydrogen obtained from water-gas made from the coke. The hydrogenation yields oil, ammonia, and a gas of high calorific value, which is combined with the gaseous fraction poor in hydrogen and some water-gas to give towns' gas.

A. B. MANNING.

Heat-treatment of briquettes. R. LESSING (B.P. 289,932, 31.1.27).—Briquettes are subjected to heat-treatment while embedded in a granular solid, e.g., powdered anthracite, aluminium granules, etc., whereby the danger of damaging the briquettes during the process is minimised. The temperatures employed may lie in the neighbourhood of 250°, or may approximate to those used in the carbonisation of bituminous coal. Steam or gas may be passed through the granular material to produce more uniform heating, and, if desired, a catalyst may be added to the briquette to reduce swelling of the material or binder. A. B. MANNING.

Fuel composition. A. B. LOW (U.S.P. 1,669,341, 8.5.28. Appl., 26.1.26).—A slow-burning composition comprises finely-divided iron pyrites, a nitrate, and a small quantity of a carbohydrate. L. A. COLES.

Production of carbon black. W. K. LEWIS, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,669,618, 15.5.28. Appl., 2.1.25).—Air, heated to 500—1800°, is passed through coke so as to produce carbon monoxide at a temperature above the decomposition point of methane, and is then introduced into a thermally-insulated passage. Methane is passed into the latter in a relatively cool condition, and the products are cooled and separated. F. G. CLARKE.

Adsorption carbon. W. J. SMIT (B.P. 264,854, 20.1.27. Holl., 25.1.26).—Low-grade, unstable, adsorption carbons may be improved and stabilised by heating them with alkali hydroxides or carbonates or with calcium chloride at 200—300°, leaching out the soluble matter, washing, drying, and grinding in the usual way. [Stat. ref.] A. R. POWELL.

Activation of carbon or carbonaceous material. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,170, 24.1.27).—An apparatus for the activation of carbon comprises an inner porous fireclay tube and an outer wider iron tube. The carbon to be activated is placed either in the inner tube or in the annular space between the tubes, and steam is passed either through the annular space or through the inner tube, so that it diffuses through the fireclay walls into the activating chamber, and the water-gas produced in the process diffuses back into the steam chamber, where it is burnt to provide heat for the operation and to regenerate steam for further use. A. R. POWELL.

Production of hydrogen from gases obtained in the production of coke. R. BATTIG (B.P. 271,491, 19.5.27. Ger., 20.5.26).—The hydrogen present in coke-oven gases is separated from the other constituents by liquefying the latter. The liquefied gases are then fractionated, and a further supply of hydrogen is obtained by the thermal decomposition of the methane fraction. The decomposition is carried out by passing the methane over highly-heated coke in a generator or in the chambers of a coke oven. If the decomposition is incomplete the resulting gases may be again subjected to separation by liquefaction. A. B. MANNING.

Gas producers. WELLMAN SMITH OWEN ENGINEERING CORP., LTD., and A. V. KEMP (B.P. 289,963, 5.2.27).—The blast in a producer provided with a central tuyère surmounted by distributing hoods is regulated by means of a sleeve valve in the tuyère which is operable from the exterior of the producer. A. B. MANNING.

Gas producer plant. POWER-GAS CORP., LTD., and N. E. RAMBUSH (B.P. 290,070, 20.7.27).—The annular water-jacket surrounding the fuel bed of a gas producer is combined with the waste-heat boiler in such a way that they have a common steam drum which is connected with the water-jacket and boiler by circulating pipes. The hot gases preferably pass downward through the tubes of the boiler. In one arrangement the steam drum takes the form of an annular jacket on the upper part of the waste-heat boiler. A. B. MANNING.

Carbonising plant used in the manufacture of gas. R. W. BROADHEAD (B.P. 289,123, 17.1.27).—The gas producer is installed in a shed built on to the walls of the retort house and is connected with a dust-extracting device, such as a whirl or cyclone, which removes the dust from the gas before it enters the insulated flues leading to the regenerator. Means are provided for transferring the incandescent coke from the retort house to the producer without appreciable loss of heat. A. R. POWELL.

Manufacture of producer gas. H. L. DOHERTY (U.S.P. 1,670,102, 15.5.28. Appl., 8.9.20).—A column of fuel passes down a shaft through the sides of which air is blown, the air supply being so controlled as to maintain the highest gas-making temperatures in the upper part of the column; combustion in the lower part of the column is controlled by introducing flue gases thereto in such a way that the fuel falls in temperature as it advances below the mid-portion of the column. A. B. MANNING.

Manufacture of oil-gas. C. A. PHELPS, Assr. to A. J. ANDERSON and M. G. BARMORE (U.S.P. 1,669,172, 8.5.28. Appl., 26.3.24).—A part of the oil is completely burned and the heat is utilised to volatilise and crack the remainder and also to heat up a checker-brick chamber. The volatilisation and cracking of the oil is effected out of contact with the combustion gases, but when this operation has been completed the products are mixed with the combustion gases and the mixture is passed into the heated chequer chamber where reaction takes place and a uniform combustible gas is produced. F. G. CLARKE.

Gas purification. J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,665,013, 3.4.28. Appl., 22.1.27).—Coke-oven or other fuel gas is freed from acidic impurities, such as hydrogen sulphide and hydrogen cyanide, by washing with an alkaline liquor. The latter is regenerated, after being heated, by treatment in counter-current with an upward stream of a portion of the purified gas. The fouled gas is utilised for heating the ovens or in some other part of the plant where the impurities are not objectionable. F. G. CLARKE.

Apparatus for extraction of oil from shale. R. CRAWSHAW (U.S.P. 1,666,488, 17.4.28. Appl., 5.2.27).—The apparatus comprises a heating unit which can be pushed into a mass of the shale and which is provided with pipes whereby compressed air may be forced into the shale and oil withdrawn therefrom. A. R. POWELL.

Extraction of oil from shale. W. RHOADES, Assr. to RHOADES SHALE OIL Co. (U.S.P. 1,668,820, 8.5.28. Appl., 3.12.23).—The volatile products obtained by the distillation of shale are rapidly removed from the retort by suction through a pipe which extends into the mass of shale and has adjustable openings. C. O. HARVEY.

Plant for the manufacture of liquid fuels. Soc. INTERNAT. DES PROC. PRUDHOMME HOUDRY (B.P. 267,512, 8.3.27. Fr., 9.3.26).—In processes similar to that described in B.P. 238,931 (B., 1925, 950) the whole train of catalysts etc. is suitably maintained at tem-

peratures adequate for the prevention of tar condensation in the vital parts of the apparatus.

C. O. HARVEY.

Production of liquid fuel for use in internal-combustion engines. P. G. SOMERVILLE and W. H. HOFFERT (B.P. 289,347, 26.10.26).—The rate of formation of gummy resinous substances in liquid fuels which are, immediately after refining, free from these substances, but which contain polymerisable substances, is inhibited (as shown by accelerated tests involving exposure to ultra-violet light) by the addition of small proportions of aromatic hydroxy-, nitro-, or amino-compounds; acids, aldehydes, and mercaptans appear to assist resin formation. The inhibitors employed include cresols, aniline, etc., and they must be added in the optimum proportions, due allowance being made for the phenolic and other substances already present in the fuel, the time during which the fuel is to be stored, etc. The amounts used are of the order of 0.05%; the use of more than the optimum percentage may assist resin formation.

C. O. HARVEY.

Fuel for internal-combustion engines and motors. C. A. WALTER and F. B. MUHLENBERG (U.S.P. 1,669,181, 8.5.28. Appl., 10.5.24).—The fuel consists of a petroleum product which has been agitated with ammonium chloride and to which has been added small quantities of copper and zinc sulphates.

C. O. HARVEY.

Production of a combustible charge for use in an internal-combustion engine. D. BALACHOWSKY and P. CAIRE (B.P. 265,186, 20.1.27. Fr., 27.1.26).—A hydrocarbon fuel is atomised, mixed with a quantity of air not exceeding one half of that required for combustion, and passed through a chamber heated by means of exhaust gases to 300–350° and containing a metal capable of effecting the catalytic decomposition of hydrocarbons. A further quantity of air is added before the mixture enters the cylinder of the engine.

C. O. HARVEY.

Manufacture of hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 270,705, 2.5.27. Ger., 10.5.26).—Hydrocarbons (mainly liquid) are produced by heating metallic carbonyls such as those of iron, cobalt, nickel, or molybdenum, with a proportion of hydrogen insufficient for the production of methane, and, if required, in the presence of catalysts such as finely-divided metals, alkalis, ammonia, etc. Gases containing hydrogen such as water-gas may be used, and the proportion of hydrogen may be varied according to the class of product required. Thus reduction of the proportion of hydrogen employed increases the molecular complexity of the products and *vice versa*.

C. O. HARVEY.

Manufacture of valuable hydrocarbons and derivatives thereof from coal, tars, mineral oils, etc. I. G. FARBENIND. A.-G. (B.P. 272,190, 20.5.27. Ger., 1.6.26).—The starting material is exposed, in a coherent thin layer, to the action of hydrogen at a raised temperature, (*e.g.*, 450–475°), preferably under pressure (*e.g.*, 200 atm.), and, if required, in the presence of a catalyst. The material is spread in a thin layer on a screw-formed surface, corrugated band, or other device,

preferably metallic, of large superficial area, and is conveyed mechanically through the reaction chamber.

A. B. MANNING.

Conversion of coal into hydrocarbons. A. GAERTNER (B.P. 266,311, 27.1.27. Ger., 18.2.26).—Hydrocarbons are produced by injecting a mixture of powdered coal, steam, and finely-divided iron or other metal capable of producing nascent hydrogen from the steam, into a reaction chamber at 400–500° and under pressure. The iron also serves to retain the sulphur present in the mixture, and lime or other alkalis may be added to retain carbon dioxide.

C. O. HARVEY.

Apparatus for treatment [cracking] of oil. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,104, 15.5.28. Appl., 7.5.25).—The vapours from a battery of cracking stills are combined in a refluxing apparatus interposed between the stills and the condenser.

C. O. HARVEY.

Apparatus for treatment [cracking] of hydrocarbons. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,105, 15.5.28. Appl., 21.8.20. Renewed 12.2.27).—Five horizontal cracking stills are arranged in a regular polygonal manner in a furnace, oil being admitted to the two lower stills and the vapours passing upwards through the next two stills and finally to a refluxing dephlegmator and a condenser *via* the fifth and topmost still.

C. O. HARVEY.

Apparatus for cracking oil. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,106, 15.5.28. Appl., 3.1.21. Renewed 25.3.27).—A refluxing conduit, situated in the vapour space of a horizontal still, serves for the introduction of raw oil, which, after being used as a scrubbing medium for the vapours, is passed to the bottom of the oil in the still.

C. O. HARVEY.

Conversion of heavy hydrocarbons into hydrocarbons of lower mol. wt. A. D. SMITH and J. PERL (B.P. 289,673, 12.7.27).—In a continuous process for cracking hydrocarbon oils, the deposition of carbon in the cracking plant is avoided by checking the decomposition while the highly polymerised products, which would subsequently produce a carbonaceous deposit, are still in solution, distilling off light hydrocarbons, cooling, separating from deposited matter, and then re-cracking.

C. O. HARVEY.

Heat-conversion of hydrocarbon oils. F. A. HOWARD and N. E. LOOMIS, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,670,037, 15.5.28. Appl., 28.11.21).—The oil, forced through a heated, tubular, cracking still with sufficient rapidity to prevent the deposition of carbon, passes to a lagged reaction chamber and thence to a second chamber heated by the furnace gases previously used for heating the still. A gas containing oxygen may be supplied to the second chamber.

C. O. HARVEY.

Treatment of hydrocarbons. R. T. POLLOCK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,122, 15.5.28. Appl., 30.6.20. Renewed 11.4.27).—The oil is heated by passage through a number of horizontal tubes situated in a furnace fitted with a fire box and

baffle walls. The expansion chamber for the vapours is also heated by the upwardly flowing furnace gases.

C. O. HARVEY.

Treatment of oils and other similar hydrocarbons to promote cracking. F. B. DEHN. From M. B. SCHUSTER (B.P. 289,556, 31.1.27).—In the cracking of hydrocarbon oils under pressure, catalytic agents such as colloidal slaked lime or clayey substances of the bentonite type are ground and mixed with the oil to be cracked. These substances, which may require preliminary purification or treatment with dispersive agents (a hydrocarbon, water, or alcohol), are stated to be more universally useful as cracking agents than aluminium chloride, and the asphaltic residues, containing the used catalysts, are more nearly akin to natural asphalt. The catalytic mixtures may also be brought into a gel form prior to admixture with the oil.

C. O. HARVEY.

Cracking of vegetable and mineral oils. Soc. ANON. LE CARBONE (B.P. 290,060, 29.6.27. Fr., 14.5.27).—The oils are dispersed at atmospheric pressure and at relatively low temperatures (about 300–450°) through a mass of activated carbon.

C. O. HARVEY.

Cracking of petroleum oil. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,103, 15.5.28. Appl., 9.3.23. Renewed 15.6.27).—The oil, after being cracked under pressure, passes to a series of expansion chambers, from which the vapours are withdrawn. The unvaporised residues from the chambers are blended in the desired proportions prior to their return to the cracking zone.

C. O. HARVEY.

Cracking of petroleum oil. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,108, 15.5.28. Appl., 27.10.23).—The residuum from an expansion chamber connected with a cracking still is combined with reflux condensates, and the mixture is further distilled by injection into the vapour space of the chamber.

C. O. HARVEY.

Treatment and refining of petroleum. A. M. NASTUKOFF (B.P. 289,920, 28.1.27).—Lubricating and burning oils of good colour and free from objectionable unsaturated substances liable to form asphaltic or gummy products are obtained by treating 1 vol. of crude oil with a mixture containing 4 vols. of strong sulphuric acid and 2 vols. of 40% formaldehyde, removing the more volatile products (after dilution) by means of steam, filtering, and neutralising and washing the hardened product. This product, which is in the form of a dry powder, contains considerable quantities of absorbed oil, which is removed by and recovered from cold solvents, such as benzene. The oils obtained by this process are free from decomposition products such as are produced during cracking processes.

C. O. HARVEY.

Refining of mineral oil. H. T. MAITLAND, Assr. to SUN OIL Co. (U.S.P. 1,668,920, 8.5.28. Appl., 21.12.21).—In an acid-refining process, the sulphuric acid is retained in a mass of material composed of discrete solid particles presenting a very large superficial area. The oil passes through this material and carries with it very little acid.

C. O. HARVEY.

Refining of oils. C. R. WAGNER (U.S.P. 1,669,151 and 1,669,180, 8.5.28. Appl., [A] 17.7.22, [B] 31.5.24).—(A) Petrolatum is removed from steam-refined stocks by separation after dilution with methyl ethyl ketone and chilling. (B) A mixture of butyl alcohol and naphtha containing more than 25% of the former is used in place of the methyl ethyl ketone.

C. O. HARVEY.

Refining of oils and other liquids. O. WERNER (U.S.P. 1,669,182, 8.5.28. Appl., 10.11.25. Renewed 28.3.28).—The oil is heated by passage down a column which contains a chamber heated by mercury vapour. The mercury vapour is derived from a continuous still to which condensed mercury is returned.

C. O. HARVEY.

Distillation of mineral oil. A. E. PEW, JUN., and H. THOMAS, Assrs. to SUN OIL Co. (U.S.P. 1,668,602, 8.5.28. Appl., 23.2.24).—The oil is fractionally distilled by continuous circulation through a system of stills, vaporisers, and fractionating columns.

C. O. HARVEY.

Recovery of sulphuric acid from acid sludges. J. D. RUYSS, Assr. to SHELL Co OF CALIFORNIA (U.S.P. 1,669,102, 8.5.28. Appl., 18.4.27).—Acid sludge from the refining of petroleum is treated with acid oils derived from cracked petroleum oils.

H. ROYAL-DAWSON.

Treatment of emulsions. L. BURGESS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,668,941, 8.5.28. Appl., 20.3.23).—A sulphonic acid, obtained from sludge of hydrocarbons refined with sulphuric acid, is added to emulsions of hydrocarbon oils and water.

H. ROYAL-DAWSON.

Demulsification of hydrocarbons. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,107, 15.5.28. Appl., 7.1.21. Renewed 4.2.27).—Caustic soda, for dehydrating purposes, is injected into the vapour space of a still in which the oil is being distilled under pressure.

C. O. HARVEY.

Deodorisation of liquid naphtha distillates and mineral oils. G. PETROFF (B.P. 289,561, 1.2.27).—Mineral oils, cracked distillates, etc. are deodorised by passing air or oxygen through the hot oil contained in a series of tanks at gradually diminishing temperatures, and containing catalysts. For instance, the oil in the first tank may contain heavy-metal salts of fatty, naphthenic, or resin acids, which activate the oxygen passing to subsequent tanks of oil which contain metal turnings such as copper or lead. The oil may require a final acid and alkali treatment.

C. O. HARVEY.

[Treatment of] mineral lubricating and transformer oils exposed at raised temperatures to air. F. HOFMANN and M. DUNKEL (B.P. 262,107, 22.11.26. Ger., 24.11.25).—Small quantities of aldehydes, and their polymerisation or condensation products with ammonia or organic amines, either alone or in admixture with one another, are added to the oils to inhibit "sludge" formation.

C. O. HARVEY.

Reclaiming used lubricating and insulating oils. A. F. MESTON, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 1,661,731, 6.3.28. Appl., 23.3.25).—The used oils are heated to 65–93°, mixed with sodium phosphate solution, and repeatedly alternately centrifuged (to separate oil and phosphate solution) and cooled to

49—60°. The oil is then mixed with 1—4% of activated earth and centrifuged.

B. FULLMAN.

Oxidation of hydrocarbons [paraffin wax]. J. R. SCANLIN, Assr. to TEXAS CO. (U.S.P. 1,668,871, 8.5.28. Appl., 20.1.26).—Heated paraffin wax is exposed to the action of an oxidising gas until the acid value of the wax is 40 or below. The wax is then free from gummy products insoluble in hydrocarbon oil.

H. ROYAL-DAWSON.

Purification of montan wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 289,621, 8.4.27).—The wax is treated with chromic acid in the presence of glacial acetic acid to which small quantities of sulphuric acid and/or acid salts of sulphuric or other acids are added.

C. O. HARVEY.

Protective coverings for [aircraft petrol] tanks, pipes, etc. J. POBEREJSKY (B.P. 289,659, 2.6.27).

Metal derivatives of diketones (B.P. 289,493).—See III. **Bituminous coatings** (B.P. 289,737).—See IX. **Carbons** (B.P. 289,680).—See XI.

III.—ORGANIC INTERMEDIATES.

Catalytic oxidation of aromatic hydrocarbons and their derivatives by means of air. E. B. MAXTED (J.S.C.I., 1928, 47, 101—105 r).—Many aromatic hydrocarbons and their derivatives, including those containing halogens or nitrogen, undergo oxidation when passed with air over heated catalysts containing vanadium, the most satisfactory being tin vanadate, which becomes active at an abnormally low temperature. In a series of experiments granular tin vanadate was used as a catalyst, and the ratio of hydrocarbon to air was controlled by means of an air flowmeter and by passing a measured portion of the air supply through the hydrocarbon maintained at a constant temperature. Pure benzoic acid was obtained from toluene with the catalyst at 290° (maximum yield 57.1% of the toluene vaporised); with excess of air the yield decreased, and the latter was also found to be dependent on the rate of flow over the catalyst, high rates giving much unchanged toluene whilst low rates resulted in further oxidation. Ethylbenzene gave a maximum yield of 41.3% of benzoic acid at 280°, *o*-xylene gave 59% of phthalic anhydride at 290°, whilst from benzyl alcohol 51.5% of benzoic acid was obtained at 280°. Benzaldehyde, which yielded 45% of benzoic acid at 300°, required a slightly higher catalyst temperature than toluene, which is much more difficult to oxidise, thus indicating that this temperature depends solely on the catalyst and is independent of the substance to be oxidised. Naphthalene with tin vanadate gave 84.3% of phthalic anhydride at 280°; with bismuth vanadate the maximum yield was 81.4% at 363°. Tetrahydronaphthalene yielded 52.8% of phthalic anhydride at 280°, but little or no tetrahydrophthalic acid. The yield of product in g./hr. per c.c. of catalyst space was in all cases except naphthalene about 0.02; with naphthalene it was considerably higher (0.2), because of the high relative stabilities of naphthalene and phthalic anhydride under the conditions employed. The method was unsuccessful when oxidation of *o*-cresol and toluidine was attempted, but

halogeno- or nitro-toluenes were readily converted into the corresponding benzoic acid derivatives.

W. J. POWELL.

Ethylene dichloride as solvent. K. H. BAUER and H. LAUTH (Chem. Umschau, 1928, 35, 82—86).—The chemical, physical, and physiological properties of a specimen of rectified ethylene dichloride are described. It had d_{20}^{20} 1.250, flash point (Abel-Pensky) 12—13°, and 78% of it boiled between 82.8° and 83.3°. Its rate of evaporation at 17° was comparable with that of trichloroethylene, and six times as fast as that of benzine (b.p. 76—143°). Ethylene dichloride mixes in all proportions with methyl and ethyl alcohols, acetone, ethyl acetate, benzene, toluene, light petroleum, ligroin, benzene, pyridine, carbon tetrachloride, trichloroethylene, pentachloroethane, and cyclohexanol. With carbon disulphide, or with completely dry solvents, it gave a turbid mixture. It is 0.5% soluble in water and itself dissolves 0.1% of water at 18—19°. The qualitative solubilities of a number of oils, fats, and waxes in ethylene dichloride and in its mixtures with equal volumes of ethyl and methyl alcohols are tabulated. Nitrocellulose is readily soluble in 80% of ethylene dichloride, 15% of methyl alcohol, and 5% of ethyl acetate, and even more so in a mixture with 20% each by vol. of the solvents mentioned. Cellulose acetate is equally soluble in ethylene dichloride-alcohol mixtures. Quantitative experiments show that ethylene dichloride is stable when boiled for 8 hrs. with varying amounts of water—an important consideration in connexion with the extraction of oil seeds. It is as stable to acid or alkaline permanganate as carbon tetrachloride, and much more stable than trichloroethylene, benzene, and benzene. It is stable to neutral but not to acid dichromate. A review of the literature shows that in its physiological action it is comparable with chloroform.

E. HOLMES.

Alcohol for alcoholic potash. KICZALES.—See VII. **Ethyl alcohol.** MEZZADROLI.—See XVIII.

PATENTS.

Production of methyl alcohol by catalysis. Soc. FRANÇ. DE CATALYSE GÉNÉRALISÉE, Assees. of C. HENRY (B.P. 265,948, 27.1.27. Fr., 9.2.26).—Finely-ground mixtures of 3 or 4 atoms of metallic strontium and 1 mol. of lead monoxide, or of 3 atoms of metallic zinc and 1 mol. of bismuth oxide, are used as catalysts in the production of methyl alcohol from carbon monoxide and hydrogen. Using the mixture 3Sr+PbO, the reaction is carried out at 300° and 5 atm. or 200° and 10 atm.

B. FULLMAN.

Manufacture of organic compounds [synthetic alcohols]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 286,010, 18.8.26. Addn. to B.P. 240,955; B., 1925, 1012).—In the process and in B.P. 254,819 (B., 1926, 721) the inner parts of the apparatus with which the materials come in contact should consist of, or be coated with, metals other than iron, nickel, and cobalt, or of iron alloys stable to carbon monoxide. The catalysts and gases used must be freed from iron, nickel, and cobalt, and their volatile compounds.

B. FULLMAN.

Concentration of formaldehyde solutions. A. ZIMMERLI (U.S.P. 1,662,179, 13.3.28. Appl. 28.1.24).—Formaldehyde solutions are refluxed until equilibrium is set up between formaldehyde, its polymerides, and hydrates, and then fractionated. B. FULLMAN.

Manufacture of acetic anhydride. CONSORT. F. ELEKTROCHEM. IND., G.M.B.H. (B.P. 272,923, 15.6.27. Ger., 16.6.26).—Acetic acid vapour is heated, preferably in the presence of a catalyst, at 400–800° by passage through a vessel the heat-transfer surfaces of which are of silicon or its carbide, copper or alloys rich in it, and especially acid-resistant alloys containing chromium or nickel (chromium-iron and chromium-iron-nickel alloys). The period of heating may with advantage be shortened. B. FULLMAN.

Production of primary [aryl]amines. P. HEROLD and P. KOPPE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,662,421, 13.3.28. Appl., 20.8.27. Ger., 21.8.26).—Nitro-compounds are reduced in the presence of hydrogen or carbon monoxide, under at least 20 atm. and at 100° or over, with a solution of readily soluble sulphides insufficient in amount for complete reduction. B. FULLMAN.

Production of aromatic amines. C. W. DAVIS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,663,476, 20.3.28. Appl., 24.4.25).—Aromatic nitro-compounds are reduced to amines by iron and water in the presence of sulphuric acid and a chloride of an alkali-forming metal. B. FULLMAN.

Purification of 3:4-dichloroaniline. I. GUBELMANN, H. J. WEILAND, and O. STALLMANN, Assrs. to NEWPORT Co. (U.S.P. 1,663,251, 20.3.28. Appl., 19.8.26).—Formation, in the presence of water, of the sulphates of a mixture of dichloroanilines permits of the separation of the practically insoluble 3:4-dichloroaniline sulphate. B. FULLMAN.

Preparation of triarylguanidines. A. E. PARMELEE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,662,397, 13.3.28. Appl., 9.4.24).—A diarylthiocarbamide is treated with an arylamine and a desulphurising agent in the presence of a chlorobenzene. B. FULLMAN.

Manufacture of phenyl-*o*-tolylguanidine. R. V. HEUSER, Assr. to A. C. BURRAGE (U.S.P. 1,669,242, 8.5.28. Appl., 27.2.23).—Aniline and *o*-toluidine in molar proportions are treated with carbon disulphide to form phenyl-*o*-tolylthiocarbamide, from which sulphur is eliminated to give the guanidine. B. FULLMAN.

