

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 16, 1926.

I.—GENERAL; PLANT; MACHINERY.

Device for circulating fluids under high pressure. I. R. MCHAFFIE (Faraday Soc., Feb., 1926. Advance proof).—A small soft iron piston rod operating in a gun-metal cylinder is controlled electro-magnetically by two coils wound in the same direction and connected in series with a resistance so that they pass a current of 1 ampère. The coils are arranged so that they may be alternately short-circuited by a motor-driven commutator consisting of a brass slip ring in contact with a brass quadrant; thus the position of the magnetic field, and therefore that of the piston, is controlled by the rotation of the commutator. With a speed of 2 revolutions per second, the pump will circulate 25 litres of air per hour at atmospheric pressure; it can be operated as a lift pump or a force pump and circulates air up to 100 atm. pressure.

B. W. CLARKE.

Absorption of chemical fogs. H. REMY (Z. angew. Chem., 1926, 39, 147—150; cf. B., 1925, 40).—Differences in the extent to which dry and moist fogs are absorbed by various agents are due to the particles of the moist fog being much larger than those of the dry. Thus, filter paper retains the moist fog better than the dry, whilst with active charcoal the reverse is true. Water converts a dry into a moist fog, the enlarged fog particles becoming thereby less mobile and less easily absorbed. Increasing the viscosity of a liquid absorbent diminishes its absorptive capacity by increasing the size of the gas bubbles passing through it, and so diminishing the chance of contact between liquid and fog particles.

W. T. K. BRAUNHOLTZ.

Production of hydrogen by steam in a hot boiler tube. J. PORTER (J. Roy. Tech. Coll. Glasgow, 1925, [2], 14—18).—No hydrogen could be detected in steam from a boiler tube when the latter was maintained at a temperature below 400°. Between 400° and 500° a very slow decomposition of the steam took place, which became very rapid at temperatures above 500°.

A. R. POWELL.

Fire-bars and their behaviour in the fire. R. STUMPER (Chaleur et Ind., 1925, 6, 549—554; Chem. Zentr., 1926, I., 1277).—The life of fire-bars depends on the metallographic structure of the metal, the chemical composition of the fuel, the method of firing, and the boiler system. Phosphorus lowers the m.p. of the iron and renders it brittle at high temperatures; the presence of phosphates in the ash of the fuel causes it to sinter to the fire-bars and increases the possibility of a

reaction between the ash and the metal of the bars with the formation of the ternary eutectic, steadite. The most harmful element in the fuel itself is sulphur. Grey cast-iron is less suitable than white cast-iron for the manufacture of fire-bars, as the presence of free graphite in the grey iron renders the metal porous, and air and deleterious gases can thus readily penetrate into the interior. White cast-iron resists the chemical action of the fire better than the grey variety and, although it is possible to cause decomposition of the cementite to take place, the carbon deposited does not cause the metal to become as porous as grey cast-iron.

A. R. POWELL.

See also A., Mar., 217, **Critical potentials of hydrogen in presence of catalytic nickel and copper** (WOLFENDEN). 230, **Plasticity of amorphous and crystalline solids** (BECKER). 240, **Mechanism of ultrafiltration** (DUCLAUX and ERRERA). 250, **Catalytic activity** (CONSTABLE); **Catalysis** (ZELINSKI).

Conditioning of boiler water. HALL.—See XXIII.

PATENTS.

Apparatus for purifying, cooling, heating, mixing, or absorbing gases and vapours. E. THEISEN (E.P. 234,109, 13.5.25. Conv., 15.5.24).—The gas or vapour traverses one or more curtains of liquid discharged outwards by one or more vane wheels carried by a rotary shaft within a casing. Each wheel comprises a flat disc carrying, near the rim only, a series of oblique vanes, and the liquid is delivered to the central portions of these discs. Means may be provided for returning the liquid, discharged outwardly from one wheel, to the centre of the succeeding wheel.

H. HOLMES.

Centrifugal separators for separating solids from liquids. LAUGHLIN FILTER CORP., Assees. of W. C. LAUGHLIN (E.P. 235,916, 22.6.25. Conv., 21.6.24).—A centrifugal separator has the separating zone divided into two parts by a vertical partition, on one side of which clear liquor is discharged, whilst on the other side, scrapers geared to rotate at a speed slightly different from the bowl, remove the deposited mud to a discharge outlet. The axial feed inlet is on the same side of the partition as the discharge outlet.

B. M. VENABLES.

Dehydrating liquids. A. S. ELSENBAST, Assr. to CELITE Co. (U.S.P. 1,570,626, 26.1.26. Appl., 14.3.22).—Small quantities of water are separated from non-aqueous liquids by admixture of dehydrated diatomaceous earth, followed by filtration.

H. HOLMES.

Heat exchanger. B. M. JOHNSON, Assr. to CARBORUNDUM Co. (U.S.P. 1,570,674, 26.1.26. Appl., 9.2.24).—Banks of conducting tubes are arranged side by side above a silicon carbide radiating wall of a combustion chamber, each bank comprising metal tubes arranged above silicon carbide tubes adjacent to the wall. Means are provided for so directing the combustion gases into contact with the tubes as to subject the carbide tubes of each bank to a higher temperature than the metal tubes, and a mixing chamber is disposed between the discharge ends of the tubes of each bank and the intake ends of the tubes of the next bank.

H. HOLMES.

Treating filtration residues for re-use. P. A. BOECK, Assr. to CELITE Co. (U.S.P. 1,571,042, 26.1.26. Appl., 19.1.21).—Spent diatomaceous earth filtering material is dried, reduced to fragmentary condition, and heated for a time sufficient to increase the filtration efficiency without substantial removal of chemically-combined water.

H. HOLMES.

Drying plant. P. BARDUCCI (U.S.P. 1,571,226—8, 2.2.26. Appl., [A] 19.1.20, [B] 18.2.22, [C] 20.2.22).—(A) A container for the material to be dried is divided into a number of sections, through which separate air currents are passed intermittently and not all at once. The distributing valves or shutters for the air currents and means for operating them in pre-determined sequence are placed outside the containers for the material. (B) A drying compartment is provided in the centre with division walls which are H-shaped in plan and do not extend right across the chamber. Air-impelling means are situated opposite one or more openings in the short wall or crossbar of the H, so that air is circulated through the central space and divides right and left through the material in the side spaces, the streams re-uniting to pass through the central space again. (C) In a drying room, reciprocating means are provided for producing vertical air currents, and other means, at the ends of the room, direct air currents respectively towards and from the reciprocator.

B. M. VENABLES.

Fractionating tower. F. B. SAMUEL (U.S.P. 1,571,805, 2.2.26. Appl., 17.4.24).—Vapour is passed downwards through a number of approximately vertical tubes or flues in a heat-insulated vertical casing, to a separator for vapour and condensate at the bottom. The temperature of the tubes is regulated by an upward draught of air around them, the temperature drop within the casing being maintained constant irrespective of weather conditions.

B. M. VENABLES.

Decolorising and purifying solutions. INTERNATIONAL SUGAR AND ALCOHOL Co., LTD. (F.P. 596,919, 20.4.25. Conv., 26.6.24).—Lignin obtained by treating cellulosic materials with acids is washed and used in the same way as wood charcoal for purifying liquids. It should not be dried, but if this has occurred, it can be re-activated by steaming.

E. S. KREIS.

Carrying out and controlling reactions. E. MÜLLER, and CHEM. FABR. BUCKAU (G.P. 422,159, 1.7.24).—Instead of removing successive samples for analysis in a continuous process, or working a discontinuous process with measured quantities of reagents, the potential difference between a normal electrode and an electrode immersed in the reaction liquid is measured and compared with a standard table. In an example, the application of the process to the preparation of bleaching solutions from chlorine and caustic soda solution is described (cf. B., 1925, 628).

E. S. KREIS.

Preparation of stable, dispersible, dry material. E. TRUTZER (G.P. 422,803, 10.9.20).—The material, mixed with a dispersion medium, is ground in a colloid mill until a disperse system of colloidal character is obtained. The medium is then removed by evaporation in an atomising and drying apparatus, and the material recovered as a dust-free dry powder. The process can be used for the preparation of dry colloidal coal.

A. B. MANNING.

Rotary dryer. W. A. HARTY and F. W. MOORE (Re-issue 16,265, 16.2.26, of U.S.P. 1,531,438, 31.3.25. Appl., 16.10.25).—See B., 1925, 577.

Obtaining powders of great fineness. E. PODSZUS (U.S.P. 1,573,017, 16.2.26. Appl., 1.7.22).—See E.P. 183,134; B., 1923, 385 A.

Separation of gaseous mixtures by liquefaction and rectification. E. JORDAN, Assr. to SOC. L'AIR LIQUIDE (U.S.P. 1,574,425, 23.2.26. Appl., 24.4.23).—See E.P. 197,302; B., 1923, 961 A.

Filter. Filtering apparatus. H. S. COE, Assr. to CYCLE Co. (U.S.P. 1,574,556—7, 23.2.26. Appl., 13.8.19 and 26.1.20).—See E.P. 241,453; B., 1925, 975.

Powdered fuel [boiler] furnaces. C. HOLD (E.P. 247,858, 19.9.25).

Electrical gas purification (G.P. 422,619).—See XI.

Filter (U.S.P. 1,568,001).—See XIX.

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

Effect of sulphur in the briquetting of sub-bituminous coal. H. K. BENSON, J. N. BORGLIN, and R. K. ROURKE (Ind. Eng. Chem., 1926, 18, 116—117).—The carbonisation for 8 hr. at 700—950° of a mixture in the form of small briquettes containing 3% of crude sulphur, 8% of California asphalt, and 89% of a sub-bituminous coal from Tono, Washington, which was ground to pass 20-mesh, yielded a firm, coke-like mass which, when burned with a forced draught, retained its shape until entirely consumed. The sulphur content of the carbonised residue was found to be about 2.25% after cooling in the retort and about 1.40% after quenching in water. The briquetted mixture also retained its shape while

burning without previous carbonisation. Mixtures of coal and sulphur without the asphalt binder did not coke on carbonisation and mixtures of coal and binder without sulphur produced a friable coke which fell to pieces on burning.

A. W. HOTHERSALL.

Decomposition of humic acids at 100°. W. ELLER and A. SCHÖPPACH (*Brennstoff-Chem.*, 1926, 7, 17—20).—Humic acids decompose with evolution of carbon dioxide and water at temperatures above 80° (cf. A., 1923, i, 542). The amount of decomposition occurring at 100° has been determined for three humic acids, one extracted from a humic coal, and two artificial humic acids prepared respectively from quinol and sucrose. During preparation the temperature of the material was not allowed to rise above 80°, and the humic acids were dried first in air at 80° and then *in vacuo* at 66° to constant weight. This final drying required about 28 days. Between 1 and 2 g. of the dry material was kept at 100° in a slow current of air, the carbon dioxide and water evolved being collected and weighed at 4-hr. intervals over a total period of 52 hrs. Progressive decomposition of all three humic acids occurred, resulting in the evolution of 6—11% of water and 0.6—1.7% of carbon dioxide in 52 hrs., calculated on the original weight of dry material.

A. B. MANNING.

Examination and evaluation of coking coals.

R. KATWINKEL (*Gas- u. Wasserf.*, 1926, 69, 145—150).—The shortcomings of existing methods of determining the caking index of coal are indicated. A method of obviating them consists in subjecting a coke-button prepared from a mixture of coal and sand to a crushing force which is regularly increased by allowing lead shot to fall at a standard rate into a vessel supported on a piston resting on the coke-button. The flow of lead shot is interrupted when the button is fractured, and the amount of shot used is a measure of the caking power of the coal, due allowance being made for uncoked portions of the crucible charge. The results are interpreted in terms of Fischer's theory of coking (cf. B., 1925, 233), which, however, cannot be reconciled with coke-oven results. The influence of size of both sand and coal on caking power are discussed and methods of testing coke are described briefly.

R. A. A. TAYLOR.

Coke production. E. W. SMITH, T. C. FINLAYSON, H. M. SPIERS, and F. S. TOWNEND (*Gas J.*, Coke Number, Nov. 9, 1925, 3—16).—Only for combustion in open grates is the ignitability of coke of any importance; consequently for most purposes (1) suitable grinding and grading, (2) low moisture content, (3) low ash and sulphur contents, and (4) satisfactory combustibility and reactivity, satisfy the requirements. The terms combustibility and reactivity should be restricted to mean respectively combustion with oxygen and reaction with substances other than oxygen. The rate at which coke is consumed in a stove of given size under a constant air blast and starting with a fixed initial temperature is a measure of the combined combustibility and reac-

tivity. Generally this should be high, but for foundry work, where high combustibility and low reactivity are desired, an independent test for reactivity, such as the determination of the rate of reduction of carbon dioxide by the coke maintained at 900°, is necessary. The most important factors influencing the combustibility and the reactivity of coke appear to be its thermal treatment during and after coke formation, its physical properties, and its content of volatile matter. The four methods of operating carbonising plant to give the desired coke discussed are: the blending of coke dust with coal; carbonising blends of caking and non-caking coals; grinding, mixing, and compressing coals prior to carbonisation; and carbonising briquetted blends of coals in vertical retorts. With the adoption of these processes it becomes more necessary to clean the coals. The additional expense involved in the application of these processes would be more than counterbalanced by the enhanced value of a better and more uniform coke, the absence of breeze, greater throughput for a given plant, and the possibility of utilising cheaper coals. By further reducing the time of carbonisation a smokeless fuel containing sufficient volatile matter to render it easily ignitable in an open grate could be produced by these processes even at high temperatures. In the methods of testing coke prominence is given to Rose's method of examining coke structure (cf. B., 1925, 834).

S. PEXTON.

Ignition temperature and reactivity of carbonisation products. K. BUNTE (*Z. angew. Chem.*, 1926, 39, 132—138; *Gas- u. Wasserf.*, 1926, 69, 192—195, 217—218).—The close interdependence of the ignition temperature and reactivity towards carbon dioxide of a variety of carbonisation products (wood charcoal, semi-coke, gas coke, oven coke, coke breeze, graphite), in sizes from 0.5 mm. to 5 mm., is emphasised and illustrated graphically. The ignition temperature is determined by passing a regulated current of air through a column of the material, the temperature of which is gradually raised by means of an electric furnace, and is taken as the temperature at which a thermocouple placed just above the top of the column indicates a sudden rise in temperature. The ignition temperature is raised by increasing the size of the particles of material, and lowered by increasing the air velocity or the oxygen content of the air. It also varies inversely as the adsorptive capacity of the material, *i.e.*, as the surface area exposed per unit mass. The reactivity is measured by passing carbon dioxide over the heated material and determining the degree of reduction to carbon monoxide at different temperatures. By plotting as ordinates the temperatures at which, for the various materials in question, reduction to carbon monoxide has proceeded to an equal degree, against ignition temperatures as abscissæ, an approximately linear relation is found to hold, which enables the reactivity curve of a material to be deduced from a knowledge of its ignition temperature.

W. T. K. BRAUNHOLTZ.

Gaseous combustion at medium pressures.
I. Carbon monoxide-air explosions in a closed vessel. II. Methane-air explosions in a closed vessel. R. W. FENNING (Phil. Trans., 1926, A, 225, 331—356).—Pressure-time records have been obtained for the explosion of a series of "complete combustion" mixtures of hydrogen, carbon monoxide, and air, from 100% H₂-air to 50% H₂-air+50% CO-air, in a closed vessel at an initial pressure of 76.8 lb./sq. in., and an initial temperature of 50°. The water vapour content of the gas mixtures was less than 1 in 2000. A second series of records was made to show the effect of addition of water vapour, from 0.3 to 2.56 vols. per 100 vols. of gas mixture, on the explosion of CO-air mixtures. In a third series the effect of a constant water vapour addition (1.2% by vol.) to the first series was observed. The apparatus consisted of an electrically heated mild-steel vessel (7 in. diam. by 8 in. long) with a spark gap (½ mm.) at the centre, fitted with a diaphragm manometer and pressure-recording device. In the first series just over 2% of H₂-air in the mixture reduced the "explosion time" (time from passage of spark to production of maximum pressure) from 0.26 sec. to 0.1 sec., and 4% reduced it to 0.076 sec. The explosion time for the CO-air mixture itself varied considerably in different determinations (0.246 to 0.29 sec.), due probably to slight variations in humidity. All mixtures gave practically the same maximum explosion pressure. The interval during which a constant maximum pressure is maintained is of the order 0.004 sec. for CO-air mixtures, and varies from 0.001 sec. to zero as the percentage of H₂-air increases. Addition of water vapour has a marked influence on the explosion time of CO-air mixtures, 0.3% by vol. reducing it from 0.29 to 0.16 sec. The same addition of water vapour raises the maximum explosion pressure by more than 2%, whilst further addition up to 2.56% adds only another 1%. Almost identical records were produced by addition of 1.21% of water vapour and of 2.1% of H₂-air respectively to the CO-air mixture. Addition of water vapour in the third series reduces the explosion time when the mixture contains less than 8% of H₂-air, but slightly increases it for greater percentages. Addition of water vapour to all mixtures in which H₂-air exceeds about 8% reduces the maximum pressure by slightly under 1%. Pressure-time records of explosions in methane-air mixtures at an initial pressure of 95 lb./sq. in. and an initial temperature of 100° show that with increase in the methane-air ratio the maximum pressure passes through a maximum at 9.7—10.5% of methane, and the "explosion time" passes through a minimum at 9.7% of methane. Complete records were made for a 9.9% mixture at initial temperatures from 24° to 400°, and at initial charge densities over a range corresponding to pressures from 2 to 5 atm. at 15°. The explosion time is increased by (1) increasing the charge density, (2) lowering the initial temperature, (3) adding a diluent such as water vapour or combustion products. The rise in pressure increases linearly with initial pressure for constant initial temperature.

The effect of the variation of specific heat with temperature is apparently masked by other factors.

A. B. MANNING.

Desulphurisation of gas. A. THAU (Gas- u. Wasserf., 1926, 69, 125—128).—The history and statistics of dry purification of gas are reviewed and particulars of American practice for removal of hydrogen sulphide by washing with sodium carbonate solution are given. The disadvantage of this wet purification process is its incompleteness and an improved dry process due to Raffloer is described. Dry iron oxide in granular form is fed from hoppers over louvres through which gas leaving the purification system passes. The oxide then falls to the bottom of an incline, whence it is blown by a stream of by-passed gas up a tower down which the main gas stream passes on its way to the vessel via which the oxide enters the plant. The shower of oxide falls down the tower and collects on an inclined false bottom from which it is constantly withdrawn through a self-sealing valve and returned to the feed. Practically no trace of hydrogen sulphide leaves the plant, and the cost per year of treating 50,000 cub. m. of gas per day is M. 30,000, as against M. 275,000 by the ordinary static process. One cub. m. of "Lux" purifying material is capable of purifying 50,000 cub. m. of gas and can be worked up to 70% of sulphur in the fouled oxide.

S. PEXTON.

Holzheim shale and shale oil. K. NEUBRONNER (Petroleum, 1926, 22, 57—61).—The oil shale is found in two layers of about 1 m. and 3 m. thickness, respectively. It quickly weathers and crumbles when exposed to the air. The upper layer yields 6—7% of oil, the under layer 1—3%. The mineral is a blue-grey to blackish-blue clayey marl, *d* 2.2—2.3, contains 1—3% of pyrites, and has a content of 16.1% of organic matter in the lower layer. The residue is basic, contains 15—17% CaO, and is used in the manufacture of cement. The shale after crushing is distilled in an internally-heated retort in the presence of inert gas. 200 Cals. are required for the distillation of 1 kg. of shale, and 70—80% of the oil is recovered. The oil, *d* 0.960—0.965, is dark brown, of an unpleasant odour due to sulphur compounds, and has a large content of unsaturated hydrocarbons. The content of aromatic hydrocarbons is small, of basic substances 0.5%, of phenols 1.4%, and of paraffin wax 0.7—0.8%; 66% distils over up to 360°. The elementary analysis shows carbon 82.6% and hydrogen 10.1%. H. MOORE.

Chemical composition of Rumanian petroleum oils. N. DANAILA (Petroleum, 1926, 22, 209—210).—The determination of unsaturated hydrocarbons was carried out by the direct method by means of ozone (cf. B., 1924, 857). The aromatic hydrocarbons were determined by the use of sulphuric acid of concentration 98.33% H₂SO₄. In the fraction 65—150° (11.31%), *d* 0.7542, there were found 1.46%, 20.42%, and 78.12%; in the fraction 65—95° (3.41%), *d* 0.7294, 1.65%, 17.50%, and 80.85%; in the fraction 95—125° (4.99%), *d* 0.7594,

1.39%, 20.92%, and 77.69%; in the fraction 125—150° (2.91%), d 0.7728, 1.22%, 23.98%, and 74.80%; and in the fraction 150—300°, 1.64%, 16.89%, and 81.47% respectively of unsaturated, aromatic, and saturated hydrocarbons. H. MOORE.

Action of petroleum-refining agents on naphtha solutions of pure organic sulphur compounds. A. E. WOOD, C. SHEELY, and A. W. TRUSTY (Ind. Eng. Chem., 1926, 18, 169—171).—The refining agents used were sulphuric acid (fuming, d 1.84, and d 1.58), sodium plumbite, sodium hydroxide, silica gel, fuller's earth, and alumina. Stock solutions of sulphur derivatives (*iso*amyl mercaptan, hydrogen sulphide, methyl sulphate, methyl *p*-toluenesulphonate, carbon disulphide, *n*-butyl sulphide, *n*-propyl disulphide, thiophen, diphenylsulphoxide, *n*-butyl sulphone, and free sulphur) were made in a sulphur-free petroleum distillate, d ²⁰ 0.77. The general procedure was to treat 50 c.c. of the stock solution with two quantities of the reagent (4 and 16 c.c. respectively) for a period of 1 hour, the desulphurising efficiency being measured by the decrease in sulphur content after treatment. Under these conditions sulphuric acid of all strengths was quite ineffective towards free sulphur or carbon disulphide, but the other sulphur compounds were removed fairly easily except in the case of the weaker acid. Sodium plumbite removed hydrogen sulphide and alkyl sulphate, and to a certain extent the mercaptans as lead mercaptides. Sodium hydroxide behaved similarly to sodium plumbite. Silica gel was effective in most cases, except for free sulphur, hydrogen sulphide, carbon disulphide, and thiophen. Fuller's earth and alumina behaved similarly and were less effective than silica gel. The action of the last three agents is due to adsorption only. The effects of each agent on each sulphur compound are tabulated. S. BOWMAN.

Adsorption studies on decolorisation of mineral oils. T. H. ROGERS, F. V. GRIMM, and N. E. LEMMON (Ind. Eng. Chem., 1926, 18, 164—169).—Working with three widely different petroleum products—kerosene, wax, and naphtha solution of cylinder stock—the authors have studied the behaviour of various adsorbents such as clays, activated charcoal, and inorganic gels. The oil was agitated with the adsorbent in definite proportions at room temperature for definite periods, and after filtration colour determinations were made. For colour measurement and expression of results the true colour method of Parsons and Wilson (B., 1922, 402 A), using the Duboscq instrument, was adopted. Applying Freundlich's equation, $x/m = KC^{1/n}$, where x is the amount adsorbed by m g. of adsorbent, C the equilibrium concentration, and n and K are characteristic constants, the logarithmic values of colour units removed were plotted against equilibrium colour and gave a series of characteristic curves for each product. The use of this equation affords a convenient method of evaluating the decolorising properties of adsorbents. The colour formation in cracked distillates when treated with certain adsorbents (Dunstan, Thole,

and Remfry, J.S.C.I., 1924, 179 T) is due partly to polymerisation and partly to oxidation.

S. BOWMAN.

High-vacuum distillation of mineral oils. L. STEINSCHNEIDER (J. Inst. Petrol. Tech., 1925, 11, 514—523).—The distillation of petroleum is not accompanied by serious decomposition till the temperature exceeds 300—320°. Above this temperature decomposition rapidly increases, and affects the quality of the lubricating oil fractions. The boiling temperature may be lowered by the use of steam or by diminished pressure. The optimum amount of steam is 1.65 pts. per 1 pt. of oil distillate, but in practice about 3.5 pts. are used. Higher temperatures prevail in bottom-heated stills than in those with internal flue, in consequence of the height of the column of oil above the heating surface. Plant for vacuum distillation at absolute pressures as low as 5 to 25 mm. operates with satisfactory results. The author advocates high vacuum, minimal use of steam, active circulation, the avoidance of re-distillation and, as far as possible, of chemical treatment of the distillates. Vacuum distillation is particularly applicable to inferior heavy asphaltic oils. It effects an economy in fuel of 50%, an extra yield of 4% in distilling and 4—6% in refining, and a saving of 50—75% in sulphuric acid.

H. MOORE.

Refining of mineral oils. H. I. WATERMAN (J. Inst. Petrol. Tech., 1925, 11, 576—582).—The presence of sulphur or halogens in oils leads to the separation of dark substances containing carbon. Unsaturated substances are not, however, as harmful in this respect as was once thought. Free or combined sulphur causes kerosene to burn with a smoky flame, as also do halogen compounds, such as ethyl bromide. Kerosene heated to 240° for two days in a Carius tube with ethyl sulphide became deep brown in colour. Kerosene was attacked by chlorine at room temperature with evolution of hydrogen chloride, and after heating had the consistency of asphalt and contained free carbon. Paraffin wax of m.p. 50.6° was cracked under pressures varying from 7 to 219 atm. The content of unsaturated compounds in the product was greater in proportion as the pressure was lower; least at the highest pressure in the presence of hydrogen. Extraction with liquid sulphur dioxide divides the compounds present in petroleum into two groups, which may be subjected to separate refining. In a petroleum fraction of d 0.86—0.92, treatment with 54% of silica gel reduced the sulphur content by 20%, whilst the use of 114—117% of gel by weight brought about a reduction of 50—70%. In the use of the hypochlorite process it is important to regulate the quantity of free alkali present.