Preparation of alkyl ethers of 3'-nitro-4'-hydroxy-*o*-benzoylbenzoic acid. I. GUBELMANN, H. J. WEILAND and O. STALLMANN, Assrs. to NEWPORT Co. (U.S.P. 1,665,541, 10.4.28. Appl., 28.6.26. Cf. U.S.P. 1,654,287; B., 1928, 224).—3'-Nitro-4'-halogeno-*o*-benzoylbenzoic acid is treated with an alcohol and a caustic alkali at an elevated temperature. Instead of the free acid its alkali salts may be used. F. G. CLARKE.

Production of dye intermediates [phenyl α -naphthyl ketone]. H. A. E. DRESCHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 289,544, 28.1.27).—*o*-Carboxyphenyl α -naphthyl ketone [*o*- α -naphthoylbenzoic acid] is decarboxylated by heating the calcium or other

alkaline-earth salt under reduced pressure at 300–330°. The phenyl α -naphthyl ketone obtained has m.p. 77°.

C. HOLLINS.

Manufacture of metal derivatives of β -diketones. BRIT. DYESTUFFS CORP., LTD., and S. COFFEY (B.P. 289,493, 28.10.26).—New metal derivatives of β -diketones containing 6 or more carbon atoms, soluble in organic solvents, and useful as antidetonants, are made by treating the diketone with a metal or its oxide, hydroxide, acetate, etc. The following derivatives are described: aluminium (m.p. 46–47°), ferric (m.p. 45°), nickelous (m.p. above 240°), cobaltous (m.p. 121–122°), cobaltic (m.p. 90°), manganic (m.p. 55–56°), chromic (m.p. 76–77°), zinc (m.p. 104), thalious, ceric, and thorium (m.p. 63°) propionylacetones; ferric, cobaltous (m.p. 123°), and thorium butyrylacetones; ferric, nickelous, and cobaltous γ -methylacetylacetones; aluminium (m.p. 139°), ferric, and chromic (m.p. 132°) γ -ethylacetylacetones; aluminium, ferric, and chromic γ -butylacetylacetones; nickelous acetylmesityl oxide (m.p. 140°); and manganic (m.p. 188°), chromic (m.p. 224°), and thorium (m.p. 213°) benzoylacetones. Many of the products are intensely coloured. C. HOLLINS.

Production of derivatives of diaryl ketones. BRIT. DYESTUFFS CORP., LTD., W. H. CLIFFE, F. W. LINCH, and E. H. RODD (B.P. 289,571, 5.2.27).—The sodio-derivatives of tetra-alkyldiaminobenzophenones (B.P. 272,321; B., 1927, 598) react with compounds containing a methyl or methylene group, and the mixture of products gives on treatment with water the tetra-alkyldiaminobenzhydrol and new carbinols: $2(\text{NR}_2 \cdot \text{C}_6\text{H}_4)_2\text{CO} + 2\text{Na} + \text{CH}_2 < \text{---} (\text{NR}_2 \cdot \text{C}_6\text{H}_4)_2\text{CH} \cdot \text{OH} + (\text{NR}_2 \cdot \text{C}_6\text{H}_4)_2\text{C}(\text{OH}) \cdot \text{CH} < \text{---}$. The carbinols are readily dehydrated by boiling dilute acids to form the corresponding ethylenes. The compounds obtained from toluene, tetramethyldiaminodiphenylmethane, acenaphthene, and fluorene are described (cf. Rodd and Linch, A., 1927, 1067). C. HOLLINS.

Production of aromatic ketonic compounds. F. H. KRANZ, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,661,293, 6.3.28. Appl., 2.4.26).—4:4'-Tetramethyldiaminodiphenyl thioketone is produced by boiling under reflux, with agitation, a mixture of 10 pts. of 4:4'-tetramethyldiaminodiphenylmethane and 15 pts. of 50% sodium hydroxide solution containing 16 pts. of sulphur. B. FULLMAN.

Production of β -naphthol-1-sulphonic acid. A. E. PARMELEE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,662,396, 13.3.28. Appl., 4.3.24).— β -Naphthol is suspended in an inert liquid and treated below 16° with a solution of sulphur trioxide. B. FULLMAN.

Oxidation of ethyl alcohol. O. Y. IMRAY, From S. GOLDSCHMIDT (B.P. 290,523, 30.11.27).—See U.S.P. 1,666,447; B., 1928, 397.

Preparation of urea from carbon dioxide and synthetic ammonia. L. CASALE (M. C. SACCHI, adrix.) (U.S.P. 1,670,341, 22.5.28. Appl., 11.6.25. It., 23.12.24).—See B.P. 241,123; B., 1926, 27.

Separation of mono- and di-alkylarylamines. R. W. EVERATT and E. H. RODD, Assrs. to BRIT. DYE-

STUFFS CORP., LTD. (U.S.P. 1,670,850, 22.5.28. Appl., 9.5.27. U.K., 25.8.26).—See B.P. 273,923; B., 1927, 648.

Production of 4-nitro-2-aminophenoxy-ethanol or -propandiol [4-nitro-2-aminophenyl β -hydroxyethyl or β -dihydroxypropyl ether]. O. KNECHT, ASSR. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,669,764 and 1,670,969, 15. and 22.5.28. Appl., [A] 13.12.26, [B] 27.10.27. Ger., [A, B] 19.12.25).—See B.P. 263,191; B., 1928, 255.

Preparation of [mercaptobenz]thiazoles. L. B. SEBRELL and J. TEPPEMA, ASSRS. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,669,630, 15.5.28. Appl., 17.6.25).—See B.P. 282,947; B., 1928, 152.

Foams for fire extinction (B.P. 289,630).—See I.

IV.—DYESTUFFS.

Derivatives of sulphur dyes prepared from indo-phenols obtained from carbazole and its substitution products. E. JUSTIN-MUELLER (Sealed Notes 2126 and 2138, 6.11. and 22.12.11. Bull. Soc. Ind. Mulhouse, 1928, 94, 185—186). Report by A. RASOUMÉEFF (*Ibid.*, 186—187).—By the restricted nitration (Note 2126) of Hydron Blue R and G deep brown and olive-green dyes are obtained which yield shades very fast to washing but loose to chlorine when applied to cotton from a caustic soda-hyposulphite vat. Nitration is effected by dissolving 10 g. of Hydron R or G in 200—300 g. of sulphuric acid (d 1.842), cooling, adding a mixture of 30 c.c. of sulphuric acid (d 1.842) and 30 c.c. of nitric acid (d 1.334), and after several hours precipitating the nitro-dye by the addition of water. On prolonged nitration (Note 2138) polynitro-derivatives are obtained, Hydron Blue R yielding a dye which is more olive than that produced by restricted nitration. These nitro-dyes suffer reduction when dyed from a caustic soda-hyposulphite vat, and the resulting amino-derivatives may be diazotised and coupled on the fibre, thereby yielding shades also fast to washing but loose to chlorine. Rasoumèeff reports favourably on the processes. A. J. HALL.

Formation of lakes of alizarin. S. LIEPATOV (Textilber., 1928, 9, 496—497).—By determination with barium hydroxide of the free and combined nitroalizarin present in the barium and copper lakes obtained by treating this dye with solutions of barium and copper acetate of various concentrations as previously described (A., 1925, ii, 1059), it is shown that such lakes are not adsorption compounds, but consist of barium or copper nitroalizarates contaminated with free nitroalizarin. *E.g.*, the lakes obtained by treating nitroalizarin with 10 and 0.65% solutions of barium acetate contain 14 and 4% of barium, respectively, but both consist of barium nitroalizarate, the former being contaminated with less free nitroalizarin than the latter. It is suggested that definite chemical combination between alizarin and the mordant on cotton also occurs during dyeing. A. J. HALL.

PATENTS.

Preparation of washable aniline dyes. P. HARTMANN, ASSR. to K. SCHENZER (U.S.P. 1,662,420, 13.3.28.

Appl., 26.11.26. Renewed 25.1.28).—A permanent dye is made by evaporating to dryness mixed solutions of "aniline crystals" and boric acid. B. FULLMAN.

Manufacture of condensation products of the naphthastyril series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 289,692, 6.9.27).—Naphthastyril is condensed in presence of phosphoryl chloride, thionyl chloride, etc. with a tertiary aromatic amine or a mono- or poly-hydric phenol. Amines give basic dyes of good light-fastness (compared with triarylmethane dyes), whilst phenols give chromable dyes; both types yield acid dyes on sulphonation. Examples are dimethylaniline (blue-violet, violet wool dye by sulphonation), dialkyl- α - and - β -naphthylamines (pure blue), diethyl-*m*-phenetidine (red-violet), pyrogallol (sulphonated); yellow, brilliant red-orange chromed), tetramethyl-*o*-phenylenediamine (blue-grey), tetramethyl-*m*-phenylenediamine (reddish-blue), and 1:4-dimethyl-1:2:3:4-tetrahydroquinoxaline (blue-black). C. HOLLINS.

Manufacture of triarylmethane dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,564, 1.2.27).—An aromatic hydroxy-compound is oxidised in alkaline solution together with a *p*-hydroxydiarylmethane, or a corresponding hydrol, or a *p*-cresol. Thus, 6:6'-dihydroxydi-*m*-tolylmethane-5:5'-dicarboxylic acid [methylenedi-*o*-cresotic acid] with *R*-acid gives a chrome reddish-blue; *p*-cresotic acid with *o*-cresotic acid (2 mols.) gives a chrome reddish-violet. The oxidant may be air in presence of copper sulphate, or lead peroxide, manganese dioxide, etc. C. HOLLINS.

Extraction of colouring matter from henna leaves. C. POLLACCHI, ASSR. to COTY SOC. ANON. SURESNES (U.S.P. 1,668,603, 8.5.28. Appl., 29.6.25. Fr. 2.7.24).—See B.P. 236,557; B., 1925, 751.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Delignification of jute fibre. J. K. CHOWDHURY and R. K. DAS (J. Indian Chem. Soc., 1928, 5, 231—243).—Phenols, in particular beech-wood creosote (Merck), remove the lignone complex from jute without affecting appreciably the cellulose. When creosote alone is used, the fibre becomes brittle, but with creosote + 10% of pyridine, the tensile strength of the product is fairly good. The effect of water at high temperatures is to reduce the fibre to a pulp, whilst excess of pyridine causes a diminution in the cellulose content. Maximum delignification with creosote + pyridine is obtained at 193—195° under 28—30 atm. for 4 hrs. The β -cellulose content of the resulting fibre is still fairly high, and it is removed partly by treatment with hot 5—6% sodium hydroxide solution. The resulting fibre retains its original colour and is of good tensile strength. H. BURTON.

Does formaldehyde protect wool against moths? C. O. CLARK (J. Soc. Dyers and Col., 1928, 44, 144—145).—The statement of Trotman and others (B., 1928, 256) that the treatment of wool with formaldehyde would probably confer on it immunity against attack by moths is disputed. Tests at the Zoological Laboratory of the I. G. Farbenindustrie show that wool so treated has not the slightest resistance to attack by moth grubs. A. J. HALL.

Viscose. V. Quality of caustic soda, manufactured in Japan, from the point of view of the viscose industry. M. NUMA (*J. Cellulose Inst. Tokyo*, 1928, 4, 82—94).—The dissolving power of sodium hydroxide for the modified cellulose contained in pulp is diminished by the presence of sodium carbonate. Small quantities of both sodium sulphate and chloride behave catalytically in assisting dissolution, but in large quantities their action is deleterious; the presence of sulphate, even in small amount, renders the viscose film brittle. More than half the total quantity of β -cellulose in the original pulp is retained by the alkali-cellulose after removal of the excess sodium hydroxide. The most satisfactory film is given by use of a sodium hydroxide solution containing the minimum amounts of carbonate and sulphate and no insoluble impurities such as alumina, iron, silicates, etc. Sodium chloride present to the extent of 3—5% has a beneficial effect on the film. Specifications for the composition of sodium hydroxide to be used in the production of viscose are laid down.

B. P. RIDGE.

Pulping flax straw. IV. Hydrolysis and delignification with alkaline reagents. E. R. SCHAFER and C. E. PETERSON (*Pulp and Paper Mag.*, 1928, 26, 477—481; cf. B., 1927, 327; 1928, 185).—Detailed results are given for a further group of experimental cooks on flax straw using mixtures of caustic soda and sodium sulphide (4:1) at different concentrations, caustic soda alone, in admixture with, and alternating with sodium sulphite, and sodium sulphite alone, and alternating with a mixture of caustic soda and sodium sulphide. Analyses of the pulps prepared with caustic soda and sodium sulphide show that the non-lignin incrustants are removed much more rapidly than is the lignin, a total cooking time of 3 hrs. with 1 hr. at maximum temperature (155°) being sufficient to reduce the former to a minimum value. The sulphur in the cooking liquor does not react appreciably during the first 3 hrs. of the cook, though it is later consumed at a slow and fairly constant rate. The results of the other cooks indicate that (a) with caustic soda and sodium sulphite the reaction is mainly alkaline hydrolysis, though the sulphite radical does react—possibly with the products of alkaline hydrolysis; (b) with sodium sulphite alone, dissolution, particularly of the lignin, is due to the action of the bisulphite radical; and (c) the solubility of lignin in sodium sulphite solution is lowered by a previous alkaline hydrolysis, though pretreatment with sodium sulphite has no effect on the solubility of lignin in caustic soda. The chlorine requirements of the crude pulps as determined by a modification of Roe's method was in each case found to be approximately equal to their lignin content.

D. J. NORMAN.

Behaviour of lignin and chlorolignin in the preparation of wood pulp by means of chlorine. I. P. WAENTIG (*Z. angew. Chem.*, 1928, 41, 493—498).—In the chlorine method of cellulose isolation the chief economic problems to be solved are the high consumption of chemicals and the utilisation of the by-products. The chlorine attacks almost exclusively the lignin, since isolated lignin takes up about the same amount as that in natural lignocellulose, 1 pt. of lignin requiring about 1.4 pts. of chlorine, of which about 70% reappears as

hydrogen chloride. The heat liberated during the reaction is approximately 175 kg.-cal. per kg. of wood, or 120 kg.-cal. per kg. of grain straw. These results are approximate, since the amount of chlorine absorbed depends on the temperature and the time of reaction, but if these are kept constant the amounts of heat evolved, hydrogen chloride formed, and the yield of cellulose are the same whether chlorine water or gaseous chlorine (with moist wood) is used. Since the reaction is carried out at a comparatively low temperature, mechanical disintegration is desirable to facilitate penetration of the fibres by the liquor and thus ensure uniform chlorination, increase of pressure and the use of chlorine solvents being also advantageous from this point of view. In the absence of such aids the process is only suitable for straw, flax, hemp, and short-fibred woods. Though the amount of chlorine taken up increases with increasing temperature, the amount of hydrogen chloride formed increases at the same rate and the ratio remains constant. Preheating of the wood with alkalis reduces the amount of chlorine necessary, but the ratio of total chlorine to hydrogen chloride remains practically constant. If the chlorination is carried out in presence of aqueous hydrogen chloride (10—15%) instead of water, the total chlorine used is unchanged, but the amount of hydrogen chloride formed decreases, indicating that hydrolysis of chlorolignin takes place less readily under these conditions. Dry chlorine does not react with dry wood. In presence of a limited quantity of water, the temperature may rise above 100° and the concentration of hydrogen chloride in the water may exceed 20%. Both these conditions have a harmful effect on the resulting fibre, but by the use of water as a cooling agent and chlorine carrier the reaction is modified so that a strong fibre possessing good folding properties is obtained.

W. J. POWELL.

Effect on cellulose fibres of treatment in the papermaker's beater. J. W. ROWE (*Proc. Tech. Sect. Papermakers' Assoc.*, 1927, 8, 186—221).—The hydration of cellulose by beating has been studied by determining the rate of evaporation of water from unbeaten and beaten sulphite wood-pulp discs under constant drying conditions. The curves connecting rate of evaporation and percentage of water in the disc show three distinct breaks, viz., for an unbeaten pulp, at about 66% (point *a*), 20% (*b*), and 12% (*c*). Above *b* the water is considered to be free (the position of *a* depending mainly on the texture of the disc), between *b* and *c* to be held by surface forces only, and below *c* to be actually distributed amongst the cellulose molecules. The value at *c*, therefore, may possibly indicate the degree of hydration of cellulose, and on this basis experiments show that whilst hydration increases with beating, the beaten pulp "dehydrates" when left in contact with water for a long period, though even when "dehydration" is almost complete, as indicated by the position of *c*, the pulp would still be considered "wet" from the papermaker's point of view. In explanation of this "dehydration" effect and of the action of the beater in general, it is suggested that beating effects a partial conversion of the cellulose from a crystalline into an amorphous state, and that, on keeping, the amorphous (hydrated) variety tends to revert to the

crystalline form. Some support is lent to this theory by the observation that the viscosity of sulphite pulps in cuprammonium hydroxide solution is not lowered by any degree of beating that would be used in the manufacture of paper, which precludes the theory that the hydration of cellulose by beating is due to an increased dispersion of the cellulose aggregate. D. J. NORMAN.

Recent work on the oxidation of cellulose. J. L. PARSONS (Ind. Eng. Chem., 1928, 20, 491—493).—A review of recent work on the degradation of cellulose lends support to the view that the term "oxycellulose" cannot be applied to any individual chemical compound. "Oxidised cellulose" is to be preferred when referring to the products of the complex reactions involved in the oxidation of cellulose. D. J. NORMAN.

Cellulose resources. I. Annual wood crop. G. M. ROMMEL (Ind. Eng. Chem., 1928, 20, 494—496).—The estimated yield in cub. ft. and tons per acre per year at 30 and 60 years of age which may be expected from some of the common trees found in American forests is tabulated. The southern pines grow to pulpwood size in 15 years as against 30—50 years for northern pines, and, if an economical method of removing resin could be found, the southern pines would be available for newsprint. The possibilities of timber waste and sawmill wood-waste for pulpmaking are also outlined. D. J. NORMAN.

Bamboo. I. Composition of the bamboo "Mōsō-Chiku." Y. UEDA, K. KASAMA, and K. KIMURA (J. Cellulose Inst. Tokyo, 1928, 4, 95—98).—The Japanese bamboo "Mōsō-Chiku" contains (approx.) water 8—9%, cellulose 42%, lignin 24.5%, pentosan 23%, alcohol and benzene extract 3%, the cellulose obtained from the dried product by the chlorination method having the following characteristics: furfuraldehyde yield 4%, copper value 0.7, α -, β -, and γ -cellulose content, respectively, 79%, 18%, and 3%. The yields by acetolysis and sulpholysis are 27 and 80%, respectively, whilst hydrolysis by 7% sulphuric acid gives xylose crystals amounting to 3.7% of the weight of dry cellulose taken. B. P. RIDGE.

Bamboo [for papermaking]. W. RAITT (Proc. Tech. Sect. Papermakers' Assoc., 1927, 8, 89—98).—The claims of bamboo as a practically inexhaustible source of paper pulp are advanced. The earlier difficulties of preparing bleached bamboo pulp have been overcome and the waste due to nodes has been eliminated by crushing. By adopting fractional digestion in conjunction with the sulphate-soda process it is possible, using 16% of soda for 5 hrs. at 24 lb./in.², to obtain a 45% yield of pulp requiring only 8% of bleaching powder (yield of bleached pulp 42%). It is estimated that unbleached bamboo pulp could be delivered (c.i.f. British ports) at £11 10s. per ton. D. J. NORMAN.

Shortening of the time of cooking in the Mitscherlich process [of paper manufacture]. L. FRIEDLÄNDER (Papier-Fabr., 1928, 26, 335—337).—In place of the usual heating coils in the cooker an internal, cylindrical, copper heating chamber is recommended. The chamber is fitted with heating tubes so arranged that the inside walls of the chamber and the outsides of the tubes are heated by steam, whereas the liquor is heated

inside the tubes and by the outside walls of the cylinder. In this way a considerably increased heating surface is obtained with circulation of the liquor through the tubes, and the time of cooking is halved. B. P. RIDGE.

Ethylene dichloride as a solvent. BAUER and LAUTH.—See III. Bleaching of cellulose. RYS.—See VI. Degumming power of soaps. TSUNOKAE.—See XII.

PATENTS.

Manufacture of a substance to be used in laundries. H. H. BOCKWEG (B.P. 281,998, 2.8.27. Holl., 8.12.26).—By the use of a mixture containing 75—150 pts. by wt. of powdered ultramarine, 3—15 pts. of powdered dragonblood, and 1000—2500 pts. of sodium bicarbonate, it is claimed that laundry operations may be reduced in number, and that an economy of 50—60% of chlorine and 15—35% of soap may be effected. E. HOLMES.

Treatment of fibres, fabrics, etc. made of or containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 260,290, 22.10.26. U.S., 24.10.25).—The material is treated with a solution of tannic acid of sufficiently high concentration, e.g., about 30%, to exert a swelling effect on the cellulose derivative (acetate). The solution is preferably maintained at 50—70°, and the duration of the treatment may be $\frac{1}{2}$ —2 hrs. The tannic acid thus introduced is preferably, but not necessarily, fixed by a further treatment with a solution containing tartar emetic or stannic chloride. The weighted silk shows increased resistance to heat and to slipping and laddering. D. J. NORMAN.

Treatment of fibrous materials for pulping purposes. T. L. DUNBAR, Assr. to CHEMPULP PROCESS, INC. (U.S.P. 1,669,234, 8.5.28. Appl., 22.4.27).—Steam is introduced into the digester during the cook, and is allowed to condense and mix with the digestion liquor. Continuous circulation is maintained, and when the mixture reaches a predetermined volume the excess of liquor is transferred to an accumulator. D. J. NORMAN.

Manufacture of cellulose esters. Cellulose acetate. H. L. BARTÉLEMY, Assr. to RUTH ALDO Co., INC. (U.S.P. 1,668,482—5, 1.5.28. Appl., [A—C] 27.1.28. [D] 30.1.28. Fr., [A] 28.12.27, [B, C] 29.12.27).—(A) Dilute acetic acid containing hydrochloric and hydrofluoric acids is used for the partial hydrolysis of the primary acetate resulting from the acetylation of cellulose in the presence of a catalyst. (B) Esterification of cellulosic fibres is facilitated by pretreating them with a mixture of gaseous acetic acid and a halogen. (C) The esterification of cellulose or its conversion products is effected by a series of at least four successive partial acetylations, each of which is completed before the succeeding one is commenced. (D) Cellulosic material is treated first with an alkaline solution containing peroxides and a soap and then as in (B). Acetylation and hydrolysis are conducted as described in (C) and (A) respectively. D. J. NORMAN.

Esterification of hydrated cellulose with lower fatty acids. Reacetylation of cellulose acetate. H. T. CLARKE and C. J. MALM, Assrs. to EASTMAN

KODAK Co. (U.S.P. 1,668,945—6, 8.5.28. Appl., 12.1.27).—(A) The hydrated cellulose is treated at 100—170° and in the absence of a catalyst with a fatty acid having more than one and less than eight carbon atoms until 4 mols. of the latter combine with 1 mol. of the former, assuming the cellulose molecule to contain 24 carbon atoms. (B) Cellulose acetate, the solubility of which has been reduced by the loss of acetyl groups, is treated with acetic acid, as sole acetylating agent, at a temperature below the decomposition point of the product until the original solubility is restored.

F. G. CLARKE.

Reducing the viscosity of nitrocellulose materials. C. U. PRACHEL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,661,736, 6.3.28. Appl., 27.2.25).—Solid colloidal nitrocellulose materials are treated at atmospheric pressure with an aqueous mixed mineral acid solution, containing nitric acid, of insufficient strength to increase the nitration.

B. FULLMAN.

Manufacture of artificial silk threads. H. WADE. From N. V. BOUWONDERNEMING KETABANG IV (B.P. 289,976, 9.2.27).—The length of travel of viscose filaments in the coagulating bath is increased to such an extent, *e.g.*, from 8 in. up to 24 in. for a sulphuric acid-sodium sulphate bath, that coagulation and all further normal alterations that the filaments may undergo are practically completed within the bath itself. The filaments are then twisted and wound on a frame.

D. J. NORMAN.

Recovery of soda ash [from digestion of cellulose]. J. HOLMES, H. A. KINGCOME, and J. L. JARDINE (B.P. 288,699, 11.1.27).—The spent liquor from the digester is discharged into a closed receiver and is fed therefrom into a waste-heat boiler where it undergoes a preliminary concentration. After further concentration in an evaporator, the liquor is sprayed into an incinerator and burnt, the heat from this operation being used to heat the waste-heat boiler, which in turn supplies the low-pressure steam requirements of the digesters and soda recovery plant. To facilitate the operation of the waste-heat boiler, vigorous circulation should be maintained therein by mechanical means (cf. Holmes, B., 1928, 120).

D. J. NORMAN.

Cooking of wood to form chemical paper stock. W. H. HOWELL, JUN. (U.S.P. 1,670,156, 15.5.28. Appl., 5.3.25).—The spent liquor withdrawn from the digester after completion of the cook is immediately subjected to a vacuum to concentrate it, and is at the same time indirectly heated by steam from the digester.

D. J. NORMAN.

Safety paper. W. R. ORNDORFF, Assr. to TODD Co., INC. (U.S.P. 1,662,509, 13.3.28. Appl., 8.1.25).—The paper contains pyrogallol 1 : 3-dimethyl ether.

B. FULLMAN.

Manufacture of artificial silk. C. M. WHITTAKER, Assr. to COURTAULDS, LTD. (U.S.P. 1,670,919, 22.5.28. Appl., 1.6.26. U.K., 27.7.25).—See B.P. 254,531; B., 1926, 782.

Fire-proofed [cellulose ester] products E. C. R. MARKS. From DU PONT VISCOLOID Co. (B.P. 289,936, 1.2.27).—See U.S.P. 1,633,067; B., 1927, 775.

Separation of bark or bast from plant stalks.

E. L. A. HEIMLICH (B.P. 265,618, 5.2.27).

Treatment of fats and oils (B.P. 281,232).—See XII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching of sulphite-cellulose. L. RYS (Papier-Fabr., 1928, 26, 256—260, 269—275, 288—289).—The advantages of bleaching in higher-stuff density (increase of reaction velocity, cold bleaching, greater space efficiency, lower power consumption, etc.) are discussed, and it is shown that by a combined chlorine water-hypochlorite bleach and alkali extraction an unusual degree of purity of colour, good chemical properties of the material, and about 30% economy of chlorine are obtained. The duration of the alkali extraction depends on the temperature and alkalinity of the liquid, milk of lime giving as satisfactory results as sodium hydroxide. In the pre-bleaching about 75% of the required total chlorine is used as chlorine water, the remaining 25%, as hypochlorite, being used in the after-bleaching, which may be catalytically accelerated by the use of metallic chlorides (*e.g.*, of copper, cobalt, or nickel). The economy of chlorine is ascribed partly to the preliminary alkali extraction and partly to the use of chlorine water. By this method the α -cellulose content is never lower, and the copper number little, if any, higher than corresponding values given by other processes, whilst a better colour is obtained. The relationship between the different degrees of fluorescence in ultra-violet light and the purity of the bleached material is established, comparisons being made both visually and photographically. Pure cellulose has a bluish-violet fluorescence, whilst the less pure is the material the more its colour approaches greenish-grey to brown. Theoretical and practical grounds for bleaching with hypochlorite in low and high stuff density, and for the chlorine water bleach and alkali extraction of the chlorinated incrusting substances are discussed.

B. P. RIDGE.

Apparatus for the analysis of solutions, especially bleach liquors containing chlorine. K. HINTZMANN (Chem. Fabr., 1928, 266—267).—To determine the available chlorine in a bleach liquor an apparatus comprising a graduated tube with a lower reaction bulb and an upper stoppered bulb holding 75 c.c. of liquid is used. The reaction bulb is filled with 75 c.c. of a standard solution of indigo-carmin (1.46 g. of the 44/45% powder and 2 c.c. of sulphuric acid/litre), the stopper placed in the upper bulb, the apparatus inverted, and the burette read. The whole is turned back to the normal position and the solution to be analysed is added slowly until the colour of the mixture changes from blue to light yellow-green. The stopper is replaced, the apparatus inverted, and the burette again read to obtain the amount of bleach liquor added.

A. R. POWELL.

Discharging indigo-dyed fabric by means of nitric acid. (A) Action of certain catalysts. (B) Discharges with Flavanthrene and Thioindigo Red B. (C) Discharges with nitroso- β -naphthol. N. VOSNESSENSKI (Sealed Notes 1924—5, 2.9.09, and

1940, 18.10.09. Bull. Soc. Ind. Mulhouse, 1928, 94, 187—189, 189—190, 190—191). Report by L. PAULUS, (*Ibid.*, 191—192).—(A) In the presence of small quantities of catalysts such as potassium sulphite, sodium bisulphite or hyposulphite, powdered aluminium, zinc, or copper, and particularly sodium nitrite, the discharge of indigo-dyed fabric may be effected by means of nitric acid, using a lower concentration of acid and temperature of treatment than would be required otherwise. For example, a mixture containing 100 g. of sodium nitrate and 20 g. of sodium nitrite produces discharges equal to those obtained with 200 g. of sodium nitrate, and sulphuric acid, (d 1.261) may be used instead of acid of d 1.372. (B) Coloured discharges on indigo may be obtained with dyes of the flavanthrene and thioindigo series, very fast to light and washing, provided that the discharge is effected by means of nitric acid instead of the more usual chromate method. (C) Clear olive discharges on indigo grounds, not obtainable by the use of albumin colours and the usual chromate method of discharge, may be produced by the nitrate discharge using nitroso- β -naphthol and iron or chromium nitrate, and a basic dye if desired. Paulus confirms the originality and usefulness of the above processes. A. J. HALL.

Effect of light on indigo-dyed [cotton] fabric.

R. HALLER, J. HACKL, and M. FRANKFURT (Textilber., 1928, 9, 415).—Claim is made for the independent discovery (cf. Hibbert, B., 1927, 840) that isatin is formed during the fading of indigo-dyed cotton by exposure to sunlight. During exposure under glass for 4 months indigo-dyed fabric changed to an olive colour, and the faded fabric yielded yellow extracts with hot water or dilute alkalis; the aqueous extract gave a blue colour when treated with pyrrole and sulphuric acid, and formed crystals (m.p. not determined) identical with those of isatin. The faded portions of the fabric also contained oxycellulose which was sufficient to reduce indigo in the presence of caustic soda. It appears that the fading of indigo is a process of oxidation, and is thus in marked contrast to the fading of cotton dyed with Indanthrene Yellow. Under similar conditions of exposure undyed cotton shows no appreciable deterioration, so it is concluded that the indigo assists the oxidation of cotton during exposure to light. A. J. HALL.

Theory of leather dyeing. H. SALT (J. Soc. Dyers and Col., 1928, 44, 134—135).—Acid and direct dyes, but not basic dyes, are readily absorbed by chrome leather, but if the chrome leather is tanned with vegetable tannages before dyeing its affinity for acid and direct dyes is reduced and its affinity for basic dyes increased proportionally to the amount of tannin fixed by the hide. Vegetable-tanned leather has a considerable affinity for basic dyes, this affinity being reduced by treating the leather with basic chromium salts before dyeing. These facts support the theory that the acid groupings of hide are "occupied" (neutralised) by mineral tanning agents, and the basic groupings by vegetable tanning agents. The usual preliminary treatments of hides before tanning increase the reactivity of the acid and basic groupings and so increases the affinity of the hides for dyes. It is suggested that this affinity of a vegetable leather for acid dyes is

due to partial replacement of the vegetable tannic acid combined with the hide by the acid dye, and since the leather-colour acid compound is insoluble, gradual exhaustion of the dye liquor occurs. If the vegetable tannic acid is fixed in the hide by drying or by treatment with tartar emetic before dyeing, the hide has no affinity for acid dyes. The degree and rate of replacement of vegetable tannic acid by an acid dye is dependent on the acidity of the dye liquor, considerable and rapid replacement occurring in a liquor of p_H 2, so that the leather is superficially and unevenly dyed, whereas in a dye liquor of p_H 6 the affinity of the leather for acid dyes is small, so that the resulting dyeings are even and well penetrated. It is usual to obtain less even shades by dyeing chrome leather with acid or direct dyes than by dyeing vegetable leather with basic dyes, but more even and better penetrated dyeings may be obtained by the former method provided that the acidity of the dye liquor is maintained at about p_H 6 by the suitable use of sodium or ammonium acetate and sulphuric acid in the dye liquor. Indigosol and Soledon dyes (water-soluble esters of leuco-vat dyes) may be dyed on leather by the methods used for acid dyes.

A. J. HALL.

Wetting-out and emulsifying agents. A. NOLL (Papier-Fabr., 1928, 26, 318—326).—The properties of a number of commercial preparations for use as wetting-out and emulsifying agents are described. Comparisons are made between their percentages of ash, its sulphate content, the percentage of the dry substance both extracted by and precipitated by alcohol from aqueous solution, and the colours of the fluorescence of their aqueous solutions. The stability of the substances is examined by treating one part by wt. of the compound and of a cyclic alcohol or ketone (*e.g.*, cyclohexanol or cyclohexanone), with one or more parts of water and observing the conditions under which a non-homogeneous mixture is obtained; the results of the examination are tabulated. B. P. RIDGE.

Coloured reserves under sulphur dyes [by printing]. H. FLEICHER (Sealed Note 1941, 23.10.09. Bull. Soc. Ind. Mulhouse, 1928, 94, 195—197). Report by V. SCHWARTZ (*Ibid.*, 197—198).—A process for obtaining coloured reserves under sulphur dyes consists of printing on cotton fabric previously prepared with a 3.5% solution of potassium ferricyanide, a reserve paste containing a solution of a basic dye in phenol, a solution of zinc tungstate dissolved in any suitable organic acid, zinc chloride, and a thickening consisting of gum Senegal and china clay, then drying, steaming in a Mather-Platt for 4 min. at 102—108° (the most satisfactory conditions of steaming are those commonly used for discharging azoic colours with Rongalite), and afterwards padding with a sulphur dye liquor, oxidising by means of an "air run," and immediately washing in cold water, souring with sulphuric acid (d 1.036) at 50°, washing with cold water, and soaping. Schwartz reports that the resulting coloured reserves are much faster to washing than those obtained previously by methods in which the basic dye is fixed with zinc ferro- or ferri-cyanide alone, this increased fastness being due to the additional presence of zinc

tungstate which is capable of forming fast lakes with basic dyes. The process suffers from the disadvantage that the reserved portions of the cotton fabric are tendered by the tartaric acid used in the reserve paste, but this may be avoided by adding sodium tungstate to the "prepare" liquor and omitting zinc tungstate from the reserve paste, the use of tartaric acid thus being rendered unnecessary. A. J. HALL.

Production of [coloured] photographic prints on cellulosic materials [cotton fabrics]. M. MICHELS (Bull. Soc. Ind. Mulhouse, 1928, 94, 124—135). Report by P. BRAUN (*Ibid.*, 135—138).—A continuous and economic process for producing coloured impressions from photographic negatives on cotton and especially viscose silk fabrics consists of padding the fabric with a sensitising solution (100 g. of green ammonium ferric citrate, 50 g. of silver nitrate, 30 g. of an organic acid, *e.g.*, tartaric, citric, or lactic acid, and 1 litre of water), drying, exposing the fabric under a photographic negative to light, washing with water, treating with a 1—2% solution of sodium chloride to convert unremoved silver nitrate into silver chloride, again washing with water, and treating with a solution containing lead ferricyanide and acetic acid, whereby silver present in the image on the fabric is converted into a mixture of the ferrocyanides of lead and silver which, after washing and treatment with a solution of cupric chloride, are converted into cupric ferrocyanide and the chlorides of lead and silver. After further treatment to remove silver chloride the fabric is immersed first in a solution containing sodium sulphite, sodium carbonate, and 1% of quinone (a catalyst), whereby the cupric ferrocyanide of the image (brown) is reduced to cuprous ferrocyanide (nearly colourless), and then for 10—15 min. in a solution containing acetic acid and a basic dye at 80—90°; washing with warm water, soaping, again washing, and then drying completes the process. About 98% of the silver used is recovered. Braun reports favourably on the process since the resulting coloured images have excellent fastness to light and washing, the shades being equal in brightness to those obtainable by means of basic dyes on a tannin mordant. Using an ordinary photographic negative an exposure of $\frac{1}{2}$ —1 hr. in full sunlight or $\frac{3}{4}$ —1 $\frac{1}{2}$ hrs. under a mercury vapour lamp was necessary to obtain satisfactory prints on cotton fabric.

A. J. HALL.

Colouring of rubber. SANDERSON.—See XIV.

PATENTS.

Dyeing of pelts, hairs, feathers, and the like. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 289,730, 30.11.27).—The pelts etc. are treated in successive baths first with a *m*-aminophenyl-naphthylamine salt and then with nitrous acid. Examples are *m*-aminophenyl- β -naphthylamine (yellow to pink), 2'-amino-*p*-tolyl- β -naphthylamine (yellow to pink), 7-(2-amino-*p*-tolyl)amino- β -naphthol (reddish-brown). The last-mentioned compound is obtained from 2:7-dihydroxy-naphthalene and *m*-tolylenediamine in presence of bisulphite. C. HOLLINS.

Dyeing of animal fibres, textile fabrics, or substances of a protein nature. S. W. WILKINSON (B.P. 289,578, 12.2.27. Cf. B.P. 242,027; B., 1926, 11).—

The affinity of wool, silk, etc. for dyes is increased by treatment with an oxidisable acid, *e.g.*, nitrous or hypochlorous acid, followed by ozone or ozonised air.

C. HOLLINS.

Dye-printing of fabrics with a free non-repeat design. R. S. BRACEWELL, Assr. to MARVELLUM Co. (U.S.P. 1,668,934, 8.5.28. Appl., 18.5.25).—A fluid mixture of oil and a finely-divided insoluble dye capable of giving a soluble leuco-compound is distributed on the surface of water or other non-solvent liquid to form a floating film of mutable design. The fabric is brought into contact with the colour film, and the colour is then reduced on the fibre to its leuco-compound, which penetrates the material and is finally re-oxidised.

C. HOLLINS.

Apparatus for the wet-treatment of textile fibres and yarns in hanks. O. SINDL (B.P. 284,984, 11.5.27. Ger., 7.2.27).

Dyeing and like machines. C. S. BEDFORD (B.P. 290,138, 31.12.27).

Bleaching of fur skins (U.S.P. 1,668,875).—See XV.

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

Contact process for sulphuric acid. W. H. DE BLOIS (Trans. Canad. Inst. Min. and Met., 1927, 30, 929—949).—A review of the development and principles of the contact process for the manufacture of sulphuric acid with a brief description of the application of the process to the gases produced in the bessemerising of nickel-copper matte at Sudbury. The gas from the converters contains about 14% SO₂, but is diluted with air before passing through the purification plant. The catalyst used is platinised asbestos, and, by the use of heat exchangers, the heat of the issuing gases from the catalytic chamber is sufficient to preheat the incoming gases nearly to the requisite temperature.

A. R. POWELL.

Contact process for the manufacture of sulphuric acid from zinc blende roaster gases. S. ROBSON (Trans. Canad. Inst. Min. and Met., 1927, 30, 950—981).—The roaster gases obtained from zinc ores containing more than 2% Pb carry a large quantity of lead sulphate fume as well as arsenic and other impurities. These are removed in modern plants by passing the gases through a series of baffled dust-settling chambers, thence through Glover towers and cooling pipes to an electrostatic separator, from which they are passed through coke filters and washing and drying towers to the contact plant. For efficient working of the electrostatic separators the temperature of the gases should not exceed 500° and the velocity must not be greater than 1 ft./sec. Numerous modern types of washing, drying, and purifying towers are illustrated diagrammatically, and brief notes of their method of working and efficiencies with various impurities are given. The conversion is effected in two vessels, each of which is provided with a heat-interchanger. The first converter operates at a higher temperature (*e.g.*, 530°) than the second, and converts the greater part of the sulphur dioxide into trioxide at a rapid rate; the second converter, serving to effect complete conversion,

is required only in districts in which sulphur dioxide discharged into the atmosphere will affect surrounding industries. Magnesium sulphate is the most suitable contact mass for high temperatures, and asbestos for the lower temperatures.

A. R. POWELL.

[Mechanical] developments in the potash industry. R. EHRHARDT (Chem. Fabr., 1928, 277—278).—The much larger outputs which are necessary to render potash manufacture profitable to-day have involved alterations in the methods of handling. Wagons of crude salt are now moved and tipped mechanically. The salt is broken in mills in which it falls upon beaters striking upwards. These produce grains of about 4 mm. diam. with little fines, and are built with throughputs of up to 250 tons per hr. With high outputs it is better to reduce to a certain size in beater mills and to finish the crushing with rollers. To economise power, the feed to these secondary mills is passed through a vibrating sieve, one design of which is described and illustrated.

C. IRWIN.

Purification of alcohol for the preparation of alcoholic potassium hydroxide. S. KICZALES (Ind. Eng. Chem., 1928, 20, 493).—2.5—3 g. of lead acetate dissolved in 5 c.c. of distilled water are thoroughly mixed with 1 litre of alcohol and to this mixture is added, without stirring, a solution of 5 g. of potassium hydroxide in 25 c.c. of warm alcohol. After 1 hr. the whole is well shaken, allowed to settle, and the decanted liquor filtered and distilled. This method is more efficient than the silver oxide method owing to the fact that the lead oxide-aldehyde additive product is less soluble than the corresponding silver oxide product.

D. J. NORMAN.

Cyclic processes involved in the manufacture of sodium nitrate from Chilean caliche. M. A. HAMID (J. Indian Chem. Soc., 1927, 4, 515—524).—The results of the investigation at 25° of the ternary system, water-sodium sulphate-sodium nitrate previously obtained (A., 1926, 245) are applied to the leaching of sodium nitrate from Chile saltpetre. Isothermal addition of water to an equimolecular mixture of sodium nitrate and sodium sulphate at first only results in the formation of darapskite ($\text{Na}_2\text{SO}_4 \cdot \text{NaNO}_3 \cdot \text{H}_2\text{O}$) until the whole solid phase consists of this compound. Further addition of water causes dissociation of this compound, the whole of the sodium nitrate so produced passing into solution while the proportion of sodium sulphate in the solid phase increases until the darapskite is completely dissociated. Throughout these changes the composition of the solution remains constant (c) and represents the most concentrated solution of sodium nitrate which can be obtained, still further addition of water causing the composition of the solution to change in the direction of diminishing sodium nitrate content. The same solution is the most concentrated, with respect to sodium nitrate, which can be obtained when water is added to a mixture in which the weight of sodium nitrate present is less than 60% of that of the sodium sulphate present (number of mols. of sodium sulphate is greater than the number of mols. of sodium nitrate). When the number of mols. of sodium nitrate in the dry mixture exceeds the number of mols. of sodium sulphate by x , it is possible to obtain a quantity of a solution (d) of constant composition

(containing sodium nitrate and darapskite) corresponding to x , whilst the solid phase consists of pure darapskite which, if separated from the solution (d), and again treated with water, yields the solution (c) of constant composition. The phase reactions involved in the Guggenheim leaching process (cf. B.P. 188,634 and 192,032; B., 1924, 94), which involves the system water-sodium sulphate-sodium nitrate-magnesium sulphate-magnesium nitrate, are discussed, and it is shown that by maintaining a sufficient quantity of magnesium salts in the leaching solution and in the raw material the whole of the sodium sulphate-nitrate compound can be decomposed, the sodium sulphate combining with the magnesium sulphate to form astrakhanite ($\text{Na}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 4\text{H}_2\text{O}$). Cooling the solution so obtained to a temperature not sufficiently low to cause the separation of the magnesium nitrate present will cause the precipitation of practically pure sodium nitrate.

J. W. BAKER.

Reduction of sodium sulphate to sodium sulphide, particularly by hydrogen and carbon monoxide in the presence of catalysts. P. P. BUDNIKOV and E. SHILOV (J.S.C.I., 1928, 47, 111—113 T).—Sodium sulphate was not reduced by carbon monoxide in the absence of catalysts, at least below 850°. In the presence of carbon reduction was rapid, and it is considered that the carbon acts catalytically and not as a direct reducing agent. Nickel chloride aided the reduction by carbon monoxide, but to a much less extent than carbon. In contradistinction to carbon monoxide, sodium sulphate was reduced by hydrogen in the absence of a catalyst, the reaction beginning at about 700° and increasing rapidly accompanied by secondary decomposition reactions at higher temperatures. The reaction is endothermic. Nickel and nickel compounds exercised a catalytic action on the reduction and also on the decomposition of the reduction products, the yield of sulphide at temperatures above 900° being less in the presence of the catalyst than without it. Loss of sodium sulphide occurred through interaction with sodium sulphate at 1000° or higher. (Cf. B., 1928, 446.)

C. A. KING.

Explosion and fire risks with potassium and ammonium persulphates, and the most suitable methods of packing and storage. G. AGDE and E. ALBERTI (Chem.-Ztg., 1928, 52, 229—232).—To study the extent of possible risk of explosion or firing of these salts, they were heated at a steady rate and the gas evolution was measured at regular intervals. It was found that neither the pure persulphates nor mixtures containing 10% of organic impurity would explode or cause rapid combustion of the organic matter, which was simply charred. The amount of gas evolved at any particular temperature was always greater in the presence of impurity, and the ammonium salt was less stable than that of potassium. Iron cases, with asbestos covering, are therefore considered unnecessary for packing, and wooden boxes of which the surface has been coated with paraffin are recommended.

R. H. GRIFFITH.

Chemistry and technology of zirconium oxide. H. TRAPP (Chem.-Ztg., 1928, 52, 365—367).—In the preparation of zirconia from zircon, the mineral is decomposed by fusion with an alkali and, after leaching and

washing, the residue consists of hydrated zirconia containing after ignition 90% ZrO_2 . The moist residue may be converted into the pure oxide by stirring it in paste form with ammonium hydrogen oxalate, removing the insoluble material, and crystallising out the ammonium zirconyloxalate. This compound forms large, dense crystals which yield pure zirconia on ignition in air. Oxide ores, *e.g.*, zirkite, are decomposed by fusion with acid sulphates or by heating with concentrated sulphuric acid, and from the solution of zirconium sulphate so obtained zirconium phosphate is precipitated by addition of sodium hydrogen phosphate. The precipitate is washed and dissolved in ammonium hydrogen oxalate solution, and the ammonium phosphate formed is separated by crystallisation from the ammonium zirconyloxalate, which is then recovered by further evaporation and purified by recrystallisation. An alternative treatment of the sulphate solution comprises evaporation and partial neutralisation to obtain a liquor containing 8–10% ZrO_2 which is treated with potassium sulphate and oxalic acid. The resulting potassium zirconyloxalate is separated from the mother-liquor, purified by recrystallisation from hot water, and decomposed with alkali carbonate or hydroxide. For the recovery of zirconia from baddeleyite ores on a large scale some modification of the basic sulphate process appears to be the only economical procedure. A. R. POWELL.

Crushing and grinding. I. Surface measurement of quartz particles. J. GROSS and S. R. ZIMMERLEY (*Amer. Inst. Min. Met. Tech. Pub.*, 1928, 46, 16 pp.).—Crushed quartz, free from iron, was agitated with 3.66*N*-hydrofluoric acid at constant temperature for 30 min. or longer, the residual particles then being ignited and weighed. The initial rate of dissolution was determined by the expression $(A + at)/(1 + kt)$, where A is the initial rate, t is the time in hrs., and a and k are constants. Three different specimens had the same rate of dissolution; a quartz crystal does not, however, dissolve equally rapidly in all directions. CHEMICAL ABSTRACTS.

Rates of absorption of sulphurous gases by alkaline solutions. P. RIOU and P. A. BÉRARD (*Compt. rend.*, 1928, 186, 1433–1436).—The rate of absorption of the vapours produced by the combustion of sulphur by milk of lime and by solutions of lime water of various concentrations, increases from the moment of separation of solid matter at a rate proportional to the concentration of the latter, and decreases gradually with rise in the temperature. J. GRANT.

Determination of sulphur dioxide in small amounts in the atmosphere. R. J. MCKAY and D. E. ACKERMAN (*Ind. Eng. Chem.*, 1928, 20, 538–542).—The method described is suitable for the determination of amounts of sulphur dioxide varying from 0.05 to 20 parts per million, and has been thoroughly checked on synthetic mixtures. The sample of air is drawn by an evacuation method into a large bottle containing a dilute starch-iodine mixture, which is then withdrawn and brought to the same intensity of blue as a blank by addition of a dilute standard solution of iodine. The apparatus, method, and the essential precautions are described in considerable detail. With alteration in

the size of sample and strength of solutions the method is applicable to the determination of sulphur dioxide in flue gases. If hydrogen sulphide is present in appreciable amounts, it must be determined separately and the necessary correction made. J. S. CARTER.

Catalytic refining of bromine. P. T. DANILITSCHENKO and M. RAVITSCH (*J. Russ. Phys. Chem. Soc.*, 1927, 59, 953–968).—Bromine is passed over heated activated charcoal and iron oxide, which act as catalysts and absorb organic impurities. By this method at a temperature of 115° bromine can be practically completely freed from chlorine. A. RATCLIFFE.

Analysis of bleach liquors etc. HINTZMANN.—See VI. **Chrome alum liquors.** SCHINDLER and KLANFER.—See XV. **Limestones for Bordeaux mixtures.** CARR and BEMILLER.—See XVI. **Silver halide emulsions.** SHEPPARD and CROUCH.—See XXI. **Effluents from potash works.** EMMERLING.—See XXIII.

PATENTS.

Separation of ammonia from gases and mixtures containing it.—F. UHDE (B.P. 272,929, 15.6.27. Ger., 15.6.26).—Liquid ammonia separated from the gaseous mixture by cooling is evaporated under low pressure in a heat-exchanger and led in counter-current to the cooled gas mixture to cool it further and obtain more ammonia. With a mixture containing too small an amount of ammonia to obtain the requisite low temperature, a portion of the evaporated ammonia is compressed and added to the mixture to be cooled. W. G. CAREY.

Ammonia condenser. S. O. LOKEY (U.S.P. 1,669,943, 15.5.28. Appl., 6.11.26).—The cooling medium is circulated through conduits in the still, and the product to be condensed is mixed with condensate and sprayed against the cooling surfaces. C. O. HARVEY.

Manufacture of ammonium chloride crystals. J. W. MOORE, W. G. POLACK, and CASTNER-KELLNER ALKALI CO., LTD. (B.P. 290,045, 18.5.27. Addn. to B.P. 273,093; B., 1927, 652).—In the interaction of synthetic ammonia and gaseous hydrochloric acid at 230–310°, gases saturated with moisture at ordinary temperature may be used, the heat of reaction preventing condensation of moisture. W. G. CAREY.

Production of aluminium oxide or products containing it from material containing aluminium sulphide. T. R. HAGLUND (B.P. 289,639, 3.5.27).—Slags containing aluminium sulphide are treated with air, sulphur dioxide, or other oxidising agent in such proportions as to liberate in the elementary state the greater part of the sulphur content at such a temperature that the liberated sulphur is distilled out of the mass. The addition of a small quantity of steam to the oxidising gas accelerates the reaction, which may be further hastened by introducing hydrogen chloride together with the steam. The treatment with sulphur dioxide may also be carried out in an aqueous suspension of the finely-divided slag either with or without heat and pressure; in this case the reaction product is aluminium hydroxide if a deficiency of sulphur dioxide is used, and aluminium sulphite if an excess is used. When carbon dioxide or carbon monoxide is employed as the oxidising

agent at 800–900°, the reaction products contain carbonyl sulphide and carbon disulphide.