H. MOORE.

Edeleanu process [of petroleum refining]. H. I. WATERMAN and J. N. J. PERQUIN (J. Inst. Petrol. Tech., 1925, 11, 560—570).—Kerosene distillates, transformer oils, Russian and medicinal oils, were treated by the Edeleanu process with

liquid sulphur dioxide. In the case of kerosenes this treatment was followed by treatment with 0.5% of sulphuric acid and 2% of oleum, with excellent results. Sulphur dioxide has great desulphurising power, the sulphur content of kerosenes being reduced from 0.29% to 0.019%, from 0.32% to 0.01%, and from 1.13% to 0.038% respectively by the above treatment. Silica gel treatment and the hypochlorite process did not remove sulphur and unsaturated constituents to the same extent. Treatment with 2% and 5% of sulphuric acid *d* 1.84, and refining with silica gel does not considerably raise the aniline point (cf. Tizard and Marshall, J.S.C.I., 1921, 20 T). Sulphur dioxide treatment raises it, *e.g.*, from 52° to 65.2°. This treatment gives favourable results as regards colour, and colour after exposure to a quartz mercury vapour lamp. By sulphur dioxide and acid treatment the sludge value of transformer oil was reduced from 1.3% to 0.5%. Sulphur dioxide may also be used in refining vegetable and animal oils and fats. H. MOORE.

Separation of the components of petroleum.

Bromination of Persian petroleum fraction, b.p. 60–80°. P. F. GORDON, D. BAIRD, and T. G. HUNTER (J. Roy. Tech. Coll. Glasgow, 1925, [2], 53–63).—When the fraction of Persian petroleum boiling between 60° and 80° is treated with bromine, drop by drop, in the presence of an excess of iron at 16° smooth bromination occurs without any side-reactions. The product is a plastic mass, the bulk of which dissolves in ether leaving a white crystalline residue. After distillation of the ether solution the residual liquid separates into two immiscible layers, and a small amount of a white crystalline substance (m.p. 164°) is precipitated. The heavy liquid has the empirical formula $C_5H_7Br_3$ and the lighter liquid the formula $C_6H_{10}Br_4$. Both liquids are viscous and have a tendency to decompose with evolution of hydrogen bromide. The ether-insoluble crystals can be fractionated from ethylene dichloride into five crystalline products having the following melting points in order of increasing solubility: 293.5°, 273.5°, 299.8°, 283°, and 305°. The second and third have the empirical formulæ $C_7H_6Br_4$ and $C_5H_4Br_4$ respectively. All five compounds are soluble in carbon disulphide and in acetone and are not decomposed by an alcoholic solution of potassium hydroxide. The first bromide on shaking with benzoyl chloride and pouring the product into water containing a little sodium carbonate, gives a white crystalline precipitate, soluble in water, alcohol, and ether. After recrystallisation from alcohol it melts at 120°. A. R. POWELL.

Extension of method of critical temperatures of solution to analysis of petrols. M. AUBERT and E. AUBREY (Compt. rend., 1926, 182, 577–579; cf. Simon and Chavanne, A., 1919, ii, 267, 432, 433).—In the earlier work the temperature above which mixtures of aniline and hydrocarbons yielded homogeneous mixtures no matter what the composition, served as a basis for summary analysis, *i.e.*, the evaluation of percentages of aromatic, saturated cyclic and acyclic hydrocarbons present in a given

petroleum. The method necessitated the nitration of aromatic substances and re-determination of the critical temperature of solution. It has now been shown that benzyl alcohol may be substituted for aniline and similar data obtained. By means of solving simultaneous equations obtained by using aniline and benzyl alcohol the composition of petrol may be determined without recourse to nitration.

R. A. MORTON.

Ignition of carburetted mixtures [motor fuels] by adiabatic compression. A. PIGNOT (Compt. rend., 1926, 182, 376–377; cf. A., 1907, ii, 18, 946; J.C.S., 1914, 2027–2036).—Mixtures of dry air and *n*-heptane, *n*-hexane, *n*-octane, cyclohexane, benzene, toluene, *m*-xylene, mesitylene, and ethyl alcohol have been subjected to a fixed volumetric compression and the initial temperature necessary for ignition has been determined. By plotting ignition temperature against percentage of hydrocarbon or alcohol, curves are obtained which sharply differentiate between aromatic and saturated hydrocarbons. With a volumetric compression of 9 benzene and absolute alcohol give similar curves. Increasing amounts of water in the alcohol result in a flattening of the curve and the disappearance at 40% of water of the minimum which is observed in this and other cases; cyclohexane–benzene mixtures show no anomalies in the curves. Addition of 10% of toluidine or ethyl sulphide (which are known as very active anti-detonators) to cyclohexane exerted negligible effects on the characteristic curves.

R. A. MORTON.

Report of Surgeon General's committee [U.S. Public Health Service] on lead tetraethyl. (Ind. Eng. Chem., 1926, 18, 193–196).—Some two hundred men were examined to determine whether lead tetraethyl blended with petrol had any effect as regards lead absorption. The average time of exposure to the substance was two years. The methods employed were the determination of the lead content of faeces of workers, the estimation of the number of stippled red cells in the blood of workers, and the measurement of the strength of the extensor muscle of the forearm. Drivers of cars using the blended fuel showed no signs of lead absorption. Garage workers handling the fuel showed very slight signs of lead absorption, but these were extremely small compared with the effects on workers known to be exposed to lead poisoning risks in other industries. The committee concludes that no reason exists for prohibiting the use of lead tetraethyl but proposes to prolong its investigations. W. N. HOYTE.

Potential public health hazards from the use of "ethyl-gasoline." F. B. FLINN (J. Ind. Hyg., 1926, 8, 51–66).—Absorption of lead tetraethyl occurs when "ethyl-gasoline" is applied to the skin of rabbits, guinea pigs, and goats, and also when animals are exposed to the fumes resulting from the evaporation of the gasoline in a gas chamber; similar results were obtained when the substance was allowed to evaporate in the open air. Exposure to the vapour results in more rapid absorption than does

application to the skin, and the rate of excretion of the lead is less than the rate of absorption.

H. J. CHANNON.

Portable combustion apparatus for detection and measurement of small quantities of petroleum vapour. C. A. NEUSBAUM, P. L. DE VERTER, and E. W. DEAN (*Ind. Eng. Chem.*, 1926, 18, 183—185).—General requirements and known standard apparatus for the measurements are discussed. The authors describe an apparatus depending on the shrinkage in volume of a known volume of air and petroleum vapour after exposure to a red hot platinum wire. The apparatus is calibrated by means of mixtures of known composition. W. N. HOYTE.

Cracking solid paraffins with formation of low-boiling products, using activated carbon. H. HERBST (*Z. angew. Chem.*, 1926, 39, 194—196).—A solid paraffin (m.p. 50—52°), distilled in contact with activated carbon at about 300° under atmospheric pressure, yielded 22% of a light oil, b.p. 45—150°, 13.4% of a heavier oil, b.p. 150—200°, and 21.2% of oil of b.p. 210—300°, reckoned by weight on the solid raw material. Further quantities of light oil could be obtained by subjecting the oily distillate distilling above 300° and the unchanged paraffin to a further distillation with activated carbon, giving a total yield of 27% of light oils distilling below 150°. This compares very favourably with the yield of 4—12% obtained by cracking crude petroleum. The process can be carried out at atmospheric or reduced pressure for paraffins boiling above 300°, which simplifies the plant required in practice, while no appreciable quantities of coke or asphalt are produced, thereby prolonging the life of the equipment. Materials with a b.p. below the decomposition temperature must be distilled under a pressure (10—25 atm.) sufficient to raise the b.p. to the required temperature, generally about 300°. B. W. CLARKE.

Lubricating oil for electricity meters. D. HOLDE and R. SCHACHENMEIER (*Petroleum*, 1926, 22, 161—168).—Types of meters and the characteristics of a satisfactory lubricating oil are discussed. A viscosity of 20° Engler and an iodine value of 6 are suggested as upper limits. Experiments are being continued as regards volatility, resinification, oxygen absorption, and surface tension.

W. N. HOYTE.

Determination of the melting points of asphalts. I. J. LAGERQVIST and M. FREDMAN (*Svensk Kem. Tidskr.*, 1925, 37, 266—276; *Chem. Zentr.*, 1926, I., 1339—1340).—The Kraemer-Sarnow method of determining the m.p. of asphalts (*B.*, 1903, 291) gives discrepant results with only slight changes in the experimental conditions. The modification due to Barta (*B.*, 1911, 1367) is recommended. A uniform rate of heating of 1° per min. should be used. The time allowed to lapse between filling the tube and making the determination, and cooling of the sample after filling the tube have little influence on the result. The apparent m.p. rises linearly with the depth of the layer of material in the tube. The internal diameter of the tube should not vary more than ± 0.2

mm. from the standard (6.5 mm.). The quantity of mercury used has considerable influence, and should therefore be accurately measured.

A. B. MANNING.

Reactivity of coke. J. P. AREND and J. WAGNER (*Fuel*, 1926, 5, 106—116).—See *B.*, 1924, 973.

See also A., Mar., 246, **Reaction provinces (JORISSEN)**. 250, **Union of carbon monoxide and oxygen in contact with nickel, copper, and their oxides (BONE and ANDREW)**. 267, **Detection and determination of gaseous olefines (SOROKIN and BELIKOV)**. 286, **Naphtenic acids (NAPHTALI)**.

Expansion of pitches and asphalts. MALLISON JACOBSON, and SARRE.—See III.

Petroleum insecticides. GRAY and DE ONG.—See XVI.

PATENTS.

Manufacture of artificial fuel. E. C. MARCESCHE (E.P. 239,474, 27.11.24. Appl., 8.9.24).—Anthracene or naphthalene oil or paste is atomised by means of a jet of air and steam and projected on to a mixture of fuel and pitch binder, or on to the pitch binder alone, the pulverised fuel being added subsequently. The mixture is briquetted.

R. A. A. TAYLOR.

Method of treating coal products. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,570,103, 19.1.26. Appl., 23.9.19. Renewed 18.7.25).—Coal, in a very finely divided state, is mixed with a fluxing agent, and fed into a moving fluid current. The mixture is conveyed through a heating zone, in which the volatile by-products are removed from the coal and the ash unites with the fluxing agent. The carbon content of the coal, free from its volatile matter, is then collected.

A. B. MANNING.

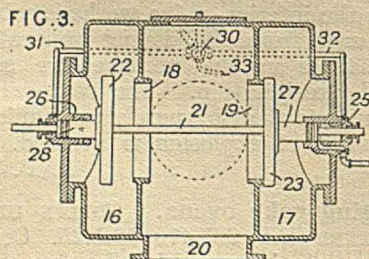
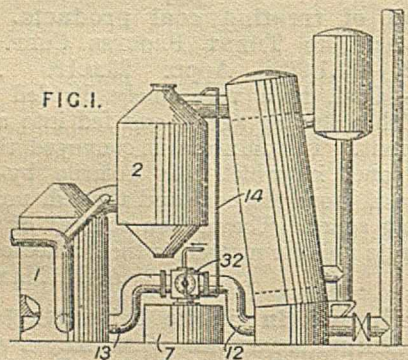
Dust removal in lignite briquetting works. SIEMENS-SCHUCKERTWERKE G.M.B.H., Asses. of C. HAHN (G.P. 422,148, 16.3.22).—The dry dusty air from the interior of the works is mixed with the moist fumes from the drying apparatus, and the solid particles in the mixture are separated by electrical precipitation. The removal of dust is more complete than by purely mechanical processes, and the risk of explosion is avoided.

A. B. MANNING.

Charging coke ovens. COLLIN UND Co., and J. SCHAEFER (E.P. 227,470, 12.1.25. Conv., 12.1.24).—A dense coke is formed by charging coal into ovens from a greater height than usual, by suitably disposing the hoppers, or by providing a hopper which can be raised. The charge openings form such a large proportion of the cross-section of the ovens that the coal loses none of its momentum in passing through and packs tightly in the ovens. In addition fireclay slabs connected to the under side of the charging doors in such a way that they are free to move vertically, bear down upon the coal charge during coking. S. PEXTON.

Retorts [for carbonising]. MERZ AND MCLELLAN, and E. G. WEEKS (E.P. 246,319, 4.2.25).—An apparatus for charging and levelling the fuel in vertical retorts is described. It consists of a rotary frame carrying fixed scrapers in a horizontal plane at the upper level of the fuel. One scraper is plough-shaped and throws part of the fuel over against the retort wall and the remainder inwards to the centre of the retort. The second scraper is separated from the first by an angle approximating to 180° and describes a circle of smaller radius than the plough. An inclined charging shoot which is carried by the frame and into which fuel is fed by a worm, through a gas-tight roller valve, drops coal into the retort in advance of the plough scraper. The fuel height is indicated by a pointer connected by a lever system to a metal flap which is connected to the rotating frame like a flag which trails over the fuel surface. S. PEXTON.

Apparatus for manufacture of water-gas. HUMPHREYS AND GLASGOW, LTD., Assees. of C. S. CHRISMAN (E.P. 245,688, 19.8.25. Conv., 18.4.25).—An automatically operated gas valve obviating the use of all hot valves in water-gas manufacture, has three gas connexions, one, 20 (Fig. 3), to the wash-box on which it stands and the other two, 13 and 12, to the bottom of the generator and waste-



heat boiler outlet respectively. The direction of gas flow is governed by the position of a common spindle, 21, carrying two disc valves, 22 and 23. The spindle is moved by the steam pressure acting on one or other of the two pistons, 26 and 27. A hand-operated four-way steam cock, 30 (Fig. 3), controls the supply of steam to the plant. When the cock, 30, is in the left-hand position shown in Fig. 3, steam travels along 32, 25, and 14, and through the hot chequered chamber to the top

of the water-gas generator and down through the latter. Valve, 23, remains seated because of the differential pressure between the top and bottom of the generator, and the water-gas made enters the wash-box via 13, 18, and 20. When the cock, 30, is in the right-hand position steam passes along 31, moves the piston and valve spindle to the right until when valve, 22, is closed, steam enters 16 and passes via 13 to the bottom of the generator. Valve, 22, remains closed because of the differential pressure across the fuel bed, and the water-gas made having passed the chequered chamber and waste-heat boiler enters the wash box via 12 and 20. S. PEXTON.

Apparatus for purifying gas. H. T. GRAFTON, C. W. WEESNER, and F. J. HOFFMAN (U.S.P. 1,572,599, 9.2.26. Appl., 30.3.21).—The gas is passed into a preheater arranged in a furnace so as to be heated by the furnace gases, and is delivered therefrom to purification chambers suspended from the furnace roof. T. H. BURNHAM.

Purification of coke-oven gas. SOC. ANON. DES ANC. ÉTABL. EGROT ET GRANGÉ (F.P. 593,565, 28.4.24).—The gases are led up through a vertical scrubber down which passes cold ammonia liquor containing tar. The gases extract the ammonia and tar, and pass then through a system of condensers where the tar, water, and part of the ammonia are separated. The liquor separating in the condensers is treated with lime and returned to the scrubber. A. B. MANNING.

Separation of condensable products [from distillation gases] by electrical precipitation. SIEMENS-SCHUCKERTWERKE G.M.B.H. (G.P. 422,121, 13.12.21).—Distillation gases are led in succession through a series of electrical precipitation chambers, which are maintained at temperatures corresponding to the dew point or condensation temperature of the products to be separated therein. Before entry into each chamber, the gas is mixed with some of the product previously condensed therein. This forms a simple means of maintaining the required temperatures in the chambers. A. B. MANNING.

Gas purifying agent capable of regeneration. SIRIUS WERKE A.-G., and J. BRUNNER (G.P. 422,630, 14.3.25).—Pipe-clay impregnated with oil is used as a purifying agent for gas, in particular for the removal of naphthalene. A. B. MANNING.

Purifying agent for acetylene. CHEM. FABR. GRIESHEIM-ELEKTRON (F.P. 596,840, 16.4.25).—Basic calcium or magnesium hypochlorite, mixed with water and a binding material, is used as a purifying agent for acetylene. A. B. MANNING.

Ascertaining the proportion of a constituent in a fluid. [Determining carbon dioxide in flue-gas.] E. X. SCHMIDT and U. O. HUTTON, ASSRS. to CUTLER-HAMMER MANUF. Co. (U.S.P. 1,569,682, 12.1.26. Appl., 30.1.24).—The ends of a conducting wire, the middle portion of which is

coiled over a roller, pass down centrally through two vertical tubes placed side by side and are maintained in a state of tension by springs at the lower ends of the tubes. Air is passed up one tube and the flue-gas under examination up the other, both gases being first brought to the same temperature, and either dried, or saturated with water vapour. The wire is heated by passing an electric current through it. Owing to the relatively low heat conductivity of carbon dioxide, the portion of the wire in the tube through which the flue-gas is passing becomes hotter than the portion in the other tube and expands, in consequence, to a greater extent, so that its tension becomes less than that of the other portion. The roller turns to equalise the tensions, and by affixing an indicator to the roller and noting its movements a measure can be obtained of the percentage of carbon dioxide in the flue-gas. T. S. WHEELER.

Motor fuel. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING, Assees. of P. ROTH (G.P. 421,814, 18.12.23).—The acetals of aldehydes and alcohols, in particular those of acetaldehyde and formaldehyde, or of ethyl and methyl alcohols, are used as motor fuels, either alone or mixed with other organic liquid fuels. They form a substitute for benzene or benzene of about equal value. A. B. MANNING.

Cracking hydrocarbon oils. H. WOLF (E.P. 215,336, 16.4.24. Conv., 1.5.23).—Heavy hydrocarbon oil is passed at a pressure of 20–300 atm. through a steel coil in which it is subjected to a temperature of 400–450° for 30–80 secs. The cracked vapours are expanded through a valve into a fractional condensing apparatus, in which the oil to be treated is preheated directly or indirectly. Any high-boiling oil separated is returned to the cracking apparatus. The apparatus is rapid and safe in action, and there is little production of coke and gas. In one experiment 115 litres of oil per hr. were passed at a linear speed of 2.5 m. per sec. under a pressure of 300 atm. through a coil in which the oil was heated to 430° for 50 sec., when 33 litres of benzene boiling up to 150° were obtained. Linear oil speeds of at least 1 m. per sec. are necessary to prevent deposits in the coil. T. S. WHEELER.

Cracking hydrocarbons. F. B. KOONTZ, Assr. to COSDEN AND CO. (U.S.P. 1,567,062, 29.12.25. Appl., 8.5.24).—Petroleum oil is passed at a pressure of about 200 lb./in.² through a series of pipes heated to 470°, and the cracked vapours are led directly into a long cylinder in which carbonaceous matter and high-boiling oil are separated. The uncondensed vapours pass up a long vertical pipe, where any entrained carbon separates and falls back into the cylinder, and thence into a reflux condenser. The vapours coming from the top are cooled by cold oil to be treated, the condensing action being controlled so that about 75% of the vapours passing to the condenser are liquefied and withdrawn from the bottom. While in the condenser they are mixed with 33% of their weight of fresh preheated oil from the top cooler. The mixture is passed back to the cracking apparatus. The vapours from the cooler on

top of the reflux condenser are condensed and recovered. The continuous removal of carbonaceous matter in the cylinder, whence it is discharged at intervals, enables the apparatus to be run continuously for long periods without cleaning.

T. S. WHEELER.

Distilling and blending hydrocarbons. D. L. NEWTON (U.S.P. 1,567,457–8, 29.12.25. Appl., 19.7.24).—(A) An absorbing medium containing hydrocarbons from casing-head gas is treated for the production of a stabilised motor fuel by partial distillation, followed by repeated counter-current contact between the liquid medium and a mixture of the distilled vapours with steam. (B) An apparatus for the above purpose comprises a tower with a distilling chamber at the top and a succession of combined distilling and blending chambers below, each of the latter being provided with perforated bottoms having perforated re-circulation tubes over the perforations. From the distilling chamber at the top the liquid runs downwards step by step through the tower. The vapour is mixed with steam and conducted to the bottom of the tower, up which it passes step by step, being exhausted just below the distilling chamber. B. M. VENABLES.

Oil [cracking] still. J. PRIMROSE, Assr. to POWER SPECIALTY Co. (U.S.P. 1,568,182, 5.1.26. Appl., 2.7.21).—Oil is cracked by passing it through a pipe coil heated in a cracking chamber which is partially separated from the combustion chamber of the furnace by a bridge wall. The oil after passing through the coil in the cracking chamber passes through a series of pipes placed just inside the roof of the cracking chamber and then through a series of pipes built into the roof of the combustion chamber. The temperature of the roof is thus reduced and overheating of the cracking coil with consequent carbonisation of the oil, due to radiation of heat from the roof, is minimised. T. S. WHEELER.

Refining mineral oils. H. STINNES-RIEBECK MONTAN-U. OELWERKE A.-G. (G.P. 421,858, 4.12.24).—Mineral oils are refined by the action of tin tetrahalides in a finely divided or diluted form. The tin tetrahalide may be used precipitated on a finely divided material. After the reaction a basic material is added, which serves to remove the acid formed and to bleach the oil. By this process too rapid a reaction and the consequent formation of gums is avoided. A. B. MANNING.

Fuel briquettes. J. F. O'DONNELL (E.P. 247,272, 10.11.24).—See U.S.P. 1,557,320; B., 1925, 949. The temperature specified is 800–1200°.

Process of manufacturing fuel. W. E. TRENT (E.P. 228,862, 17.11.24. Appl., 9.2.24).—See U.S.P. 1,512,427; B., 1925, 62.

Pulverising, feeding, and burning fuel. J. CRITES, Assr. to RAYMOND BROS. IMPACT PULVERIZER Co. (U.S.P. 1,573,040, 16.2.26. Appl., 8.3.24).—See E.P. 230,434; B., 1925, 870.

Apparatus for distilling carbonaceous materials. C. A. GRIFFITHS (U.S.P. 1,573,824, 23.2.26. Appl., 31.5.24).—See E.P. 228,749; B., 1925, 275.

Gas producer. C. W. LUMMIS and G. H. ISLEY, Assrs. to MORGAN CONSTRUCTION Co. (Reissue 16,262, 16.2.26, of U.S.P. 1,251,064, 25.12.17. Appl., 13.7.23).—See B., 1918, 117A.

Method and means for using motor fuels. T. MIDGLEY, JUN., AssT. to GEN. MOTORS CORP. (U.S.P. 1,573,846, 23.2.26. Appl., 15.4.22).—See E.P. 196,237; B., 1924, 165.

Distilling volatile products [mineral oils]. L. GRANGER, C. MARILLER, and C. PRACHE (U.S.P. 1,574,622, 23.2.26. Appl., 9.1.20).—See E.P. 154,558; B., 1922, 4A.

Washing minerals or coal. G. RANWEZ (E.P. 237,928, 4.8.25. Conv., 2.8.24).

Charging of coal into coke-ovens. COLLIN UND Co., and J. SCHAEFER (E.P. 234,053, 21.1.25. Conv., 15.5.24. Addn. to 227,470).

See also pages 256, Dispersible dry material (G.P. 422,803). 264, Purification of hydrocarbons (E.P. 246,210); Purification of oils (G.P. 421,909). 276, Bituminous compositions (E.P. 246,548); Bituminous emulsions (U.S.P. 1,567,061). 284, Electrode carbon (G.P. 422,235). 299, Lead tetraethyl (U.S.P. 1,567,159).

III.—TAR AND TAR PRODUCTS.

Determination of the coefficient of cubical expansion of pitches and asphalts. H. MALLISON, F. JACOBSON, and K. SARRE (Z. angew. Chem., 1926, 39, 154).—A flask of about 200 c.c. capacity, carrying a ground-in stopper of elongated, tubular shape, is filled to a mark on the stopper with pure, air-free water at 15° and weighed. It is then emptied, dried, and about 100 g. of the sample are accurately weighed into it, water again being added to make up to the mark. This is done at 15° and 60°, and the flask and its contents are weighed on each occasion. The coefficients of cubical expansion for samples of coal-tar pitch and petroleum asphalt, between 15° and 60°, were found to be 0.00045 and 0.00062 respectively. W. T. K. BRAUNHOLTZ.

PATENTS.

Treatment of low-temperature tar from lignite. SOC. ANON. HYDROCARBURES ET DÉRIVÉS (F.P. 596,143, 3.7.24).—The dry tar is treated with a solvent, such as 90% alcohol, which dissolves the phenols and part of the light hydrocarbons. The mixture is filtered from the residue which contains paraffin, and the filtrate is fractionated to separate the solvent, phenols, and light oils. The crude phenols may be fractionated to give phenol, cresols, xylenols, etc., or may be hydrogenated to cyclohexanols. The light oils are cracked or hydrogenated in the presence of a desulphurising agent,

giving motor fuels similar to benzol. The residue is separated into a paraffin and non-paraffin fraction by treatment with acetone. The crude solid paraffins are pressed and purified by treatment with superheated steam. From the second fraction lubricating and fuel oils are obtained by distillation in vacuum.