A. R. POWELL.

Manufacture of titanium compounds. H. WADE. From TITAN Co. A./S. (B.P. 289,111, 17.11.26).—The precipitate of metatitanic acid or of a basic titanium salt obtained by hydrolysis of solutions of quadrivalent titanium at elevated temperatures is rendered soluble in dilute mineral acids and in concentrated solutions of organic acids by digesting it at 150° with a concentrated solution of an alkali hydroxide, whereby the titanium is converted into a fine crystalline powder of an alkali titanate. This powder dissolves readily in oxalic, lactic, and tartaric acids, and may be used for the manufacture of potassium titanoxalate etc.

A. R. POWELL.

Apparatus for production of gaseous ozonides. E. J. BAGNALL (A. M. BAGNALL, extrix.), Assr. to KNOX TERPEZONE Co., INC. (U.S.P. 1,668,884, 8.5.28. Appl., 22.11.22. Renewed 30.7.27).—The apparatus comprises an ozone generator, an evaporator, and a dryer in communication with one another, and means for supplying air to the dryer and for air-cooling the generator.

A. R. POWELL.

Manufacture of diammonium phosphate. R. GRIESSBACH, O. BALZ, and A. RÖSSLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,670,504, 22.5.28. Appl., 19.2.26. Ger., 20.2.25).—See B.P. 256,137; B., 1926, 821.

Manufacture of persalts. F. NOLL (U.S.P. 1,669,997, 15.5.28. Appl., 21.8.23. Ger., 28.8.22).—See B.P. 202,985; B., 1924, 980.

Manufacture of zinc oxide. J. A. SINGMASTER, F. G. BREYER, and E. H. BUNCE, Assrs. to NEW JERSEY ZINC Co. (U.S.P. 1,670,169, 15.5.28. Appl., 27.3.26).—See B.P. 268,301; B., 1927, 916.

Extraction of krypton and xenon from air. A. J. A. BLARINGHEIM, Assr. to SOC. ANON. D'ÉCLAIRAGE ET D'APPLICATIONS ELECTRIQUES (U.S.P. 1,670,014, 15.5.28. Appl., 26.5.24. Fr., 29.6.23).—See B.P. 218,266; B., 1925, 242.

Hydrogen from coke-oven gases (B.P. 271,491).—See II. **Recovery of soda ash** (B.P. 288,699).—See V. **Plastic lime** (U.S.P. 1,664,598).—See IX. **Activation of oxygen** (B.P. 265,636).—See XI. **Treatment of phosphates** (B.P. 264,867). **Fertilisers** (B.P. 290,075).—See XVI.

VIII.—GLASS; CERAMICS.

Causes and removal of certain heterogeneities in glass. L. W. TILTON, A. N. FINN, and A. Q. TOOL (U.S. Bur. Standards, Sci. Paper No. 572, 1928, 22, 719–736).—Refractive indices of six well-annealed samples of a barium flint optical glass from the same melt revealed individual deviations from the mean having a total spread from -37×10^{-6} to 26×10^{-6} . Thermal expansion and heat absorption measurements indicated a commencement of the critical temperature zone at about 545°, and a careful re-annealing was then given in which the samples were heated to 560° and cooled at the rate of 80° per day, with a break at 486°, during which

the temperature was held constant for 13 days. The range of the deviations was so reduced to 21×10^{-6} . A second re-annealing with greater precautions to reduce furnace gradients brought this range down to 5×10^{-6} .

A. COUSEN.

[Cause of opacity of] white enamel. R. D. COOKE (J. Amer. Ceram. Soc., 1928, 11, 126–130).—A white enamel is heterogeneous, being an intimate mixture of two or more transparent substances of different indices of refraction. Opaque frits contain crystals of fluorine compounds, such as sodium and calcium fluorides, having low indices of refraction compared with the silicate glass. Whiteness is defined as the ratio of the total light energy received on a surface to the total light energy reflected from it. In order to formulate a satisfactory test of whiteness, a standard enamel, with additions of 0, 4, 8, 12, and 16% of tin oxide, was milled. Sample plates were sprayed with each of these enamels in various thicknesses. It is shown that the reflexion factor increases with increase in the amount of tin oxide and with the thickness. By measuring the thickness and the reflexion factor, the intrinsic whiteness of the enamel can be determined.

A. T. GREEN.

Determination of iron in glass sand. G. E. F. LUNDELL and H. B. KNOWLES (J. Amer. Ceram. Soc., 1928, 11, 119–125).—Methods for determining small quantities of iron in sands are considered and their relative accuracies discussed. Of these, the electrometric titration method, the hydrogen sulphide reduction method, and the gravimetric method are described in detail. The colorimetric procedure gives low and the others high results. The electrometric titration method is both rapid and satisfactory provided platinum is excluded and the blank correction properly carried out.

A. T. GREEN.

Physical properties of artificial aluminous abrasives. R. C. PURDY and A. E. MACGEE (J. Amer. Ceram. Soc., 1928, 11, 192–203).—Test pieces of artificial corundum differently bonded were made by the normal puddled process and turned to a size satisfactory for testing. The firing, which varied from 70 hrs. at cone 12 to 120 hrs. at cone 10, was conducted in commercial kilns at various plants. On testing, it was found that porosity, specific gravity, elasticity, and thermal expansion decrease, whilst resistance to heat shock, impact strength, modulus of rupture, crushing strength, and mechanical endurance increase, with increasing percentages of bond and resulting hardness of grade. Above 300° the expansion of the bond is less than that of the artificial corundum, whilst below this temperature the reverse holds, the expansions of the two constituents tending towards equality. The properties of similarly constituted abrasives vary with variations in plant practice.

A. T. GREEN.

Measurements of the expansion of refractory bricks at 1600°. K. ENDELL and W. STEGER (Arch. Eisenhüttenw., 1927–8, 1, 721–724; Stahl u. Eisen, 1928, 48, 722).—After a critical discussion of the merits of the methods used for determining the expansion of refractories at high temperatures by previous workers, results obtained by a new method devised by the authors are recorded. Fireclay bricks with a medium or high

content of alumina expand only slightly but evenly over the range 20—1300° and then begin to contract. Fire-clay bricks with a high content of quartz exhibit the usual irregularities in expansion caused by the various transformations which take place on heating quartz. Magnesite bricks expand considerably but evenly up to 1500°, then begin to contract. Silica bricks of d 2.35 retain a constant volume between 600° and 1600°; those having d 2.40 are constant up to 1450°, and those having d 2.45—2.54 expand considerably up to 1250° and at 1600° their total linear expansion is 2.85—3.35% of the original length.

A. R. POWELL.

PATENTS.

Pressing of articles of fused silica. QUARTZ & SILICE (B.P. 283,174, 28.12.27. Fr., 6.1.27).—Blocks of silica are heated electrically on a furnace bed of "zirconium" or graphite at 2200° to cause volatilisation of silica and so prevent adherence to the bed; the mass is then rapidly pressed in moulds heated at 1000° and annealed in a closed chamber heated at 1200°.

W. G. CAREY.

Manufacture of refractory compositions. J. S. WITHERS. From VITREFRAX Co. (B.P. 289,560, 1.2.27).—Material containing about 72% Al_2O_3 and 28% SiO_2 , and resembling mullite, is prepared by fusing suitable quantities of a mixture of precipitated alumina and material such as cyanite, which is preferably concentrated to about 95% purity. The charge is fused in a three-phase electrical furnace under such conditions that the bath is kept in a fluid condition so that heavy impurities sink to the bottom and, like the crust at the top, can subsequently be stripped from the product. A more compact material suitable for lining furnaces containing glasses, enamels, etc. contains up to 3% K_2O and 0.25% P_2O_5 , but not more than 1% CaO and MgO .

L. A. COLES.

[Soda lime] glass. B. LONG, Assr. to SOC. ANON. DES MANUF. DES GLACES & PROD. CHIM. DE ST.-GOBAIN, CHAUNY, & CIREY (U.S.P. 1,669,908, 15.5.28. Appl., 12.1.26).—See B.P. 264,490; B., 1927, 602.

Annealing of glass articles. UNITED GLASS BOTTLE MANUFERS., LTD., E. A. C. PRYOR, and F. A. HURLBUT (B.P. 289,501, 29.10.26 and 26.8.27).

IX.—BUILDING MATERIALS.

Action of sulphates on the components of Portland cement. T. THORVALDSON, V. A. VIGFUSSON, and R. K. LARMOUR (Trans. Roy. Soc. Canada, 1927, [iii], 21, III, 295—310).—The effect of sulphate solutions on cement may be observed by following the expansion of a bar of cement mortar, and the decrease in tensile strength with time, when immersed in the sulphate solution. In this way the action of sulphates on tricalcium silicate, β -dicalcium silicate, and tricalcium aluminate, which, according to Shepherd, Rankin, and Wright (A., 1911, ii, 725; 1915, ii, 50), are the chief constituents of normal Portland cement, has been studied. Mortars made from pure tricalcium silicate and pure β -dicalcium silicate are not disintegrated in solutions of sodium sulphate, but expand and are disintegrated in solutions of magnesium sulphate. If tricalcium aluminate is added to either, the resulting

mortar disintegrates rapidly in solutions both of sodium and of magnesium sulphate. A mortar made from a composite cement containing 21.5% of tricalcium aluminate, 52.7% of tricalcium silicate, and 25.8% of β -dicalcium silicate mixed with Ottawa sand in the proportion of 1 : 7, has the usual tensile strength of a similar mortar made from normal Portland cement, and is affected by solutions of sodium, magnesium, and calcium sulphates in exactly the same way. This is strong evidence in favour of Shepherd, Rankin, and Wright's views (*loc. cit.*) as to the composition of normal Portland cement clinker. Steam treatment has been found to increase enormously the resistance of cement mortar to the disintegrating action of sulphate solutions.

M. S. BURR.

Deterioration of concrete by corrosive waters. R. GRÜN (Chem. Fabr., 1928, 281—283, 294—295).—The most important damage is caused by sulphuric acid either combined in sea-water or free in natural acid water. Ordinary concrete can be made resistant by producing a dense non-porous aggregate of uniformly sized material. At least 400 kg. of cement/m.³ must be used. Painting with bitumen is beneficial and better than covering with clinker and cement. Aluminous cement with 7—50% Al_2O_3 is very resistant to magnesium sulphate, but its behaviour in presence of sodium sulphate is doubtful. The resistance of pozzuolana to corrosion is very great, as is shown by the existence of water mains in good condition to-day constructed by the Romans of this material. The effect of its addition to concrete is to increase the density and to liberate lime, which acts protectively. It can only be used with slow-setting concrete. Cements prepared from blast-furnace slag fall into the same class; or a mixture of blast-furnace slag and Portland cement clinker may be used. Those made from acid slags have not a high initial strength, but if the clinker content is low they are very resistant to salt water. Aluminous slags have the best "hydraulic" qualities of any, but are not so resistant to corrosion as might be expected. Basic and magnesian slags also make good cement, but it is not resistant to salt water. Generally speaking, all these cements have less initial strength than Portland cement, but greater resistance to corrosion.

C. IRWIN.

Examination of cements and plastic masses, with especial reference to their behaviour in electrotechnique. I. Binding power. W. NAGEL and J. GRÜSS (Wiss. Veröff. Siemens-Konz., 1928, 6, [2], 150—173).—The binding power of a cement or plastic mass is defined as the weight in kg. required to pull a metal rod 8 mm. in diam. from a hole, 10 mm. deep and 10 mm. in diam., in which the rod is embedded in a porcelain plate by means of the cement under test. For zinc oxychloride cement with borax to retard setting, maximum binding power is obtained with $4ZnO : 1ZnCl_2$ when the zinc oxide used has been well burnt. For magnesium oxychloride cement with or without a filler the maximum binding power is obtained with $4MgO : 1MgCl_2$. The greatest strength is obtained with sand as a filler; calcite is almost as good, but kaolin is appreciably inferior. The quantity of alum used in mixing Keene's cement has little effect on its binding

power, but with 10% a slight maximum occurs in the curve. The binding power of artificial resins such as "Glyptal" or coumarone resin increases roughly in proportion to the amount of filler used; natural colophony behaves somewhat similarly.

A. R. POWELL.

Effect of decay on the chemical composition of wood. L. F. HAWLEY, L. C. FLECK, and C. A. RICHARDS (Ind. Eng. Chem., 1928, 20, 504—507).—Analyses of the sapwood of two hard woods and two soft woods before and after attack by a white-rot fungus (*Polystictus hirsutus*) and a brown-rot fungus (*Lenzites striata*) indicate that the pentosans in the cellulose are attacked more rapidly in proportion to the amount present than are the hexosans; also that the more readily hydrolysed constituents of the cellulose, as determined by the hydrolysis number (cf. B., 1927, 598), are, in general, more rapidly attacked than is the more stable cellulose, though the two processes appear to start simultaneously. In the early stages of decay the action of the white-rot fungus, *P. hirsutus*, resembles that of the brown-rot fungi in attacking preferentially the cellulose component of the lignocellulose complex.

D. J. NORMAN.

PATENTS.

Manufacture of cement. H. O. MOEBIUS, Assr. to AMER. MIAG CORP. (U.S.P. 1,680,269, 15.5.28. Appl., 2.8.27).—The pulverised raw material is burnt to clinkers, then partly cooled, crushed, and agitated in a current of cool air.

H. ROYAL-DAWSON.

Manufacture of coloured Portland cement. AMME-LUTHER WERKE BRAUNSCHWEIG DER "MIAG" MÜHLENBAU & IND. A.-G. (B.P. 284,295, 27.1.28. Ger., 27.1.27).—A metal pigment, e.g., an oxide of chromium, nickel, cobalt, or copper, and a salt capable of forming a coloured melt with it on calcination, e.g., a phosphate, borate, or halide, are added to raw materials used for making Portland cement. Reducing agents are also added if the raw material contains iron oxide, so that this is reduced and can be removed.

L. A. COLES.

Cementitious material. H. E. BROOKBY and C. K. ROOS, Assrs. to U.S. GYPSUM CORP. (U.S.P. 1,668,548, 8.5.28. Appl., 23.4.24).—The material consists of powdered raw calcium sulphate blended, without calcination, with sodium thiosulphate.

H. ROYAL-DAWSON.

[Production of cellular] concrete. J. A. THOMPSON (B.P. 272,479, 24.5.27. Austral., 12.6.26).—Concrete containing material for the generation of gases during setting so as to produce a cellular mass is allowed to set in hermetically sealed moulds in which the pressure is reduced to about $14\frac{1}{2}$ — $10\frac{1}{2}$ lb./in.², according to the depth or thickness of the mass.

L. A. COLES.

Manufacture of plastic hydrated lime. H. DITTLINGER (U.S.P. 1,664,598, 3.4.28. Appl., 25.6.26).—Hydrated lime that gives a "putty" of the requisite plasticity immediately on mixing with water, and does not therefore require seasoning, is made by shattering the dry hydrate by causing it to impinge upon a beater rotating at high speed within a casing. The hydrate may be prepared by using a dilute aqueous sodium thiosulphate solution for slaking, in order further to increase the plasticity of the product.

F. G. CLARKE.

Transformation of slag-forming waste materials (such as domestic or industrial refuse, gutter residues, etc.) into slag-sand of various grain-size. MUSAG GES. F. DEN BAU VON MÜLL- U. SCHLACKEN-VERWERTUNGSANLAGEN, A.-G., and A. GROTE (B.P. 280,902, 9.11.27. Ger., 20.11.26).—The waste materials are burned in the front portion of an obliquely mounted rotary furnace, and the resultant slag is superheated in the remaining portion of the furnace in such a way that the softened lumps of slag are broken up and granulated.

C. JEPSON.

Wood preservation. H. W. WALKER (U.S.P. 1,670,086, 15.5.28. Appl., 25.7.24. Renewed 12.4.27).—The wood is impregnated with the product of a water-soluble dye and an organic constituent of the wood which is toxic to wood-boring organisms and has a solubility in cold water slightly in excess of the minimum toxic concentration.

H. ROYAL-DAWSON.

Bituminous coating compositions. G. A. WELLINGS and E. JOHNSTONE (B.P. 289,737, 1.11.26).—A solution of bitumen containing gold size and a solution or suspension of sulphur with or without a filter.

L. A. COLES.

Manufacture of asbestos bodies. H. GERDIEN, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,670,659, 22.5.28. Appl., 7.11.25. Ger., 17.10.24).—See B.P. 241,576; B., 1927, 389.

[Press for] the manufacture of bricks. H. ACKERMANN, and SCHEIDHAUER & GIESSING A.-G. (B.P. 267,949, 17.3.27. Ger., 17.3.26).

Production of slabs for walls, partition walls, etc. A. P. LAURIE (B.P. 290,042, 12.5.27).

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

Rotating tube drying furnace. J. A. L. ORTLEPP (J. Chem. Met. Soc. S. Africa, 1928, 28, 210—213).—A rotary drying furnace particularly designed for drying tin concentrates out of contact with flue dust and excessive draughts consists of a steel tube 8 ft. long and 8 in. in diam. inclined at a slope of 1 in 12. The tube is heated externally, excess air being admitted to prevent burning of the metal. Material to be dried is fed from a top drying plate into a hopper to the tube, from which it is discharged through a suitable screen into a box, which also regulates the current of air and steam through the drying tube.

C. A. KING.

Smelting titaniferous ores of iron. A. STANSFIELD (Trans. Canad. Inst. Min. and Met., 1927, 30, 802—818).—Titaniferous iron ores with a high percentage of titania can be smelted in blast furnaces under suitable conditions to obtain a pig iron and a fusible slag containing about 20% of titanium oxide in the form of a lower oxide, probably Ti_2O_3 . Under too highly oxidising conditions no reduction of the titania takes place and pasty slags are formed, whereas under very highly reducing conditions titanium carbide and cyanonitride are obtained and the slags are fusible only at very high temperatures.

A. R. POWELL.

Phosphorus and arsenic in steel and the substitution theory. A. E. CAMERON (Trans. Canad. Inst. Min. and Met., 1927, 30, 858—868).—The solid solubility of phosphorus and of carbon in γ -iron is about 1.7%, and the effects of the two elements on the mechanical properties of iron are approximately the same. Carbon, however, increases the tensile strength owing to the insolubility of the carbide in α -iron and its separation on cooling together with the resistance to slip afforded by the finely-crystalline state of the eutectoid, whereas phosphorus can cause hardening only by producing distortion in the lattice of α -iron. This distortion of the body-centred lattice of α -iron produces the well-known cold-shortness of phosphoretic steels. Most steels contain arsenic up to about 0.1%, but as the solid solubility of arsenic in iron is 6.8% the hardening and, therefore, the embrittling effect of arsenic on steel is much less marked than that of phosphorus. An embrittling effect may, however, be caused by segregation of the arsenic due to the presence of carbon in solid solution producing areas in which the arsenic content is very much higher than the average. The metal in these areas would then have a lower m.p. than the remainder, and during heat-treatment may be in a semi-plastic state, thus causing serious weakness and possibly breakdown during forging.

A. R. POWELL.

Behaviour of basic open-hearth steel sheets during rolling. E. POHL (Stahl u. Eisen, 1928, 48, 649—656).—The mechanical properties of a basic open-hearth mild steel having a tensile strength at 20° of 49 kg./mm.² have been determined between 20° and 500°, and the effect on these properties of a tensile strain above and below the elastic limit has been ascertained. A strain below the elastic limit has no effect on the mechanical properties, but a strain above that limit induces an increase in hardness and tensile strength, but a decrease in impact resistance, especially at temperatures between 200° and 300°. It is recommended, therefore, that steel boilers should be constructed to work at temperatures above 350° so as to avoid the range of "blue brittleness" between 200° and 300°.

A. R. POWELL.

[Mechanical properties of] boiler-plate [steels].

A. POMP (Stahl u. Eisen, 1928, 48, 681—689).—The effects of temperature, ageing, and recrystallisation on the mechanical properties of 14 samples of boiler-plate steels have been determined and are recorded graphically. All the samples tested contained 0.05—0.2% Cu, 0.02—0.04% S, 0.02—0.04% P, 0.4—0.6% Mn, and 0—0.2% Si. The carbon content in the plain steels was 0.05, 0.15, or 0.25%, and in the nickel steels the effect of 3% and 5% of nickel was determined. The elastic limit, limit of proportionality, and permanent strength decreased in all cases with rise of temperature, the actual values depending on the carbon, manganese, and nickel content. The tensile strength increased to a maximum between 200° and 400°, the elongation and reduction of area decreased to a minimum at 100—200° and then rose sharply, and the impact resistance rose abruptly to a maximum at 50—150° and then fell rapidly. Prolonged ageing of the worked plates at 200° resulted in a slight increase in the tensile strength and elastic limit with a corresponding decrease in the elongation; the

impact strength of the mild steel plates was considerably decreased after this treatment, and that of the steel plates containing 0.25% C fell appreciably, whereas the nickel steel plates showed only a slight decrease in strength. Annealing just below the A3 point caused a small decrease in the tensile strength and a large decrease in the impact strength of the plain carbon steels, but a slight increase in both these values of the nickel steels. The superiority of the latter for boiler plates is thus established.

A. R. POWELL.

Impact resistance of steels at low temperatures.

J. F. MORRISON and A. E. CAMERON (Trans. Canad. Inst. Min. and Met., 1927, 30, 839—857).—The impact resistance of hypoeutectoidal steels is decreased by exposure to low temperatures. At -30° the impact resistance of a normalised steel containing 0.1% C is about one tenth that of the same steel at 20°, whilst a normalised steel with 0.34% C has an impact strength at 20° three times as great as at -30° . The same steels in the quenched and fully tempered condition have an impact strength at -30° which is only slightly lower than that at 20°. For structural purposes in cities where a low winter temperature is experienced, the best resistance to shock is obtained by quenching from 900° followed by tempering at 610°.

A. R. POWELL.

Determination of oxides in steel. F. WILLEMS (Arch. Eisenhüttenw., 1927—8, 1, 605—608; Stahl u. Eisen, 1928, 48, 623—624).—Manganous oxide may be satisfactorily determined in steel by shaking fine turnings or filings of the metal with a solution of iodine in absolute alcohol. The operation is carried out in a closed flask from which the air has been completely displaced by nitrogen, and, when dissolution is complete, the residue is collected on a "Cella" filter, washed with absolute alcohol, and dissolved in hydrochloric acid. Manganese is determined in this solution by any of the ordinary methods. Silica and alumina inclusions in steel may also be satisfactorily determined by this process, but the copper ammonium chloride and the bromine methods of Oberhoffer and Ammann (B., 1927, 967) give equally satisfactory results for these constituents, and are more easily carried out.

A. R. POWELL.

Precious metals in the Sudbury [nickel] ores and their recovery.

C. LANGER, SEN., and C. JOHNSON (Trans. Canad. Inst. Min. and Met., 1927, 30, 903—908).—The residues from the carbonyl volatilisation of nickel in the Mond process of treating copper-nickel sulphide ores contain practically the whole of the precious metals contained in the original ore and represent a concentration of these metals of 16,000:1. A typical analysis of these residues is 1.85% Pt, 1.91% Pd, 0.56% Au, 0.39% Ir, Ru, and Rh, and 15.42% Ag, together with antimony, lead, nickel, copper, arsenic, sulphur, selenium, and tellurium. The residues are roasted to expel volatile constituents, and smelted with litharge and alkaline fluxes to obtain a lead regulus. This is cupelled with more silver to give at least six times as much silver as other precious metals in the resulting bullion, which is then electrolysed in a silver nitrate bath. Pure silver is thus obtained at the cathode and a rich slime at the anode, from which the

platinum metals and gold may be recovered by the usual procedure.

A. R. POWELL.

Recrystallisation of α -brass. A. BASS and R. GLOCKER (*Z. Metallk.*, 1928, **20**, 179—183).—The temperature at which 70:30 brass commences to recrystallise is dependent on the nature of the deformation to which the metal has been subjected, but the size of the new crystals and their orientation are not affected by the nature of the previous work. The size of the new crystals is greater the higher the annealing temperature and the smaller the degree of deformation, and their orientation, as determined by X-ray examination, is similar to that of annealed silver after severe deformation. The presence of lead up to 1.2% has no influence on the tensile strength, elongation, or grain size after annealing, but a very small iron content suffices to restrain the grain growth very considerably below 700°. Above this temperature iron causes a rapid increase in grain size with further rise in the annealing temperature. With 63:37 brass the presence of β -grains may considerably affect the shape of the recrystallisation diagram.

A. R. POWELL.

Industrial control of cold-hardening and annealing [of brasses] by light-load ball tests. P. NICOLAU (*Rev. Mét.*, 1928, **25**, 155—168).—A discussion of tests suitable for the industrial control of low-temperature annealing of brass cartridge case collars after deformation, in order to prevent season cracking. The mercury or mercuric salt test and microscopical examination are impracticable, but a Brinell hardness test with a 1.58 mm. ball under a load of 10 kg. applied for 15 sec. gives consistent results, and the method is suitable for unskilled labour. A suitable apparatus is described, and results for 67/33 and 72/28 brasses are discussed in detail. In each case the strain is relieved by annealing at temperatures between 300° and 350°, but between 350° and 400° the hardness curves show a point of inflexion not previously noticed. Severe working lowers the temperature at which strain is removed by low-temperature annealing, but increases the final hardness of the annealed metal.

W. HUME-ROTHERY.

Structure of brass tubes. Surface faults in bent tubes and their prevention. F. OSTERMANN (*Z. Metallk.*, 1928, **20**, 186—188).—Some characteristic crystal structures of the surface of extruded and of cold-drawn and annealed brass tubes are illustrated by photomicrographs. The copper content of brass for extruding tubes should not be below 61.3%, and the extrusion temperature must not exceed 600° if satisfactory bends are to be obtained. A higher temperature leads to the formation of coarse needle-shaped crystals which cause a rough, hackly surface, or even fracture, to be produced during bending.

A. R. POWELL.

Metallurgy of ancient bronzes. D. BUTESCU (*Bul. Soc. Romane Stiin.*, 1928, **30**, 83—98).—The metal of a number of bronze implements found at Drajna-de-Jos, Rumania, and probably dating from 1100—1400 B.C., has been analysed. In addition to copper and tin, small amounts of iron, manganese, and lead were found. There was some evidence that rapid quenching had been employed to secure hardness.

R. CUTHILL.

Aluminium alloys in motor construction. H. STEUDEL (*Z. Metallk.*, 1928, **20**, 165—178).—A review of the preparation, properties, and uses of various age-hardening aluminium alloys showing how advantage is taken of these properties in the construction of cylinders, piston rods, and other parts of motors, especially for aviation purposes.

A. R. POWELL.

Extraction of vanadium and its salts from residues containing vanadium. N. N. EFREMOV and A. ROSENBERG (*J. Russ. Phys. Chem. Soc.*, 1927, **59**, 689—699).—The powdered dry waste residue from the working of vanadium ores is treated with boiling nitric acid and heated on the water-bath for 2 hrs. The precipitate thus formed consists mainly of vanadic oxide, and is dissolved in boiling sodium hydroxide and converted into sodium metavanadate. The contaminations present remain undissolved and are removed by filtration. The filtrate contains sodium vanadate, sodium aluminate, and traces of sodium silicate. The aluminate is removed by acidifying with nitric acid and subsequent boiling with a strong solution of sodium carbonate. The precipitate formed is collected, the filtrate made faintly acid with nitric acid, boiled to drive off carbon dioxide, and precipitated with an excess of ammonia solution. The filtrate, containing pure sodium metavanadate, is boiled and ammonium metavanadate precipitated with an excess of a hot saturated ammonium chloride solution. Uranium and further quantities of ammonium vanadate are recovered from the first filtrate. Vanadic acid precipitated on asbestos fibre is determined by treating the asbestos with a solution of sodium hydroxide, hot water, and hydrogen peroxide, filtering, acidifying the filtrate with sulphuric acid, reducing vanadic acid to a vanadyl salt with sulphur dioxide, boiling in a stream of carbon dioxide to remove excess of sulphur dioxide, and titrating with permanganate.

A. RATCLIFFE.

Orientation of the crystallites of rolled and drawn metals. Crystallographic significance of the mechanism of slip during drawing, pressing, and rolling of metals. G. TAMMANN and A. HEINZEL (*Arch. Eisenhüttenw.*, 1927—8, **1**, 663—667; *Stahl u. Eisen*, 1928, **48**, 693—694).—In metals with a face-centred cubic lattice (*e.g.*, silver, copper, gold, and aluminium) slip can take place along four octahedral planes, whereas in metals with a body-centred cubic lattice it may take place along twelve eicositetrahedral planes in a direction parallel to the line bisecting the obtuse angle of the eicositetrahedral face. In drawing, slip takes place first along that plane which contains the largest component of the tractive force; friction then causes this plane to rotate, and slip ensues along the second plane and so on until eventually all the slip planes are oriented in the direction of the pull. It follows that, under the action of tractive forces, the maximum thrust occurs along the slip planes of hard-drawn metals when a threefold axis of symmetry in the case of face-centred metals, and a twofold axis in the case of body-centred metals, falls in the direction of drawing; the converse is true for compressive forces. The effect of rolling differs from that of drawing or hammering in that after a certain degree of reduction a complete re-orientation of the crystallites takes place;

in the first stage of rolling the change in the orientation is similar to that caused by drawing, and in the second stage it is similar to that caused by hammering. For hard-rolled metals, therefore, the orientation of the crystallites in the plane of rolling is a combination of the effects produced by drawing and hammering.

A. R. POWELL.

Electrolytic copper. W. KÖSTER (Z. Metallk., 1928, 20, 189—191).—When hard-rolled electrolytic copper is annealed the hardness falls more or less proportionately with the rise in temperature up to 500°, which temperature is necessary for complete annealing even with only 3% deformation. If electrolytic copper is subjected to a heat treatment which causes recrystallisation to take place previous to rolling, the reduction of hardness during subsequent annealing takes place within a range of 25—50° at temperatures varying from about 100°, after 93% reduction in thickness, to about 350° for a 3% reduction. This difference in behaviour is ascribed to the elimination of occluded hydrogen during recrystallisation, the copper used in the tests having given up 0.00026% when heated *in vacuo* at 500°. Fire-refined copper does not alter in sp. gr. after cold rolling and annealing, but the value of *d* for electrolytic copper decreases from 8.92 after annealing above 600° to 8.6 after annealing at 1000°. Prolonged annealing at 780° causes a gradual reduction in *d* to 8.42 after 70 hrs. These changes in volume are due to the liberation of hydrogen along the grain boundaries with the consequent formation of fine pores which grow in size with the temperature and time of annealing.

A. R. POWELL.

Potentiometric analysis of binary, ternary, and quaternary tin alloys. H. BRINTZINGER and F. RODIS (Z. Elektrochem., 1928, 34, 246—250).—The reduction of the solutions of tin-antimony alloys by chromous chloride solution has been studied potentiometrically, as far as the production of metallic antimony. Simultaneous separation and determination of the two metals may be effected by converting the sodium thioantimonate and thiostannate, present in the solution after removal of copper and lead, into antimony pentachloride and stannic chloride, respectively, by addition of hydrogen peroxide and hydrochloric acid. The simultaneous separation and determination of copper and tin requires the presence of considerable calcium chloride and of hydrochloric acid in the solution; dissolved oxygen is removed by passing a current of carbon dioxide through the hot solution, the titration being conducted at 90—100°. The first break in the titration curve indicates the reduction from the cupric to the cuprous state, and is easily determined; the second break, corresponding to the reduction from stannic to stannous tin, can only be obtained accurately by allowing a 3 min. interval between the addition of chromous chloride and the potential reading. The two breaks in the bismuth-tin titration curves are due to the reduction first from stannic to stannous and then of the trivalent bismuth ion to metal, whilst those of the iron-tin curve are caused by reduction first of iron and then of tin. Iron, tin, and bismuth, and copper, tin, and bismuth in ternary mixtures are reduced in the order named. The same holds for antimony, copper, and tin mixtures, but owing to the close proximity of

the first two breaks, a fourth break, corresponding to the reduction of trivalent antimony to metal, may be employed. The presence of lead in antimony-copper-tin alloys does not influence the titration, and the amount present may be determined by difference.

H. F. GILLBE.

Application of electrical resistance measurements to the study of atmospheric corrosion of metals. J. C. HUDSON (Proc. Physical Soc., 1928, 40, 107—131).—A description is given of experimental procedure for quantitative field tests on the atmospheric corrosion of metals, based on the determination of the change in resistance of wire specimens produced on exposure. The resistance changes produced in duplicate specimens agree to within 5%. The results obtained are in agreement with those given by determinations of change in weight. For copper the percentage change in resistance was inversely proportional to the wire diameter, which makes it possible to obtain results with thin wires in a few weeks.

C. J. SMITHELLS.

Sulphuric acid. DE BLOIS; also ROBSON.—See VII.

PATENTS.

Rabble furnace. A. N. JETTE (U.S.P. 1,670,038, 15.5.27. Appl., 8.9.26).—The bricks, composing an insulating mantle surrounding a central shaft and carried by supporting rings thereon, are kept in position by vertically-disposed reinforcing rods passing through the mantle and engaging the rings. J. S. G. THOMAS.

Froth-flotation concentration of ores. C. P. LEWIS, ASSR. to MINERALS SEPARATION N. AMER. CORP. (U.S.P. 1,668,917, 8.5.28. Appl., 28.7.25).—The pulp of the oxidised ore is agitated with a xanthate and a soluble silicate under conditions to produce a mineral-bearing froth, which is then separated. H. ROYAL-DAWSON.

Refining of iron. I. G. FARBENIND. A.-G. (B.P. 268,770, 26.3.27. Ger., 1.4.26).—Carbon, sulphur, and phosphorus may be almost completely removed by melting steel scrap with iron oxide obtained by the oxidation of iron carbonyl as described in B.P. 258,313 (B., 1926, 979). A. R. POWELL.

Production of non-ferrous coated billets of steel or the like. J. M. ROTH (B.P. 290,112, 7.11.27).—Steel billets are faced, coated with a flux, and electrically heated while centred in a mould, into which the coating metal is poured. The composite billet may then be rolled, drawn, or otherwise reduced to rods or wire. C. A. KING.

Proofing of iron and steel against rust. W. H. COLE (B.P. 289,906, 1.1.27).—A corrosion-resisting deposit is formed by treating iron or steel with a solution containing phosphates of aluminium, zinc, iron, and chromium, to which solution may be added "flux" salts, *e.g.*, diammonium phosphate, sodium diborate, or potassium dichromate. C. A. KING.

Alloy steel. F. H. ROGERS. FROM REMY-STÄHLWERKE STAHLSCHEMIDT & Co., G.M.B.H. (B.P. 289,990, 28.2.27).—A high-speed tool steel contains 17% W, 7% Ta, 5% Cr, 1.5% V, and 0.9% C, and, if desired, 2% Mo. F. G. CROSSE.

Treatment of copper minerals. L. F. CLARK, ASST. to UNION CARBIDE SALES CO. (U.S.P. 1,670,021, 15.5.28. Appl., 29.10.23).—Ores containing copper minerals are treated, prior to flotation, with acetylene and other oxidisable substances the oxidation of which results in the formation of insoluble substances. The particles of copper mineral are thus coated with a mixture of cuprous acetylide and the insoluble oxidation products which assist the subsequent flotation process.

A. R. POWELL.

Recovery of lead and zinc from zinc waste. E. BURY and F. W. WALKER (B.P. 290,035, 3.5.27).—A mixture of 10 pts. of zinc waste, 2 pts. of coke dust containing 2% S, 3 pts. of lime, and 2 pts. of coal is heated in a cement kiln by means of a flame of pulverised fuel burnt in an excess of oxygen, whereby the zinc is volatilised as oxide and the lead as sulphate. The residue in the kiln is suitable after grinding for the manufacture of hydraulic cement.

A. R. POWELL.

Metal [tungsten and molybdenum] alloys. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. of W. P. SYKES (B.P. 279,490, 21.10.27. U.S., 22.10.26).—Age-hardening alloys of iron with tungsten or molybdenum or both are prepared by heating together intimate mixtures of the constituent metals in a powdered form, such as is produced by the reduction of the oxides in hydrogen. The mixture is compressed into bars which are sintered in hydrogen above 1440° until diffusion is complete; the bars are then quenched and aged for several days at a temperature somewhat above 600°. The solid solubility of tungsten in iron at 1525° is about 33% and at 600° about 8%; that of molybdenum in iron is 24% at 1440° and 7% at 600°. The hardness of the quenched alloys is about 200–250 and that of the aged alloys, after annealing under the correct conditions, varies from 500 to 650 according to the composition.

A. R. POWELL.

Treatment of ores containing platinum. S. C. SMITH (B.P. 289,220, 10.3., 26.4., and 21.5.27).—Nickel-copper sulphide concentrates derived from South African norite deposits are roasted "sweet" and the product is heated with 70% sulphuric acid at 212° to dissolve substantially the whole of the copper and iron and the greater part of the nickel. The residue from this treatment is thoroughly washed and heated with 25–30% hydrochloric acid and a chlorate or manganese dioxide, whereby the platinum, palladium, gold, and part of the iridium and rhodium are dissolved and may be recovered by precipitation with zinc. Platinum is extracted from dunite ores by heating them at 500° and leaching with hydrochloric acid and an oxidising agent other than nitric acid.

A. R. POWELL.

Powder for welding castings, particularly of grey cast iron. F. POLITZ (U.S.P. 1,669,840, 15.5.28. Appl., 4.9.26. Ger., 21.10.25).—A small percentage of iron suboxide is added to the welding powder, as a flux.

F. G. CLARKE.

Soldering of metals or metal alloys. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 289,553, 31.1.27).—Tungsten or molybdenum may be soldered to other metals or alloys by means of a solder consisting of an alloy of copper with 2–3% Si or Al.

Borax or glass may with advantage be used as flux in the process.

A. R. POWELL.

Plant for extracting gold from sea-water. B. ČERNÍK and B. STOCES (B.P. 289,638, 3.5.27).—A screen or lattice supported at some distance above the bottom of a natural or artificial reservoir for sea-water is covered with material capable of combining with or retaining gold, *e.g.*, pyrites or active charcoal, and channels are provided below the lattice to regulate the passage of the water through the material. A modified form for use in the open sea consists of an inverted U-tube containing a pump in one limb and the material in the other.

L. A. COLES.

Radioactive material. A. FISCHER (B.P. 278,347, 21.9.27. Austr., 29.9.26).—Oxides or other compounds of radioactive elements are mixed with an organic reducing agent, *e.g.*, tartaric acid or sugar, and an organic binder, and the mixture is applied to thin sheets or wires of a metal or alloy such as platinum or irido-platinum. The whole is then heated to such a temperature that the organic material is burnt off, and the radioactive coating is then incorporated in the surface of the carrying metal by mechanical treatment, *e.g.*, rolling, drawing, or hammering. Alternatively, the metal base may be used as a cathode in a fused electrolytic bath containing a compound of the radioactive element. For coating iron, steel, or silver sheet or wire the radioactive material may be incorporated with one of the usual mixtures used in producing a coating of fused enamel on the metal. The radioactivity of the resulting products is such that not only the γ -rays, but also the α - and β -rays, are fully utilised, and the products are capable of sterilisation by any of the usual methods.

A. R. POWELL.

Purification of metals. KAISER-WILHELM-INST. F. EISENFORSCHUNG (B.P. 265,213, 27.1.27. Ger., 27.1.26).—The metal to be purified is melted in a high-frequency induction furnace under a slag adapted to remove the impurities. Thus, nickel is refined under a slag containing lime, fluorspar, and nickel oxide, which removes sulphur, carbon, manganese, silicon, and aluminium, and is then deoxidised under a lime-fluorspar slag by the addition of the requisite quantity of silicon or manganese. Iron and iron alloys may be refined under a layer of hammer-scale and suitable fluxes. The rapid circulation of the metal caused by the high-frequency currents ensures a rapid and complete removal of impurities.

A. R. POWELL.

Production of aluminium by fusion electrolysis. I. G. FARBENIND. A.-G. (B.P. 283,949, 19.12.27. Ger., 21.1.27).—Alumina for addition to the electrolytic bath is rendered denser by grinding it under pressure as described in B.P. 272,109 (B., 1927, 591).

A. R. POWELL.

Case hardening [of iron]. A. LENNARTZ (U.S.P. 1,671,034, 22.5.28. Appl., 25.1.27. Ger., 15.2.26).—See G.P. 444,126; B., 1928, 57.

Production of [multi-coloured] gold strip or sheet. A. WHITEHOUSE (B.P. 289,660, 7.6.27).

Coating of tiles, sheets, etc. of iron or ferrous metals. A. REY (B.P. 270,298, 26.4.27. Fr., 27.4.26).

XI.—ELECTROTECHNICS.

Maintaining or restoring the electric capacity of the negative plates of lead accumulators. L. GAGNAUX (Sealed Note 1910, 30.6.09. Bull. Soc. Ind. Mulhouse, 1928, 94, 192—194). Report by E. BANDERET (*Ibid.*, 194—195).—The original capacity of the plates is maintained or may be restored (after reduction due to changes in the spongy condition of the lead forming the plate) by the addition to the electrolyte of the products obtained by the action of concentrated sulphuric acid on organic substances such as oils, fats, aromatic hydrocarbons, phenols, and resins; about 2—4 g. of the sulphonated product is required per litre of electrolyte. Alternatively, the organic substances may be incorporated in the negative plate before use so that they may be sulphonated during charging. Banderet reports favourably on the process. A. J. HALL.

Cements and plastic masses. NAGEL and GRÜSS.—See IX. **Corrosion of metals.** HUDSON.—See X. **Electrolytic apparatus for analysis of insecticides etc.** HOLLAND and GILLIGAN.—See XVI. **Electrocolorimeter.** STOCKHAUSEN and WINDISCH.—See XVIII.

PATENTS.

[Electric] induction furnace. J. M. WEED, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,669,109, 8.5.28. Appl., 19.4.23. Renewed 18.8.27).—A primary winding and a secondary formed of a liquid charge contained in a looped cavity in a refractory receptacle are structurally related so that the spacing from the primary of sections of the secondary in planes passing through the axis of the primary increases progressively from one end to the other. A passage is provided external to the secondary through which a charge may circulate between portions of the secondary spaced at different distances from the primary. J. S. G. THOMAS.

Production of electrical heating elements. E. L. and A. P. WIEGAND, Assrs. to E. L. WIEGAND CO. (U.S.P. 1,669,385, 8.5.28. Appl., 4.6.27).—A resistor is supported within, by, and between opposed parts of a sheath containing refractory electrical insulating material, and the sheath is reshaped so as to cause the insulating material to be distributed about the resistor after these opposed parts have been withdrawn therefrom. J. S. G. THOMAS.

Magnetic materials. WESTERN ELECTRIC CO., INC., Asses. of J. W. ANDREWS, R. GILLIS, A. F. BANDUR, C. P. BEATH, H. M. E. HEINECKE, and J. C. KARCHER (U.S.P. 1,669,642—9 and 1,669,665, 15.5.28. Appl., [A, B, E, F] 17.4.26, [C] 24.4.26, [D] 3.1.27. Renewed 16.2.28, [G] 3.1.27, [H] 10.4.26, and [I] 17.8.26).—(A—G) A series of compositions containing various granular magnetic materials (*e.g.*, nickel-iron alloys) which are moulded and bound together by coatings of different insulating materials applied in successive layers (*e.g.*, silicic acid and water-glass or ferric oxide, chromic acid and an alkali, a nitrate, kaolin and a flux, etc.). (H—I) A heat-treatment for rendering the above alloys brittle etc. is claimed. J. S. G. THOMAS.

Magnetic core. G. W. ELMEN, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,669,658, 15.5.28. Appl.,

11.9.26).—A compressed mass of insulated particles of magnetic material having an initial permeability greater than that of iron is heat-treated to improve its magnetic properties. J. S. G. THOMAS.

Storage-battery plate. J. H. CALBECK and J. A. SCHAEFFER, Assrs. to EAGLE-PICHER LEAD CO. (U.S.P. 1,668,801, 8.5.28. Appl., 26.7.24).—The formed paste of a storage-battery plate of high capacity, long life, and high porosity contains substantial amounts of fumed basic lead sulphate and of its derivatives formed in the development of the plate. J. S. G. THOMAS.

Dry cell. E. H. McCABE, Assr. to FRENCH BATTERY CO. (U.S.P. 1,670,040, 15.5.27. Appl., 22.12.24).—A metal film is applied to the end part of a carbon electrode by means of an air blast directed upon the molten point of a metal wire, so that particles of metal freed from the wire are embedded in the pores of the carbon. J. S. G. THOMAS.

Manufacture of carbons for electrotechnical uses. M. OSWALD (B.P. 289,680, 27.7.27).—Rough rupture surfaces bounded by smooth surfaces on carbon rods are provided with an adherent metallic or non-metallic covering. J. S. G. THOMAS.

Battery electrolyte compound. Manufacture of storage batteries. L. R. STEELE (U.S.P. 1,668,740—1, 8.5.28. Appl., [A] 2.3.22, [B] 1.5.23).—(A) A suspension in water of a powdered compound formed from a soluble silicate and ammonium sulphate is used in place of sulphuric acid in storage batteries of the Planté type. (B) Sulphuric acid electrolyte is removed from a Planté-type cell after discharge to a voltage of about 1.7 and the electrolyte described under (A) is substituted. J. S. G. THOMAS.

Manufacture of electrodes for arc welding. BRIT. THOMSON-HOUSTON CO., LTD., Asses. of E. W. SCHWARTZ and F. R. KAIMER (B.P. 283,582, 4.1.28. U.S., 15.1.27).—Steel welding wire is boiled for 5—8 hrs. in a bath consisting of, *e.g.*, water (100 pts.), sodium silicate (5 pts.), red oxide of iron (30 pts.), and lime (40 pts.) by wt. J. S. G. THOMAS.

Manufacture of an electron-emitting cathode. E. E. SCHUMACHER, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,668,734, 8.5.28. Appl., 12.7.23).—Current is passed through a metallic filament supported in an agitated evacuated vessel containing powdered thermionically-active material. J. S. G. THOMAS.

Manufacture of luminous electric discharge tubes. J. B. J. M. ABADIE (B.P. 276,018, 13.8.27. Fr., 14.8.26).—A discharge tube of glass containing salts of uranium or other fluorescent salts is filled with mercury and neon or helium. J. S. G. THOMAS.

Devices for generating ultra-violet rays and activating oxygen etc. H. GEFFCKEN and H. RICHTER (B.P. 265,636, 8.2.27. Ger., 8.2.26).—Ultra-violet radiation is produced by a glow discharge between mercury electrodes in a discharge vessel of material, *e.g.*, quartz, transparent to the radiation. J. S. G. THOMAS.

Absorption of non-precious gases in manufacturing electric discharge tubes. E. SCHRACK and R. ETTENREICH (RADIOWERK E. SCHRACK) (B.P. 272,843, 18.11.26. Austr., 17.6.26).—Non-precious gases, *e.g.*,

oxygen, nitrogen, are removed from the tubes by heating therein to about 150° an alloy of rare-earth metals and a relatively large amount of heavy metals, e.g., an alloy containing 50% Ce, 25% Fe, 20% La, and 5% Di. J. S. G. THOMAS.

[Electric] induction furnace having unidirectional circulation. J. R. WYATT, Assr. to AJAX METAL CO. (Re-issue 16,967, 15.5.28, of U.S.P. 1,312,069, 5.8.19).—See B., 1919, 687 A.

Electrode mass for self-baking electrodes. C. W. SÖDERBERG, Assr. to NORSKE A./S. FOR ELEKTROKEMISK IND. (U.S.P. 1,670,052, 15.5.28. Appl., 22.7.24. Norw., 25.7.23).—See Swiss P. 111,653; B., 1926, 284.

Manufacture of battery plates. W. J. PLEWS, Assr. to PLEWS & HUTCHINSON CORP. (U.S.P. 1,670,046—7, 15.8.28. Appl., [A] 27.6.21, [B] 29.12.27).—See B.P. 207,606; B., 1924, 101.

Aluminium (B.P. 283,949). Radioactive material (B.P. 278,347).—See X. **Electrodeposition of rubber (B.P. 289,965).**—See XIV.

XII.—FATS; OILS; WAXES.

Determination of the degumming powers of soaps [for boiling-off silk]. R. TSUNOKAE (J. Soc. Dyers and Col., 1928, 44, 142—144).—The degumming action of a soap on raw silk being proportional to its degree of hydrolysis in aqueous solution, the degrees of hydrolysis shown by a number of commercial soaps as determined by the salting-out, titration, toluene, and electro-conductivity methods were compared and correlated with their degumming powers. The salting-out method proved unsatisfactory, but the other methods yielded comparable results, except that the toluene method, in which the free fatty acid liberated by hydrolysis is extracted with toluene and afterwards weighed, yielded somewhat low results. The toluene method yields unreliable values for hydrolysis when applied to soaps containing oxidised fatty acids. The degumming powers and degrees of hydrolysis of sodium stearate, palmitate, and oleate decrease in the order named.

A. J. HALL.

Oils of the chaulmoogra group. Gorli fat. E. ANDRÉ and D. JOUATTE (Bull. Soc. chim., 1928, [iv], 43, 347—360).—The fat obtained in 39—53% yield from seeds of the *Oncoba echinata* (gorli) plant after 18—20 recrystallisations from acetone and light petroleum affords a solid glyceride, m.p. 51°, $[\alpha]_D +49.33^\circ$, saponif. value 194°, iodine value 72.5, together with a mixture of liquid glycerides, $d_{20}^{16} 0.9482$, $n_D^{16} 1.4828$, $[\alpha] +46.4^\circ$, saponif. value 194, iodine value, 108. Comparison of the properties of the solid glyceride with those of synthetic *trichaulmoogrin*, m.p. 45°, $n_D^{20} 1.4764$, $d_4^{20} 0.9437$, $[\alpha] +54.83^\circ$, saponif. value 190.6, iodine value 87.3, indicates that the solid glyceride is probably a molecular compound of *trichaulmoogrin* and *palmitodichaulmoogrin*, palmitic acid being present to the extent of about 15%. The crude fat contains 10—12% of palmitic acid, about 80% of chaulmoogric acid, and about 10—12% of a liquid unsaturated acid which appears to contain two ethylenic linkings, and for which the name *gorlic acid* is proposed. The new acid, which

has not been obtained perfectly pure, has $d_{20}^{18} 0.9364$, $n_D^{18} 1.4783$, $[\alpha] +50.3^\circ$, iodine value 169.6 (Cheery 181.4) (cf. Dean and Wrenshall, B., 1921, 89 A; 1925, 928). The *amide*, m.p. 95°, *chloride*, *diethylamide*, *lithium salt*, and *gorlicohydroxamic acid* have been prepared. *Gorlic acid* contains the pentamethylene ring characteristic of the acids of the chaulmoogric group, and it is probable that one ethylenic linking is present in the ring system and one in the side chain. *Trichaulmoogrin* could not be isolated by extraction of gorli fat, and its m.p. constitutes an exception to the rule that a triglyceride melts at a higher temperature than the constituent fatty acid. R. BRIGHTMAN.

Rate of dissolution of essential principles during percolation. II. Extraction of raffinose from cotton-seed cake. M. BRIDEL and (Mlle.) M. DESMAREST (J. Pharm. Chim., 1928, [viii], 7, 433—447, and Bull. Soc. Chim. biol., 1928, 10, 510—521; cf. B., 1928, 388).—Raffinose (300 g.) is best obtained from cotton-seed cake (10 kg.) by threefold percolation with 60% alcohol (3000 c.c. each time) at the ordinary temperature followed by removal of the alcohol, dilution with water, and defecation with lead salts. The extraction removes 92.93% of the total raffinose (7.2%) in the first 5 hrs., and is complete in 10 hrs. Inversion of the sugars in the final liquors shows that other optically active substances are present (cf. Hudson and Harding, A., 1914, i, 1166). G. A. C. GOUGH.