A. B. MANNING.

Purification of benzol and similar hydrocarbons. K. COX and P. J. McDERMOTT (E.P. 246,210, 21.10.24).—The refining of benzol, petrol, and other light distillates is carried out by distillation in the presence of an adsorbent material such as bauxite, kaolin, soapstone, and similar micro-porous materials. The adsorbent material, ground to pass a 200-mesh sieve, is placed in the still without any previous treatment, agitation being effected by ebullition or by mechanical means. This combination of distillation and refining is claimed to eliminate the gum-forming constituents without removing the low-boiling unsaturated hydrocarbons. The spent adsorbent earth can be reactivated by roasting or acid treatment. S. BOWMAN.

Purification of oils. F. HOFMANN and M. DUNKEL (G.P. 421,909, 12.12.23).—Mineral and tar oils and low-temperature tars are refined by heating under pressures of less than 100 atm., or without pressure, in the presence of catalysts, or in vessels with catalytically active walls. The products are stable in air and light, and in most cases do not require further treatment with sulphuric acid.

A. B. MANNING.

Recovery of acetone from light oils. ZECHÉ M. STINNES, Asses. of A. WEINDEL and H. KIEMSTEDT (G.P. 421,910, 7.2.24).—Benzol first washings are washed with a quantity of water which is used repeatedly until it has dissolved a suitable quantity of acetone, when the acetone is expelled by steam. The distillate is treated with sodium bisulphite solution, whereby evil-smelling impurities are driven off by the heat of reaction, and the acetone-bisulphite compound is worked up in the usual manner.

L. A. COLES.

Continuous distillation of tars and oils. E. BLÜMNER (U.S.P. 1,573,370, 16.2.26. Appl., 11.4.21).—See G.P. 340,991; B., 1922, 407A.

IV.—DYESTUFFS AND INTERMEDIATES.

Red and green shades of Indanthrene Blue RS and GCD. A. ZIMMERFELDT (Chemicals, 1925, 24, 19—20; Chem. Abstr., 1926, 20, 293).—Experiments are given supporting the view that the redder shade of Indanthrene RS and GCD is caused by the oxidation of the carboxylic hydrogen atom, whilst the greener shade is due to that of the imino-hydrogen atom of the hydrazine group.

A. A. ELDRIDGE.

Effect of dyes used in paper-making on the animal life of water. O. HAEMPEL (Z. Unters. Nahr. Genussm., 1925, 50, 423—426).—Experiments

have been carried out to determine the toxicity to the animal life of water of eleven solutions of dyes used in paper-making. Dilutions of 1 part of dye to 100,000, 500,000, and 1,000,000 parts of water, respectively, were taken and their effects noted daily in each case for 10 days. The dyes have been placed in four classes according to their toxicities, Orange II. and Safranin being harmless, whilst Victoria Blue, Brilliant Green, and Benzol Green are the most poisonous investigated.

J. GRANT.

Four cases of poisoning by chloronitrobenzene and one by acetanilide, occurring in a chemical works. A. RENSHAW and G. V. ASHCROFT (J. Ind. Hyg., 1926, 8, 67—73).—Exposure to trays into which a mixture of *o*- and *p*-chloronitrobenzene was run at 90° and allowed to cool, caused poisoning usually within three days, apparently due to absorption of vapour through the lungs; similar symptoms were observed in a case of acetanilide poisoning, and it is suggested that aniline may be the toxic substance (produced in the former case by the reducing action of the hæmoglobin of the blood), and that the formation of a stable compound with hæmoglobin impairs the oxygen-carrying capacity of the blood.

H. J. CHANNON.

PATENTS.

Manufacture of new [azo] dyestuffs. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (E.P. 246,394, 7.8.25).—Mordant, acid, and direct cotton dyestuffs are obtained from β -naphthylamine-4:5-dicarboxylic acid (known in the free state only in the form of its anhydride), either by coupling it with a diazo compound or by diazotising it and coupling it with a suitable component. The dyestuffs may be converted into yellow, orange, and violet acid or/and mordant dyestuffs containing chromium by treatment with chromium compounds, and green mordant dyestuffs of particular importance are similarly obtained from those dyestuffs produced by coupling β -naphthylamine-4:5-dicarboxylic acid with *o*-hydroxydiazo-compounds. For example, an acid dyestuff which yields brick-red shades on wool very fast to washing, and also to potting when after-chromed, is obtained by coupling (after diazotisation) 8.5 pts. of aniline-4-sulphonic acid with 10.7 pts. of β -naphthylamine-4:5-dicarboxylic anhydride dissolved in 150 pts. of water containing 10 pts. of 30% caustic soda and 10 pts. of soda ash. A green dyestuff suitable for printing on cotton mordanted with chromium or for dyeing fast green shades on wool from an acid bath is prepared from the azo dyestuff obtained from diazotised 4-nitro-2-aminophenol-6-sulphonic acid and β -naphthylamine-4:5-dicarboxylic acid, by boiling 47.6 pts. with 950 pts. of water and chromium fluoride (equiv. to 22.6 pts. of Cr₂O₃), the product being purified by solution in dilute caustic soda, and precipitation by subsequent addition of acetic acid.

A. J. HALL.

Vat dyes of the thioindigo series. A. W. JOYCE, ASSR. to CHEMICAL FOUNDATION, INC. (U.S.P. 1,568,458, 5.1.26. Appl., 28.1.25).—

6-Chloro-2:3-diketo-4-methyldihydro-1-thionaphthen or one of its 2-keto-equivalents is condensed, for example, by heating in a solvent such as glacial acetic acid, benzene, nitrobenzene, or the like, with or without addition of a condensing agent such as zinc chloride or acetic anhydride, with a 3-oxy-1-thionaphthen compound, e.g., its alkoxy- or alkoxy-halogen derivatives, to yield dyestuffs which give on animal and vegetable fibres, in an alkaline hyposulphite vat orange-red to bluish-red shades fast to light, chlorine, acids, and alkalis. These dyestuffs can be further halogenated to give bluer shades. For example, 6-chloro-3-keto-4-methyldihydro-1-thionaphthen-2-oxime is heated in glacial acetic acid at 100° with 4-bromo-3-oxy-6-ethoxy-1-thionaphthen to give a product dyeing soaped cotton in a vat bluish-red shades. For halogenation the dyestuffs are dissolved in sulphuric acid and treated directly with chlorine or bromine.

T. S. WHEELER.

Carbocyanine dyes [infra-red sensitizers]. G. O. GUTENKUNST, ASSR. to EASTMAN KODAK Co. (U.S.P. 1,568,667, 5.1.26. Appl., 29.9.21).—A β -naphthaquinoline alkyl halide is treated in alcoholic solution with a strong base, such as potassium hydroxide or potassium ethoxide, in presence of chloroform, iodoform, or formaldehyde, to give by condensation on itself or with added quinoline or phenanthroline alkyl halide a carbocyanine dyestuff which is of value as an infra-red sensitiser for photographic emulsions. For example, β -naphthaquinoline ethiodide is treated with alcoholic potassium hydroxide containing iodoform, when the corresponding carbocyanine dyestuff separates. It sensitises emulsions down to 740 μ .

T. S. WHEELER.

Manufacturing a perylene vat dye. A. ZINKE and H. SHOEPFER, ASSRS. to F. BENSA (U.S.P. 1,569,111, 12.1.26. Appl., 17.10.23).—1 pt. of perylenequinone is chlorinated in about 20 pts. of nitrobenzene and the chlorine additive product formed, with or without separation from the nitrobenzene, is boiled for 10 min. with 2—10 pts. of aniline or other base, when hydrogen chloride is split off and chloroperylenequinone is formed. It separates on cooling in orange-red needles and dyes cotton in the vat fast brilliant greenish-yellow shades.

T. S. WHEELER.

Vat dyes of the perylene series. COMP. NAT. DE MATIÈRES COLORANTES ET MANUF. DE PROD. CHIM. DU NORD RÉUNIS (ÉTABL. KUHLMANN), and H. PEREIRA (F.P. 589,643, 25.11.24. Conv., 30.11.23).—Bromoperylene are treated with aromatic acids or their halogenides, anhydrides, or salts in the presence of condensing agents such as aluminium chloride. For example, dibromoperylene, m.p. 276—278°, when heated with benzoic acid and aluminium chloride at 150—170° gives a reddish-violet vat dye for cotton; bromoperylene, m.p. 189°, and benzoyl chloride give a blue dye. Dibromoperylene with benzoyl chloride gives a blue, with salicylic acid a brownish-red, with phthalic

acid a brownish-violet, and with naphthoyl chloride a reddish-violet dye. Dinitroperylene with benzoic acid gives a brown dye, with salicylic acid, a greenish-brown. The dye from chloroperylene and benzoic acid is reddish-violet.

A. COULTHARD.

Vat dyes. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING (F.P. 593,117, 9.2.25. Conv., 16.2 and 27.10.24).—Dyes are formed by condensing *p*-hydroxyphenazines, aminophenazines, arylaminophenazines, alkylaminophenazines, or naphthasultam-3:4-phenazines with 1:4-quinones (containing no halogens) or with *o*-hydroxy-1:4-quinones in the presence of substances which combine with water. The dyes formed in this way, may be oxidised with sodium hypochlorite if required. The dye (brown needles) from chloronaphthasultam-3:4-phenazine and 1:4-naphthaquinone dyes cotton and wool in yellowish-brown shades from the vat; treatment with sodium hypochlorite gives a greenish-yellow vat dye. Naphthasultam-3:4-phenazine and benzoquinone give a dye, brownish-red prisms, which dyes cotton in khaki shades. Treatment with hypochlorite results in a dye suitable for the production of lakes. The dye from α -naphthol-3:4-phenazine and benzoquinone, green needles, dyes cotton and wool in fast green shades, that from α -naphthylamine-3:4-phenazine and 1:4-naphthaquinone dyes cotton yellow. The dye from α -naphthol-3:4-phenazine and β -naphthaquinone gives greenish-yellow shades on wool and cotton. Similar dyes are obtained with β -naphthaquinone and naphthasultam-3:4-phenazine, 2-chloro- α -naphthol-3:4-phenazine, or phenyl- α -naphthylamine-3:4-phenazine.

A. COULTHARD.

Manufacture of vat [benzanthrone] colouring matters. J. Y. JOHNSON. From BADISCHE ANILIN U. SODA FABR. (E.P. 247,261, 6.11.24).—See F.P. 589,323; B., 1926, 148.

Green sulphurised dyestuff. E. REBER and J. FRÖLICH, ASSRS. to SOC. OF CHEM. IND. IN BASLE (U.S.P. 1,568,622, 5.1.26. Appl., 9.7.23).—See E.P. 201,540; B., 1924, 976.

Compounds for dyeing (U.S.P. 1,567,731).—See VI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of alkali with special application to wool. H. R. HIRST and A. T. KING (J. Text. Inst., 1926, 17, T 94—100).—Since many faults in woollen goods are due to retention of alkali, the determination of the latter is important. It is shown that common analytical methods are inadequate, but that wool gives up the whole of its alkali, whether in the form of hydroxide, carbonate, or soap, to terephthalic acid in aqueous suspension. The terephthalic acid which goes into solution can be determined by precipitation and weighing, but this method, though suitable for special cases of dyed wools, gives somewhat high results. Titration with bromophenol-blue as indicator is more con-

venient, and the authors prefer to add an excess of acid to the sodium terephthalate filtrate, filter, and titrate with sodium hydroxide. Calcium soaps also react with terephthalic acid, so a separate determination of calcium is necessary when they are present.

J. C. WITHERS.

Determination of sulphuric acid in wool. H. R. HIRST and A. T. KING (J. Text. Inst., 1926, 17, T 101—103).—The wool is wetted and added to a measured volume of standard sodium terephthalate solution. The unchanged terephthalate is titrated in the filtrate (cf. preceding abstract). A better method for dyed wool, which is easily stripped, is to leave the material in a suspension of magnesium carbonate, filter, and precipitate the sulphate formed as barium sulphate.

J. C. WITHERS.

Tensile strength and elongation of artificial silk. P. KRAIS (Z. angew. Chem., 1926, 39, 196).—The tensile strength of a single fibre of artificial silk (viscose) is reduced by 25% in a damp atmosphere (humidity 86%) compared with a dry atmosphere (humidity 52%), while the elongation before breaking is increased by 30%. The figures are 40% and 20% respectively when the fibre is completely soaked in water. For a strand composed of 27 fibres, the loss in tensile strength of the wet silk is 57%, while no change occurs in the elongation.

B. W. CLARKE.

Banana fibre. R. O. BISHOP (Malay. Agric. J., 1925, 8, 376—381).—Fibre from the stem of cultivated varieties of banana is superior to that from the native wild varieties, and although the yield is relatively small the quality resembles that of medium grades of "partially-cleaned" manila hemp.

D. F. TWISS.

Chemical examination of fibre from the bark of Terap, Tutor, and Baru. R. O. BISHOP (Malay. Agric. J., 1925, 8, 382—386).—The fibres from Tutor (*Artocarpus Kunstleri*, King), Baru (*Hibiscus macrophyllus*, Roxb.), and Terap (*H. floccosus*, Mast.) barks have similar chemical characteristics; the last, however, has a longer ultimate fibre and after treatment could probably be used as a substitute for hemp; the first two are alike in microscopic features and appear suitable for making paper of good quality.

D. F. TWISS.

Action of concentrated sulphuric acid on cellulose and extraction of cellulose from wood. K. ATSUKI and T. MINAKI (Cellulose Ind., Tokyo, 1926, 2, 3—10; cf. B., 1926, 8).—Sulphuric acid at concentrations between $H_2SO_4.H_2O$ and $H_2SO_4.3H_2O$ dissolves cellulose at the ordinary temperature without charring. Electrical conductivities of such hydrated forms indicate that the water is not entirely in combination. Dissolution of the cellulose is started by the combination of its OH groups with the sulphuric acid, followed by swelling and peptisation of the fibre. The rotation-time curve of the solution shows two points of inflection, as in the case of hydrochloric acid (*loc. cit.*), and the viscosity decreases rapidly until the first

point of inflection and then remains nearly constant as the cellulose changes from colloid to crystalloid products. The cellulose recovered by dilution is in a highly hydrated condition, drying to a horny product, and the amount obtained after 30 min. contact is over 100%. The quantity recovered by dilution decreases and its copper value increases at quicker rates the higher the concentration and temperature of the acid. α -Cellulose may be extracted from wood by treatment with 64–66% acid at 10–15° or with 66–70% acid at 0–10°, with but little loss. The wood, however, must be very finely powdered and passed through sieves of 64–120 meshes per sq. cm., and the precipitation of the cellulose by dilution must take place as soon as possible after solution. Japanese spruce yielded 45.2% of α -cellulose with copper value 1.30, free from furfuroids but sometimes slightly contaminated with lignin.

J. F. BRIGGS.

Producing cellulose from bamboo. G. FESTER and G. MAIDANA (*Z. angew. Chem.*, 1926, 39, 75–76).—Cellulose may be obtained by boiling bamboo from the Argentine Republic under pressure with 2.5% sodium hydroxide or a dilute solution of causticised wood ashes. The yield is about 45%, and it bleaches to a good white and is free from lignin.

A. GEAKE.

Alcoholysis of wood. B. HOLMBERG and S. RUNIUS (*Svensk Kem. Tidskr.*, 1925, 37, 189–197; *Chem. Zentr.*, 1926, I., 136–137).—If the cellulose and lignin in wood are combined to form an ester, with lignin as the acid, treatment with an ethoxide or with alcohol and an acid will produce ethoxylignin, which will be saponifiable with alkali. If the lignin–cellulose compound is an acetal, it will not be decomposed by ethoxides. Treatment with alcohol and acid will produce lignin, if this is the alcoholic component; or an unsaponifiable ethoxy-lignin, if the lignin is the carbonyl component of the acetal. Experiments with a coniferous wood favoured the last theory.

A. GEAKE.

Bleaching wood celluloses. H. WENZL (*Papier-Fabr.*, 1926, 24, 81–88).—Hypochlorous acid oxidises more rapidly than does hypochlorite ion, and strongly hydrolysed hypochlorites, such as those of zinc and aluminium, are therefore more active than calcium hypochlorite. When cellulose is bleached in a hollander at 35° the product obtained with sodium hypochlorite has a lower ash content and copper number and a higher proportion of α -cellulose than that obtained with magnesium and, especially, calcium hypochlorite. The hydrate copper number is higher, which may be due to a greater swelling power of the sodium salt. It is possible that the impurities dissolve more readily as sodium than as calcium salts. The pentosan content is nearly the same in each case. Neutral salts may be expected to have an adverse effect on bleaching by diminishing the solubility of impurities. Cellulose bleached with a bleaching powder containing 34.6% of active chlorine and 2.4% of chloride chlorine is whiter than when bleached with a powder containing 25.4% of

active chlorine and 9.2% of chloride chlorine. The white produced by sodium hypochlorite is adversely affected by additions of sodium chloride, sodium hydrogen carbonate, aluminium sulphate, or magnesium chloride. The addition of calcium carbonate to the bleaching powder used results in a higher copper number and pentosan content. When cellulose is bleached with sodium hypochlorite the active chlorine diminishes less rapidly than when the calcium salt is used. The addition of sodium carbonate to sodium hypochlorite greatly decreases the rate at which it is reduced, whereas sodium hydrogen carbonate and sodium hydroxide have only a small effect. The rate is also diminished by sodium and aluminium sulphates and potassium chlorate and nitrate, and is increased by calcium chloride, magnesium sulphate, and sodium acetate; sodium and magnesium chlorides have little effect. The addition of sodium acetate, and probably salts of other lower fatty acids, renders the bleaching inefficient.

A. GEAKE.

Oxycellulose. H. KAUFFMANN (*Textilber.*, 1925, 6, 591–592; *Chem. Zentr.*, 1926, I., 536–537).—The depth of dyeing of material with Methylene Blue does not depend on its oxycellulose content; goods containing oxycellulose dye equally deeply after this has been completely removed by boiling. Oxycellulose is determined by measuring the “boiling-off number,” and the “oxygen number.” The former is the number of c.c. of 0.1N-potassium permanganate reduced by the organic substances extracted from 1 g. of the material by boiling with aqueous sodium hydroxide. The latter is the number of c.c. required to oxidise 1 g. of the material to carbon dioxide and water. It is concluded that oxycellulose has the formula $C_{12}H_{20}O_{15}$. In the hypochlorite bleach the most active constituent is free hypochlorous acid.

A. GEAKE.

Polysaccharides, XXXIV. Hydro- and oxycelluloses. P. KARRER and T. LIESER (*Cellulosechem.*, 1926, 1, 1–6).—Cotton wool is completely dissolved when it is steeped in an 84% solution of phosphoric acid for several hours at the ordinary temperature and then heated to 35° for $\frac{1}{2}$ hr. By the addition of water a hydrocellulose is precipitated which is completely soluble in 10% sodium hydroxide, and which has a copper number (Schwalbe) of only 3. This hydrocellulose is readily methylated in cold sodium hydroxide solution by methyl sulphate. After four methylations a maximum methoxyl content of 42.5% is attained. As in the cases of normal cellulose (Irvine and Hirst, B., 1923, 710 A) and lichenin (Karrer and Nishida, B., 1924, 373) this value cannot be exceeded, although the theoretical value is 45.5%. This methylated hydrocellulose behaves like methylated lichenin in that it dissolves in cold water and is reprecipitated on boiling, and in being difficult to acetylate. The alkali-solubility of hydrocellulose is not due to carboxyl groups, and no alkali is used up; it is probably a case of peptisation or of the formation of additive compounds with alkali hydroxides. The electrical conductivity of the hydroxide is somewhat reduced. Oxycelluloses may be distinguished from hydrocelluloses by their acidity, but there is no

relation between the acidities of oxycelluloses and their copper numbers. The phosphoric acid hydrocellulose does not yield isosaccharic acid when boiled with lime. If oxycellulose is separated by dilute sodium hydroxide into soluble and insoluble portions, it is only the former that yields isosaccharic acid. Cellulose xanthate solutions may be purified by dialysis and the solution thus obtained contains 1 atom of sulphur and 1 of sodium for each 6 carbon atoms. The pure solution is not stable and when kept the cellulose is almost completely precipitated. The colours of the precipitates obtained with heavy metal salts are somewhat different from those given by impure solutions, and are as follows: copper, brown, turning yellow; bismuth, lemon-yellow; lead, white, turning yellow to brown; nickel, yellow, becoming greenish; iron, yellow. A. GEAKE.

Determination of lignin. S. VENKATESWARAN (J. Indian Chem. Soc., 1925, 2, 253—260).—Before determining the lignin in straw it is necessary to extract the finely powdered substance for 3—4 hrs. with ether in order to remove all fats and resins. The absorption of phloroglucinol by the straw is about $2\frac{1}{2}$ times as great as that of the lignin isolated from it. Experiments show that this difference is due to the presence of furfuraldehyde-yielding constituents in the straw and not to loss of lignin by decomposition during extraction. For the isolation and determination of lignin the following method is recommended: 1 g. of ether-extracted straw is hydrolysed for 20 hrs. at room temperature with 15 c.c. of 72% sulphuric acid. The acid is diluted to 3% concentration and the solution boiled under reflux for 1 hr. The lignin is filtered off, washed, dried at 105°, weighed, and ignited. The hydrochloric acid method of hydrolysis, though satisfactory, is not so convenient as the sulphuric acid method, and Wenzl's method (B., 1924, 464) is unsatisfactory as the reagent attacks the lignin molecule. R. W. WEST.

Determination of the acidity of paper. S. KOHLER and G. HALL (Paper Ind., 1925, 7, 1059—1063; Chem. Abstr., 1926, 20, 286).—The undesirable "internal" acidity (due to aluminium sulphate employed as rosin precipitant) and the harmless "external" acidity (due to aluminium sulphate added to preserve the glue) are determined by shaking 5 g. of the ground material with 250 c.c. of boiling water in small portions, heating for 1 hr. on the steam-bath under an air condenser, and repeating the extraction twice after filtration at the pump. After cooling, the extracts are separately titrated, using phenolphthalein, with 0.01*N*-sodium hydroxide. The "external" acidity alone is determined by shaking 10 g. in small cut pieces for 2 min. with 100 c.c. of cold water, filtering at the pump without washing, and titrating as before. A. A. ELDRIDGE.

Removal of printers' ink from old paper. E. BERL and W. PFANNMÜLLER (Z. angew. Chem., 1925, 38, 887—889).—Experiments were made on the removal of printers' ink from old paper by shaking the shredded paper with a mixture of water and an organic liquid. Since cellulose is hydrophile and

lampblack is hydrophobe a separation by flotation was to be expected. Trials were made with benzene, machine oil, carbon tetrachloride, and tetralin, the last-named proving the most satisfactory. Better results were obtained by combining a physical and chemical method of separation. The paper was first boiled with an excess of dilute sodium hydroxide solution and then treated with the water and tetralin. T. S. WHEELER.

Effect of dyes used in paper-making on animal life in water. HAEMPEL.—See IV.

PATENTS.

Producing plastic masses from cellulose derivatives. P. BALKE and G. LEYSIEFFER (E.P. 229,674, 18.2.25. Conv., 18.2.24).—Plastic masses are produced by the process described in E.P. 154,157 (B., 1922, 383A) but replacing cellulose derivatives by waste materials containing cellulose derivatives, which may be combined with gelatinising agents, e.g., celluloid or cellulose acetate waste. A. GEAKE.

Manufacture of viscose solution. MASCHINENBAU-ANSTALT HUMBOLDT (G.P. 418,056, 3.12.21).—Cellulosic material is dissolved in the usual solvents (e.g., zinc chloride, a mixture of strong acids, concentrated hydrochloric acid and a bisulphate, or zinc chloride and a bisulphate), separated from insoluble matter, precipitated, and washed. The purified cellulose is dissolved in the calculated amount of dilute caustic alkali and converted into viscose. Concentrated solutions may thus be obtained, suitable for the manufacture of plastic masses, artificial filaments, films, etc. A. GEAKE.

Manufacture of artificial threads, films, and the like from viscose. N. V. NEDERLANDSCHE KUNSTLIJDEFABR. (G.P. 421,506, 18.1.22).—Carbon disulphide is added to ripened or unripened viscose solution and the mixture spun at once or after further ripening. A. GEAKE.

Manufacture of threads, films, and the like from viscose. H. Voss (G.P. 421,800, 7.11.22).—The precipitating bath consists of neutralised sulphite-cellulose waste liquor and ammonium salts. The latter may be added as such, or may be produced in the waste liquor or the precipitation bath. A. GEAKE.

System for boiling cellulose. ZELLSTOFF-FABR. WALDHOF, A. SCHNEIDER, and C. HANGLEITER (E.P. 239,191, 15.8.25. Conv., 26.8.24).—In regenerating waste heat and gases (sulphur dioxide) from sulphite-cellulose boilers, by blowing the gases into fresh sulphite liquor (cf. G.P. 350,471; B., 1922, 855A), undesirable back-pressure effects are avoided by a system of injectors, pumps, and valves. The gases are drawn from the boilers by pumping sulphite liquor through injectors, the vacuum chambers of which are connected to the tops of the boilers. The liquor then passes through a

further pump and check valve into a storage vessel. The liquor may be withdrawn from the bottom of the vessel for further circulation, or fresh liquor may be used continuously. A. GEAKE.