Flour from linseed from which the oil has been expressed. E. ANDRÉ (J. Pharm. Chim., 1928, [viii], 7, 481—486).—Possible methods of utilising to the best advantage linseed grain are discussed, special reference being made to the use in pharmacy of the flour obtained after expressing the oil and separating the husk. W. O. KERMACK.

Auer's conception of the drying of oils. A. EIBNER (Farben-Ztg., 1928, 33, 2165—2169).—The "gas-coagulation" theory is criticised mainly on the grounds that in its evolution Auer has ignored the work of earlier investigators on the individual characteristics of various oils, the hydrophilic nature of linseed oil films, etc. Some of the author's experiments in this field are briefly recapitulated. S. S. WOOLF.

Standardisation of methods of testing oil. Measuring flasks for Engler's viscosimeter. H. SCHLÜTER (Chem.-Ztg., 1928, 52, 367).—For the standardisation of the Engler viscosimeter with water the collecting flask is usually graduated to deliver a certain volume so that several tests can be done successively. This graduation is useless when testing oils, as the amount which remains in the flask on draining varies with the oil. Hence it is recommended that these flasks should be graduated for collecting and for delivering a standard volume. A. R. POWELL.

Stearic acid standards. ANON. (Soap, 1927, 3, 37).

Ethylene dichloride as a solvent. BAUER and LAUTH.—See III.

PATENTS.

Degreasing of bones and other materials containing moisture. BRIT. GLUES & CHEMICALS, LTD.,

and R. B. DREW (B.P. 289,569, 3.2.27).—By means of the apparatus described it is possible to degrease bones with hot vapour of a volatile solvent, at the same time vaporising the water content of the bone. By an arrangement of several extracting vessels the mixed solvent and water vapours are used for further extractions until its solvent properties are exhausted by the increasing proportion of water vapour, after which the solution of fat is separated from the water and the solvent evaporated for further use. E. HOLMES.

Treatment of fats and oils. H. T. BÖHME A.-G. (B.P. 281,232, 7.10.27. Ger., 25.11.26).—Sulphuric acid which is extensively used in preparing water-soluble fatty sulphonates or alkali sulphonates may be replaced by phosphoric acid in preparing compounds of value in the textile, leather, and pharmaceutical industries. Thus, the oil or fat is treated with phosphoric anhydride, washed to remove the excess phosphoric acid, and then preferably converted into the alkali salt. The reaction may be assisted by the addition of acetic anhydride or acetyl chloride, or by the employment of acetylphosphoric acid. It is claimed that the process gives a product of much better colour than the sulphuric acid process. E. HOLMES.

Manufacture of soap. A. E. HATFIELD, E. A. ALLIOTT, and ACHILLE SERRE, LTD. (B.P. 289,582, 15.2.27).—Soaps of especial value in dry-cleaning work may be prepared by wholly or partially saponifying a fatty acid of setting point below 2°, preferably below -0.5°, with aqueous ammonia or solutions of potassium or sodium carbonates or hydroxides, an emulsifying agent being added at some stage of the manufacture. Various emulsifying agents, as di- and tri-chloroethylene and pentachloroethane, may be used, whilst if cyclohexanol be employed the setting point of the original acid may be as high as 10°. Practically pure oleic acid is suggested as a suitable starting material. E. HOLMES.

Production of preparations capable of being emulsified and forming soaps. G. PETROFF (B.P. 289,934, 1.2.27).—Sulpho-aromatic fatty acids of high mol. wt. are obtained by treating mixtures of liquid aromatic hydrocarbons of low mol. wt. and saturated fatty acids or stearine with sulphuric acid containing free sulphur trioxide. The products emulsify in the presence of dilute mineral acids, and give light-coloured soaps. [Stat. ref.] B. FULLMAN.

Production of cleansing and degreasing agents and emulsifying agents. R. SAJITZ and E. POTT (CHEM. FABR. POTT & Co.) (B.P. 266,746, 25.2.27. Ger., 25.2.26).—Aromatic alcohols which are difficultly soluble or insoluble in water, and in particular benzyl alcohol, are treated alone or in combination with hydrocarbons (*e.g.*, solvent naphtha) with alkali salts of fatty acids, sulpho-fatty acids, naphthenic acids, sulphonates of aromatic or of hydroaromatic hydrocarbons, or corresponding acids. Benzyl alcohol does not affect the frothing capacity of such agents, and has the advantage of possessing only a slight odour. E. HOLMES.

Generation of foam from foam-developing substances. R. SCHNABEL, ASSR. to MINIMAX A.-G. (U.S.P. 1,669,213, 8.5.28. Appl., 23.4.27. Ger.,

30.9.25).—A saponin solution is supersaturated under pressure with a gas which is readily absorbed and which, on removal of the pressure, is liberated again with the development of foam from the liquid. A. R. POWELL.

Melting [blocks of] fatty substances. SOC. ANON. ANC. ÉTABL. G. PELLERIN (B.P. 269,873, 24.3.27. Fr., 22.4.26).

Manufacture of soap flakes. MASCHINENFABR. ELITE A.-G. (B.P. 268,320, 9.3.27. Ger., 29.3.26).

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

The red lead question. L. SCHERTEL (Farben-Ztg., 1928, 33, 2101—2104).—Mainly polemical with Rahder (*cf.* B., 1928, 99). Accelerated tests indicate that "disperse" red lead is in no way inferior to the "classic" red lead as a protective pigment. S. S. WOOLF.

Production of flat oil varnishes. F. WILBORN (Farben-Ztg., 1928, 33, 2169—2170).—Various substances are added to oil varnishes to produce a "matt" finish, *e.g.*, waxes, aluminium hydroxide, magnesium carbonate, kaolin, aluminium stearate, palmitate, resinates, zinc stearate, etc. The properties and suitability of the various types are discussed. S. S. WOOLF.

"Cellosolve" [ethylene glycol monoethyl ether] and its derivatives in nitrocellulose lacquers. E. W. REID and H. E. HOFMANN (Ind. Eng. Chem., 1928, 20, 497—504).—The properties of "cellosolve," "cellosolve acetate," and ethylene glycol monobutyl ether are described, and the advantages these compounds possess over other nitrocellulose solvents in the manufacture of nitrocellulose lacquers are discussed. Resins and combinations of resins suitable for use with these solvents are indicated, also the factors to be considered in the selection of solvent mixtures and thinners. Typical working formulæ are given for nitrocellulose lacquers for special purposes. D. J. NORMAN.

Plastic masses in electrotechnics. NAGEL and GRÜSS.—See IX.

PATENTS.

Manufacture of non-thickening paints and lacquers. W. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 289,968, 7.2.27. Addn. to B.P. 289,153; B., 1928, 456).—The process of the prior patent is extended to paints and lacquers having a pigment base of zinc compounds other than lithopone. L. A. COLES.

Manufacture of lacquers, impregnating materials, etc. W. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 289,523, 26.1.27. Addn. to B.P. 286,789; B., 1928, 376).—Cellulose esters and ethers, especially in the manufacture of films etc., are dissolved in compounds of the acetal type of polyhydric alcohols in the absence of water. B. FULLMAN.

Manufacture of lithopone. W. H. LANDERS (U.S.P. 1,669,476, 15.5.28. Appl., 21.2.25).—Undried lithopone is fed into the higher end of a slightly inclined rotating tubular furnace in which it passes successively through drying, preheating, and calcining zones. Combustible gas is introduced into the furnace at the lower end and is burnt in the presence of insufficient air for complete

combustion, whilst more air is introduced between the calcining zone and the higher end of the furnace.

L. A. COLES.

Manufacture of crude lithopone. J. H. CALBECK, Assr. to EAGLE-PICHER LEAD Co. (U.S.P. 1,669,857, 15.5.28. Appl., 21.1.26).—Aqueous solutions of zinc sulphate and barium sulphide are charged continuously in equivalent proportions into a mixing tank, and the overflow passes into a settling tank. L. A. COLES.

Preparation of phthalic anhydride-glycerol resins. J. H. SCHMIDT, Assr. to BAKALITE CORP. (U.S.P. 1,663,183, 20.3.28. Appl., 28.4.24).—The colourless product obtained by the action of phthalic anhydride on a polyhydric alcohol below 175° is heated, at temperatures below the formation of inherent colour, until it is hard, tough, and infusible. B. FULLMAN.

Manufacture of a phenol[formaldehyde] resin. C. J. ROMIEUX, Assr. to AMER. CYANAMID Co. (U.S.P. 1,669,674, 15.5.28. Appl., 29.4.25).—A reactive resin is prepared by reaction between a phenol of b.p. above 202° and formaldehyde, in the presence of alkali (less than 0.1% based on the phenol present).

B. FULLMAN.

Production of artificial resin. C. KULAS (U.S.P. 1,669,831, 15.5.28. Appl., 16.10.25).—Phenol-aldehyde resins, after separation from the reaction mixture by cooling and settling, are centrifuged in the cold condition. The layers of partially dehydrated resins which subsequently separate are subjected to rotation while supported upon a heated surface. The resin layers finally separating are centrifuged cold.

F. G. CLARKE.

Production of solid, finely-divided material [litharge]. L. SCHERTEL and W. LÜTY, Assrs. to T. GOLDSCHMIDT A.-G. (U.S.P. 1,670,049, 15.5.28. Appl., 14.11.22. Ger., 15.11.21).—See B.P. 189,132; B., 1924, 343.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Effect of heat on raw rubber. C. R. PARK, C. M. CARSON, and L. B. SEBRELL (Ind. Eng. Chem., 1928, 20, 478—483).—Heating with water in an autoclave for 24 hrs. at 158° plastifies rubber to approximately the same extent as passage five times between tight rolls. The proportion of the acetone-soluble matter becomes increased, as also do its acid number and its nitrogen content; these changes are attributable to hydrolysis of protein and fatty constituents and to depolymerisation of rubber itself. The rate of vulcanisation of the rubber is increased for unaccelerated mixings and for mixings containing mercaptobenzthiazole, but is not appreciably altered for hexamine, diphenylguanidine, and ethylideneaniline; the stress-strain curves of the vulcanised products, relative to similar products from unheated rubber, are more convex to the strain axis. The heat-treatment has no adverse effect on the ageing qualities and sometimes improves them. It is probable that amines, nitrogen bases, and alkaloids are the substances mainly responsible for the characteristics of heated rubber. D. F. TWISS.

“Normal ageing” of compounded rubber. R. H. MCKEE and H. A. DEFEW (Ind. Eng. Chem., 1928, 20,

484—491).—Nine vulcanised mixtures of different composition have been examined after ageing under various conditions. Storage in a wet atmosphere gives a softer stress-strain curve than storage in a dried atmosphere; drying after a long exposure to a moist atmosphere causes an increase in tensile strength. Over-vulcanised rubber deteriorates in tensile strength and also oxidises more rapidly in a dry than in a wet atmosphere; under-vulcanised rubber decreases in tensile strength less rapidly in dry air than in moist, but the rate of oxidation is approximately the same in both. Paraffin wax exerts a protective action against sunlight with samples under strain; zinc oxide also is a protective agent against sun-cracking, doubtless because of its opacity. Storage of mixed rubber for 1½ or 3 years causes marked reduction in rate of vulcanisation and slight decrease in tensile strength. D. F. TWISS.

Influence of fatty acids on vulcanisation [of rubber]. G. S. WHITBY and B. A. EVANS (J.S.C.I., 1928, 47, 122—126 r).—Accelerators requiring zinc oxide for activation need this in a soluble form; the presence of organic acids is not immaterial in the presence of a plentiful supply of zinc oxide (cf. Martin and Davey, B., 1925, 602). Mercaptobenzthiazole produced no vulcanisation at 126° in the absence of the resin acids even with desaturated rubber and zinc oxide in equal proportions, whereas with the addition of oleic acid (1½ pts. : 100 pts. of rubber) good vulcanisation was obtained in 30 or 40 min. Stearic acid also exercised as favourable an effect on the activity of anhydroacetaldehyde-aniline in the presence of 20 pts. as of 5 pts. of zinc oxide. In the case of accelerators, such as hexamethylenetetramine and diphenylguanidine, which are capable of inducing good vulcanisation in the absence of zinc oxide, the presence of organic acids is relatively unimportant. Of the three fatty acids naturally present in rubber, the effectiveness in assisting vulcanisation decreases in the order: stearic, oleic, linoleic; the lower effectiveness of the unsaturated acids may be attributable to their combination with sulphur and consequent reduction of its active mass. D. F. TWISS.

Rubber vulcanisation accelerators in ultra-violet light. R. DIMAR and W. DIETSCH (Chem.-Ztg., 1928, 52, 388—389).—Some organic accelerators give characteristic fluorescence when exposed to the light from a quartz-mercury lamp after passage through a “uviolet” filter; fluorescence is also exhibited by raw or vulcanised mixings containing certain accelerators. As an analytical method, however, for purposes of identification, the method would be of value only in conjunction with other tests. D. F. TWISS.

Colouring of cold-cured rubber. W. E. SANDERSON (J. Soc. Dyers and Col., 1928, 44, 137—140).—The use of organic accelerators (e.g., tetraethylthiourea disulphide, zinc diethyldithiocarbamate, and zinc isopropylxanthate) in the vulcanisation of rubber allows brighter shades to be obtained from dyes previously used for colouring rubber, and has extended the range of dyes suitable for this purpose. Cold-cured rubber goods may be coloured by immersion in aqueous solutions of basic dyes at 88° and drying by drumming in warm air with the addition of sawdust for removal of the loose

colour, or by dipping in a solution of oil-soluble dyes in benzene or solvent naphtha containing a small amount of stearic acid, and then evaporating the solvent. Many basic, direct, sulphur, and vat dyes are readily absorbed by rubber latex from their aqueous solutions on the addition of acetic acid, it being desirable to add tannic acid before the acetic acid when basic dyes are used; the colour of the resulting coagulated latex resists subsequent cold-cure processes, the acid dyes being generally most resistant. Dyes or colour lakes used for rubber goods vulcanised by the cold-cure process should be fast to acids, since free acid is formed during this process by the action of moisture on the sulphur monochloride used. Lake pigments are generally suitable for "spread work" such as rubber-coated fabrics, and comprise acid dyes precipitated by barium chloride; basic dyes precipitated by tannic acid, Katanol (a thiophenol), Tamol (a condensation product of naphthalenesulphonic acid and formaldehyde), or phosphotungstic or phosphomolybdic acid (these lakes have superior fastness to light); the insoluble azoic pigments; alizarin dyes precipitated on alumina; and insoluble earth colours. Aluminium bronzes are used in producing metallic effects.

A. J. HALL.

Thermal and calorimetric magnitudes of caoutchouc and of similar substances. M. LE BLANC and M. KRÖGER (Z. Elektrochem., 1928, 34, 241—244).—The specific heats of raw and vulcanised rubber in both the normal and stretched conditions have been determined at temperatures from about -170° to 40° . The significance of the irregular temperature-specific heat curves is discussed.

H. F. GILLBE.

PATENTS.

Preparation of latex products for rubber manufacture. J. E. NYROP (B.P. 289,583, 15.2.27; cf. B.P. 259,922; B., 1928, 41).—Using the spraying apparatus described earlier, latex is completely or partly dried, anti-coagulating agents such as potash soap and also, if desired, softening agents, *e.g.*, oils, and other compounding ingredients, being simultaneously sprayed without previous mixing with the latex. The products are capable of being redispersed or softened with water and are suited to further treatment in rubber manufacture.

D. F. TWISS.

Electrodeposition of rubber and the like from latex. W. A. WILLIAMS (B.P. 289,965, 7.2.27).—Into alkaline latex containing an added electrolyte such as ammonium chloride are introduced two suitable electrodes which are conductively connected outside the liquid; the resulting galvanic action causes electro-negative rubber globules to deposit on the anode; *e.g.*, commercial preserved latex, approximately 0.5*N* with respect to ammonia, is mixed with twice its volume of a saturated solution of ammonium chloride, and the electrodes consist, respectively, of a zinc plate and a carbon element surrounded with manganese dioxide. Compounding ingredients may be added to the latex so as to be deposited with the rubber.

D. F. TWISS.

Dispersion of rubber into a colloidal substance. H. L. TRUMBULL and J. B. DICKSON, Assrs. to B. F. GOODRICH Co. (U.S.P. 1,668,879, 8.5.28. Appl., 23.4.26).

—Rubber, containing a tacky softener, is milled into a viscous paste of a substance such as glue, casein, or gluten, so as to produce a dispersion of this in rubber, the dispersion being subsequently inverted so as to bring the paste into the continuous phase.

D. F. TWISS.

Vulcanisation of rubber to leather. L. B. CONANT (B.P. 275,194, 5.7.27. Can., 29.7.26).—Leather after being preheated in air to approximately the vulcanisation temperature is assembled, while still hot, with the rubber composition, which is then vulcanised under pressure by heat.

D. F. TWISS.

Rubber articles. L. C. PETERSON (B.P. 283,122, 3.1.28. U.S., 3.1.27).—Rubber sheets, with one side vulcanised and the other side raw, suitable, *e.g.*, for tyre patches, are prepared by superposing a layer of a mixture of rubber including a vulcanising agent and an accelerator on a layer of rubber mixture free from vulcanising ingredients and containing an acidic substance such as sodium hydrogen sulphate. When the composite sheet is heated to vulcanise the backing the sodium hydrogen sulphate prevents the raw facing from becoming vulcanised by any sulphur which may migrate from the backing.

D. F. TWISS.

Manufacture of reinforced abrasive articles [containing rubber]. W. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,668,475—6, 1.5.28. Appl., [A] 18.11.22, [B] 16.9.25. Renewed 11.2.28).—(A) A mixture of comminuted old rubber with abrasive materials, sulphur, and latex is dried, moulded, and vulcanised. (B) An abrasive article is formed of granular abrasive material held together by a cementing layer which also binds irregularly disposed reinforcing fibres; the cementing layer also comprises a continuous bond of rubber.

D. F. TWISS.

Production of rubber compositions. H. L. FISHER, Assr. to B. F. GOODRICH Co. (U.S.P. 1,668,235—7, 1.5.28. Appl., [A] 14.1.24. Renewed 14.3.28, [B] 17.1.24, [C] 14.4.24. Renewed 14.3.28).—(A) Raw rubber is treated with *p*-toluenesulphonic acid. (B) Concentrated sulphuric acid (2—25 pts.) mixed with an inert carrier is milled into rubber (100 pts.) and the mixture is heated to reaction temperature. (C) A mixture of rubber (100 pts.) with a sulphonic acid or chloride (8 pts.), *e.g.*, *p*-toluenesulphonic acid, and with sulphuric acid (2 pts.) is heated so as to cause a pronounced exothermic reaction.

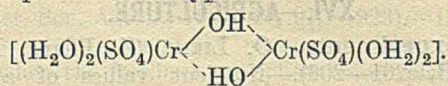
D. F. TWISS.

Rubber composition. F. C. DYCHE-TEAGUE (U.S.P. 1,670,599, 22.5.28. Appl., 19.2.26. U.K., 10.12.25).—See B.P. 268,853; B., 1927, 452.

XV.—LEATHER; GLUE.

Chrome tanning. VI. Properties and behaviour of various pretreated chromium sulphate solutions. E. STIASNY and O. GRIMM (Collegium, 1928, 49—71; cf. B., 1927, 393).—The p_H value of a 1% solution of hexa-aquochromium sulphate, 0% basicity, which had not been heated was 2.81 and gradually diminished to 1.98 on being kept for 4 months. The "verolung," which increases the p_H value, is outweighed by the effect of the migration of the sulphate residues

in the chromium complex. The addition of hydrochloric acid to the above solution restrained the sulphate residue migration, and the acidity of the liquors was less than the calculated value. When the chromium sulphate solution was boiled for 5 min. the p_H value fell to 1.21, but increased to 1.81 after being kept for 4 months. The diminution in p_H value was much greater than for chloride solutions and pointed to much greater "verolung" with sulphate solutions, but the increase in p_H value on keeping was also greater with chromium sulphate than with chromium chloride solutions. The "verolte" basic sulphate returns to its original state more easily than the chloride. There was evidence on adding hydrochloric acid to the boiled chromium sulphate solution that the (SO_4) ions exercise a neutral salt effect and slightly diminish the p_H value. Boiling the chromium sulphate solution for longer periods did not cause much further change; thus, the p_H value after 60 hrs. was 1.20. In this it differs from chromium chloride liquors. On adding hydrochloric acid to the boiled solution and keeping it, the p_H value increased due to the reversal of the "verolung" process. Dilution caused a diminution in the acidity of cold prepared solutions and a slight diminution in that of both boiled solutions which can be explained by "verolung." The acidity of a 33% basic chromium sulphate solution which had not been heated increased on being kept. This is explained by "verolung" of the monohydroxosulphate which disturbs the hydrolysis equilibrium and so forms more acid. The addition of hydrochloric acid to the basic chromium sulphate solution caused a diminution in the acidity which diminished still more on ageing, this again being due to reversal of the "verolung" process. Boiling the solution caused an increase in the acidity due to greatly increased hydrolysis, progressive "verolung," and diminished buffering action by the (SO_4) ion, but the acidity gradually fell to that obtained on ageing the cold solution. These and other researches show that "verolung" plays the greatest rôle in chromium sulphate solutions. There is no evidence of the formation of oxygen bridges, as with chromium chloride solutions, owing to the greater tendency of the (SO_4) residues to form complexes of the type.



The migration of the acid radical is only found in 0% basic chromium chloride solutions, but it is different with chromium sulphate solutions. The buffering action of the chlorine ion is not observed with basic salts, but the (SO_4) ion always exerts a buffering effect due to the formation of (SO_4H') ions. Frequently this effect exceeds all the other factors affecting acidity. Dialysis tests, hide powder tests, and determinations of the precipitation value throw little light on the properties of the liquors.

D. WOODROFFE.

Chrome tanning. VII. Hydrolysis and tanning action of sulphato-chromium sulphates. E. STIASNY and D. BALÁNYI (Collegium, 1928, 72—97).—It is shown that sulphato-chromium sulphates are less hydrolysed, and therefore less acid, than hexa-aquo-

chromium sulphates. "Verolte" basic sulphato-chromium sulphates are obtained on boiling hexa-aquo-chromium sulphate solutions. These solutions are more acid than those of the hexa-aquochromium sulphates owing to the increased acidity consequent on the "verolung." (SO_4) radicals always enter into the chromium complex. This migration is always hastened by adding alkali, *i.e.*, making the liquor basic, thus showing that "verolung" and masking of the (SO_4) groups are parallel processes. The amount of both masked and ionised (SO_4) groups combined with chromium diminishes with increasing degree of basicity. The relative amount of sulphate groups in the complex remains fairly constant meanwhile. If a solution of chromium sulphate is rendered basic with sodium carbonate instead of with sodium hydroxide, no dihydroxo-chromium sulphate is precipitated, and a much greater basicity is attained before precipitation commences. On adding sodium carbonate, carbonate radicals enter the chromium complex as well as hydroxo- and "ol" groups. These carbonate groups stimulate "verolung," and in consequence of the increased "verolung" there is much more migration of (SO_4) groups into the chromium complex. The increased number of such groups represses the carbonate groups, and finally, in a chrome liquor rendered highly basic with sodium carbonate, there are very few carbonate groups in the complex. When a solution of hexa-aquo-chromium sulphate was boiled, "verolung" took place and a portion of the sulphate radicals combined as ions migrated into the complex. This process was very rapid at b.p. owing to the disturbance of the hydrolysis equilibrium by "verolung." On cooling, there is a reversal of the "verolung" process due to the acid formed, and some of sulphate radicals in the complex become sulphate ions again. The acid present reacts with the latter forming (HSO_4) ions, and thus the acidity diminishes. This latter process is only subsidiary in feebly basic solutions, but if the boiled solution is immediately cooled rapidly and rendered basic, the free acid will be neutralised and the reversal of the "verolung" on keeping is hindered. This effect is obtained with liquors of 16.6% basicity, which are prepared by adding the necessary amount of sodium hydroxide which corresponds to the amount of acid liberated. Consequently, the p_H value of the solution increases only slightly on ageing. The small increase is due to the acid formed by the hydrolysis of the basic sulphato-sulphate, which acid has a slight tendency to reverse the "verolung." The proportion of sulphate radicals in the complex to those present as ions is not affected to the same extent by rendering the liquor basic. Large amounts of sulphate radicals entered the chromium complex as a result of boiling, but they gradually returned to the ionic form on being kept. Since the temperature coefficient of the formation of sulphate complexes is large, many sulphate groups gradually return to the ionic form, though a number of masked sulphate groups remain even after seven weeks. In a basic liquor the free acid formed by hydrolysis is neutralised, and there is therefore less reversal of the "verolung" process. The presence of hydroxo-groups in basic salts does not diminish the masked (SO_4) groups

([SO₄] in the chromium complex), and these are the same in both the basic and normal liquors. Tanning tests with different solutions of chromium sulphate show that the chromium complexes in the tanned hide powder contain more sulphate groups than those in the chrome liquor. This is attributable to the stronger tanning power of the chromium complexes containing more sulphate groups, and which are at the same time more strongly "verolten." Both types of hydroxy-groups, "verolten" and not "verolten," are necessary to tannage.

D. WOODROFFE.

Determination of sulphato-groups in chrome-tanned leather. H. B. MERRILL, J. G. NIEDERCORN, and R. QUARCK (J. Amer. Leather Chem. Assoc., 1928, 23, 187—208).—The curve showing the rate of removal of sulphuric acid from chrome-tanned leather by pyridine solution indicates that some acid combined with the chromium is also removed, hence Gustavson's method (B., 1927, 261) of determining the acidity of chrome leather is wrong. Experiments with chrome leather and water at different p_H values respectively have shown that p_H 5.4 or slightly lower is the maximum acidity at which protein-bound acid can be removed completely, and the following method, based on this, is recommended. A sample (2 g.) of the chrome leather is weighed out into a shake bottle, covered with 100 c.c. of distilled water, shaken for 1 hr., and allowed to remain overnight. Next day, 2 drops of 0.5% methyl-red solution are added and the contents are titrated with 0.02N-sodium hydroxide until the colour changes from clear red to salmon, *i.e.*, p_H 5.2—5.4. The mixture is then shaken for 15 min., restored to the proper shade for p_H 5.3 by adding alkali, and shaken for the rest of the day. The shade is examined every 15 min. for the first 2 hrs. and every 30 min. after that. After being kept overnight, it is neutralised to p_H 5.3 four times more at intervals of 1 hr. The entire neutralisation should occupy less than 48 hrs. The solution is poured off, and the leather transferred to a Wilson-Kern extractor, washed with running distilled water for 1 hr., again transferred to a beaker, and the sulphate remaining in the sample determined by the phosphate displacement method, thus obtaining the chromium-bound sulphate.

D. WOODROFFE.

Glue testing. Report of the Commission of the German Association for testing technical materials. O. GERNGROSS (Collegium, 1928, 130—143).—Ten samples of commercial glues were tested. The moisture was determined by heating 3—5 g. of the finely-divided material at 100—105° for 18 hrs., or to constant weight under atmospheric pressure. Phosphorus pentoxide should be used in all desiccators. The results showed that the best glue contained 17% of water. The toluene distillation method decomposes the glue. The p_H value of a 1% solution was determined colorimetrically, but did not give a constant potential in the electro-metric method. Good agreement was obtained in the viscosity determinations on a 17.75% solution at 40°, using the Engler viscosimeter. The viscosity was also determined by the Ostwald apparatus on 3% and 10% solutions. The report favours the Vogel-Ossag apparatus for gelatin and glue solutions. The jelly strength as determined by the American apparatus

showed fairly large individual variations, and is rejected as being too crude. The Greiner apparatus (A., 1927, 19) was used on 10% solutions which had been left 16 hrs. at 18—20°, and gave better concordance. The adhesive properties of the different glues, tested on pieces of wood, depended on the glue concentration, and variations were attributed not only to molecular and micellar phenomena, but also to the diffusibility of glue into the wood. A good, highly viscous, quick-setting glue will diffuse less into the wood than poor thin glue. The poorer glue will compare more favourably in concentrated solutions. Glues should be diluted to 30—40% strength and their adhesive properties tested by the Rudeloff process (cf. B., 1918, 743 A). The Bechhold-Neumann test (B., 1924, 759) is criticised. Some of the glues tested presented anomalies of bad viscosity and joint strength, with very good jelly strength. One such glue was derived from chrome-tanned leather shavings. Results with the different glues showed that the Greiner jelly-strength test (*loc. cit.*) showed better agreement with the joint-strength tests than did the viscosity determinations.

D. WOODROFFE.

Theory of leather dyeing. SALT.—See VI. **Gelatin for ice cream.** DAHLBERG and others.—See XIX.

PATENTS.

Preparation of fish skins for making "sha-green."

H. V. ANDREW, and G. BETJEMANN & SONS, LTD. (B.P. 289,213, 7.3.27).—Skins of ray fish and other fish are cleaned, dyed, or stained, brought to an even thickness, and a stencil pattern is applied to the back of the skin, which is then sprayed with pigment or colour. The front of the skin is finished or polished as usual.

D. WOODROFFE.

Treatment [bleaching] of fur skins and the like.

H. STEIN, W. E. AUSTIN, and I. LIEBOWITZ, Assrs. to STEIN FUR DYEING Co., INC. (U.S.P. 1,668,875, 8.5.28. Appl., 14.10.25).—The skins are treated in a solution containing ammonia, a soap soluble in water, and more than 3% of hydrogen peroxide.

A. R. POWELL.

Treatment of fats and oils (B.P. 281,232).—See XII. **Rubber on leather** (B.P. 275,194).—See XIV.

XVI.—AGRICULTURE.

Soil experiments. O. LIEHR (Z. Pflanz. Düng., 1928, 7B, 201—206).—Nutrient values of a large number of soils were determined by the Neubauer seedling method and by that of Lemmermann and König. No general relationship existed between these values and the soil reaction. A general lack of phosphate in German soils is indicated.

A. G. POLLARD.

Relative lime needs of ammonium sulphate and sodium nitrate, and of different crops. B. L. HARTWELL and S. C. DAMON (J. Amer. Soc. Agron., 1927, 19, 843—849).—Each 100 g. of ammonium sulphate requires about 120 g. of limestone more than is required by 128 g. of sodium nitrate to attain equal soil reaction. In general, crop yields are similar for the two sources of nitrogen and the same reaction.

CHEMICAL ABSTRACTS.

Utilisation of nitrogen by grass land in the North Sea marshlands. F. TRILLING (Z. Pflanz. Düng.,

1928, 7B, 226—234).—Considerable variation in the efficiency of nitrogenous manures in marshes is recorded. Newer soils respond more markedly to treatment with ammonium sulphate than do older ones. Increases in hay yields following nitrogenous fertilisation are greater in soils having little clover in the herbage. Botanical examination shows this increase is largely in the proportion of meadow grass, with a diminution in that of dogstail. The relatively small effect of ammonium sulphate on rich clover land is ascribed to the influence of the different type of herbage on the physical and chemical properties of the soil. A. G. POLLARD.

Organic food reserves in relation to the growth of lucerne and other perennial herbaceous plants. L. F. GRABER, N. T. NELSON, W. A. LUEKEL, and W. B. ALBERT (Wisconsin Agric. Exp. Sta. Res. Bull., 1927, [80], 128 pp.).—The maturity and quantity of top growth, the root growth, and longevity of lucerne plants are generally associated with a high content of carbohydrate and reserve nitrogen in the roots. Susceptibility to winter injury is increased by low percentages of dry matter and low concentrations of carbohydrates and reserve nitrogen in the roots at winter dormancy.

CHEMICAL ABSTRACTS.

Effects due to spraying of fruits. P. R. V. D. R. COPEMAN (S. African J. Sci., 1927, 24, 198—203).—It has been noted that there is a deterioration of flavour in oranges sprayed with lead arsenate. Determinations of acidity, sucrose, and total sugar in the juice showed a marked reduction of acidity in sprayed fruits and a diminution in the ratio of sucrose to total sugar (presumably due to the conversion of sucrose into invert sugar), though the total sugar content is not affected. It is considered that the decrease of both acidity and sucrose accounts for the development of the insipid flavour. The action appears to be confined to arsenical sprays. A similar reduction of acidity occurs in apples and pears sprayed with lead arsenate, but the magnitude of the change is much less than in the case of oranges, and is not sufficient to alter the flavour.

C. T. GIMINGHAM.

Influence of fertiliser treatments on maturity and yield of cotton. J. W. MUSGRAVE and D. C. COE (J. Amer. Soc. Agron., 1927, 19, 910—923).—High percentages of nitrogen delay maturity. The yields are larger when the fertiliser is applied before planting.

CHEMICAL ABSTRACTS.

Burnt limestones in relation to quality of Bordeaux mixtures. R. H. CARR and L. N. BEMILLER (Ind. Eng. Chem., 1928, 20, 514—516).—Experiments with limestones of known composition and with mixtures of pure calcium and magnesium oxides showed that the proportions of the two oxides in the lime used in preparing Bordeaux mixtures have a marked and consistent effect on the alkalinity of the liquid above the precipitate, on the colour and rate of subsidence of the precipitate, and on the length of time the mixture will retain its colloidal nature and be suitable for spraying. Alkalinity of the liquid decreased as the proportion of magnesium oxide was increased, and characteristic changes in the colloidal state of the precipitate were also noted. C. T. GIMINGHAM.

Manufacture of manure from straw and calcium cyanamide. F. ZUCKER (Z. Pflanz. Düng., 1928, 7B, 206—215).—Calcium cyanamide was used successfully in making artificial farmyard manure from straw. The best results were obtained when the heaps of straw were about 90 cm. in height. Careful control of moisture conditions was necessary to ensure complete rotting through the stack with minimum losses in dry weight. Pot experiments served to show the value of the manure, but, in general, this was somewhat below that of ordinary yard manure. A. G. POLLARD.

Liquid manure, urine, and drainage water. STEHR and M. BERGOLD (Z. Pflanz. Düng., 1928, 7B, 216—226).—The addition to cattle urine of sulphuric or phosphoric acid in amounts corresponding to its total nitrogen content reduces nitrogen losses by checking fermentation. The loss of nitrogen during the fermentation of liquid manure is traced almost entirely to the volatilisation of ammonia, no free nitrogen being liberated. Drainage from "hot fermented" manure (Krantz) differs from ordinary liquid manure in that a large proportion of its total nitrogen is in organic combination. It is also richer in potash and phosphate, the latter existing mainly in colloidal suspension. A. G. POLLARD.

Utilisation of waste water from sugar factories. M. GREVEMEYER (Z. Ver. Deut. Zucker-Ind., 1928, 135—139).—Pots containing the following materials were sown with mustard: (a) loamy soil, (b) similar soil saturated with waste water, (c) soil with lime sludge, (d) soil with nitrogen as ammonium sulphate and lime as chalk to correspond with sludge, and (e) completely manured soil. The fresh weights of the crops (in grams) were (a) 128.5, (b) plants all died down, (c) 132.1, (d) 224.5, (e) 265.2. Oats sowed subsequently in the same pots yielded 38, 252.5, 45, 45.5, and 46 g. as fresh plant weight respectively. The failure of mustard in (b) is attributed to the possible presence of harmful salts and the slimy condition of the soil. In the case of oats, soil (b) was less exhausted by the previous crop, and nitrogenous substances may have broken down to a more available form. To other series of pots soil which had been irrigated by waste water was applied as manure at a rate corresponding to 10 tons/acre. The materials used, when extracted with 10% hydrochloric acid, yielded 0.109—0.299% K₂O, 0.305—0.484% N, 0.038—0.332% P₂O₅, 0.70—0.84% CaO, 0.054—0.123% MgO, and with barley gave increased yields of about 10%. With mustard the increase was less marked.

F. E. DAY.

Artificial farmyard manure. M. CARBERY and R. S. FINLOW (Agric. J. India, 1928, 23, 80—85).—The successful preparation of artificial farmyard manure from sugar-cane waste, straw, weeds, etc. is described. Heaps of the material were wetted with diluted cattle urine and sprinkled with bone meal. Better results were thus obtained than when ammonium acetate was employed as a "starter." Field trials demonstrated the satisfactory nature of the material. A. G. POLLARD.

Insecticidal action of esters of halogenated fatty acids in the vapour phase. R. C. ROARK and R. T. COTTON (Ind. Eng. Chem., 1928, 20, 512—514).—The

minimum lethal dose for the rice weevil of various esters of chloroformic, monochloro-, dichloro-, and monobromo-acetic, and chloro- and bromo-propionic acids in the vapour phase has been determined under laboratory conditions. The monochloroacetates appeared to possess interest as possible fumigants and were tested in a fumigation vault on a large scale. The methyl compound was effective against various stored-product insects at the rate of 1 lb. per 1000 cub. ft., and the ethyl compound at 2 lb. at a temperature of 27° or above. The monochloroacetates are, however, injurious to seed wheat.

C. T. GIMINGHAM.

Electrolytic apparatus for determination of copper in insecticides and fungicides. E. B. HOLLAND and G. M. GILLIGAN (Ind. Eng. Chem., 1928, 20, 533).—A form of apparatus with fixed electrodes is described and figured.

C. T. GIMINGHAM.

Feeding of sorghum silage and concentrate to Scindi calves. F. J. WARTH and S. K. MISRA (Mem. Dep. Agric. India, Chem. Series, 1927, 9, 125—153).

PATENTS.

Treatment of phosphates or phosphate-containing substances decomposed by sulphuric acid. STOCKHOLMS SUPERFOSFAT FABR. AKTIEBOLAG (B.P. 264,867, 21.1.27. Swed., 23.1.26).—The product obtained by decomposition with sulphuric acid of the phosphate or phosphatic substances, which may be of low grade with high content of iron, alumina, or carbonates, is treated in suspension with milk of lime or limestone powder, and then stirred at about 40° for not more than 6 hrs. with ammonium carbonate or ammonia and carbon dioxide, a saturated solution of ammonium sulphate being used as a carrier for the latter reagents. In this way di- and tri-calcium phosphates soluble in citrate or citric acid solutions are precipitated, and the sulphuric acid used for the decomposition is converted into ammonium sulphate. The liquid is heated to dissolve the ammonium sulphate, the calcium salts are separated by filtration, and the ammonium sulphate formed during the process is recovered by cooling the filtrate.

F. R. ENNOS.

Manufacture of fertilisers. D. TYRER (B.P. 290,075, 2.8.27).—The potassium sulphide present in the product obtained by heating a mixture of calcium phosphate and potassium sulphate or bisulphate under reducing conditions is decomposed by treatment with an acid or acid salt containing an acid radical of fertilising value, e.g., phosphoric acid or nitric acid, and, if desired, the acid salts in the product are neutralised with ammonia.

L. A. COLES.

Manufacture of insecticides, disinfectants, and fertilisers. V. CASABURI (B.P. 289,498, 24.11.26).—A process for the manufacture of solutions, powders, and semi-solid mixtures of various known insecticides or disinfecting agents with hemicelluloses obtained from locust-tree beans is claimed. The hemicelluloses are colloidal and have high adhesive powers. Ground fertilisers may be added to the mixtures.

C. T. GIMINGHAM.

XVII.—SUGARS; STARCHES; GUMS.

Correct procedure in sulphitation of thin [sugar] juice. I. Sulphitation. E. THIELEPAPE and P. MEIER (Z. Ver. deut. Zucker-Ind., 1928, 233—241).—In order to test the relative merits of sulphitation before the second carbonatation and of simultaneous treatment with sulphur dioxide and carbon dioxide, the amount of sulphite remaining in solution in either case has been studied. Solutions of lime, with and without sugar, and limed juices, were treated with known amounts of sulphur dioxide and carbon dioxide by both methods. Successive treatment removed a distinctly larger amount of sulphite than simultaneous treatment.

F. E. DAY.

Gilchrist process of [sugar juice] clarification. J. J. SEIP (Internat. Sugar J., 1928, 30, 82—84).—Some advantages of the Gilchrist process (in which the juice is limed while hot with calcium saccharate under definite hydrogen-ion control; cf. B.P. 247,542; B., 1927, 952) as compared with ordinary liming are: a more intense flocculation with inclusion of fine particles, a better elimination of colloids, a lighter coloured juice, less lime consumption, and, in general, a better control and more rapid operation. The mud from the settler has pH 6.9—7.3, which is considered ideal for filter-press work in giving a well-exhausted cake.

J. P. OGILVIE.

Factors influencing the filtration of raw sugar solutions. R. H. KING (The Planter, 1927, 79, 221—223, 242—244, 264—267).—A readily filtering sugar demands good clarification in its manufacture, the phosphoric acid content of the juice being the major factor in securing this. Suspensions which impede filtration are the result of poor defecation, solubility of certain substances which are precipitated by concentration in the clarified juice, redissolution of the settlings during washing, and also the introduction of insoluble matter from the low-grade sugar re-melted. Suspensions capable of forming precipitates that retard filtration are formed as the result of the digestion of fine particles of bagasse during treatment with lime at a high temperature.

J. P. OGILVIE.

Bleaching of off-colour sugar crystals. J. P. OGILVIE and R. G. W. FARNELL (Internat. Sugar J., 1928, 30, 92—93).—Experiments on the effect of various gases on "off-colour" sugars showed a distinct improvement in the case of beet sugars with chlorine and ozone, but with cane crystals none of the gases applied (which also included sulphur dioxide and oxides of nitrogen) effected any noteworthy bleaching, due possibly to the fact that the discoloration of this class of sugar is due largely to insoluble tannin matter.

J. P. OGILVIE.

Value of balancing the total soluble solids of the cane sugar factory. E. E. BATTELLE (Internat. Sugar J., 1928, 30, 89—91).—In appraising the relative technical efficiency of several cane factories, in addition to the usual balance-sheet relating to the polarisation of the various products, it is advantageous to extend this to include a statement of the total soluble solids as well. By this means losses are often disclosed which may not be apparent in the present scheme of reporting.

J. P. OGILVIE.

Treatment of effluents from beet sugar factories.

B. J. OWEN (Internat. Sugar J., 1928, 30, 75—79).—A Brackett 2-stage, cup-type screen placed beneath the outlet of the washer was found to remove the whole of the rootlets, leaves, small gravel, and earth, and subsequently a Wyllie gravel filter left the effluent clear and bright. It is concluded that the problem of dealing with conveying and washing waters seems capable of easy solution by this method, as they can be discharged direct to the river or recirculated. J. P. OGILVIE.

Nature of the neutralisation precipitate and its effect on the recovery of milk sugar [lactose] from grain-curd casein whey. R. W. BELL and P. N. PETER (Ind. Eng. Chem., 1928, 20, 510—512).—Acid wheys, such as grain-curd casein whey, prepared by coagulation with dilute acid, contain nearly all the salts of milk and on neutralisation yield a precipitate of calcium phosphate and protein material, which increases the viscosity of the concentrated whey and prevents crystallisation of the lactose. The quantity of this precipitate and the ratio of calcium phosphate to protein material increases with a decrease in the titratable acidity of the liquid. The complete removal of this precipitate is an essential to the successful recovery of lactose from the acid whey. F. R. ENNOS.

Yeast gum. Y. HASHITANI (Bull. Agric. Chem. Soc. Japan, 1927, 3, 2—14; cf. B., 1927, 567).—Yeast gum forms a benzoyl derivative, $C_6H_5O_5Bz_2$, m.p. 223—225°, decomp. 240°, and an acetyl derivative, $C_6H_7O_5Ac_3$, m.p. 178—182°; depolymerisation of yeast gum with glycerol at 210—220° yields α -yeast gum, $[\alpha]_D^{20} +80.08^\circ$, mol. wt. 808 (dibenzoyl derivative, m.p. 198—199°; triacetyl derivative, m.p. 158—159°).

CHEMICAL ABSTRACTS.

Sugar factory waste waters. GREVEMEYER.—See XVI. **Glucose.** MEZZADROLI.—See XVIII.

PATENT.

Clarification of liquids [sugar juices]. A. VALLÉE Y GUMÁ (U.S.P. 1,665,167, 3.4.28. Appl., 7.11.21. Cuba, 21.10.21).—Sugar juices, after the usual liming, are boiled vigorously to remove air and vapours. The subsequent clarification by settling is facilitated by maintaining the temperature at 100°. Other liquids, boiling above 100°, may also be kept, during clarification, at those temperatures at which the differences between the densities of the liquids and their suspended solids are greatest. F. G. CLARKE.

XVIII.—FERMENTATION INDUSTRIES.

Taka-invertase. II. R. WEIDENHAGEN and B. B. DEY (Z. Ver. Dent. Zucker-Ind., 1928, 242—261; cf. B., 1928, 423).—Comparison of the rates of inversion of sucrose by taka-invertase and by invertase from a top-fermentation yeast showed that as the reaction proceeded the velocity coefficient decreased in the former, but increased in the latter case. These departures from the unimolecular form of the reaction may be characteristic of invertases from the two sources. Contrary to the statement of Leibowitz and Mechlinski (A., 1926,

865), taka-invertase hydrolysed raffinose more than twice as fast as did yeast invertase of a similar sucrose-inverting power. This reaction closely followed the unimolecular form. The action of taka-invertase on sucrose and raffinose was markedly inhibited by α -glucose, less by β -glucose, and only slightly by lævulose; in fact, lævulose was without influence in the case of sucrose. The action of yeast invertase was most strongly inhibited by β -glucose; α -glucose had least effect on its hydrolysing action on sucrose, lævulose least on its action on raffinose. Since the inhibiting effect of the hexose was found to increase with decreasing amounts of substrate, it is concluded that the effect is due to affinity between hexose and enzyme.

F. E. DAY.

Comparison of acid production of *B. Delbrücki* and the cold lactic acid bacteria (*B. lactis acidii* and *B. cucumeris fermentati*, Henneberg). STAIGER and M. GLAUBITZ (Z. Spiritusind., 1928, 51, 109—110).—Solutions of sugars of 5% and 10% concentration, containing 1.5% and 3% of yeast extract, respectively, were employed. Molasses of 10% and 20% concentration, equivalent to 5% and 10% of sucrose, were also used, with and without yeast extract. One set was inoculated with *B. Delbrücki* and incubated at 45° for 18 days, the other with equal amounts of *B. lactis acidii* and *B. cucumeris fermentati* and kept at room temperature for a similar period. 250 c.c. of each culture medium were used, and the two kinds of bacteria gave the following amounts (c.c.) of *N*-lactic acid, respectively. In sucrose 5%, 17.5, 26.25; 10%, 21.25, 36.25; dextrose 5%, 8.75, 23.75; 10%, 10.00, 33.75; maltose 5%, 12.25, 28.75; 10%, 12.25, 35.00; molasses and yeast extract 10%, 28.75, 45.00; 20%, 35.75, 57.50; molasses without yeast extract 10%, 11.25, 18.75; 20%, 12.50, 20.00. F. E. DAY.

Saccharification of amylaceous substances and manufacture of ethyl alcohol and dextrose in an antiseptic medium by means of oriental bio-enzymes. G. MEZZADROLI (Zymologica, 1928, 3, 37—39).—If a sample removed from an aseptic saccharification vessel is passed through a filter and the filtrate, treated with a few drops of toluene, xylene, thymol, formaldehyde, fluoride, etc., is kept at the same temperature as the original vessel, the formation of sugar proceeds *pari passu* with that in the vessel. The mucor (amylo) process may, therefore, be modified as follows: The grain, broken into three or four, is placed in a bath at 50—60° containing about 8 pts. per 1000 of hydrochloric acid (d 1.16), left for about 2 hrs., and heated in a cooker, preferably horizontal, for 40—60 min. at 4 kg. pressure. When the liquor begins to filter well through filter-paper, the mass is discharged into a closed, aseptic vessel, cooled and aerated with sterile air, and at 40° seeded with a flask of sporulating mucor 105. After 15—18 hrs. from the time of seeding the mycelia begin to appear and soon assume their normal appearance, and when saccharification has produced 30—50 g. of sugar per litre, toluene or fluoride is added in sufficient amount and the mash is discharged into open vats. When the sugar reaches 50—80 g./litre, yeast acclimatised to the antiseptic is introduced. The claims for this procedure

are cheaper plant, lower coal consumption, acceleration of the various operations, and diminished chances of failure. T. H. POPE.

Electrocolorimeter for the photo-electric measurement of malt colours. F. STOCKHAUSEN and F. WINDISCH (*Woch. Brau.*, 1928, 45, 231—232).—The light from a small lamp passes through a cell containing the malt extract to be examined, and impinges on a potassium photo-electric cell. No details are given of the means by which the current from this is magnified to operate the pointer-type voltmeter on which the colour is read directly. The scale zero and light intensity are adjusted by two variable rheostats. Determination of colour can be carried out in a few seconds.

F. E. DAY.

Linear or logarithmic calculation of colour in malt analysis. BERMAN and L. LAUFER (*Woch. Brau.*, 1928, 45, 183). H. K. SCHINDLER (*Ibid.*, 183—185). F. DUCHÁČEK and V. L. ŽILA (*Ibid.*, 236—238). F. KUTTER (*Ibid.*, 238—239).—It is proposed that in calculating the observed colour of a laboratory wort of about 8.5% concentration to that of the 10% wort, the proportion should be $\log 10 / \log 8.5$ instead of $10/8.5$. An actual example is given in which the observed colours were: 8.6% 0.16; 9.95% 0.175. The linear calculation from the former to the latter gave 0.185, the logarithmic 0.171.

A reply to Bermann and Laufer (preceding abstract). From a mathematical consideration of the Weber-Fechner law it is concluded that the linear relationship should be used in the calculation. Actual examples of wort prepared at different concentrations are: Concentration 8.40%, colour (observed) 0.20; concentration 9.95%, colour (observed) 0.24, by calculation (linear) 0.237, (logarithmic) 0.216; calculating from strong to weak wort, concentration 8.35%, colour (observed) 0.46; concentration 4.175%, colour (observed) 0.24, by calculation (linear) 0.23, (logarithmic) 0.31. The case quoted by Bermann and Laufer shows too little difference to be significant.

The correctness of the linear calculation on the basis of the Lambert-Beer law of absorption is discussed, and its practical accuracy verified by colour determinations on worts prepared at various concentrations from the same malts.

Extracts of varying concentration from the same malt had colours which were approximately in direct linear proportion to the concentration. Though the linear calculation is only approximate, the logarithmic calculation is quite erroneous. F. E. DAY.

Simple graphical method for determining the course of the natural distillation process. H. BRANDES (*Chem. Fabr.*, 1928, 261—262).—The construction of a curve showing the relation between the percentages of alcohol in the vapour and in the residual liquid in the flask during the course of distilling a mixture of alcohol and water without the aid of a fractionating column is described, and a geometrical construction is given for deriving from this curve a curve showing the percentage of alcohol in the residue at any stage of the distillation. A. R. POWELL.

Yeast gum. HASHITANI.—See XVII. **Essential oil of hops.** CHAPMAN.—See XX.

PATENTS.

Manufacture of beverages. H. MACKESON (B.P. 289,601, 9.3.27. Addn. to B.P. 267,236; B., 1927, 375).—The addition either of the priming solution containing malt extract after fermentation or of the malt extract before fermentation is omitted.