Plant where heat-consuming apparatus operates with a number of feed conduits. [Utilising heat accumulators in connexion with cellulose digesters.] J. RUTHS, K. INGERÖ, and H. SCHRENK, ASSYS. to AKTIEBOLAGET VAPORACKUMULATOR (U.S.P. 1,568,420, 5.1.26. Appl., 7.12.22).—A cellulose digester is connected with the low-pressure steam supply (heat accumulator), but as soon as the pressure in the digester equals that in the low-pressure main a valve opens automatically and admits steam from the high-pressure main while communication with the low-pressure main is cut off. In this way the best use is made of the low-pressure steam, although the pressure at which it is supplied may vary, and little supervision is required even when operating a large number of digesters. T. S. WHEELER.

Treating seed hulls [to produce paper pulp]. R. MORGENIER (U.S.P. 1,570,389, 19.1.26. Appl., 7.12.20).—Rice hulls are boiled for 2 hrs. with sodium hydroxide of 10° B. (d 1.07), the liquor is removed, and the residue beaten to a pulp. A. GEAKE.

Manufacture [drying] of paper. F. GREWIN (E.P. 223,927, 24.10.24).—The drying is assisted by blowing air from the outside into spaces enclosed between the frame of the machine, the web of paper or cardboard and the dryer felt. The air is introduced through nozzles, which may be alternately on either side of the machine, and passes at a high velocity through the defined spaces in such a manner and direction that it does not impinge on the paper web but acts rather as an ejector for the accumulated water vapour. J. F. BRIGGS.

Paper making process. E. P. R. MARKS. From DRYING SYSTEMS, INC. (E.P. 246,048, 10.8.25).—The moist sheet or web is partly dried by blowing steam into or through it while supported by wire gauze or other suitable porous material. Water is mechanically expelled and the web is heated ready for further drying operations. The steam may be supplied from a box contained in part of the suction roller drawing the web from the pulp vat. A. GEAKE.

Washing artificial silk. A. PINEL (U.S.P. 1,573,859, 23.2.26. Appl., 26.4.23).—See E.P. 199,355; B., 1924, 330.

Drying of textile and similar materials. H. L. JULIEN (E.P. 247,783, 14.4.25).

Manufacture of paper. [Pulping waste paper.] S. LEICESTER and L. B. HOLMAN (E.P. 247,638, 13.11.24).

Tanning preparations from sulphite-cellulose waste lye (U.S.P. 1,571,873).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Use of reduction methods of analysis, particularly determination of the copper number, in determining bleaching damage. H. WENZL (Woch. Papierfabr., 1925, 56, 994—997, 1024—1027; Chem. Zentr., 1926, I., 272).—The copper number of bleached cotton material is not an accurate criterion of the deterioration produced by bleaching unless considered together with tests of tensile strength. The solubility of oxycellulose increases with the alkalinity of the bleaching liquor. The most rapid and trustworthy method for determining copper number is that of Schwalbe and Hägglund (Fehling's solution is used), and those modifications involving the use of ferric sulphate or simplified apparatus (cf. Knecht and Thompson; B., 1920, 718A) are less satisfactory. The changes which occur when Fehling's solution is heated may be very much reduced by the use of pure reagents and apparatus by which the overheating of the solution is avoided. A. J. HALL.

Comparison of the effects of oxidation before and after the mercerisation of the cotton fibre. E. KNECHT and E. F. MULLER (J. Soc. Dyers and Col., 1926, 42, 46—53).—The investigations were carried out on bleached American 2/50's cotton yarn, oxidation being effected by means of potassium permanganate (cf. Knecht and Thompson; B., 1920, 718A) and bleaching powder, and mercerisation by means of caustic soda of 50° Tw. (d 1.25) and nitric acid of 83° Tw. (d 1.415) without tension. Cotton yarn mercerised with caustic soda shrinks less, becomes weaker, and has a smaller copper number and less affinity for dyestuffs than yarn mercerised by means of nitric acid; mercerisation increases the copper number of cotton. Mercerised cotton is more susceptible to oxidation and deterioration by exposure to heat and light than ordinary cotton, the yarn mercerised with caustic soda being more susceptible. Dyed mercerised yarns fade more rapidly than similarly dyed ordinary cotton, and this property is shown to a greater extent with cotton mercerised with caustic soda. Cotton yarns mercerised by caustic soda and nitric acid after either a moderate or high degree of oxidation suffer a decrease and a slight increase in textile strength respectively, but whereas nitric acid increases the copper number of the oxidised yarn slightly, caustic soda, owing to its solvent action, decreases the copper number considerably. Yarns weakened by oxidation suffer less than their usual shrinkage when mercerised with nitric acid. The loss of tensile strength produced by mercerisation of oxidised cotton with caustic soda decreases as the degree of oxidation is decreased and *vice versa*, and when the degree of oxidation is less than 0.0064 atom of oxygen per $C_6H_{10}O_5$ unit, mercerisation results in an increase of tensile strength, thus confirming the conclusion of Micheli (B., 1903, 1290). The ultimate effect of mercerisation with caustic soda is the resultant of weakening and strengthening actions due to solvent action and mercerisation respectively.

Although mercerised cotton yarns after oxidation with potassium permanganate have considerably higher copper numbers than similarly oxidised ordinary yarn, these differences do not occur when oxidation is carried out *in vacuo*. Under similar conditions, mercerised cotton is oxidised more quickly than ordinary cotton. Successive determinations of the copper number of the same sample of oxidised yarn yielded a series of diminishing values whose aggregate totals in the case of mercerised (with caustic soda or nitric acid) and ordinary cotton subsequently oxidised to the same degree of oxidation, become nearly equal; the copper numbers were determined by the method of Knecht and Thompson (*supra*). Cotton yarns mercerised by means of various strengths of caustic soda and subsequently oxidised, have copper numbers which decrease as the strength of the mercerising liquor decreases. Approximately equal rises of temperature occur during the mercerisation of cotton with caustic soda and nitric acid under similar conditions. The affinity of mercerised cotton for oxygen and for moisture varies with the degree of mercerisation. The drying of mercerised yarn decreases its susceptibility to oxidation, but mercerised yarn maintained moist by physical methods shows no increased affinity for oxygen. The amount of moisture retained by completely mercerised yarn corresponds to the formula $2C_6H_{10}O_5 \cdot H_2O$. A. J. HALL.

Application of nitrobenzyl cellulose ethers to the dyeing of cotton. D. H. PEACOCK (J. Soc. Dyers and Col., 1926, 42, 53).—Cotton is dyed in shades fast to washing by esterification with *m*- and *p*-nitrobenzylphenyldimethylammonium chloride (Leucotrope is phenylbenzyl dimethylammonium chloride), subsequent reduction of the nitro-group and then diazotisation and coupling with suitable components. For example, 1 g. of cotton boiled for 10 min. with 100 c.c. of a 1% solution of the nitro-derivative of Leucotrope containing a slight excess of sodium carbonate, is washed and reduced by treatment with an aqueous solution of sodium hyposulphite at 60–70°, is then diazotised at 5–7° in the usual manner and developed by immersion in aqueous solutions containing β -naphthol (for a rose shade), *R*-salt (rose), Schaffer's acid (red), *H*-acid (violet), chromotropic acid (red-violet), *m*-phenylenediamine (orange-brown), *J*-acid (red), phenyl-*J*-acid (red), carbonyl-*J*-acid (orange-red), γ -acid (acid coupling, red; alkaline coupling, bluish-red), or phenyl- γ -acid (violet-brown). A. J. HALL.

Comparison of the affinity of cotton, wool, and particularly cellulose acetate silk for azo-compounds [dyes] containing sulphonic, carboxyl, arsenic, and stibinic acid groups. A. J. HALL and M. I. AISCHE (J. Text. Inst., 1926, 17, T104–110).—In the hope of discovering dyes soluble in water, but applicable to cellulose acetate without the agency of soaps or other solubilising substances, some azo-compounds containing arsenic or stibinic acid groups have been prepared. The dyes, the properties of which are summarised in a

table, were prepared by diazotising and coupling from sulphanilic, *p*-aminobenzoic, *p*-arsanilic, and *p*-stibanilic acids. None of the compounds was suitable as a dye for cellulose acetate silk, but the results indicate that it should be possible to prepare azo-dyes containing arsenic and stibinic acid groups capable of satisfactory application to cellulose acetate. The stibinic acids are not fast to light, but the other dyes are about equally fast. The affinity for cellulose acetate increases in the order; sulphonic, arsenic, stibinic, carboxylic; and β -naphthol derivatives are more effective than α -naphthol compounds. J. C. WITHERS.

Bleaching wood cellulose. WENZL.—See V.

PATENTS.

Bleaching hair, bristles, and the like. B. VENEZIANI (Austr. P. 100,709, 31.3.24).—Bristles are treated with hydrogen peroxide solution and then made alkaline with gaseous ammonia. The treatment may be repeated after drying, and the material may be subjected to a vacuum before the first treatment to remove air bubbles. A. GEAKE.

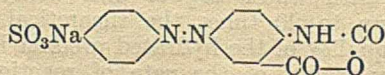
Rendering mercerised cotton, ammonium cuproxide [cuprammonium] silk, viscose silk and like products refractory against the further absorption of direct dyes. CHEM. FABR. VORM. SANDOZ (E.P. 241,854, 9.1.25. Conv., 24.10.24).—The processes previously described (cf. G.P. 346,883 and E.P. 233,704; B., 1922, 214 A; 1925, 956) for preparing "immunised" cotton (cf. E.P. 246,609; following abstract) are similarly used for immunising mercerised cotton and cellulose artificial silks, suitable esterifying agents being the chlorides and anhydrides of aliphatic and aromatic carboxylic acids, and chlorides of aromatic sulphonic acids. For example, immunised viscose silk is obtained by immersing 1 kg. of viscose silk for 5 min. at 20° in a 10% alcoholic solution of caustic soda, centrifuging, then immersing the silk for 5 min. at 65° in 10 kg. of a 20% solution of acetyl chloride in toluene, afterwards centrifuging and soaping, washing, and drying at 50°. Similarly to immunise or mercerise cotton, 1 kg. of the cotton is immersed for 30 min. at 20–25° in 10 kg. of a 14% alcoholic solution of caustic soda, then centrifuged, immersed for 2 hrs. at 40–45° in 10 kg. of a 20% solution of *p*-toluenesulphochloride in toluene, again centrifuged, then soaped, washed, and dried at 50°. A. J. HALL.

Dyeing of ["immunised"] cotton materials. SANDOZ CHEMICAL CO., LTD., and A. E. WOODHEAD (E.P. 246,609, 17.11.24).—"Immunised" cotton (*i.e.*, cotton resistant to direct dyestuffs) prepared by treatment of ordinary cotton with alcoholic caustic soda and *p*-toluenesulphochloride by the method described in E.P. 195,619 (cf. E.P. 233,704; B., 1925, 956) is dyed by means of insoluble or difficultly soluble dyestuffs which have been solubilised by pre-treatment with suitable dispersing agents; *e.g.*, higher fatty acids or their sulpho-derivatives, their alkali-metal or ammonium salts. The methods of dyeing are similar to those generally used for

cellulose acetate except that the temperature of dyeing is preferably 75–95°. Claim is made for use of the following dyestuffs: azo dyestuffs prepared from nitroanilines, dinitroanilines, nitrotoluidines, nitroxylidines, anisidines, chloroanisidines, nitroanisidines, phenetidines, chlorophenetidines, nitrophenetidines, or chloronitroanilines diazotised and coupled with aniline, methylaniline, dimethylaniline, ethyl- or di-ethylaniline, methylethylaniline, diphenylamine, methyl- or ethyl-diphenylamine, *m*-toluidine, or *p*-xylydine; aniline, chloroanilines, dinitroanilines, toluidines, xylydines, nitrotoluidines, nitroxylidines, anisidines, chloro- or nitro-phenetidines diazotised and coupled with α - or β -naphthylamine or their methyl, dimethyl, ethyl, diethyl, methylphenyl, or ethylphenyl derivatives; α - or β -naphthylamine or their chloro- or nitro-derivatives diazotised and coupled with aniline or the alkyl- or aryl-aniline or naphthylamine derivatives mentioned above, or α - or β -naphthylamine; naphthols, resorcinol, β -hydroxynaphthoic acid and its anilide, toluidide, and similar derivatives coupled with diazotised aminobases such as *m*-nitroaniline and α -naphthylamine. The method of dyeing allows the production of two-colour effects in union fabric containing ordinary cotton and other fibres, together with immunised cotton.

A. J. HALL.

Compound for dyeing. BADISCHE ANILIN-
U. SODA-FABR., Assees. of F. GÜNTHER (U.S.P.
1,567,731, 29.12.25. Appl., 8.6.25).—An aromatic
o-aminocarboxylic acid other than an uncoloured
anthranilic acid derivative is treated with carbonyl
chloride or ethyl chloroformate to give a derivative
of isatoic anhydride which combines with cellulose
or other carbohydrates forming a kind of ester of
an aminoarylcaryboxylic acid so that the dyed
substance is a part of the dye molecule. If suitable
groups are present the carbohydrate compounds
can be coupled with diazo-compounds or diazotised
and coupled with azo-dye components. For example
diazotised sulphanilic acid is coupled with anthranilic
acid and the product is treated with carbonyl
chloride in feebly alkaline solution to give a
substance with the formula



which dyes cellulose in an alkaline bath yellow
shades. If the coloured fibre is diazotised and
coupled with β -naphthol a bright red shade results.
2:3-Naphthylaminecarboxylic acid and carbonyl
chloride yield a compound which when combined
with cellulose gives no colour but the fibre can be
diazotised and coupled with azo-dye components.
If ethyl chloroformate is used it is boiled with the
substance under treatment. T. S. WHEELER.

**Apparatus for the liquid treatment of textile
materials upon rollers.** J. BRANDWOOD, A.
STOCKER, and TWYVER WORKS, LTD. (E.P. 247,661,
21.11.24).

Carrying out and controlling reactions.
(G.P. 422,159).—See I.

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

**Gypsum-sulphuric acid process (of the
Farbenfabr. vorm. F. Bayer & Co.).** W. J.
MÜLLER (Z. angew. Chem., 1926, 39, 169–174).—
Calcium sulphate in the form of gypsum or an-
hydrite is reduced by carbon in the form of coke,
at 800° according to the equation $\text{CaSO}_4 + \text{C} =$
 $\text{CaO} + \text{SO}_2 + \text{CO}$. The lime must be recovered in a
marketable form to make the process economically
possible, and clay is therefore added to the raw
materials in the requisite proportions to obtain
Portland cement. The operation is carried out in
a rotary kiln fired with pulverised fuel. The
materials must be dried before entering the kiln
in order to obtain a sufficiently high concentration
of sulphur dioxide in the issuing gases. An
oxidising atmosphere must be maintained in the
kiln to prevent the formation of carbon oxysulphide
in the gases and calcium sulphide in the cement
clinker. In practice, about half the theoretical
quantity of coke is used, to prevent too rapid
reduction and the formation of calcium sulphide
in the clinker. The Portland cement produced
is of good quality and is ground with blast-furnace
slag and sold as blast-furnace cement. The gases
from the kiln contain 6–7% SO_2 ; after electrical
separation of dust and washing, they are used to
make sulphuric acid, by the contact or chamber
process.

B. W. CLARKE.

**Determination of hydrocyanic acid in gaseous
mixtures.** G. E. SEIL (Ind. Eng. Chem., 1926,
18, 142–143).—The gas containing hydrogen
cyanide is caused to react with 0.1*N*-iodine solution
in the presence of starch solution saturated with
sodium hydrogen carbonate in a special 500-c.c.
Tutwiler burette. The hydriodic acid is thus neu-
tralised as formed. The iodine is added until the
starch solution just turns blue, and the liquid is
is then withdrawn from the burette, which is washed
down with distilled water or sodium hydrogen
carbonate solution. Standard sodium thiosulphate
solution (1 c.c. = 0.2288 mg. per litre of HCN gas
on a 500-c.c. sample) is used for exact titration of
the excess of iodine and enough hydrochloric acid
is then added to give an acid reaction (amount
previously ascertained), whereby cyanogen
iodide and hydriodic acid interact to yield
hydrocyanic acid with liberation of iodine. If a
blue colour appears after keeping for 0.5–1 minute
hydrocyanic acid is present, and the solution is
titrated to the point of disappearance of colour.
The difference between the two burette readings
 $\times 10$ gives the grains of hydrocyanic acid per 100
cub. ft. from 500 c.c. of sample. D. G. HEWER.

**Determination of strength of liquid hydro-
cyanic acid by specific gravity.** M. WALKER
and C. J. MARVIN (Ind. Eng. Chem., 1926, 18,
139–142).—The strength of hydrocyanic acid
solutions may be rapidly determined to an accuracy
within 0.2% by taking the specific gravity by means
of hydrometers with a scale from 0.650 to 0.750,

with 50 divisions on each, extending over a stem length of 106.4 cm., so graduated that readings may be taken to the third decimal place. The liquid is drawn up by suction into a glass cylinder containing the hydrometer and the thermometer passes through the cylinder stopper. The table of strengths of acid corresponding to specific gravities given was prepared by determinations on liquids of different strengths analysed by the gravimetric method of absorption in caustic soda and titration with silver nitrate.

D. G. HEWER.

[Manufacture of solid] calcium cyanide.

F. J. METZGER (Ind. Eng. Chem., 1926, 18, 161—163).—Calcium cyanide, previously known only in dilute solution, is now being manufactured in the solid state by the action on calcium carbide of liquid hydrogen cyanide containing 2% of water calculated on the weight of carbide. $\text{CaC}_2 + 4\text{HCN} = \text{Ca}(\text{CN})_2 + 2\text{HCN} + \text{C}_2\text{H}_2$. The reaction is carried out in a jacketed vacuum filter to which the carbide is gradually added from a gas-tight hopper with cooling. The acetylene evolved contains a small proportion of hydrogen cyanide, which is removed by washing with alkali. On completion of the reaction the sludge is warmed, the hydrogen cyanide distilling off and the calcium cyanide being obtained as a light tan-coloured powder which is immediately filled into airtight containers. This product, which contains about 55% of cyanogen, is very rapidly decomposed by atmospheric moisture, with liberation of hydrogen cyanide, and it has been termed "powdered hydrocyanic acid." It offers many advantages over liquid hydrocyanic acid in the fumigation of citrus trees against scale and in other similar applications.

C. IRWIN.

Nitrogen fixation by means of barium carbonate and coal in an electric oven. A. SCHWEITZER (Z. Elektrochem., 1926, 32, 98—105).—

A prepared mixture of barium carbonate and coal was coked and then treated with nitrogen in a graphite crucible heated to 1300°. An absorption of 6.6% of nitrogen was obtained. The product, consisting of 3 pts. of barium cyanide and 1 pt. of barium cyanamide, was treated with water in an autoclave at 7—8 atm. pressure for $\frac{1}{4}$ hr. The gases, consisting of ammonia and hydrogen, were led through sulphuric acid, and the ammonia was determined. In the technical process, the finely powdered coal is mixed with barium carbonate, a little coal tar and water, and formed into cylindrical pieces in a hydraulic press. The coking takes place in a retort oven at 800° for 1½ hr. For the nitrogen absorption an electric furnace of special construction is used, having graphite electrodes, single-phase alternating current being employed at 45—65 volts. Great economy of power may be effected at this stage, which can be further improved by additional protection against loss of heat. The heated mass is treated first with producer gas, and later with 99% pure nitrogen. The reaction product is treated with steam at 7 atm. pressure in an autoclave twice, the gases being collected in sulphuric acid. The

nitrogen absorption is about 4%. The explanation of the reactions adopted is that the barium carbonate becomes reduced first to oxide and then to carbide, which reacts with nitrogen giving barium cyanide or barium cyanamide and carbon. The stoichiometric relations require 1 mol. $\text{BaCO}_3 : 4\text{C}$, but it is better in practice to use carbon greatly in excess, in the approximate proportion 1 mol. $\text{BaCO}_3 : 18\text{C}$. The action of water on barium cyanide and barium cyanamide yields ammonia in each case, together with barium formate and barium carbonate respectively. The thermal decomposition of barium formate yields hydrogen and barium oxalate; the latter on further heating yields barium carbonate, which is thus recovered for subsequent operations.

C. H. D. CLARK.

Technology of calcium cyanamide. P.

DOLCH (Z. Elektrochem., 1926, 32, 68—70).—The method of determining carbide in a finely-powdered sample by dropping saturated salt solution upon it mixed with salt and examining the evolved gas is replaced by that of adding the carbide in small quantities at a time to water. The error in the older method lies in the retention of acetylene in the lime sludge, in a polymerised form. This was proved by evaporating the ether extract, when a tarry residue remained. No decomposition of carbide occurs during grinding. The influence of foreign gases in lessening the nitrogen absorption by finely-milled carbide in a heated oven is very considerable. The efficiency of the process is greatly increased by sweeping out foreign gases periodically by means of a current of nitrogen. If in a series of ovens the outgoing gas from one oven passes into the next, the proportion of foreign gases leaving each oven becomes progressively greater, with corresponding decrease of nitrogen absorption. Hydrogen is formed during the heating by decomposition of acetylene.

C. H. D. CLARK.

Hydration of lime. W. G. WHITMAN and G. H. B. DAVIS (Ind. Eng. Chem., 1926, 18, 118—120).—

The effects of the hydration of lime at various temperatures with water vapour and with water in amounts up to 20 times the theoretical quantity upon the quality of the hydrated product have been studied by means of the rate of reaction of the product with acid, its rate of settling in water, and its appearance under the microscope. In general, the quality as judged by any one of these methods agrees closely with the ratings obtained by the other two. Hydration with the theoretical amount of water or with water vapour tends to produce excessively high temperatures locally in the lime, thus delaying the completion of the reaction and allowing time for the agglomeration of the particles with the result that a low-grade product containing coarse grains is formed. The three most important factors are the ratio of water to lime, the temperature of hydration, and the degree of agitation, the best product being obtained by hydrating with a large excess (20 times the theoretical amount) of water in boiling solution.

A. W. HOTHERSALL.

Fusion of alumina with soda [sodium carbonate]. G. FELD (Z. angew. Chem., 1926, 39, 174—175).—Bauxite or aluminium hydroxide precipitated from aluminium chloride by ammonia, when fused with sodium carbonate at 900—1200°, gives the sodium aluminate $\text{Na}_2\text{O}, \text{Al}_2\text{O}_3$. Aluminium hydroxide precipitated by carbon dioxide from a solution of sodium aluminate, at temperatures below 950° forms the aluminate $2\text{Na}_2\text{O}, \text{Al}_2\text{O}_3$, which at higher temperatures is converted into the aluminate $\text{Na}_2\text{O}, \text{Al}_2\text{O}_3$. The different behaviour of these various forms of alumina is assumed to be due to a difference in structure of the hydroxides. B. W. CLARKE.

Quick method for the determination of ozone. H. B. McDONNELL (Ind. Eng. Chem., 1926, 18, 135).—Low concentrations of ozone may be measured by allowing the ozonised air to flow from a nozzle with an outlet of definite size (e.g., 1.5 mm.) through a glass tube into a testing solution consisting of potassium iodide and starch, and an amount of sodium thiosulphate solution equivalent to 5 c.c. of 0.01% iodine solution. The gas is passed until the blue colour appears and the time noted with a stop-watch. The method is also applicable to dilute mixtures of chlorine and air. D. G. HEWER.

See also A., Mar., 261, **Determination of sulphites and of ferrous iron (BONNER and YOST); Volumetric determination of sulphates (ATKINSON).** 263, **Use of uranous sulphate in volumetric analysis (VORTMANN and BINDER).**

Determination of perchlorate in Chile saltpetre. HOFMANN, HARTMANN, and HOFMANN.—See XVI.

PATENTS.

Production of phosphoric acid. CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 241,903, 20.10.25. Conv., 23.10.24).—Phosphorus pentoxide fume, which is very difficult to condense, is easily dissolved by water or phosphoric acid solution near its boiling point. To produce phosphoric acid, phosphorus or a mixture containing it may be burnt in a tower down which water trickles, the heat of combustion of the phosphorus heating the water to the required temperature to dissolve the fume. E. S. KREIS.

Manufacture of sulphuric acid. W. CARPMAEL. From F. BAYER U. Co. (E.P. 245,903, 5.12.24).—In the manufacture of sulphuric acid by the combined contact process and chamber process, the amount of contact catalyst is reduced to $\frac{1}{8}$ — $\frac{1}{10}$ of that ordinarily used. The sulphur dioxide is transformed into sulphur trioxide by the usual contact process and the remaining sulphur dioxide is converted into sulphuric acid by the chamber process, e.g., as described in E.P. 149,648, 187,016, and 184,966 (cf. B., 1921, 693 A; 1922, 982 A, 858 A). E. S. KREIS.

Production of hydrocyanic acid. E. EICHWALD (F.P. 595,399, 18.6.24).—Catalysts for use in the production of hydrocyanic acid from gas mixtures containing nitrogen and carbon compounds, e.g.,

from ammonia and carbon monoxide, consist of metal compounds containing oxygen, which, under the conditions of the reaction, are not, or are only partially reducible, e.g., they may contain ferric oxide and alkali or alkaline-earth oxides. The catalysts are preferably deposited upon material such as pumice stone, asbestos, or wood charcoal.

L. A. COLES.

Mechanical salt-cake furnaces. FARBW. VORM. MEISTER, LUCIUS, u. BRÜNING (E.P. 225,548, 24.11.24. Conv., 29.11.23).—A mechanical salt-cake furnace, in which the process is completed in one stage, is constructed of acid-resisting brick or stone, instead of cast iron. It is preferably fired with producer gas with recovery of the waste heat. The advantages are greater durability and the possibility of the use of higher temperatures. C. IRWIN.