F. R. ENNOS.

Manufacture of a dry urease preparation. HENKEL & Co. G.M.B.H. (B.P. 277,644, 30.8.27. Ger., 15.9.26).—A stable, soluble urease preparation is obtained by adding substances such as phosphates to a filtered or centrifuged aqueous extract of soya or jack beans or similar urease-containing parts of plants, and then atomising the solution in a dry, heated current of air or gas; or the extraction may be made with solutions of the phosphates etc. above-mentioned.

B. FULLMAN.

Dehydration of alcohol. J. A. STEFFENS (U.S.P. 1,670,053, 15.5.28. Appl., 16.8.23).—See B.P. 220,606; B., 1925, 185.

Fermenting products (B.P. 289,978).—See XIX.

XIX.—FOODS.

Action of cold on the fats of milk. G. QUAGLIA-RIELLO (*Atti R. Accad. Lincei*, 1928, [vi], 7, 75—78).—The lowering of the surface tension occurring when milk is cooled for 30 min. at below 10° is due to passage of triglycerides of the lower fatty acids from the fat globules to the interglobular liquid, in which they dissolve. The phenomenon is not shown by homogenised milk. T. H. POPE.

Grading of commercial gelatin and its use in the manufacture of ice cream. II. A. C. DAHLBERG, D. C. CARPENTER, and J. C. HENING (*Ind. Eng. Chem.*, 1928, 20, 516—526).—Methods used for grading gelatins, both commercial and high-grade varieties, are compared as to their suitability for the manufacture of ice cream. In weak solutions a reduction in the ageing temperature and the presence of the serum solids of milk caused an increase in the extent of gelation. The minimum percentages of each gelatin required to give the desired body and texture to the ice cream, as well as the requisite character after melting, were also determined, and a simple practical test is described for this purpose. Provided the correct amount was used, the quality and volume yield of ice cream did not appear to be affected by the kind or strength of the gelatin. The principal property of gelatin which is of value in ice cream manufacture appears to be its ability to re-form a gel at low temperatures after being whipped to a fluid condition; the agar gels do not behave in this way. F. R. ENNOS.

Adsorption phenomena [with wheat]. G. TESTONI (*Rev. gén. Colloïd.*, 1928, 6, 8—15).—In an attempt to decide whether gluten is already existent in wheat flour, or whether it is produced during the mixing with water, the author has studied the colours produced in a

wheat grain by adsorption of various natural and artificial organic colouring matters. It is concluded that gluten is not pre-existent in wheat flour, but is formed under the influence of calcium salts, when mixed with water. The sensitivity of indicators is diminished by adsorption of the indicator at a surface, and measurement of the decrease in sensitivity provides a means of characterising the adsorbent surface. The values are expressed as "adsorption values," and some of the proteins of wheat have been characterised in this way. Ferric and cupric salts are adsorbed by gluten and gliadin, and the colourless products fail to give reactions for the respective metal ions except towards potassium ferrocyanide. The product of adsorption of nickel salts fails to give a reaction for nickel ions with any reagent. It is suggested that reaction can take place only when the second substance also is adsorbed, and that this condition is realised in the case of potassium ferrocyanide. E. S. HEDGES.

Thermophilic and thermoduric micro-organisms, with special reference to species isolated from milk. II. Thermal resistance of micro-organisms. A. H. ROBERTSON (Vt. Agric. Exp. Sta. Bull., 1927, 274, 1—27).

PATENTS.

Manufacture of fermenting products. BRIT. ARKADY CO., LTD., R. WHYMPER, and H. HEWITT (B.P. 289,978, 12.2.27).—Bread is made by adding to the dough or incorporating with the flour and other ingredients used in making the dough a fermenting product comprising yeast and a fungus such as *Aspergillus oryzae* or *Penicillium*, together with the products obtained by the previous action of either or both of these micro-organisms on rice, barley, or vegetable substances rich in proteins. C. RANKEN.

Treatment of dried fruit. E. C. R. MARKS. From SUN-MAID RAISIN GROWERS OF CALIFORNIA (B.P. 289,722, 11.11.27).—Raisins are subjected to opposing blasts of highly ozonised air and steam as they fall through a vertical chute. They are then quickly dried at 400° and the heated vapours removed. F. R. ENNOS.

Preservation of fodder. E. C. R. MARKS. From M. FLUBACHER (B.P. 285,939, 22.10.26).—The fodder plants are cut and packed by pressing in air-tight containers, each layer being subjected to heavy pressure in order to prevent injurious fermentation, self-heating, or breathing of the plants. A 3% solution of a mixture composed of sodium chloride 50%, calcium chloride 10%, sodium phosphate 10%, and ferrous lactate 30% is used to spray the fodder either before or after cutting, or during the packing into the containers, whereby butyric fermentation is prevented. F. R. ENNOS.

Preparation of a composition for coating substances [foods] liable to putrefaction. G. VAN DER VEEN and G. J. P. H. A. DE BEUS (B.P. 286,096, 14.3.27).—Rosin, purified by fusion with soda, is melted with solid paraffin in such proportion as to reduce the m.p. to 25—30°. Activated charcoal, oxide or carbonate of magnesium, and, if desired, magnesium chloride, may be added to the melted mixture. F. R. ENNOS.

Sterilisation of clear and turbid liquids [foods] by means of ultra-violet rays. E. O. SCHEIDT (U.S.P. 1,670,217, 15.5.28. Appl., 24.10.25. Ger., 5.9.25).—See B.P. 257,956; B., 1928, 106.

Production of a [grated] almond milk food product. H. HOFER-MASSARD (B.P. 282,088, 9.12.27. Switz., 10.12.26).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Composition of tragacanth-paraffin oil emulsions. E. SCHULEK and G. VASTAGH (Pharm. Zentr., 1928, 69, 275—276).—The emulsion is first separated by warming with alcohol and the paraffin oil is extracted with light petroleum. The petrol extract is evaporated and dried at 120°. The presence of saccharin in the aqueous-alcoholic residue was demonstrated. The results of two analyses are appended. H. BURTON.

Nicotine in tobacco. II. A. HEIDUSCHKA and F. MUTH (Pharm. Zentr., 1928, 69, 305—307; cf. B., 1927, 570).—Using the methods of Rasmussen (A., 1916, ii, 359) previously employed, and that of Pfyl and Schmitt (B., 1927, 955), determinations were made of the nicotine content of tobacco and smoke from various German cigarettes, including special brands advertised as harmless or poor in nicotine. The presence of nicotine could be confirmed in all cases by determination of the m.p. of the dipicrate. The amount of nicotine found in the tobacco and smoke respectively, calculated in each case on the weight of dry tobacco, was: ordinary cigarettes, 1.11—1.31% and 0.16—0.23%; special brands, 0.64—1.22% and 0.09—0.16%. R. K. CALLOW.

Determination of alkaloids in ergot. F. WESSEL (Pharm.-Ztg., 1928, 73, 354—355).—The procedure recommended in the German Pharmacopœia, 6th Ed., is very troublesome, emulsions being formed which render filtration difficult; the titration of the alkaloids isolated is also inaccurate. It is better to remove fats by a preliminary extraction with light petroleum, and to precipitate the alkaloids from the hydrochloric acid solution obtained with ammonia, instead of with sodium carbonate. The precipitated alkaloids are dried and weighed. S. I. LEVY.

Volumetric determination of aluminium in pharmaceutical preparations. H. MATTHES and P. SCHÜTZ (Pharm.-Ztg., 1928, 73, 353—354).—Details of the procedure for the precipitation of the 8-hydroxyquinoline compound of the metal, and the determination of the hydroxyquinoline obtained from the precipitate by treatment with acid, are given. The latter is based on the formation of the dibromo-substitution product by the action of potassium bromide and bromate; the excess of bromate used is determined iodometrically. S. I. LEVY.

Higher-boiling constituents of essential oil of hops. A. C. CHAPMAN (J.C.S., 1928, 1303—1306).—Distillation of the higher-boiling constituents of hop oil gave seven fractions, b.p. 87—97° to 180—200°/4 mm., d_{20}^{20} 0.8948—0.9560, n_D^{20} 1.4769—1.4975. The first fraction, after hydrolysis with alcoholic potassium

hydroxide and subsequent removal of alcohols with phthalic anhydride, yielded a ketone, $C_{13}H_{22}O$, having b.p. 74—76°/3 mm., d_{20}^{20} 0.8861, n_D^{20} 1.485, $[\alpha]_D^{20}$ -0.4°, when regenerated from its semicarbazone, m.p. 98°. The name *luparone* is suggested for the ketone. From the fractions of b.p. 110—150°/4 mm., after successive treatment with alcoholic potassium hydroxide and phthalic anhydride, there was obtained an unsaturated alcohol (*luparenol*), $C_{15}H_{24}O$, having one double linking, b.p. 125—128°/3 mm., d_{20}^{20} 0.9738, n_D^{20} 1.5023, $[\alpha]_D^{20}$ -3.7° (*phenylcarbimide* derivative, m.p. 157°). The residue, when purified by successive treatment with phthalic anhydride and alcoholic silver nitrate, gave a phenolic ether (*luparol*), $C_{16}H_{26}O_2$, b.p. 122—124°/2 mm., d_{20}^{20} 0.9170, n_D^{20} 1.4942. When luparol is boiled with 50% aqueous potassium hydroxide it undergoes gradual decomposition into isovaleric acid, also obtained by oxidation with potassium permanganate, and a phenol, probably $C_{11}H_{16}O_2$, b.p. 115—117°/4 mm., d_{20}^{20} 0.9448, n_D^{20} 1.4670. The fractions of b.p. 150—200°/4 mm. consist chiefly of luparol and luparenol. isovaleric acid is a constituent of the original oil. H. BURTON.

Essential oils of *Eucalyptus micrantha* (D.C.) and *E. haemastoma* (Smith). I. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. New South Wales, 1927, 61, 267—268).—Steam distillation of 6 lots of leaves and terminal branchlets of *E. micrantha* (D.C.) gave yields of oil varying from 0.62 to 0.9% and having d_{15}^{15} 0.8883—0.9122, α_D^{20} -25.5° to +5°, n_D^{20} 1.4701—1.4892, ester value 6.2—23.1, ester value after acetylation 41.9—95.2, solubility in 80% alcohol 1 vol.—8 vols. The principal constituents of the oils were *l*- α -phellandrene (moderate to large quantities), sesquiterpenes, terpineol (?) and piperitol and their hexoic esters, *d*- α -pinene, cineole, sesquiterpene alcohols, and traces of aromatic aldehydes. Piperitone was probably present and eudesmol was present in one oil. Leaves and terminal branchlets of *E. haemastoma* (Smith), collected at the same time and place as those of *E. micrantha*, on steam distillation gave 0.25—0.5% of oil having d_{15}^{15} 0.9295—0.9571, α_D^{20} +3.3° to +16.8°, n_D^{20} 1.4861—1.4957, ester value 8.6—12.0, ester value after acetylation 72.5—116.9, solubility in 70% alcohol 1.7—2.0 vols., and containing eudesmol, sesquiterpenes (aromadendrene and probably eudesmene), *d*- α -pinene, cineole (10—15%), and a very small quantity of phellandrene not detectable by the B.P. test.

E. H. SHARPLES.

Essential oils of two species of *Baeckea*. A. R. PENFOLD (J. Proc. Roy. Soc. New South Wales, 1927, 61, 285—295).—Steam distillation of the leaves and terminal branchlets of 2 lots of *Baeckea brevifolia* (De Candolle) collected in 1925 and 1927, gave 1.6% and 1.02% of oils having the following respective constants, d_{15}^{15} 0.9110, 0.9257; α_D^{20} -3.9°, —; n_D^{20} 1.4839, 1.4888; ester value 3.2, 5.4; ester value after acetylation 75, 109. Both oils were soluble in 0.6 vol. of 80% alcohol and insoluble in 10 vols. of 70% alcohol, and the principal constituents were: α - and β -pinene, the latter predominating, cineole, eudesmol (30—45%) (*allophanate*, m.p. 174°), and small quantities of phenolic substances.

In a similar manner the leaves and terminal branchlets of *Baeckea linifolia* var. *brevifolia* (Mueller) gave 0.5—0.82% of mobile oil having d_{15}^{15} 0.8917—0.9035, α_D^{20} +6.85° to +10.85°, n_D^{20} 1.4752—1.4791, ester value 8.3—21.2, ester value after acetylation 44.7—60.9, solubility in 80% alcohol (by wt.) 4.6—5.0, and consisting of α - and β -pinene, cymene, cineole (18%), sesquiterpene and sesquiterpene alcohol, an unidentified alcohol (d_{15}^{15} 0.915, α_D^{20} +7.3, n_D^{20} 1.4806; naphthylurethane, m.p. 120°), a mixture of phenols belonging to the tasmanol group, and isobutyric and isovaleric acids present as esters.

E. H. SHARPLES.

Essential oil from timber of rosewood (*Dysoxylon fraserianum*). A. R. PENFOLD (J. Proc. Roy. Soc. New South Wales, 1927, 61, 337—346).—Three separate steam distillations of the shavings of rosewood gave a yield, on the wet wood (41—46.13% moisture), of 1.2—2.7% of pale to dark blue, viscous oil having d_{15}^{15} 0.925—0.9398, α_D^{20} -5°, n_D^{20} 1.5035—1.5046, ester value 2.2—10.6, ester value after acetylation 40.4—62.5, and was insoluble in 10 vols. of 90% alcohol (by wt.). The oil consisted of a mixture of sesquiterpenes, possibly two, which are apparently hydroazulenes, as on dehydrogenation with sulphur azulene was obtained, a third sesquiterpene, *dysoxylonene* (b.p. 136—137°/10 mm., d_{15}^{15} 0.9236, α_D^{20} nil, n_D^{20} 1.5063; *dihydrochloride*, m.p. 108—109°, α_D nil) evidently related to the cadinene group. Cadinene was found to be the principal sesquiterpene in the first two distillations. Azulene, unidentified phenols, and probably copaene and aromadendrene were present in the oils.

E. H. SHARPLES.

Determination of eugenol. W. A. N. MARKWELL and L. J. WALKER (Perf. Ess. Oil Rec., 1928, 19, 169).—For the determination of eugenol in a number of samples of oil the following method is quicker, more convenient, and necessitates a smaller quantity of oil than that in which Hirschsohn flasks are used. 2 c.c. of the oils are measured into each of several Gerber milk tubes containing 16 c.c. of 5% potassium hydroxide solution and the tubes are shaken at 5 min. intervals for 30 min. A further 4 c.c. of the potassium hydroxide solution are added to each and the tubes are centrifuged for 5 min., the pear-shaped bulbs being placed inwards. By adjustment of the stopper the uncombined oil is raised into the graduated stem. The percentage of eugenol is obtained from the formula: % = 100 - 50Vf, where V is the volume of uncombined oil and f is the factor required to convert divisions on the Gerber scale into c.c.

E. H. SHARPLES.

Flour from linseed. ANDRÉ.—See XII.

PATENTS.

Preparation of emetine. E. BURCKHARDT and M. STÄRKLE, Asss. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,670,059, 15.5.28. Appl., 8.6.27. Switz., 27.5.27).—Cephæline is methylated with phenyltrimethylammonium hydroxide.

C. HOLLINS.

Production of killed hog cholera virus. J. REICHEL (U.S.P. 1,661,233, 6.3.28. Appl., 16.2.25).—Live viru-

lent hog cholera virus in the form of defibrinated blood is treated with 0.5% of phenol, and then kept at 5–10° for 30–90 days, and at about 37° for 30 days. The product is used for immunising hogs against cholera.

B. FULLMAN.

Manufacture of hydrocyclic ω -aminoalkyl compound. H. RUPE, ASSR. TO SOC. CHEM. IND. IN BASLE (U.S.P. 1,670,990, 22.5.28. Appl., 2.9.25. Switz., 1.10.24).—See B.P. 240,814; B., 1926, 27.

Manufacture of unsaturated aldehydes. H. RUPE, ASSR. TO SOC. CHEM. IND. IN BASLE (U.S.P. 1,670,825, 22.5.28. Appl., 11.3.27. Switz., 22.3.26).—See B.P. 267,954; B., 1927, 571.

Manufacture of basic bismuth salts of aryl-arsinic acids. R. W. E. STICKINGS, ASSR. TO MAY & BAKER, LTD. (U.S.P. 1,669,542, 15.5.28. Appl., 2.12.26. U.K., 5.12.25).—See B.P. 266,820; B., 1927, 349.

Treatment of oils (B.P. 281,232).—See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Optical sensitising of silver halide emulsions. I. Adsorption of orthochrome-T to silver bromide. S. E. SHEPPARD and H. CROUCH (J. Physical Chem., 1928, 32, 751–762, and Phot. J., 1928, 68, 273–280).—The partition coefficients for an isocyanine-orthochrome-T bromide between aqueous solutions and chloroform have been measured spectrophotometrically and show higher values in weaker buffer solutions of the same p_H . The partition of the dye in the aqueous layer is lowered by the addition of soluble halides. The absorption spectra of orthochrome-T bromide at p_H 4.8–5.8 show that as the colourless form of the dye increases the ultra-violet absorption increases. In chloroform and in methyl alcohol the absorption maximum is slightly affected. Data on adsorption of the dye at 50° by silver bromide grains are given, and two alternative hypotheses are advanced to explain the results obtained. Experiments on the diffusion of the dye into gelatin cylinders indicated a lowering of diffusivity with rising p_H in agreement with the tendency of the coloured form of the dye to form colloidal micelles of low diffusivity, and, further, that the amount of dye adsorbed at saturation is apparently much greater than the amount taken up by silver halide for optimum sensitising. A mechanism for optical sensitising is suggested.

L. S. THEOBALD.

Photographic prints on fabrics. MICHELS.—See VI.

PATENTS.

Production of photographs in natural colours. W. LANGGUTH and C. HUMMEL (B.P. 274,129, 11.7.27. Ger., 9.7.26).—In the process in which three component pictures are successively printed on the same paper support using azo dyes, the support saturated with the dye components is exposed while moist under the negatives in a cooled frame. The order of printing is blue-green, green-yellow, and finally purple. The separate

component images are printed under filters of colours complementary to the colours being printed. Suitable dye components are, for blue-green: dianisidine and 1:8-aminonaphthol-2:4-disulphonic acid; for the green-yellow: 2-methylbenzidine and ethyl acetoacetate; for the purple picture: *o*-anisidine and β -hydroxynaphthoic acid or its derivatives; in each case the amine is diazotised in presence of sodium sulphite.

W. CLARK.

Production of coloured photographic pictures. J. H. CHRISTENSEN (B.P. 289,175, 26.1.27).—Colour photographs to be viewed by reflected light consist of screen unit colour images of a layer of colour elements covered by a layer containing silver, the film being brought into contact with a coherent white layer reflecting the main part of the light within a depth from its surface not exceeding the average diameter of the coloured elements. Suitable white layers consist of a layer of cellulose derivative penetrated by numerous air-filled fissures, or a cellulose derivative containing a white pigment.

W. CLARK.

Production of photozincograph printing surfaces. F. ULLMANN (B.P. 288,023, 24.3.27).—The zinc plate is treated with the salt of a metal, *e.g.*, aluminium, having a tendency to form double salts, before the application of the sensitive chromate layer. The action between the zinc and the chromate preparations is minimised.

W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Metal derivatives of diketones (B.P. 289,493).—See III.

XXIII.—SANITATION; WATER PURIFICATION.

Stream-flow sewage-treatment process. H. N. JENKS and M. LEVINE (Eng. News-Rec., 1928, 100, 808–813).—Laboratory and large-scale experiments are described in which the supply of oxygen necessary for the operation of the activated sludge system of sewage purification is obtained by passing the mixed liquor in a shallow stream along an open channel. This is succeeded by a quiescent period in which the oxygen so obtained is utilised biologically in purifying the sewage, which is then again passed over the channel by means of a pump, re-aerated, and the cycle repeated. The frequency of re-aeration can be adjusted so as to conform with the oxygen demand at different stages of the process and thus give increased efficiency. It is claimed that strong domestic sewage can be purified in 6 hrs. with a minimum expenditure of energy for aeration.

C. JEPSON.

Modern aspects of chlorination of water. N. J. HOWARD (J. Amer. Water Works' Assoc., 1928, 19, 546–552).—Recent research suggests that the sterilising action of chlorine is due not to the production of nascent oxygen, but to the formation of some toxic substance which prevents cell division. The substitution of chlorine for alum when the water is physically good results in

considerable saving in the maintenance cost of the filters. A mixture of chlorine and ammonia is considered to possess many advantages over straight chlorine, particularly in a delayed action when dealing with spore-forming bacteria and after-growths in mains. It is said to be tasteless, and in some cases the use of ammonia prior to chlorination has prevented iodoform tastes previously present. This taste is sometimes due to the presence of traces of phenol, cresol, anisole, or xylenol. The taste due to chlorine is likely to occur if the amount used is the minimum necessary for sterilisation, but by adding excess of chlorine and giving a certain period of contact before removal of the residual chlorine by sulphur dioxide all taste can be avoided.

C. JEPSON.

Method for phenol determinations [in water]. J. R. BAYLIS (J. Amer. Water Works' Assoc., 1928, 19, 597—604).—Gibbs' method (cf. A., 1927, 688) in which use is made of the blue indophenol colour produced with dibromobenzoquinonechloroimide is suggested. A sample of the water (800 c.c.) is taken and distilled at a rate of 8—10 c.c./min. The distillate is collected in five successive lots of 100 c.c. each, and the phenol determined by comparison of the indophenol colour produced. This amount of distillate contains 85% of the phenol in the original sample. If the total quantity is between 15 and 5 pts./billion it is desirable to concentrate by taking two or more samples each of 800 c.c., distilling over 50% of the sample (containing 75% of the original phenol), and determining the phenol in the combined distillates. It is considered that the smallest quantity of phenol which will cause noticeable chlorophenol tastes is 5 pts./billion. Before adding the dibromobenzoquinonechloroimide, the distillates should be buffered so as to have p_H 9.6—10.0 as this influences the rate of indophenol formation.

C. JEPSON.

Test for phenolic tastes and odours in water after chlorination. F. W. SPERR, JUN., W. H. FULWEILER, F. E. DANIELS, and O. O. MALLEIS (J. Amer. Water Works' Assoc., 1928, 19, 605—606).—A sample (500 c.c.) is made acid to litmus by means of sulphuric acid and distilled. The distillate (250 c.c.) is collected in a 500 c.c. graduated flask, made up to 500 c.c. with distilled water, and diluted as follows: 1 to 10; 1 to 100; 1 to 1000, etc. 200 c.c. of the distillate and a like amount of each dilution are then taken and treated with a slight excess of chlorine water (a total of 0.3 p.p.m. is usually enough). After 15 min. the excess chlorine is boiled off and the odour test made on the hot liquid, after which it is allowed to cool and tasted. Results should be expressed as the lowest dilution in which the taste and odour are negative. C. JEPSON.

Volumetric method for determination of sulphate ion [in potable waters etc.]. F. G. GERMUTH (J. Amer. Water Works' Assoc., 1928, 19, 607—609).—For determining sulphate present in potable waters or other materials, in which the amount present is small, a slight excess of 0.02*N*-barium chloride is added, with vigorous stirring, to a measured volume of sample slightly acidified with hydrochloric acid and at room temperature. After 5 min. it is made alkaline to phenol-

phthalein by means of ammonia, and 10 c.c. of 5% ammonium acetate are added. The excess barium chloride is titrated back with 0.02*N*-potassium chromate, using lead nitrate as an outside indicator, and the amount of sulphate present is calculated from the volume of barium chloride used. C. JEPSON.

Fumigation tests with ethylene dichloride-carbon tetrachloride mixture. L. F. HOYT (Ind. Eng. Chem., 1928, 20, 460—461).—The new fumigating mixture of 3 vols. of ethylene dichloride and 1 vol. of carbon tetrachloride proposed by Cotton and Roark (cf. B., 1927, 862) gave a complete kill of all insects present (meal-moth and clothes-moth larvæ, and flour beetles) when used in a 500 cub. ft. vault at the rate of 14 lb. per 1000 cub. ft. for 24 hrs. at 28°. In an 8000 cub. ft. room the results were also satisfactory, though the insects were killed less rapidly. The mixture is easy to use and appears to be a valuable and safe fumigant.

C. T. GIMINGHAM.

Natural separation of salts from rivers charged with [magnesia-containing] effluent from potash works. EMMERLING (Chem.-Ztg., 1928, 52, 398—399).—The river Wipper, which is charged with magnesia derived from potash works, was found during a part of its course, where it passed through a limestone formation, to deposit a mud containing up to 3% MgO as against 0.4% higher up. The limestone itself was non-magnesian, and any reaction between calcium carbonate and magnesium chloride was out of the question. The limestone is, however, argillaceous, and a suspension of the finely-powdered material in water when shaken with magnesium chloride solution deposited a flocculent precipitate containing magnesia. Gelatinous silicic acid is without effect, and the presence of lime is necessary. It was also found that humic acids precipitate magnesia more readily than lime, and water plants take it up in relatively large quantities from waters containing it.

C. IRWIN.

Colliery waters. SIMPKIN.—See II. **Halogenated fatty acids as insecticides.** ROARK and COTTON.—See XVI. **Effluents from beet sugar factories.** OWEN.—See XVII.

PATENTS.

Softening and purification of water. HÖGANÄS-BILLES HOLMS AKTIEBOLAG and NORDISKE NATROLITH A./S. (B.P. 279,788, 6.7.27. Swed., 29.10.26).—A base-exchange, water-softening material, depending on the action of siliceous gels, may be produced by calcining "Keuper" clay at not exceeding 800° and subsequently crushing it to the desired size. If the calcination has been carried too far the activity of the gels may be re-established by boiling with a solution of an alkali salt, e.g., sodium chloride. After being used for softening water, the effectiveness of the clay may be regenerated by treatment with hydrochloric acid.

C. JEPSON.

Water-softening filters, and regeneration and purification of same. C. HUFSCHMIDT (B.P. 290,064, 5.7.27).

Slag-sand from refuse (B.P. 280,902).—See IX. **Disinfectants** (B.P. 289,498).—See XVI.