Separating a mixture of hafnium and zirconium compounds. N. V. PHILLIPS' GLOEILAMP-ENFABR. (E.P. 226,180, 20.11.24. Conv., 12.12.23).—A mixture of the phosphates, phosphites, arsenates, arsenites, antimonates, or antimonites of hafnium and zirconium is introduced into a medium containing hydrofluoric acid or an alkali bifluoride. The complex acids or their salts produced are then fractionally crystallised. The ammonium salts are preferred, but examples are also given of the use of lithium and of barium salts. C. IRWIN.

Making chlorides [aluminium chloride]. G. L. PRITCHARD and H. HENDERSON, ASSRS. to GULF REFINING Co. (U.S.P. 1,568,181, 5.1.26. Appl., 17.11.21).—A mixture of bauxite or other aluminous material and coke is introduced into the top of a heat-insulated tower divided into two sections. In the upper section it is preheated to 560° by the direct or indirect action of furnace gases and in the lower it is subjected to the action of chlorine or hydrochloric acid. Aluminium chloride is formed and volatilises, passing to a condenser by an exit pipe at the top of the lower section. Means are provided for passing the material from the upper to the lower section while preventing gas from passing in the opposite direction, and also for impeding the fall of the material down the tower. To provide the heat necessary to complete the formation of aluminium chloride, a small quantity of aluminium dross may be added to the material to be treated.

T. S. WHEELER.

Recovering borax from saline liquors. A. W. GAUGER and H. H. STORCH, ASSRS. to BURNHAM CHEMICAL Co. (U.S.P. 1,571,002, 26.1.26. Appl., 21.2.25).—The liquor is evaporated until other salts crystallise, leaving a concentrate of metaborate and tetraborate. This is cooled and agitated in contact with a finely divided material, when crystals of borax are rapidly formed. C. IRWIN.

Production of alkali cyanides. STOCKHOLMS SUPERFOSFAT FABR. AKTIEBOLAGET, Assees. of H. G. A. RAMSAY (Swed. P. 58,030, 23.2.24).—Mixtures of calcium cyanamide with alkali chlorides or alkali carbonates, or with both, are fused and

treated with moderately concentrated alkali hydroxide solution, preferably hot, in quantity at least sufficient to convert the whole of the calcium into calcium hydroxide. After filtration, the solution is evaporated to recover alkali cyanide. L. A. COLES.

Manufacture of oxygen. SOC. L'OXYLITHE (E.P. 240,412, 3.7.25. Conv., 25.9.24).—The coil for liquefying the oxygen in the vapours emanating from the liquid air surface is placed outside the rectifying column instead of in the lower part of the column, thus simplifying construction and allowing free trickle of liquid in the column. E. S. KREIS.

Detecting oxygen. A. H. MAUDE, Assr. to WESTINGHOUSE ELECTRIC AND MANUF. CO. (U.S.P. 1,567,534, 29.12.25. Appl., 2.1.24).—A glass cylinder contains a known volume of a standard solution of indigo-carmin reduced with a standard solution of alkaline stannite, the space above the reagent being evacuated. The cylinder is sealed below by a closed capillary tube. The space under test, for example, the gas space of a transformer, is connected to the capillary tube and the latter is broken, whereupon gas from the transformer bubbles up through the liquid in the container into the evacuated space. The time required for the liquid to turn from orange to green gives a measure of the oxygen present. The solution indicated is suitable for gases containing 1–21% of oxygen. For smaller percentages ammoniacal cuprous chloride is used. Other gases can be similarly determined, using suitable reagents.

T. S. WHEELER.

Purification of radium emanation. V. F. HESS and E. D. LEMAN, Assrs. to UNITED STATES RADIUM CORP. (U.S.P. 1,570,834, 26.1.26. Appl., 14.5.23).—The emanation, after a preliminary partial purification, is passed into a tube containing an absorbent capable of removing the remaining impurities. The part of the tube containing the impurities is then sealed off from the remainder which contains purified emanation. An apparatus for carrying out the process comprises an explosion chamber, an expansion chamber, an absorption bulb, an auxiliary trap chamber, a capillary tube, and a vacuum pump with the necessary connexions.

C. IRWIN.

Treatment of earthy minerals. W. FELDENHEIMER (U.S.P. 1,573,385, 16.2.26. Appl., 26.5.25).—See E.P. 242,358; B., 1926, 55.

Recovery of caustic hydroxides from waste solutions. F. H. GRIFFIN, Assr. to VISCOSE CO. (U.S.P. 1,573,703, 16.2.26. Appl., 27.6.23).—See E.P. 237,468; B., 1925, 757.

Carrying out and controlling reactions (G.P. 422,159).—See I.

Drying and calcining gypsum (U.S.P. 1,571,189).—See IX.

Portland cement and sulphuric acid (F.P. 596,423).—See IX.

Electrolysis of alkali chlorides (F.P. 596,918).—See XI.

VIII.—GLASS; CERAMICS.

Action of molten glass on refractory materials. O. BARTSCH (Glastech. Ber., 1925, 3, 249–265; Chem. Zentr., 1926, I, 1469).—Pots are actively attacked, but for a short time only, by the batch materials, and the attack of the glass itself is milder, though of longer duration, being a process of solution. For this reason glazing of the walls and rapid melting are necessary. The vitreous ground mass of the clay passes into the glass, and the mullite structure of the wall grows. The quaternary eutectic of composition SiO_2 65.27%, Al_2O_3 16.98%, CaO 5.08%, K_2O 12%, melts at 1180° and may pass into the glass. Still greater amounts of alumina can be dissolved, but this is opposed by the slow rate of solution. In the bounding layer between pot and glass a highly aluminous, viscous mass is formed containing mullite and sometimes corundum, especially in the case of highly aluminous clays, and this layer protects the walls. If the density of the bounding layer deviates from that of the melt, the layer tends to move either to the top or bottom, in which case further pot surface is exposed, whilst the upward movement tends to produce hard cords. For this reason clays low in alumina are specially suitable for pots. Bond clay and grog should have the same composition. Cavities in the pot body increase the possibility of attack. Homogeneous pots are most readily prepared by casting. A. COUSEN.

Silica bricks. Thermal analysis. TRAVERS and DE GOLOUBINOFF (Rev. Mét., 1926, 23, 27–47).—The thermal expansion of the various forms of silica was investigated using the dilatometer designed by Chevenard. The test-pieces were prepared by moulding cylinders of the finely-powdered material mixed with 3–4% of bonding material (potassium silicate or lime) and slowly heating to 800° . The results obtained with quartz confirm those of Le Chatelier except that the reversible expansion at 575° is found to be 0.27, Le Chatelier's figure being 0.42. A new point in the tridymite curve at 440 – 450° , is evidence of an undescribed δ -tridymite. Above 440° , the expansion of tridymite is very small but is always positive. Very pure cristobalite gave a greater thermal expansion than that used by Le Chatelier, the total expansion between 20 and 210° being 1.55, and the expansion at 210° being 1.05, Le Chatelier's figures being 1.2 and 1.00 respectively. The transformation $\alpha \rightarrow \beta$ -cristobalite is shown to be rapid and reversible.

P. B. ROBINSON.

PATENTS.

Glasses. JENAER GLASWERK SCHOTT UND GEN. (E.P. 219,972, 25.7.24. Conv., 3.8.23).—A series of glasses are claimed which all contain as ingredients SiO_2 , Al_2O_3 up to 10% (if present), B_2O_3 from 5 to 18%, K_2O and Na_2O together from 3 to 15%, the Na_2O not exceeding 1% and being at most one-fifth of the percentage of the K_2O . They possess the advantages that they can be worked for optical purposes

without devitrifying or becoming turbid, whilst they have a high thermal endurance. Such glasses have a small dispersion and a comparatively high refraction. Other ingredients of the glasses may be (1) PbO , Sb_2O_3 forming together with the K_2O and Na_2O from 3 to 35% of the glass; (2) BaO , CaO , MgO , ZnO , which together with the K_2O and Na_2O form at most 35%; (3) PbO , Sb_2O_3 , BaO , CaO , MgO , ZnO which together with the K_2O and Na_2O form at most 35% of the glass. A. COUSEN.

Cord-free glass. COMP. DES LAMPES (F.P. 594,354, 23.1.25. Conv., 25.1.24).—Cord-free glass for optical purposes is prepared within a vertically-arranged cylindrical container, which either consists entirely of a platinum-iridium alloy or of tungsten with an inner lining of platinum-iridium alloy. The upper portion of the cylinder is widened to a funnel shape, the lower part narrowed. The truly cylindrical portion is provided with two superimposed heating-wire coils, the narrow portion with a third heating coil. In the upper portion of the cylindrical tube is a stirring device, movable in a vertical direction. Beneath the exit opening is a dish capable of rising and falling in order to receive molten glass. The apparatus is filled to the upper rim with the batch, which is melted in the cylindrical part, with stirring. The lower portion of the cylinder is more strongly heated, and then the exit freed by the melting of the batch. When sufficient glass has flowed out the opening is again closed by discontinuing the heating. A. COUSEN.

Tunnel ovens for ceramic and chemical purposes. H. T. PADELT (E.P. 245,005, 16.6.25).—A tunnel oven has mixing chambers for gas and air disposed in rows one above the other and side by side in the oven wall, and air and gas flues having separate control devices open into the mixing chambers. The mixing chambers and burners are arranged in groups having separate gas and air flues. The gas supply may be increased or decreased at will in accordance with the temperature and output required, and the regulation can also be so performed as to obtain a higher or lower temperature at the higher or lower part of the tunnel as required.

P. B. ROBINSON.

Melting magnesite-sand. C. FIEDLER (G.P. 421,428, 24.7.24. Conv., 26.7.23).—The sand is bonded with a magnesium chloride cement and the pieces, together with natural lump magnesite, are fired in suitable furnaces. A. COUSEN.

Stone- or porcelain-like mass. H. MORINKROP (Swiss P. 103,400, 13.11.22; G.P. 421,422, 13.11.23).—The material consists of 84–78.5% SiO_2 , 1.54–1.47% Al_2O_3 , 5.85–5.5% CaO , 1.13–1.02% K_2O , and 7.26–13.5% Na_2O . It is obtained by melting a mixture of sand, a natural fluoride (as fluorspar), a double silicate of aluminium (felspar), and soda ash. It is highly acid-resisting and a good electrical insulator. A. COUSEN.

Red glazes and enamels. PATENT-TREUHAND GES. FÜR ELEKTRISCHE GLÜHLAMPEN, Asses. of

H. NACHOD (G.P. 421,424, 27.5.24).—Red glazes and enamels are prepared by adding chromates, particularly dichromates, and one or more opacifying agents to a basic glass. A layer of the glaze 0.05 mm. thick is coloured, by this means, red throughout.

A. COUSEN.

Fuel feeding arrangement for tunnel furnaces. J. B. MONNIER (E.P. 232,929, 10.3.25. Conv., 25.4.24).

IX.—BUILDING MATERIALS.

Aluminous cements. G. AGDE and R. KLEMM (Z. angew. Chem., 1926, 39, 175–186).—Bauxite from the Vogel mountains can be used as a raw material for aluminous cement, the chemical composition of the product falling well within the range of these cements on the Gibbs triangular system. Microscopical examination shows the cements to consist of an amorphous ground-mass together with crystals of mellith; on setting, similar effects are observed to those obtained with Portland cement, the amorphous material being attacked first by the water and converted into a mass of interlocking crystals which probably account for the quick-setting properties of the cement. Subsequently, lime and alumina go into solution, and then colloidal alumina and silica are deposited in a reactive "gel" form, which is converted into calcium silicates, aluminates or aluminosilicates, thus causing the hardening of the cement. A range of products can be obtained in which a gradual transition from aluminous to Portland cement occurs. Materials with a very high lime content appear to set very rapidly, but develop no subsequent strength. Replacement of a large proportion of the alumina in aluminous cements by ferric oxide reduces the strength, since calcium ferrite has no binding powers. Aluminous cements are very resistant to the attack of acids (10% potassium bisulphate solution), unless the iron content is high, and to magnesium sulphate solutions.

B. W. CLARKE.

Design of concrete mixtures. D. A. ABRAMS (Bull. I., Struct. Materials Res. Lab., Lewis Inst., Chicago, 1918. Revised 1925. 20 pp.).—The strength of concrete depends on the ratio of water to cement used, an increased water-ratio giving decreased strength. To produce concrete of a given strength with the minimum of cement, the aggregate must be selected and graded to produce a workable concrete with the minimum quantity of water. The "fineness modulus," *i.e.*, the sum of the percentages by weight of the material retained by successive sieves in the Tyler standard series, forms a rational basis for proportioning the aggregate. Tables are given for determining the relative proportions of fine and coarse aggregate required to produce an aggregate of the desired "fineness modulus," which will determine the consistency of the concrete used. Concrete of a given strength, *i.e.*, water-ratio, may therefore be produced by an infinite variety of mixtures of fine and coarse aggregate and cement, and the selection of the mixture to be used will depend on the degree

of plasticity or workability required, cost and availability of various types of aggregate, and other properties desired. Thus maximum density of concrete (*i.e.*, lowest percentage of voids) is not always produced by the grading of aggregate giving maximum strength, this discrepancy being greater for richer mixes. Tables and charts are given whereby the best concrete mixture can be designed for any given conditions.

B. W. CLARKE.

Blast-furnace slag as a building material.

R. GRÜN (Deuts. Bergwerks.-Ztg., Festnummer Bauwesen u. Baustoffind. Reprint. 4 pp.).—Brief descriptions are given of the preparation and use of crushed slag, slag sand (granulated slag), slag stone, slag cement (from granulated slag and lime), and iron Portland cement and blast-furnace cement (from slag and Portland cement clinker).

B. W. CLARKE.

Mercuric chloride [corrosive sublimate] for timber impregnation. R. FALCK and S. MICHAEL (Z. angew. Chem., 1926, 39, 186—193).—Mercuric chloride, although apparently taken up in large amounts by timber, is in reality only adsorbed by the wood substance, and does not form any chemical compound with the wood. The mercuric chloride can be recovered almost quantitatively as such by extracting the wood with water, and the use of mercuric chloride for timber preservation is therefore useless in exposed conditions from the point of view of durability and also dangerous. Further, owing to the adsorption of the sublimate by the fibres, the penetration into the wood itself is very small, the mercuric chloride remaining concentrated on the surface. A mixture of sodium fluoride and mercuric chloride does not give improved results, since no chemical compound is formed analogous to that obtained with sodium chloride and mercuric chloride, and the constituents therefore react independently. Further, the mercuric chloride is liable to be reduced in contact with metal fittings etc. to mercury and mercurous chloride, which has no poisonous or disinfectant properties.

B. W. CLARKE.

Gypsum-sulphuric acid process. MÜLLER.—See VII.

PATENTS.

Bituminous mixtures for road construction etc. UNIVERSAL RUBBER PAVIORS (MANCHESTER, 1923), LTD., and A. E. BROWN (E.P. 246,186, 4.4.25).—Clay, ground so that 95—98% passes a 200-mesh sieve, and dried at 300—400° to remove hygroscopic water, is agitated relatively slowly with bitumen at 80—100°, forming a colloidal dispersion of the clay, which is not liable to settle out on storing and is suitable as a grouting or covering material for road work and the like.

B. W. CLARKE.

Bituminous mixtures for road construction. UNIVERSAL RUBBER PAVIORS (MANCHESTER, 1923), LTD., and A. E. BROWN (E.P. 246,065, 4.4.25. Addn. to 246,186; preceding).—Residual products of gutta-percha, balata, or low-grade rubber-like materials, *e.g.*, gutta-percha pitch, are added to

bituminous mixtures prepared as described in the original patent, forming a composition which adheres well to concrete and road surfaces, and is stable under all conditions of climate and traffic.

B. W. CLARKE.

Bituminous compositions. W. J. MELLERSH-JACKSON. From G. C. WARREN (E.P. 246,548, 22.10.24).—Rubberised fabric is mixed with mineral aggregate (gravel, crushed stone, etc.) at a temperature (120—200°) sufficient to soften the rubber, and after agitating violently a molten bituminous cementing material is added (generally at 120—180°) and thoroughly amalgamated. The rubber gives resiliency and the fibres of the fabric give strength and durability to the composition, which can be used for road surfacing, flooring, roofing, and the like.

B. W. CLARKE.

Making [non-adhesive] bituminous emulsions. L. KIRSCHBRAUN (U.S.P. 1,567,061, 29.12.25. Appl., 17.1.21. Renewed 31.3.24).—In the preparation of emulsions of asphalt dispersed in water containing colloidal clay (*cf.* U.S.P. 1,302,810; B., 1919, 494 A) the tendency of the clay to surround the asphalt particles and render the whole non-adhesive is increased by addition of an acid flocculating agent such as aluminium sulphate or sodium hydrogen phosphate.

T. S. WHEELER.

Drying and calcining gypsum. C. DRESSLER, Assr. to AMER. DRESSLER TUNNEL KILNS, INC. (U.S.P. 1,571,189, 2.2.26. Appl., 25.2.20).—The material is transported through a long horizontal heating chamber, terminating at one end in a depending portion, by a number of conveyor belts arranged longitudinally one above the other, so that at the discharge end the upper belt projects beyond the belts beneath it and at the opposite end the lower belt projects beyond the belts above it.

B. W. CLARKE.

Manufacture of Portland cement and sulphuric acid. F. G. GOETZMANN (F.P. 596,423, 3.4.25).—Gypsum or anhydrous calcium sulphate is mixed with clay, sand, and a small quantity of coal, and the mixture so finely ground that, at the most, 8% of residue is left on a 4900-mesh per sq. cm. sieve. The mixture is slightly moistened and then heated at 1400—1500° in a rotary kiln. The sulphur dioxide evolved is converted into sulphuric acid.

A. COUSEN.

Cement from blast-furnace slag. R. GRÜN (G.P. 421,776, 11.3.23).—A cement resistant to salt water is made by grinding blast-furnace slag with highly aluminous clinker or with fused aluminous cement in the same proportions as are usual with blast-furnace and iron-Portland cements. The products are sufficiently resistant for all building purposes and they are more resistant to salt water, and cheaper than aluminous cement.

A. COUSEN.

Magnesia cement. FARBENFABR. VORM. F. BAYER UND Co., Assees. of H. VON KÉLER and J. DRUCKER (G.P. 421,987, 21.2.24).—An aqueous

suspension of crystalline magnesium chloride or an aqueous solution of the chloride is mixed with magnesia in desired proportions and the resulting powder is heated to about 250°. The magnesium chloride solution and magnesia may be mixed by simultaneous spraying and the mixture dried by hot gas. Bonded magnesia cement or materials prepared from magnesia cement may be regenerated by heating to somewhat above 250°. A. COUSEN.

Refractory cement and concrete. P. J. F. KESTNER (U.S.P. 1,573,072, 16.2.26. Appl., 12.3.25).—See E.P. 231,141; B., 1925, 719.

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

Production of iron from silicates and pyrites. G. TAMMANN and G. BÄTZ (Z. anorg. Chem., 1926, 151, 129—139).—When hornblende or basalt is mixed with wood charcoal and a small proportion of lime or sodium carbonate and heated first at 600° and then at 1400°, the iron is reduced almost quantitatively. In the absence of alkali, or when heated directly to 1400°, the reduction is less complete, and it is further diminished by the addition of quartz. When heated with precipitated and ignited silica, ferric oxide, magnetite, and ferrous oxide each begins to react at 800°, but reaction with quartz does not begin below 950°. More heat is evolved with the silica than with quartz. Solid ferric oxide and magnetite do not react directly with silica, but only after loss of oxygen and formation of ferrous oxide; this begins at 800°. When iron silicates, prepared by heating ferric oxide and silica, are reduced with hydrogen for one hour at 850°, the proportion of the iron reduced falls from 24.4% when the molecular fraction of silica is 0.375 to 0.3% when it is 0.600. When the silica content is further increased the proportion of the iron reduced rapidly increases, and is 38.4% when the molecular fraction of silica is 0.920. When iron pyrites is heated a halt in the heating curve occurs at 684°, due to loss of sulphur. In the presence of lime no halt is observed; the liberated sulphur combines with the lime to form calcium sulphate and sulphide. A mixture of pyrites and lime is only slightly reduced when heated with wood charcoal. By the interaction of lime and pyrites a difficultly reducible calcium iron oxysulphide is formed, and only a small quantity of ferrous oxide.

A. GEAKE.

Influence of alternating currents on the electrolytic corrosion of iron. A. J. ALLMAND and R. H. D. BARKLIE (Faraday Soc., Feb., 1926. Advance proof).—Experiments on the corrosion of iron in alkaline solutions by direct current confirmed those of other workers, the presence of chlorine ions activating the metal. When alternating current was used, change in the alkali concentration of the electrolyte had no particular influence, but lowering the current frequency produced an increased corrosive effect, and there seems little doubt that the discharge of hydrogen ions during the cathodic

impulse activates the iron by decreasing the oxygen content in the surface layer of metal. The effect of alternating current superposed on direct current was a marked increase in corrosion, and where an anode remained passive either at a D.C. intensity of 0.125 amp./cm.² or at an A.C. intensity of 0.375 amp./cm.² and 400 frequency, the anode became definitely yellow in two minutes under the combined effect. A high AC:DC ratio (more than 2) was favourable to corrosion, as was also an increase in current density and low frequency. Typical sub-soil drainage liquid saturated with carbon dioxide yielded similar results. C. A. KING.

Widmannstätten structure in iron-carbon and iron-nickel alloys and in meteorites. T. KASÉ (Sci. Rep. Tôhoku, 1925, 14, 537—558).—Hypoeutectoid steel gives the Widmannstätten structure on annealing above 1000° or on heating it to partial melting and cooling in air from a temperature above the Ar₃ point. Hypereutectoid steel must be cooled from above the Ac_m point. Quenching is not an effective method and cooling slowly through the A₁ transformation point destroys the characteristic structure. The Widmannstätten structure is obtained in iron-nickel alloys by a long annealing above the Ac₃ point and then quenching in water, and the formation of this structure in meteorites may be due to a long exposure to a very high temperature and subsequent rapid cooling. C. A. KING.

Self-magnetisation of steels under torsion. R. CAZAUD (Compt. rend., 1926, 182, 467—468).—A study of the self-magnetisation of steels under torsion suggests that such measurements may be used to obtain rapid indications of composition and of thermal treatment. Test-pieces of steels containing from 0.3 to 1.0% of carbon were placed, under torsion, in the magnetic field of a doubly-wound bobbin. With a constant current flowing through the primary winding of the bobbin, any deformation of the test-piece through torsion causes a variation of the magnetic flux, and sets up induced currents, recorded photographically, in the secondary coil. Torsion produces a negative variation of the magnetic flux in mild steels containing less than 0.5% of carbon, and a positive variation with steels in which the carbon content is more than 0.5%. With extra hard steels, cast steels, and certain special steels, of martensitic structure, the variation of the flux with torsion is continuous. Repeated torsions cause the steels to approach a state of magnetic saturation. Cold working tends to increase the variations produced in the magnetic flux by torsion. F. G. TRYHORN.

Specific volume determinations of carbon and chromium steels. J. H. ANDREW, M. S. FISHER, and J. M. ROBERTSON (J. Roy. Tech. Coll. Glasgow, 1925, [2], 70—78).—The specific volume of steels containing up to 1.2% C increases as the temperature of quenching is raised to an extent which is greater the higher the carbon content. This is evidently due to expansion of the martensite. With more than 1.2% C austenite is produced in amounts which increase with rise of quenching temperature so

that the specific volume of the steel begins to decrease again. This decrease is most marked after quenching from 1100°. If, however, the same steels are heated to 1100° for a short time, allowed to cool to 1000—800°, and then quenched, the specific volumes are extraordinarily high, possibly owing to graphitisation having taken place. The increase in specific volume on quenching indicates that martensite is a solid solution of cementite in ferrite in which the iron lattice has been expanded by carbon and that the amount of this expansion produced by a definite quantity of carbon in solution exceeds the volume of the corresponding quantity of cementite. The specific volume curves for chromium steels are similar to those for plain carbon steels. The effect of tempering chromium steels with more than 1% C is first to reduce slightly the specific volume then between 200° and 300° to cause it to increase rapidly corresponding with the tempering of the austenite; above 300° simultaneous tempering of austenite and martensite results in a decrease in the specific volume. With a low-carbon chromium steel a steady fall in the specific volume takes place with rise in temperature of tempering. Austenitic carbon steels with or without chromium increase in specific volume after immersion in liquid air although the electrical properties remain unchanged.

A. R. POWELL.

Crystalline structure of metals. J. H. ANDREW (J. Roy. Tech. Coll., Glasgow, [2], 63—69).—A speculative discussion in which the author attempts to correlate the atomic structure of metallic crystals with the physical properties of the metal, with especial reference to iron and steel. The part played by surface tension at the grain boundaries of a polycrystalline substance in determining the hardness is briefly discussed with reference to the volume changes on quenching and tempering martensitic steels (cf. preceding abstract).

A. R. POWELL.

Magnetic properties of permalloy. D. BINNIE (J. Roy. Tech. Coll., Glasgow, 1925, [2], 5—7).—The initial permeability of annealed permalloy (78.5% Ni, 21.5% Fe) is 30 times that of the best soft iron and a field as low as that of the earth will saturate the alloy to a magnetic intensity comparable with that of soft iron. The magnetic properties are, however, very sensitive to strain, which causes a marked diminution of the susceptibility. Thus, a thin strip of permalloy after coiling and uncoiling exhibited magnetic properties similar to those of steel.

A. R. POWELL.

Cementation of ferrous alloys by tungsten. J. LAISSUS (Compt. rend., 1926, 182, 465—467; cf. B., 1925, 635).—The action of finely powdered ferrotungsten as a cementing agent for electrolytic iron and for steels containing from 0.15% to 0.8% of carbon, has been studied. The temperature of cementation varied from 800° to 1100°, and its duration from 2½ to 10 hrs. Photomicrographs of sections showed the existence of two zones: an inner one of solid solution, well developed during the longer heats, and a bright outer layer, consisting probably of tungsten carbide. The thickness of the latter

increased with the time and with rise of temperature. The cementation zone decreased in thickness with increase in carbon content of the steel. Steels cemented with tungsten are more resistant to an oxidising atmosphere at 900° than uncemented steels, up to a period of 5 hrs. Beyond that time, the cemented layer rapidly disintegrates by scaling. The cemented steels have considerable resistance to corrosion in water and sulphuric and hydrochloric acids, but are very rapidly attacked by nitric acid.

F. G. TRYHORN.

Determination of vanadium in ferrovandium. K. SOMBYA (Sci. Rep. Tohoku, 1925, 14, 577—591; cf. B., 1925, 29).—The sample (0.5 g.) is dissolved in nitric acid, the excess expelled completely by evaporation with sulphuric acid, the insoluble material filtered off after addition of 50 c.c. of water, and the solution oxidised with permanganate, nearly neutralised with ammonia, cooled, and diluted to 120 c.c. A few drops of diphenylamine solution and an excess of ferrous sulphate are added, followed by potassium dichromate solution in excess of that required to oxidise the ferrous sulphate. After addition of 10 c.c. of concentrated hydrochloric acid and 15 c.c. of phosphoric acid mixture the excess of dichromate is titrated with ferrous sulphate.

A. R. POWELL.

Wet method for the determination of silver in lead. B. S. EVANS (Analyst, 1926, 51, 79—81).—When cupellation is impracticable silver may be determined in lead by the following procedure: 50 g. of metal are dissolved in nitric acid, ammonia is added until the solution is alkaline, followed by acetic acid until just acid, and the liquid is boiled for 2 hrs. with a coil of copper foil. The precipitated silver and excess of copper are filtered off, washed with hot water, and redissolved in nitric acid. The solution is made just alkaline with sodium hydroxide and again acid with hydrochloric acid, adding a slight excess of the latter to precipitate all the silver. After boiling for 15 min., the solution is kept overnight and filtered. The precipitate is dissolved in 10—15 c.c. of 1:1 ammonia, the solution diluted to 200 c.c., an excess of standard potassium cyanide solution added, followed by a few crystals of potassium iodide, and the excess cyanide titrated with a standard silver solution as in the case of nickel.

A. R. POWELL.

Constitution of the alloys of silver and tin. A. J. MURPHY (Inst. Metals, Mar., 1926. Advance copy, 18 pp.).—A re-examination of the equilibria in the system silver-tin by microscopical, thermal, and electrical resistance methods has confirmed the shape of the liquidus and the composition of the eutectic (96.5% Sn, m.p. 221°) as found by Petrenko (B., 1907, 615) but the constitution of the solid alloys differs widely from that found by earlier investigators. The solid solution of tin in silver contains a maximum of 13.3% Sn at 724° and this slowly falls to just below 11% at 100°. This α solution reacts with liquid at 724° to form a second solid solution, β , which is the sole constituent of alloys containing 12.5—19% Sn at 20° and of alloys containing 13—24%

Sn at 480°; at 724° it contains 14.5% Sn. The $\alpha + \beta$ field is a narrow belt extending over the range 13.3—14.5% Sn at 724° and 11—13% Sn at 20°. The γ constituent is the compound Ag_3Sn , which exists in a narrow range of less than 1% about 26% Sn, the $\beta + \gamma$ field occupying the space between the β and γ ranges mentioned above. Alloys containing more than 27% Sn are all composed of γ and tin containing less than 0.1% Ag in solid solution. The α solid solution is characterised by prolific twinning accompanied by the development of finer marking following the course of cleavage planes in the crystals. The β solid solution consists entirely of polygonal crystals without any surface markings and the compound Ag_3Sn , after prolonged annealing, of polygonal crystals with fine cross-hatch markings. The inversion in this compound at 232° recorded by Petrenko has not been confirmed, but by electrical resistance measurements a transformation of unknown nature has been found to take place at 60°. The addition of even very small quantities of silver to tin suppresses both the allotropic transformations, 0.1% Ag preventing the tin from changing to the grey variety even after 3 weeks at -78°. A. R. POWELL.

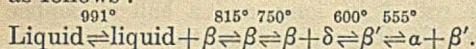
Influence of gases on copper at high temperatures. I. A. G. LOBLEY and D. JEPSON (Inst. Metals, Mar., 1926. Advance copy, 13 pp.).—A special type of electric resistance furnace used in the experiments is described. The furnace can be evacuated or filled with any desired gas. The crucible containing the copper can be lowered from the heating zone into a water-cooled chamber, thus giving a rapid and reproducible rate of cooling. The behaviour of the copper while molten and during solidification was observed through observation windows in the top and side of the furnace. The charge consisting of 220 g. of pure copper was heated in nitrogen, hydrogen, and carbon monoxide at atmospheric pressure to various temperatures between the melting point (1083°) and the boiling point (2310°) for 30 min., after which the crucible was lowered into the cooling chamber and rapidly cooled. The resulting ingots were examined for blowhole volume (by apparent density), hardness, and macrostructure. The Brinell hardness numbers were in no case affected by the treatment. The period and intensity of ebullition on cooling were observed and the weight of copper thrown up as spray was determined, and from these data a confirmation was obtained of the relation between the amount of gas held in the cavities of the solidified ingot to the total amount driven out. Up to temperatures of 1900° and 1700° respectively, nitrogen and carbon monoxide are not absorbed by molten copper in excess of that soluble in the solid metal. Hydrogen is absorbed to a varying degree at all temperatures between the melting point and 2200°, and is ejected on solidification, forming blowholes. The blowhole volume-temperature curve indicates a fall from the melting point to a minimum at approximately 1350°, rising to a maximum at about 1750°, and then falling as the temperature rises to approximately 2200°, the last fall being

probably connected with the increasing vapour pressure of the copper. A. W. HOTHERSALL.

Determination of zinc oxide in brass. B. S. EVANS and H. F. RICHARDS (Inst. Metals, Mar., 1926. Advance copy, 7 pp.).—Purified hydrogen is passed over a sample of the brass weighing about 80 g. at a temperature of 980° (at which the brass is molten but the volatilisation of zinc is not excessive), and the water formed is absorbed and weighed. The reverse reaction by which the water formed is reduced in the cooler parts of the furnace by the volatilised zinc is suppressed by the insertion on either side of the combustion boat of a tightly fitting copper gauze roll, 5 in. long, and a ball of copper wire rolled to fit the combustion tube. Both the rolls and balls are deoxidised prior to the determination by heating to 1000° for some hours in a current of hydrogen. The hydrogen gas is passed at a measured rate of about one litre per hour, both the blank and the actual test being continued until a constant increase in the weight of the absorption tube is obtained per litre of gas passed. The accuracy of the method was established by reduction of weighed amounts of zinc oxide, the preparation of a standard sample of brass containing a known amount of zinc oxide having been found impracticable. The amount of oxygen present in a series of commercial brass ingots was found to vary from 0.002% to 0.005%. A. W. HOTHERSALL.

Copper-rich aluminium-copper-tin alloys. D. STOCKDALE (Inst. Metals, Mar., 1926. Advance copy, 27 pp.).—The liquidus curve of aluminium-copper alloys falls to a minimum at 8.3% Al and then rises to a maximum at 12.5% Al (cf. B., 1922, 818 A); addition of tin reduces the melting points of these alloys in such a way that if the temperature is plotted against the atomic percentage of copper, the liquidus curve of any series of alloys containing a constant proportion of tin is nearly parallel to that of any similar series, i.e., the minimum at 82 at. % Cu and the maximum at 75 at. % Cu are independent of the kind of foreign atom present. From these results it is shown by van't Hoff's law that the first solid to separate at the maximum contains no tin and that the latent heat of solidification of a solution of one atom of aluminium in three atoms of copper is the mean of its constituents, indicating that no combination to Cu_3Al takes place. Up to 9% Sn the eutectic line of the solidus becomes much depressed so that a valley runs through this part of the ternary model; on the left of this valley the α solid solution of aluminium and tin in copper separates out and on the right the β solid solution is formed from the liquid and α phases. The limit of the α solid solution at room temperature is very nearly a straight line on the ternary triangular diagram. The field of the β phase is rendered extremely complex by the addition of tin, decreasing progressively as the tin increases up to 9%. The eutectoid transformation temperature is raised considerably in the alloys richest in copper by adding tin, which also reduces the apparent velocity of the transformation. This change takes place at approximately constant

temperatures for alloys containing the same percentage of aluminium but varying amounts of tin, e.g., the alloys containing 7% Al with 5, 7, or 9% Sn all undergo the transformation at 600–610°. Alloys comparatively rich in aluminium and tin and consisting of the β phase at high temperatures undergo complicated changes on cooling; at first the δ phase separates, but, on further cooling, this appears to redissolve in the remaining β or reacts with it to form a new phase which cannot be distinguished microscopically from β but which appears to be polymorphous with it. The phase changes on cooling an alloy with 9% Al and 9% Sn may be represented as follows:



A. R. POWELL.

Mechanical properties at high temperatures of an alloy of nickel and copper with especial reference to "creep." H. J. TAPSELL and J. BRADLEY (Inst. Metals, Mar., 1926. Advance copy, 19 pp.).—Tensile, torsion, impact, fatigue, and hardness tests at various temperatures up to 800° are recorded for an alloy consisting of 68.74% Ni, 28.16% Cu, 2.35% Mn, 0.56% Fe, 0.10% C, 0.008% S. The ultimate stress falls slightly with increase of temperature from 37 tons per sq. in. at 15° to 33 tons per sq. in. at 400° and thereafter rapidly to 7.5 tons per sq. in. at 800°. The elongation is 46% at 15°, 38–35% at 100°–400°, 19% at 500°, 30% at 700°, and 20% at 800°; the reduction in area remains fairly constant at about 70% up to 300°, then falls rapidly to a minimum of 21% at 500°, increasing again to 30% at 700° and once more falling with further rise in temperature, whereas the limit of proportionality falls steadily with increase of temperature. The limiting "creep" stress decreases at about the same rate as the ultimate tensile strength from 24 tons per sq. in. at 400° to less than 1 ton per sq. in. at 700°, while the modulus of rupture remains constant to 300°, then falls rapidly, and the resistance to impact of a notched bar falls slowly throughout the range. The impact hardness number remains nearly constant at about 234–224 up to 300°, then falls slowly to 185 at 400°, remaining constant at this figure up to 600° and falling to 151 at 700°. The limiting fatigue range is ± 15 tons per sq. in. at 15° and ± 11.7 tons per sq. in. at 500° and the mean coefficient of expansion between 0° and 800° is 0.000017.

A. R. POWELL.

Sand-cast aluminium-manganese alloys. S. DANIELS (Ind. Eng. Chem., 1926, 18, 125–130).—The properties of a number of sand-cast aluminium-manganese alloys containing up to 9.6% Mn have been investigated. The tensile strength was raised from 5.25 tons/in.² for the alloy containing no manganese to 7.6 tons/in.² with 1.02% Mn, but the elongation (on 2 in.) fell rapidly from 38.5% to 22.6% and then to 2.6% with no manganese, 1.02% and 2.88% Mn respectively. The Brinell hardness numbers showed a gradual increase from 20 to 48 with increasing manganese content. The presence of manganese increased the shrinkage and promoted

porosity when present in appreciable quantity. The alloy containing 4.6% Mn machined like cast iron, probably owing to the presence of the hard and brittle compound MnAl₃. The alloys were readily attacked by distilled water (30 days, immersion), but showed only slight corrosion after 100 hrs. in salt spray. Heat treatment at 580° for 96 hrs. followed by quenching in water and ageing at 149° for 2 hrs. did not materially alter either the physical or mechanical properties or the corrodibility. The metallography is discussed and a number of photomicrographs are reproduced.

A. W. HOTHERSALL.

Physical properties of very light magnesium alloys. A. PORTEVIN and F. LE CHATELIER (Compt. rend., 1926, 182, 382–384).—For magnesium alloys, the following formulæ express the specific volume (V) and electrical resistance (R) as functions of x (the percentage of added metal) $V = 0.575 - K_v x$ in cm.³/g. and $R = 4.5 + K_r x$ in microhms/cm³/cm. The values for K_v and K_r found for various added metals, the limiting values of which are given in brackets, are: Al (7%), 0.0022, 1.34; Cd (6%), 0.0047, 0.15; Cu (13%), 0.0052, 0.01; Mn (4%), 0.004, 0.13; Ni (15%), 0.00538, 0.06; Pb (5%), 0.0056, 0.58; Zn (5%) 0.0043, 0.28. For aluminium and zinc the values given in brackets represent the limiting solid solution and the limit of homogeneity respectively; in other cases they represent the limiting alloy examined. The coefficient of expansion is in no case greatly different from that of magnesium; zinc and lead cause an increase whilst copper, nickel, and especially silicon cause a decrease. Ternary alloys have also been investigated. Taking into account the mechanical properties it is concluded that the binary magnesium alloys with nickel and copper, and the ternary alloys with aluminium-nickel and aluminium-copper promise to be most useful. For certain applications such as motor pistons they give the most suitable values for density, elastic limit, hardness, heat conductivity, and expansion.

R. A. MORTON.

Recent developments in metallurgical research. New facts developed by high-power metallography. F. F. LUCAS (J. Roy. Microscop. Soc., 1925, 393–404).—The examination at high magnifications of austenite containing a network of martensitic figures shows that the martensitic needle is always characterised by a central mid-rib or axis which etches darker with picric acid than the remainder of the needle. The directions of the axes of martensitic needles within any similarly oriented portion of an austenite grain indicate that martensite results from decomposition along the crystallographic planes of austenite. No case was found of a martensitic needle crossing a grain boundary or a twinning plane of austenite. Multiple twinning bands of the order of 0.00001 in. are often found in martensitic needles. Two varieties of troostite, nodular and flocculent, are recognised. The former may develop directly from austenite, or from an austenite-martensite matrix, in which case it has its origin in a martensitic needle either along the mid-rib or at the junction of two needles.

After the nodule has formed, the excess constituent is rejected to the grain boundaries and at the same time stratification sets in and the radial grain of the troostitic nodule is transformed into a grain of pearlite. Whereas nodular troostite responds to a test for crystallinity the flocculent variety does not, probably because the particles composing it are not within the range of resolution. Evidence has been obtained that the precipitation of ferrite marks the very early stages of the transformation of austenite and martensite into pearlite under definite conditions of heat treatment. The amount of flocculent troostite formed is a measure of the excess constituent present. A steel of eutectoid composition yields little or no troostite and the austenite and martensite form pearlite without passing through a troostitic stage. Photomicrographs at 3950 diameters magnification illustrate the different stages in the decomposition of austenite in a specimen of previously normalised 1% C steel which was heated to a high temperature at one end while the other end was maintained at room temperature, and the whole then quenched in iced brine. The effects of cold work and the mechanics of the process of regranulation by annealing were studied in a billet of permalloy (78.5% Ni, 21.5% Fe) cold-rolled from 1-in. to $\frac{3}{8}$ -in. rod, one end of which was subjected to a long anneal and the other maintained cold. In addition to slip-bands, incipient fissures were observed. Twinned areas developed gradually by reorientation of distorted metal. Small areas within the same grain developed a more pronounced orientation towards the annealed end of the rod. At low magnification the large elongated grains produced by cold work persisted in phantom outline after the metal had regranulated on annealing.

M. COOK.

High-power metallography. F. F. LUCAS (J. Franklin Inst., 1926, 201, 169—216; cf. preceding abstract).—The application and limits of high magnification in metallurgical photomicrography are discussed. The process of the decomposition of austenite to pearlite in a 0.5% carbon steel is illustrated with photomicrographs at a magnification of 3230 diameters. The extreme and intermediate stages are obtained in one specimen by suitable heat treatment. The hardened end of the specimen consists of a network of martensitic needles in austenite. On approaching the abrupt transformation troostite appears along some of the martensitic needles and ferrite soon appears in the centre of the troostitic areas. As the troostite increases it forms a border enclosing areas of martensite and austenite. It continues to increase until it reaches a maximum and then decreases, leaving large areas of austenite and martensite. The central areas of ferrite increase with the troostite, and when no more of the latter is formed the ferrite increases at its expense until it disappears except for a small border surrounding large areas of ferrite. This residual troostite is of eutectoid proportions and stratifies, assuming the orientation of adjoining grains. At the same time small cubic pits develop in the austenite-martensite

and increase in number along the cleavage planes of the austenite. These pits unite to form long bands of ferrite, showing that stratification takes place in austenite and martensite areas without these areas passing through troostite. The troostite which is formed is a rough measure of the excess ferrite present. Nodular troostite was obtained in 0.5% carbon steel quenched under uniform conditions. The phenomenon of regranulation on annealing after cold work is illustrated with photomicrographs at high magnifications.

M. COOK.

Liquation in molten alloys and its possible geological significance. S. W. SMITH (Inst. Min. Met., Feb., 1926. Advance copy, 48 pp.).—The liquation phenomenon in molten alloys known as inverse segregation is discussed at length, and it is shown that this behaviour is determined by differences in temperature between different portions of a homogeneous molten mixture before solidification commences and that the constituent which liquates towards the cooling surface is that which, if added in further quantity to the mixture under consideration, would lower its freezing point. Reasons are also advanced for considering that the Ludwig-Soret effect in solutions is identical with liquation in molten alloys and that the liquation phenomena observed in alloys are applicable to molten matter in general. Preliminary experiments on copper matte containing 29—3% Cu have been carried out to test this theory and distinct evidence of the liquation of copper towards the chilled surface has been obtained. On the basis of these results the mode of primary copper enrichment in certain pyritic ore-bodies is discussed.

A. R. POWELL.

Volatilisation of lead during lead burning, using different flames. H. ENGEL and V. FROBOESE (Arch. Hyg., 1925, 96, 69—101; Chem. Zentr., 1926, I., 1479).—Volatilisation of lead during such processes as lead burning and applying homogeneous lead coatings, takes place to the greatest extent with the oxy-hydrogen flame, using an excess of oxygen, but is reduced considerably by carburising the gas, or by using coal gas instead of hydrogen; it is a minimum when a pale, non-sooting acetylene flame is used. Decrease in the volatilisation when hydrocarbons are present in the gas appears to be due to the formation of a carbon monoxide-hydrogen zone in the flame. No lead hydride was detected in the combustion gases, the volatilised material appearing to consist principally of lead monoxide mist in a sufficiently fine state to remain in suspension in the air for a long time.

L. A. COLES.

Composition of copper mattes. B. BOGITCH (Compt. rend., 1926, 182, 468—470; cf. B., 1926, 194).—Equilibrium conditions have been determined in the system Cu-S-Fe, in the liquid state, and at temperatures near the solidification point. Ferrous sulphide and iron are miscible in all proportions in the liquid state, but addition of copper, in amounts more than 3%, causes a separation into two layers, the upper high in sulphur content, the lower very low in sulphur content. As the amount

of copper is increased, most of the metal passes into the upper layer, until its copper content reaches about 50%. Copper added above this amount tends to collect in the lower layer, until this contains 94–95% Cu. Beyond this point, additional copper tends to collect in the upper layer. The limiting system $\text{Cu}_2\text{S}-\text{Cu}$, free from iron, consists, in the liquid state, of two conjugate solutions. The bearing of the phase diagram for the above system on the metallurgy of copper is discussed.

F. G. TRYHORN.

Successive potentiometric titration of copper and iron in metallurgical products. T. F. BUEHRER and O. E. SCHUPP, JUN. (*Ind. Eng. Chem.*, 1926, 18, 121–124).—0.3–0.4 g. of the sample is dissolved in aqua regia and the solution is evaporated to dryness with excess of strong hydrochloric acid. The residue is digested with a minimum of water acidified with 1–2 c.c. of strong hydrochloric acid and the solution is filtered. The filtrate is acidified with 5–10 c.c. of strong hydrochloric acid and is covered with a layer about 1 in. thick of kerosene, previously purified by successive treatment with several charges of concentrated sulphuric acid. 10–15 c.c. of chromous chloride solution in excess of that required to turn the solution red with reduced copper are added, the reduction of both copper and iron being prompt and complete if the solution is kept agitated. The solution, the volume of which should be limited to 50–70 c.c., is titrated with potassium dichromate solution, galvanometer readings being taken at intervals of 0.1 or 0.2 c.c. around the inflection point, and the true end point obtained by interpolation. The chromous chloride solution, which is kept under a layer of kerosene, maintains a fairly constant normality whether in acid or nearly neutral solution or in contact with rubber. It is prepared by boiling potassium dichromate in hydrochloric acid solution with excess of zinc free from arsenic and iron until reduction to the light blue solution is complete. Quantitative and reproducible results were obtained for copper and iron simultaneously by this method and also by direct titration with chromous chloride solution, the time taken being less than half that ordinarily required. Reproducible results could not, however, be obtained either by direct titration with stannous chloride or with titanous chloride or by titration with potassium dichromate after reduction with stannous mercuric chloride (Penny's method for iron), stannous chloride alone, or with titanous chloride. Molybdenum, selenium, arsenic, and antimony might interfere with the method if present in considerable quantity. The method has not been applied to products very low in copper such as slags and tailings.

A. W. HOTHERSALL.

Critical studies on the fusion of rare metal ores. II. Separation of tantalum and columbium [niobium]. G. W. SEARS (*J. Amer. Chem. Soc.*, 1926, 48, 343–348; cf. A., 1925, ii., 580).—A method is given for the separation of tantalum and niobium by the pyrosulphate fusion of tantalite

and columbite. The temperature of fusion has a great influence on the subsequent solubility of the tantalum and niobium compounds, the best temperature for the separation by means of concentrated sulphuric acid (1:1 acid dissolves niobium completely, whilst the tantalum is left in the residue) being 835–875°. The proportion of pyrosulphate used in the fusion mixture and the time of heating have little or no effect on the solubility. Concentrated sulphuric acid is a more effective differentiating solvent than hydrofluoric acid. The compounds formed by the fusion are probably tantalates and niobates rather than sulphates or double sulphates.

W. THOMAS.

Corrosion of iron. W. G. WHITMAN (*Chem. Reviews*, 1926, 2, 419–435).

See also A., Mar., 229, **Electrical conductivity of silver-zinc alloys** (PETRENKO). 230, **Plasticity of amorphous and crystalline solids** (BECKER). 231, **Thermal expansion of tungsten** (HIDNERT and SWEENEY). 233, **Vapour tensions of mercury, cadmium, and zinc** (JENKINS). 248, **Cathodic pulverisation of alloys** (MAZUR). 254, **Solid metal-hydrogen compounds** (HÜTTIG). 261, **Electrometric determination of tellurium** (SCHRENK and BROWNING); **Determination of potassium as perchlorate** (YOE); **Determination of rubidium and caesium** (STRECKER and DIAZ). 262, **Separation of lead and silver** (VOETMANN and HECHT); **Determination of thallium** (STRECKER and DE LA PENA); **Electrolytic separation of copper from cadmium** (JILEK and LUKAS); **Titration of mercury with potassium cyanide, lead with arsenate, and iron with thiosulphate** (JELLINEK and CZERWINSKI). 263, **Confirmatory test for aluminium** (ESTILL and NUGENT); **Separation of iron and aluminium from zirconium** (LESSING). 264, **Distillation of mercury containing gold** (RIESENFELD and HAASE).

Inductive heating. NORTHROP.—See XI.

PATENTS.

Production of castings containing silicon. RHEINISCHE EISENGIESSEREI UND MASCHINENFABR. A.-G. (E.P. 237,554, 27.2.25. Conv., 26.7.24).—High-silicon iron castings are produced in preheated moulds as in the pearlite-casting process. The temperature of the moulds varies from 500° for a 10% silicon content to about 150° for a 20% silicon content, the temperature also being lower the greater the wall thickness. T. H. BURNHAM.

Magnetic [iron-silicon] alloys. V. B. BROWNE (U.S.P. 1,570,229, 19.1.26. Appl., 19.3.25).—Iron is refined and additions of iron ore are made to the bath until no further reduction of the carbon content is obtained. Air or other oxidising gas is then caused to permeate the bath until substantially all of the remaining carbon is removed. The metal is removed from the bath and the necessary silicon addition made to adapt the silicon-iron for use as a magnetic alloy, the carbon content being not over 0.03%. T. H. BURNHAM.

Treatment of iron or steel for preventing oxidation or rusting. T. W. COSLETT (E.P. 247,071, 26.6.25).—Iron or steel surfaces are provided with a corrosion-resisting coating by subjecting them to the action of a solution such as is described in E.P. 8667 of 1906 and 28,131 of 1909 (B., 1907, 207; 1911, 92), *e.g.*, acid zinc phosphate solution, to which is added a compound of boron, *e.g.*, boric acid or borax. Before treatment the iron or steel may be immersed for a short time in a solution of boric acid. During treatment an electric current may be passed from the article as anode to a zinc or iron cathode. T. H. BURNHAM.

Degassing and deoxidising steel. S. PEACOCK and N. E. COOK (U.S.P. 1,570,176, 19.1.26. Appl., 3.3.25).—Anhydrous trisodium aluminate is added to molten iron or steel, whereby compounds with any eutectoids present are formed which melt in the bath of metal, and any compounds of oxygen and carbon associated with the eutectoids are liberated. T. H. BURNHAM.

Alloy steel. W. P. WOODSIDE and C. N. DAWE, ASSTS. to STUDEBAKER CORP. (U.S.P. 1,572,458, 9.2.26. Appl., 24.8.23).—The steel contains 0.10—0.23% C, 0.30—0.60% Mn, 1.40—1.75% Ni, 0.20—0.30% Mo, and 97—98.1% Fe. T. H. BURNHAM.

Annealing iron articles. H. T. GRAFTON and C. W. WEESNER (U.S.P. 1,572,600, 9.2.26. Appl., 30.3.21).—The process consists in the use of purified producer gas substantially free from oxygen and containing an insufficient amount of moisture to stain the iron articles. T. H. BURNHAM.

Deposition of aqueous mineral suspensions. I. TRAUBE (E.P. 246,361, 7.5.25).—The deposition of ores from their aqueous suspensions or slimes is accelerated by the addition of an extract of a plant such as Carrageen moss or Iceland moss, with or without a small proportion of a salt or other electrolyte such as an alkaline-earth sulphate or carbonate. H. HOLMES.

Reverberatory [puddling] furnaces. E. C. LOWNDES (E.P. 246,681, 19.2.25).—A reverberatory furnace, particularly for puddling, is fired with pulverised fuel which is projected with a limited amount of air into a small preliminary combustion chamber where ash is deposited and the organic matter gasified; additional air enters as the gases pass over the fire-bridge into the main part of the furnace. B. M. VENABLES.

Nickel-copper alloy. J. W. LEHR, ASSR. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,567,066, 29.12.25. Appl., 28.8.23).—A copper-nickel alloy containing Ni 20—40% (preferably 35—40%), Sn 1—6% (2.5%), Pb 1—8% is cheaper than Monel metal while possessing all the good qualities of the latter. T. S. WHEELER.

Alloy. J. H. L. DE BATS, ASSR. to DE BATS METALS Co. (U.S.P. 1,572,593, 9.2.26. Appl., 24.1.24).—The alloy contains 15—30% of nickel

and the balance two metals of the chromium group, each not less than 15%. T. H. BURNHAM.

Carbonising box. C. E. MACQUIGG, ASSR. to ELECTRO METALLURGICAL Co. (U.S.P. 1,568,174, 5.1.26. Appl., 21.11.21).—Ferrochromium alloys containing about 20% Cr and 5—7% Cu resist the action of both oxygen and carbon at high temperatures, and on this account are of value in the manufacture of boxes for use in case-hardening steel. The resistance to carbonising is due to the presence of copper, and to oxidation to the presence of chromium. T. S. WHEELER.

Treating metals. D. H. CROSSER, ASSR. to CHICAGO CRUCIBLE Co. (U.S.P. 1,568,271, 5.1.26. Appl., 29.8.24).—Reagents for treating metals are finely divided and briquetted with a large excess of clay to effect better dispersion of the reagents throughout the molten metal. For example, briquettes containing hydrated aluminium silicate (69.5%), nickel (10.4%), ferromanganese (5.6%), ferrosilicon (8.3%), ferrochromium (3.5%), and aluminium (2.7%) are added to iron in a cupola in the proportion of 1 pt. to each 100 pts. of iron. The process can also be applied to the treatment of iron in the blast furnace or the hearth, and to the treatment of copper or aluminium. T. S. WHEELER.

Alloy. P. GIRIN, ASSR. to SOC. ANON. DE COM-MENTRY, FOURCHAMBAULT ET DECAZEVILLE (U.S.P. 1,572,996, 16.2.26. Appl., 9.12.20).—See E.P. 159,857; B., 1922, 470 A.

Apparatus for refining or separating metals. H. HARRIS (U.S.P. 1,573,829, 23.2.26. Appl., 8.12.22).—See E.P. 199,660; B., 1923, 783 A.

Refining lead. H. HARRIS (U.S.P. 1,573,830, 23.2.26. Appl., 8.7.24. Renewed 20.10.25).—See E.P. 189,013; B., 1923, 60 A.

Cement from blast-furnace slag (G.P. 421,776).—See IX.

Zinc white (F.P. 595,396).—See XIII.

XI.—ELECTROTECHNICS.

Inductive heating. E. F. NORTHRUP (J. Franklin INST., 1926, 201, 221—244).—In ironless inductive heating, the principles of which are fully discussed, the necessity of making into ring form the substance to be heated is dispensed with, thus rendering the choice of shape unlimited and permitting of a furnace of crucible form with minimum radiation surface for a given volume. The inductor coil of a metal-melting furnace is a single-layer winding of flattened copper tube or solid copper strap several times as wide as it is thick and is wound edgewise. The "coupling" between the coil and the melt is chiefly determined by the ratio, square of the diameter of the melt divided by square of diameter of the coil. The frequency N is derived from the formula $N \geq 25 \times 10^3 / \rho D^2$, where ρ is the resistance in ohms between opposite faces of a cm. cube of the materia.

and D is the diameter in cm. of the cylindrical charge to be melted. For small furnaces the high frequency needed is best obtained by the discharge-gap oscillator. It is not usual to obtain the high-frequency current by the use of oscillators when the power required for a single unit exceeds about 25 kw. The efficiency of an ironless induction furnace theoretically quite equals that of an induction furnace with transformer iron. Success in working depends on the fact that long wave-length electromagnetic radiation passes without hindrance through any heat insulator into the mass, while short wave-length radiation can only escape with difficulty from the mass, which accounts for the heat-accumulation in the charge and the attainment of very high temperatures. Molten conducting matter acted on inductively is automatically stirred and when alloys are melted the constituents mix thoroughly. Several other advantages of inductive heating are enumerated.

M. COOK.

See also A., Mar., 229, Detection of wireless waves by crystals of galena and pyrite (GAUBERT). 247, Temperature coefficients of reference electrodes (BIRCHER and HOWELL); Measurements with quinhydrone electrode (LINDERSTRÖM-LANG); 248, Electrolysis of oxides dissolved in boric acid or in borates (ANDRIEUX); Electrolytic deposition of carbonaceous iron from complex compounds with organic acids (SCHMIDT); Mercury cathodes in electrolytic oxidation processes (STSCHERBAKOV). 251, Electrolysis of lead chloride (SAXON). 260, Formation of ozone from air under pressure; Ozonisation with high-frequency current (VON WARTENBERG and TREPPENHAUER).

Lubricating oil for electricity meters. HOLDE and VON SCHACHENMEIER.—See II.

Influence of alternating currents on electrolytic corrosion of iron. ALLMAND and BARKLIE.—See X.

PATENTS.

Electrodes for electrolytic decomposition apparatus. C. F. HOLMBOE (E.P. 246,228, 29.10.24).—A plate electrode is provided with ribs on both sides of sufficient size to provide the greater portion of the active electrode surface. The arrangement and position of the ribs may be varied as desired, *e.g.*, to allow circulation of the liquid. Such electrodes, other conditions being equal, produce greater quantities of gas and require lower voltages.

J. GRANT.

Storage-battery separator. P. E. NORRIS, Assr. to WESTINGHOUSE UNION BATTERY CO. (U.S.P. 1,567,747, 29.12.25. Appl., 1.11.23).—Wood pulp, flour, or other porous material is treated with rubber latex to form a storage battery separator. The rubber latex coats the porous material without penetrating it.

T. S. WHEELER.

Voltaic cell. M. E. CONRAD, Assr. to C. B. SHOENMEHL, INC. (U.S.P. 1,567,838, 29.12.25. Appl., 9.2.21).—Cells of the type described in U.S.P.

719,752 (B., 1903, 304) have their resistance decreased and voltage increased by forming the negative electrode of copper oxide or other suitable metal oxide which has been treated with the fumes or vapour of sulphur at a temperature above the boiling point of sulphur.

T. S. WHEELER.

Carbon electrodes for primary cells using air-depolarisation. COMP. LORRAINE DE CHARBONS, LAMPES ET APPAREILLAGES ÉLECT. (F.P. 594,697, 31.5.24).—The electrodes consist of active carbon, graphite, or retort carbon, and an oxidising catalyst, *e.g.*, a compound of manganese, copper, silver, vanadium, uranium, titanium, iron, or cobalt.

J. GRANT.

Electrolysis of alkali chlorides. F. GERLACH (F.P. 596,918, 20.4.25. Conv., 21.5.24).—In the electrolysis of alkali chlorides with mercury cathodes, the mercury liberated by decomposition of the alkali amalgam is led to the amalgam-formation chamber in presence of liquid (water, alkali chloride solution, dilute alkali lye) and, if desired, in an atmosphere of an inert gas.

Manufacture of electrode-carbon. C. EHRENBERG, H. WIEDERHOLD, C. KRUG, M. G. HOLSBOER, K. FISCHER, and STUDIENGES. FÜR AUSBAU DER IND. (G.P. 422,235, 30.11.23).—Humic acids are liberated by acid from the solution produced by treatment of substances containing humic acid (*e.g.*, humic coal, peat, etc.) with alkali, and are then carbonised.

J. GRANT.

Material for self-burning electrodes. NORSKE A./S. FOR ELEKTROCHEM. IND. NORSKE IND.-HYFOTEKBANK (Swiss P. 111,653, 23.7.24. Conv., 25.7.23).—The electrodes consist of metal casings containing a mixture of carbon particles of varying degrees of subdivision, together with so much (more than 18%) binding material (tar and pitch) that the mass when warm is no longer capable of being compressed by stamping. During heating this filling coheres to form a mass having a high density.

J. GRANT.

Electrical gas purification. SIEMENS-SCHUCK-ERTWERKE GES.M.B.H. (G.P. 422,619, 28.2.24).—The gas is led first in contact with precipitating electrodes arranged along the direction of gas flow, and then through permeable electrodes placed across the flow.

E. S. KREIS.

Manufacture of electroionic discharge tubes. EDISON SWAN ELECTRIC CO., LTD., Asses. of RADIO-RÖHREN-LAB. G. NICKEL GES.M.B.H. (E.P. 232,978, 24.4.25. Conv., 28.4.24).—In a thermionic discharge tube, the effects of space charge are limited by using an incandescent cathode (and/or an auxiliary heated electrode) which is covered with a chemical compound (*e.g.*, calcium hydride) which is highly emissive of electrons and which disintegrates gradually producing positive ions. The tube is either wholly evacuated or contains no gas other than that given off by the heated compound. Other substances either for binding and/or for the production of electrons and positive ions

may be mixed with the disintegrating compound. The grid of ions which is formed in close proximity to the incandescent filament has practically the same properties as an electrically charged stable grid. (Reference is directed, in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 230,226.) A. W. HOTHERSALL.

Deposition of chemical compounds on incandescence bodies. N. V. PHILIPS' GLOEILAMPENFABR. (F.P. 594,165, 8.10.24. Conv., 11.10.23).—Solid chemical compounds (nitride and phosphide of zirconium, carbides of molybdenum and tantalum, etc.) are produced by passing the vapours from certain of their volatile compounds, usually mixed with hydrogen or nitrogen, over a metal filament heated to 1000—1800°. J. GRANT.

Impregnated insulation. G. F. DREHER, ASSR. to GEN. ELECTRIC Co. (U.S.P. 1,574,562, 23.2.26. Appl., 9.6.23).—See E.P. 228,224; B., 1925, 249.

See also pages 256, Carrying out and controlling reactions (G.P. 422,159). 261, Dust removal in briquetting works (G.P. 422,148). 262, Separating condensable products from gases (G.P. 422,121). 274, Detecting oxygen (U.S.P. 1,567,534). 283, Treating iron or steel to prevent corrosion (E.P. 247,071). 289, Manufacture of rubber goods (E.P. 246,532). 297, Electrical apparatus for preserving fodder (G.P. 418,481). 299, Lead tetraethyl (U.S.P. 1,567,159).

XII.—FATS; OILS; WAXES.

Separation of ricinoleic acid from the mixed fatty acids of castor oil. K. INOKUCHI (J. Soc. Chem. Ind. Japan, 1925, 28, 1353—1358).—The solubilities of barium ricinoleate and barium stearate in absolute alcohol (g. per 100 g. of solution) are: 10°, 0.06 g., trace; 20°, 0.13 g., trace; 30°, 0.325 g., trace; 40°, 2.00 g., 0.014 g.; 50°, 9.372 g., 0.018 g.; 60°, 28.19 g., 0.024 g. In alcohol of d_4^{15} 0.8119 the solubility of barium stearate is trace at 20°, 0.011 g. at 30°, 0.017 g. at 40°, 0.022 g. at 50° and 0.029 g. at 60°. For preparing pure ricinoleic acid from castor oil fatty acids by the barium salt-alcohol process, it is recommended that the temperature be 50°, the time for dissolving the mixed barium salts in the solvent about 1 hr., that as solvent first 95% (by weight) alcohol, then 98%, and finally absolute alcohol be used, and that the amount of alcohol for one separation be about 100 c.c. for each 10 g. of the salts. K. KASHIMA.

Highly unsaturated acids in ox liver oil. K. KIMURA (J. Soc. Chem. Ind. Japan, 1925, 28, 1366—1373).—An oily liquid obtained by direct heating of ox liver in a pan was extracted with ether and the extract was treated with acetone and alcohol. Ox liver oil (soluble in ether and acetone) thus obtained had the following characters: d_4^{100} 0.9008, acid value 36.5, saponif. value 128.3, iodine value 113.3, n_D^{60} 1.4720, and unsaponifiable matter 40.62%. The fatty acids freed from un-

saponifiable matter had m.p. 39—42°, d_4^{100} 0.8437, neutralisation value 196.3, iodine value 104.4, n_D^{60} 1.4480, ether-insoluble bromides 19.2%. The corresponding values for the fatty acids from the portion insoluble in acetone but soluble in alcohol and from the portion insoluble in both alcohol and acetone were, respectively: m.p. 48.5—51°, 51—54.5°, d_4^{100} 0.8515, 0.8521, neutralisation value 188.6, 186.0, iodine value 132.1, 143.5, n_D^{60} 1.4522, 1.4529, ether-insoluble bromides 30.0%, 38.3%. The ether-insoluble bromides had nearly the same composition in all cases. The bromides were debrominated and the highly unsaturated acids thus obtained were converted into methyl esters and subjected to distillation. Hydrogenated products of some fractions contained arachidic and behenic acids. The presence of $C_{20}H_{32}O_2$ (cf. Hartley, J. Physiology, 1907, 17; 1909, 353) and of $C_{22}H_{36}O_2$ in the oil was proved. Acids of the $C_nH_{2n-10}O_2$ series are either absent or present only in minute amount. The odour of the highly unsaturated acids from ox liver oil resembles that of the acids from fish oil. K. KASHIMA.

Vitamin potency of cod-liver oils. XVIII. Effect on vitamin potency of cold pressing cod-liver oils. A. D. HOLMES and M. G. PIGOTT (Ind. Eng. Chem., 1926, 18, 188—189).—The stearine solidified by refrigeration in a brine-cooled tank was removed from the liquid oil. Chemical analysis of the crude and pressed oils revealed little difference between them, and tests on the vitamin potency, although not showing identical results, did not indicate any significant divergencies.

D. G. HEWER.

Examination of cacao-fat for alkalis and alkaline-earths. J. PRESCHER and R. CLAUS (Z. Unters. Nahr. Genussm., 1925, 50, 429—430).—The occurrence and determination of alkalis or alkaline-earths in animal and cacao fats are discussed, and the official method of analysis for the former case is applied to the latter. This involves the precipitation of the acidified water-extract of the fat with ammonia and ammonium carbonate solutions. The natural lime-content of cacao-fat (about 0.01%) is greater than that of animal fat. J. GRANT.

Use of sintered glass crucibles for the separation of digitoninesteride in the examination of fats for phytosterol. J. PRESCHER and R. CLAUS (Z. Unters. Nahr. Genussm., 1925, 50, 420—423).—Jena sintered glass crucibles and filter-flasks have been found to be highly satisfactory for the separation of the digitonin precipitate in the isolation of sterols (phytosterol, cholesterol) from fats, prior to conversion into the actates for identification purposes. J. GRANT.

Detection and determination of small amounts of chromium in fats. A. O. SNODDY (J. Oil and Fat Ind., 1925, 2, 20—25).—200 g. of fat are ignited in a muffle, the ash is fused with sodium and potassium carbonates, and the aqueous solution of the fused mass, after acidification with hydrochloric acid, is diluted to 200 c.c. An aliquot part of this

solution, after addition of 5 c.c. of 20% hydrochloric acid and 1 c.c. of diphenylcarbazine solution (0.1—0.2 g. in 10 c.c. of glacial acetic acid, diluted to 100 c.c. with 95% alcohol), is compared colorimetrically with potassium dichromate and carbonate solution similarly treated, the violet colour reaching a maximum intensity within 10 min. Small amounts of iron, aluminium, copper, and silicon do not interfere.

A. A. ELDRIDGE.

Autoclave fat-splitting. H. RIEMER (Z. Deuts. Oel- u. Fett-Ind., 1925, 45, 653—655, 701—703, 720—722, 736—739; Chem. Zentr., 1926, I, 1323—1324).—The author gives a brief characterisation of the evolution of industrial fat-splitting as well as the theoretical explanation thereof due to Harkins and Langmuir. The suitability of different metals for autoclave construction is discussed, cast-iron not being recommended for high-pressure work. An aluminium alloy is found especially suitable as it is not affected by fatty acids or condensed water, is not attacked by zinc oxide or magnesia, and is suitable mechanically. A detailed description of the author's high-pressure process (cf. Austr. P. 101,041; B., 1926, 200) is given, the following advantages being claimed:—Pressures of 18—25 atm. may be used; there is no anhydride formation; no previous purification of the fat is necessary; deodorisation is effected, so that there is the possibility of treating all kinds of raw materials, especially marine animal oils.

H. M. LANGTON.

Catalytic hydrogenation of highly unsaturated acids. I. Course of hydrogenation of methyl esters of highly unsaturated acids in the presence of nickel catalyst. Y. TOYAMA and T. TSUCHIYA (J. Soc. Chem. Ind. Japan, 1925, 28, 1079—1087).—The methyl esters of the highly unsaturated acids obtained from Japanese sardine oil have been hydrogenated in the presence of nickel at 185—210° under ordinary pressure. The mixed esters had b.p. 210—227°/5 mm., saponif. value 169.2, iodine value 350.6; the fatty acids obtained from the esters yielded 127.1% of ether-insoluble bromide containing 71.06% Br. The individual acids which may possibly have been present in the original sample are $C_{20}H_{30}O_2$, $C_{22}H_{34}O_2$, $C_{26}H_{38}O_2$, and $C_{22}H_{36}O_2$. Before the iodine value of the original sample fell to 11.4, portions were taken out at 12 different stages of hydrogenation and analysed. The content of fatty acids giving ether-insoluble bromide steadily decreased as hydrogenation proceeded. Thus when the iodine value fell to 173.1 the amount of ether-insoluble bromide was 1.67%, and when the iodine value was 134.9 no bromide was produced. As the bromide content of ether-insoluble bromide was more than 69% in every case, it may be concluded that no substantial amount of fatty acids giving ether-insoluble hexabromide was formed during hydrogenation. The fact that the bromine content of the ether-insoluble bromide decreased somewhat with the progress of the hydrogenation may be accounted for by partial conversion of the fatty acids with five double bonds into those with four double bonds. Until nearly all of the

fatty acids giving ether-insoluble bromide disappeared no appreciable amount of solid acid was formed, but immediately after this stage solid acids were formed in substantial amount. It is probable, therefore, that until nearly all of the fatty acids giving ether-insoluble bromide have disappeared, the hydrogenation results mainly in the conversion of highly unsaturated acids into acids with two double bonds, and that subsequently the chief change is the conversion of the acids with two double bonds into those with one double bond, with formation also of saturated acids. Even in the sample having an iodine value of 134.9, the presence of saturated acids is indicated. The solid acids with one double bond formed during the hydrogenation include acids having markedly higher m.p. than naturally occurring C_{20} and C_{22} acids of the oleic series, and it is even probable that solid acids with two double bonds are formed.

K. KASHIMA.

Naphthenic soap. J. MIKUMO (J. Soc. Chem. Ind. Japan, 1925, 28, 1121—1126).—Sodium soap, prepared from a mixture of Japanese naphthenic acids (d_4^{15} 0.9708, acid value 249.2), was examined with regard to its surface and interfacial tensions, and emulsifying, lathering, and detergent powers. Parallel tests were made with sodium soaps from Kahlbaum's oleic acid, coconut oil, and rosin. The naphthenic soap is extremely soluble in cold water, and is superior even to coconut oil soap in lathering power. It is, however, soft and is far inferior to the oleate in detergent action. It may be regarded as intermediate between fatty and rosin soaps.

K. KASHIMA.

Determining palm kernel oil and butter in margarine. ELSDON and SMITH.—See XIX.

PATENTS.

Preparation of emulsifying agents from castor oil. J. B. G. AUBRY (F.P. 592,764, 30.1.25).—Castor oil is polymerised by heating in an inert atmosphere at 240°, the product being sulphonated, allowed to remain for 48 hrs., washed with water containing formaldehyde, and then neutralised by addition of ammonia.

A. J. HALL.

Preparation of mixed esters of fatty acids. OELWERKE GERMANIA G.M.B.H., and W. NORMANN (G.P. 417,215, 26.6.20).—Mixtures of esters of fatty acids are heated at a high temperature for a prolonged period, with or without a catalyst, whereby mixed esters having a lower melting point are formed. Products closely resembling butter fat may be thus obtained. Non-esterified products may be removed from the resulting esters by special methods, e.g., distillation. Suitable catalysts include aromatic and aromatic-aliphatic sulphonic acids, tin and its salts, and alkali alkoxides. For example, a mixture of 10% of tristearin and 90% of soya bean oil having m.p. 54.5° yields when heated at 250° for 9.5, 17, and 24 hrs. products having m.p. 50.8°, 43.7°, and 34.3° respectively. A mixture of 2 mols.

of tristearin and 1 mol. of triacetin is hard, brittle, and friable but on heating for 72 hrs. at 200—250° it loses its brittleness and becomes plastic. A mixture of equal parts of neutral coconut oil and ethyl stearate heated for 18 hrs. at 250° in the presence of 1% of tin and in a stream of carbon dioxide (for removal of volatile products) rapidly yields the ethyl esters of lower fatty acids having an easily recognised odour; the distillate has a saponification value of 221, whilst the saponification values of ethyl stearate and the ethyl esters of coconut oil acids (mean mol. wt., 202) are 180 and 244 respectively. When a mixture of 20 pts. of tristearin, 80 pts. of coconut oil, and 1% of naphthalenesulphonic acid is heated for 2.5 hrs. at 250°, the product has m.p. 34.5° (m.p. of original mixture is 57.3°); when $\frac{1}{2}$ % of sodium ethoxide is used as a catalyst and the heating is carried out for $\frac{1}{2}$ hr. *in vacuo* at 140°, the product has m.p. 31.4°. A. J. HALL.

Extraction of mixtures of oils and fats by means of alcohol. K. KUBIERSCHKY (G.P. 417,964, 28.5.20).—The alcoholic extract is treated with sufficient water so that a part of the alcohol-soluble substances is precipitated together with any dissolved neutral oil; a more valuable extract and a higher yield of neutral oil are thereby obtained.

A. J. HALL.

Preparation of fats from fatty acids with an odd number of carbon atoms. FARBENFABR. VORM. F. BAYER U. Co., Assees. of S. DREICHSEL and H. WEYLAND (G.P. 422,687, 12.4.24).—A fatty acid mixture consisting mainly of lauric acid is converted into the corresponding methyl ketones, the latter are oxidised, and the resulting fatty acids are converted into glycol or glycerol esters in the usual manner. Tasteless and odourless, readily assimilable fats are obtained.

H. M. LANGTON.

Hydrolysing fats, oils, and waxes. W. SCHRAUTH (Can. P. 245,373, 10.3.24).—Fatty substances are heated with aqueous solutions of sulphonic acids of polynuclear hydrocarbons substituted with alkyl groups, e.g., isopropyl, in the nucleus.

A. J. HALL.

Chromium soap. T. T. GRAY (U.S.P. 1,567,049, 29.12.25. Appl., 3.10.22).—A mixture of an alkali soap, such as sodium stearate, with $\frac{1}{5}$ th to 5 times its weight of a chromium soap, such as chromium stearate, is non-irritant, has good lathering and detergent properties, no objectionable taste, and is particularly suitable for use as a shaving soap or as a dentifrice. The chromium stearate is prepared by the interaction of chromium hydroxide and molten stearic acid or by saponifying a fatty acid or a fat with a mixture of sodium and chromium hydroxides.

T. S. WHEELER.

Obtaining sulphonated products of wool fat. O. HERZOG (E.P. 247,714, 10.1.25).—See U.S.P. 1,543 157; B., 1925, 680.

Acetylation of fatty substances (U.S.P. 1,567,785).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Barium sulphate [heavy spar and *blanc fixe*]. C. P. VAN HOEK (Farben-Ztg., 1926, 31, 1136—1137).—The undesirable properties conferred on a paint by the presence of barium sulphate in the form of heavy spar or *blanc fixe* finds a parallel in rubber mixes. The presence of an adsorbed layer of air on the particles inhibits adequate adhesion to the oil medium in paints and is suggested as being the cause of the low opacity and the weakening of paint films.

A. DE WAELE.

Influence of number and size of particles on the covering power [of pigments]. C. KÜHN (Farben-Ztg., 1926, 31, 1131—1133).—The relative opacities of unit volumes of suspensions of burnt sienna in boiled linseed oil were determined by viewing under a low-power microscope illuminated by diffused candle light, and noting the opacity values of smoke-glass oculars necessary to obtain complete extinction of the light transmitted. It was found that the opacities were proportional to the number of particles per unit volume of suspension, further tests confirming the fact that the relative dimensions of the particles (between the limits of 159 and 283 cm.² specific surface examined) did not affect the opacities. The relationship between specific surface and opacity is similarly linear, but the rate of increase of opacity with increase in the number of particles per unit volume of suspension increases more rapidly in the case of the finer particles (cf. B., 1920, 697 A).

A. DE WAELE.

Settling and packing of mixed paints. W. C. ARSEM (Ind. Eng. Chem., 1926, 18, 157—160).—The tendency of the particles of paint pigments to flocculate and of driers and soaps in the vehicle to gelatinise is inhibited by the presence of free acid or resinous dispersing agents. On storage, a slow combination of basic pigments with the peptising agent may occur, prejudicing the stability of the vehicle sol and producing basic soaps which have very little dispersing action on a pigment derived from the same metal, thus permitting flocculation and caking. The alteration in acidity and ash of the medium in stored zinc oxide paints was observed over a period of 22 months, and the ratio of ash to total acid (calculated as oleic acid) approached the constant value required by the formation of basic zinc oleate of known constitution.

S. S. WOOLF.

Factors determining the brightness and opacity of white paints. F. H. RHODES and J. S. FONDA (Ind. Eng. Chem., 1926, 18, 130—135). The hiding power and whiteness of a film of white paint is dependent on thickness of film, refractive index of pigment and of medium, particle size of pigment, and percentage volume of pigment in the paint. Earlier determinations of opacity using films of liquid paint are adversely criticised. In the present investigation dried films of various thicknesses over black were examined for brightness by an integrating photometer (cf. Taylor, U.S. Bur. Standards, Sci.

Paper 405). The conception of "ultimate brightness"—not increased by further increase in the thickness of the film—is introduced, and a formula correlating the above factors is developed and receives support from the experimental results. An explanation is offered of the increase in opacity obtained by adding small quantities of dark pigments to a white paint.

S. S. WOOLF.

Evolution of vapours containing nitric oxide, carbon monoxide, and hydrogen cyanide in the decomposition of triolin. E. WILKE-DÖRFURT, A. SIMON, and E. GÜHRING (*Z. angew. Chem.*, 1926, 39, 196—198).—Triolin, a new form of flooring material (cf. Simon, B., 1926, 21), decomposes at about 125° with the formation of highly poisonous vapours containing 8—10% HCN and considerable proportions of nitric oxide and carbon monoxide. The hydrogen cyanide is formed by the decomposition of the triolin itself, and by a secondary reaction between carbonaceous matter and the nitric oxide first formed, as well as by the decomposition of the Prussian blue used for colouring purposes. Even at room temperatures appreciable quantities of nitric oxide are formed in a current of air passing over the triolin.

B. W. CLARKE.

See also A., Mar., 243, Swelling and dispersion of colloidal substances in ether-alcohol mixtures. (MARDLES). 259, Ferro- and ferricyanides (TARUGI).

PATENTS.

Zinc white. R. D. LANCE (F.P. 595,396, 17.6.24).—The mixture of carbon monoxide and zinc vapour, resulting from heating oxide or sulphide ores with coal, is burnt in the neck of the muffle or at the gas outlet by blowing in air. Combustion of the carbon monoxide assists that of the zinc, and the product is free from metal.

A. GEAKE.

Acetylation of fatty and other substances. [Preparation of resinous substances.] L. G. BOURGOIN (U.S.P. 1,567,785, 29.12.25. Appl., 23.10.24).—The fat or other substance under treatment is mixed with a molecular proportion of calcium carbide and heated with a salt containing water of crystallisation or treated under pressure with steam at a temperature above the melting point of the substance, e.g., 100—350°. The products are plastics and resins suitable for use as insulators, artificial rubber, or the like. For example, 5—8 pts. of carbide are mixed with 100 pts. of an animal or vegetable fat or the corresponding amount of glycerin and fatty acid and treated with steam under pressure at 180—250° for 2 hrs. According to the conditions of treatment there is obtained a more or less plastic and malleable substance which is easily moulded when hot and is insoluble in water, acetone, alcohol, and ammonia but soluble in carbon tetrachloride, carbon disulphide, and ether.

T. S. WHEELER.

Purification of spirit-soluble Manila copal. L. BLUMER CHEM. FABR. (G.P. 420,918, 29.7.24).—The pulverised copal is refluxed with limited quantities

of solvent until it is well swollen, whereupon the solvent is distilled off, the temperature raised to about 200°, and finally the mobile liquid mass is filtered. The copal may be mixed with other resins.

E. H. SHARPLES.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Hevea latex. IV. Proteins. V. Possible occurrence of a coalescing enzyme. W. N. C. BELGRAVE (*Malay. Agric. J.*, 1925, 8, 367—375; cf. B., 1925, 771).—By the successive addition of alcohol and sodium hydroxide solution to diluted *Hevea* latex coagulation is effected, but the serum retains a considerable proportion of the protein which can be precipitated by regulated acidification. Analysis of the protein reveals a fairly uniform distribution of nitrogen between the diamino-, monoamino-, and non-amino-groups. The views of de Vries (B., 1924, 797, 798; 1925, 17) as to the existence of a coalescing enzyme in latex are rejected. Protein substances rather than resins are probably associated with the coagulation phenomena, but the opposing views could be reconciled by the occurrence of a lipin-protein complex unstable towards heat.

D. F. TWISS.

Rubber coagulated with sodium silicofluoride. J. EDWARDES (*Bull. Rubber Growers' Assoc.*, 1926, 8, 34—36).—The use of sodium silicofluoride as coagulant in conjunction with that of *p*-nitrophenol as mould-preventive, prevents the development of the violet colour frequently observed in rubber prepared with the aid of the latter substance and acetic acid; it also renders unnecessary the addition of sodium bisulphite to latex for the production of pale crêpe rubber, and checks the tendency to bubble formation in smoked sheet rubber.

D. F. TWISS.

Coagulation and mould prevention of smoked sheet rubber. H. P. STEVENS (*Bull. Rubber Growers' Assoc.*, 1926, 8, 36—40; cf. preceding abstract).—Rubber prepared with the aid of *p*-nitrophenol vulcanises rather more rapidly than the standard or than rubber prepared with dinitrophenol; the retarding effect of sodium silicofluoride is therefore compensated to some extent by the simultaneous use of *p*-nitrophenol. Sodium silicofluoride prevents bubbles in smoked sheet rubber.

D. F. TWISS.

Alteration of the degree of dispersion of rubber. F. EVERS (*Kautschuk*, 1925, Nov., 8—9).—If rubber is treated in boiling chlorobenzene or xylene solution with aluminium chloride, the resulting solution on addition of alcohol yields a precipitate which gradually hardens. The product gives a benzene solution of very low viscosity and evidently represents a much lower state of "aggregation" than that of the original rubber.

D. F. TWISS.

Influence of state of subdivision [of rubber] on the period necessary for extraction with acetone [to determine resin]. S. REINER (*Kautschuk*, 1926, Jan., 5—6).—Experiments with

an apparatus especially designed to give uniform results show that the size of the fragments of Para rubber greatly influences the rate of extraction of the resins by acetone. The results indicate that the customary period of extraction is excessive.

D. F. TWISS.

Chemistry of rubber and gutta-percha. Isoprene and caoutchouc. X. H. STAUDINGER (Kautschuk, 1925, Aug., 5—9; Sep., 8—10).—Rubber, polysaccharides, proteins, and many synthetic products are "eucolloids" in which the primary colloidal particles are identical with the molecules (macromolecules), whereas with pseudocolloids, such as salts of the fatty acids, the primary colloidal particles result by association of the molecules. Association involves forces similar to those active in crystallisation, whereas in polymerisation the fundamental molecules undergo chemical change. Styrene is a "hemicolloid" containing both molecularly complex eucolloid material and also substances of low molecular weight. Hemicolloids are also obtained in the degradation of rubber. The chemical nature of *cyclocaoutchouc*, *hydrocaoutchouc*, rubber (natural and synthetic), and gutta-percha is discussed briefly as also is the chemical effect of heat on rubber.

D. F. TWISS.

Effect of accelerators on cure [vulcanisation] and quality of various rubbers. R. P. DINSMORE and A. O. ZIMMERMAN (Ind. Eng. Chem., 1926, 18, 144—157).—The two most significant properties for the comparison of various rubbers are the "stiffness" and the "tear resistance"; the "best cure" of rubber is the period of vulcanisation at a given temperature at which these properties approach the most satisfactory balance with respect to each other and with respect to their values after ageing. Slope, tensile strength, ultimate elongation, tensile product, hysteresis, permanent set, energy of resilience, and coefficient of vulcanisation are all less satisfactory as criteria of the quality of rubber and of its variation with vulcanisation. Although the difference in rate of vulcanisation may be reduced by the introduction of an accelerator into a rubber-sulphur mixture, accelerated mixings are not necessarily more uniform and two rubbers with the same rate of vulcanisation in simple admixture with sulphur may exhibit different rates of vulcanisation with the addition of an accelerator. It is therefore insufficient to use one or even two or three mixings to evaluate rubber for use with various accelerators. Pale crêpe, on the whole, is more uniform than smoked sheet rubber, but unfortunately its quality is uniformly on the low side. The mixing used for the tests was rubber (100), sulphur (variable), accelerator (variable), and zinc oxide (6). By "stiffness" is meant the increase in load necessary to increase the elongation from 600 to 800% for an unaccelerated mixing or from 500 to 700% if accelerated.

D. F. TWISS.

Estimation of fineness of mineral fillers in the rubber industry. F. KIRCHHOF (Kautschuk, 1925, Sep., 17—18).—A convenient rough method

for estimating the relative particle size of various powders intended as rubber ingredients is to place from 5 to 20 g. in a graduated 100 c.c. tube 25 cm. long and to tap until the bulk becomes constant; the calculated final volume per gram (or per gram/specific gravity) forms a comparative index of the fineness.

D. F. TWISS.

Heavy spar and blanc fixe. VAN HOEK.—See XIII.

PATENTS.

Preserving vulcanised rubber. ROBEL UND FIEDLER G.M.B.H. (E.P. 221,788, 26.8.24. Conv., 13.9.23).—The deterioration of vulcanised rubber on storage is counteracted by treatment with a mixture of glycerol and benzaldehyde. A suitable solution is prepared by adding a mixture of glycerol (172 g.), benzaldehyde (34 g.), and talcum (1300 g.) to a hot solution of rubber (60 g.) in paraffin oil (600 g.) which has been heated for 4 hrs. at 140°; phenylhydrazine (28 g.) or glucose (20 g.) may also be introduced; the intimate mixture is then stirred at 180° for 2 hrs. The cold mixture is applied with a hard brush.

D. F. TWISS.

Manufacture of rubber goods from rubber emulsions. ANODE RUBBER Co. From P. KLEIN and A. SZEGVÁRI (E.P. 246,532, 28.7.24. Addn. to 223,189; B., 1925, 46).—In addition to the protection of the rubber deposit from contact with the anode gases by mechanical means, the production of homogeneous electrophoretic deposits of rubber can be ensured by prevention of the liberation of gases. The addition of sodium chloride and pyrogallol to preserved latex, or the impregnation of a porous mould, surrounding the anode, with manganese oxide prevents the liberation of gaseous oxygen. Alternatively a similar end may be achieved by using a zinc anode with latex containing sodium chloride. By using a carbon anode with latex to which sodium thiosulphate has been added, sulphur is deposited together with the rubber. The thickness of the rubber deposit is influenced by the material of the mould; the deposit forms more rapidly on gypsum than on unglazed earthenware. Fabric-lined rubber goods can be produced by slipping over an anode-mould of the desired shape the necessary fabric lining and depositing the rubber thereon.

D. F. TWISS.

Hard resinous vulcanisation accelerator. W. SCOTT, Assr. to E. I. DU PONT DE NEMOURS AND Co. (U.S.P. 1,571,739, 2.2.26. Appl., 19.12.22).—An accelerator for the vulcanisation of rubber is formed by the action of formaldehyde on the condensation product of acetaldehyde (3 mols.) and aniline (2 mols.).

D. F. TWISS.

Acceleration of vulcanisation of rubber. RICARD, ALLENET ET CIE. (Addition 29,785, 31.5.24, to F.P. 523,417; cf. E.P. 157,050; B., 1921, 520 A).—Rubber is mixed with 0.2—1% of furfuraldehyde or other furfuraldehyde derivatives containing nitrogen, and vulcanised at 5—6 atm. for 9—18 min.

E. H. SHARPLES.

Recovery of rubber and fabric from old rubber. A. L. MARCHAL (F.P. 593,602, 26.11.24).—Rubber goods containing fabric, such as tyres, are heated in a closed vessel at 180–200° with copper turnings and a rubber solvent; the rubber can then be removed from the adhering fabric by washing with benzine. The fabric so obtained can be used again after bleaching and the rubber is to a great extent desulphurised. E. H. SHARPLES.

Bituminous compositions (E.P. 246,548).—See IX.

Storage battery separator (U.S.P. 1,567,747).—See XI.

XV.—LEATHER; GLUE.

Activities of pancreatic enzymes used in bating upon different substrates. J. A. WILSON and H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1926, 21, 50–53).—The activities of nine commercial pancreatins have been compared by seven methods, viz., the Fuld-Gross casein method, the methods previously quoted (cf. B., 1926, 205), and the Wohlgemuth fat method. In the Fuld-Gross method the activity equals the reciprocal of the smallest number of grams per litre of enzyme capable of digesting all the casein in a solution containing 1 g. per litre in 1 hr. at 40°. The Wohlgemuth method defines the activity as the c.c. of 0.1 *N*-sodium hydroxide required to neutralise the free fatty acids liberated from 10 g. of emulsified olive oil by 0.10 g. of the enzyme in 1 hr. at 40°. The Willstätter fat figure is preferred because it provides for a better control of p_H value and gives results more easily reproducible. The results show that the pancreatins used in bating possess fat-splitting powers, but these are not reliable as a measure of the activities on the skin fats. The commercial products are very variable in composition. The activities as determined by the casein method are not the same as those on collagen, elastin, and keratose. The latter should be used as substrates and not casein.

D. WOODROFFE.

Important rôle played by enzymes in bating. J. A. WILSON and H. B. MERRILL (Ind. Eng. Chem., 1926, 18, 185–188).—When skins are limed before tanning, the keratins are partially hydrolysed into keratose, which is soluble in neutral or alkaline solution, but is precipitated at its isoelectric point, p_H 4.1. If this material is left in the skin and precipitated there by the acid tan liquors, the value of the finished leather is impaired. In bating, keratose is hydrolysed by the enzymes to such an extent that it is no longer precipitable by acid. Pancreatin hydrolyses keratose in the p_H range 5.5–11.2 only, and maximum hydrolysis is obtained at p_H 7.9. The hydrolysis at 40° and p_H 7.9 has been measured as a function of the time and concentration of the enzyme and keratose. The proteolytic powers of different enzyme preparations have been measured by their keratose-digesting power (cf. B., 1926, 205).

D. WOODROFFE.

Ultrafiltration of vegetable tanning solutions. A. W. THOMAS and M. W. KELLY (Ind. Eng. Chem., 1926, 18, 136–138).—The ultrafiltration of solutions of hemlock bark extract containing 0.4 g. of tannin per 100 c.c. and varying in p_H from 2.0 to 9.0 was carried out in a Columbia type of ultrafilter using compressed nitrogen at pressures of 80, 120, and 200 lb./in.² The membranes were prepared by soaking filter paper in collodion solution and allowing to become "tacky" before immersion in water, according to the method of R. J. Browne (B., 1923, 989 A). Pronounced differences were observed in the amount of total solids held back both as a function of the p_H of the solutions and also, with identical solutions, as a result of differences in the collodion solutions in which the membranes were prepared and individual variations in the membranes. Variations in the gelatin-salt test for tannins in the ultrafiltrate give an additional indication of the unreliability of the method for separating tannins and non-tannins.

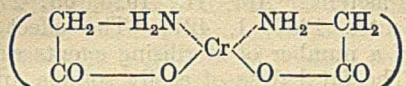
A. W. HOTHERSALL.

Aldehyde tannage. A. W. THOMAS, M. W. KELLY, and S. B. FOSTER (J. Amer. Leather Chem. Assoc., 1926, 21, 57–76).—Experiments with powdered gelatin or hide powder and formaldehyde solutions show that the tanning action is best between p_H 6.0 and 9.0. The amount of formaldehyde fixed from increasingly concentrated solutions increases but not in direct proportion. High concentrations of sodium chloride increase the rate of fixation of the aldehyde. Neutral or faintly alkaline solutions of formaldehyde are most favourable for tanning. Owing to the hydrolysis of hide powder, formaldehyde tanning tests should not be made on this material.

D. WOODROFFE.

Internal complex salt formation as mechanism of chrome tanning. K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1926, 21, 22–30; cf. B., 1924, 65; 1925, 604, 817).—With decrease in the percentage acidity of chrome liquors, there is an increased fixation of chromium by formaldehyde-collagen and for a moderately acid salt this increase is accentuated by increased concentration of the liquor. The grass-green colour of the tanned formaldehyde compound seems to show that the colour of ordinary chrome-tanned leather is due to internal salt formation between the secondary valencies of the chromium salts and the basic groups of the protein. Fixation of chromium-containing anions is decreased by pre-treatment of the collagen with formaldehyde. Hide powder tanned with sumach, quebracho, and tannic acid at p_H 2.0–6.0 shows decreased fixation of chromium from chromium chlorides and sulphates (up to 50% reduction). In concentrated basic liquors part of the tannin is replaced by chromium, probably due to the fixation of electro-negative sulphato-complexes. The oxalato-compound showed a proportionately lesser retardation in the chromium fixation by the tannin-collagen compounds than the salts in which chromium is present as cation. The degree of chromium fixation by hide substance from ordinary chrome liquors is

determined not only by the state of the acidic groups, but is also a function of the basic groups of the protein. Hide powder tanned by means of chromium cations shows no decreased affinity for vegetable tannins. Hide powder tanned with oxalato-chromium compounds shows a slight decrease in the rate of vegetable tanning. The reactivity of deaminised collagen and tannin-collagen compounds towards cathodic and anodic chromium is similar and this resemblance supports the inner complex hypothesis of chrome tanning, according to which the chrome-collagen compound is an internal complex salt of the type



Primary valency is probably concerned in the reaction of chromium salts with the acidic groups in collagen and secondary valency with the basic groups. Anodic chromium fixation from the oxalato-compound shows a maximum rate in the isoelectric zone of the proteins and is probably of the residual valency type. This reaction is distinctly different from the combination of chromium cations with collagen.

D. WOODROFFE.

Colloid chemistry in tanning. K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1926, 21, 53—57).—A reply to the criticisms of W. D. Bancroft (*ibid.*, 1925, 565) who doubts the formation of definite chemical compounds in chrome tanning (cf. B., 1925, 604).

D. WOODROFFE.

Behaviour of deaminised collagen. Further evidence in favour of the chemical nature of tanning. A. W. THOMAS and S. B. FOSTER (J. Amer. Chem. Soc., 1926, 48, 489—501).—Hide powder (100 g.) was suspended in water (1 litre), and a solution of sodium nitrite (100 g.) in water (500 c.c.) added, and then glacial acetic acid (70 c.c.). After keeping for 24 hrs. the resulting canary-yellow product was collected, washed with water, and covered with sodium chloride to reduce swelling and force out more of the acid solution, salt and acid then being removed by washing with water, the product dehydrated with 95% alcohol, and dried in a current of air (cf. Hitchcock, A., 1923, i, 1244). The material thus obtained contained 0.49% less nitrogen than the original hide powder, and its isoelectric point was $p_{\text{H}} 3.7$ — 4.2 . The p_{H} at which fixation of tannin by the deaminised material was a minimum was 3.0 — 3.5 , as compared with 4 — 5 for the original hide powder, and, at p_{H} values below 4.0 , the fixation of tannin by the deaminised material was less than that of the original. On the alkaline side of $p_{\text{H}} 3.5$, the rate of fixation by the deaminised material increased rapidly to a maximum at $p_{\text{H}} 7$ — 8 , then fell to $p_{\text{H}} 9$ — 10 . These results are in accordance with the view that tanning is a process of chemical combination between the tannin and the skin. Calf-skin, deaminised analogously, shows two points of minimum plumping, at $p_{\text{H}} 4.0$ and 8.3 , and swells,

in solutions of $p_{\text{H}} 6$ to 10 , to a considerably greater degree than the raw skin.

F. G. WILLSON.

Films of adhesives. J. W. MCBAIN and D. G. HOPKINS (J. Physical Chem., 1926, 30, 114—125; cf. B., 1925, 292).—Determinations of the tensile strengths of various adhesives have been made by a method which measures the strengths of thin films prepared by allowing them to dry on a surface (a ferrotype plate coated with a trace of vaseline) to which they do not adhere. The load is then applied to rectangular strips, $4 \times \frac{1}{2}$ in., of the films. The representative values found are as follow: a high-grade commercial glue, 12,200—12,400; high-grade commercial gelatin, 12,800; purified gelatin, 9500; refined isinglass, 9500; and glue size, 7200 lb./in.² The tensile strength of such films depends on the humidity of the atmosphere. The effect of an addition of a powdered solid on the resulting strengths of the films of glue depends on the quantity added, e.g., 10—15% by weight of lead sulphate has no effect, but an addition of 66.7% weakens the tensile strength by 31%. A similar loading of the glue by zinc oxide, aloxite, and fine water-ground flint (fired) caused an even greater weakening; of the soluble substances added to the glue, only potassium dichromate increased the strength, whilst formalin had no effect; potash alum, sodium formate, salicylate, or benzoate, and glucose reduced the strength. Films prepared from 25% solutions of glue in water or 0.1*N*-hydrochloric acid were equally tenacious, whereas those with 0.1*N*-sodium hydroxide were decidedly weaker. The hydrolytic degradation of such aqueous, acid, and alkaline solutions of glue continuously heated brings about a relatively slight loss in strength after 28 days at 55—57.5°, but at higher temperatures (up to 100°) the effect is much greater, and films, even when of high tensile strength, become very brittle. The gelatin, glue, and isinglass films, when loaded nearly to the point of fracture, developed permanent, opaque markings suggestive of slip planes or crystallisation of the adhesive. X-Ray examination of one of these films gave no indication of crystalline structure, however. The paper strip method of Millar (J.S.C.I., 1899, 16) is shown to be unsatisfactory even as a method for comparing the tensile strengths of different adhesives. The experimental error involved in the present method is generally much less than 10%, and it is claimed that the method will supplement and, to some extent, supersede the more indirect methods now in use.

L. S. THEOBALD.

PATENT.

Tanning preparations [from sulphite-cellulose waste lye]. T. BLACKADDER, Assr. to ROBESON PROCESS CO. (U.S.P. 1,571,873, 2.2.26. Appl., 10.6.24).—Sulphite-cellulose waste liquor is treated with slightly more lime than is required to neutralise it, the precipitate is separated, the liquor concentrated, and the lime in solution is precipitated with sulphuric acid. 1.25—2.0% of lime is used for a waste liquor requiring 0.5% for neutralisation.

D. WOODROFFE.

XVI.—AGRICULTURE.

Mechanical analysis of soils. Report on the present position and recommendations for a new official method. SUB-COMMITTEE OF THE AGRICULTURAL EDUCATION ASSOC. (J. Agric. Sci., 1926, 16, 123—144; cf. Robinson; B., 1922, 990A).—The method hitherto in use (J. Agric. Sci., 1906, 1, 470) does not secure a complete preliminary dispersion of the sample. The method recommended as official includes preliminary treatment with hydrogen peroxide, to remove humified organic matter, and with 0.2*N*-hydrochloric acid to remove calcium carbonate and adsorbed bases. Dispersion is effected by shaking in ammoniacal water. The actual analysis is effected by the pipette method, depending on the depth-concentration relationship. Full working details are given.

G. W. ROBINSON.

Evaporation of water from soil. B. A. KEEN, E. M. CROWTHER, and J. R. H. COURTS (J. Agric. Sci., 1926, 16, 105—122; cf. Keen, B., 1915, 146).—Further investigations on the evaporation of water from soil are reported. The drying of moist soil in shallow pans is irregular, and more readily reproducible results are obtained by exposing the soil in thin layers below glass plates. During the early stages, drying is largely confined to the peripheral portions. Evaporation of water from soil is controlled by two groups of factors depending respectively on soil-water relationships and on environmental conditions. The latter include such factors as diffusion of water vapour, bulk air movements, and temperature gradients. The precise interpretation of evaporation curves cannot be attained unless a carefully controlled and reproducible technique is used.

G. W. ROBINSON.

Relations between certain soil moisture constants and the determination of the vesicular coefficients of soils. C. H. WRIGHT (J. Agric. Sci., 1926, 16, 18—23; cf. Hardy, B., 1923, 846A).—The author discusses Wilsdon's modification of the Briggs-Shantz formula connecting the maximum water-retaining capacity with the hygroscopic coefficient (cf. B., 1925, 19). Evidence is produced to show that the content of free water in a soil at the maximum water-retaining capacity is 21%. The values obtained by the author for the vesicular coefficient are in good agreement with those obtained by Hardy from the moisture content at point of maximum plasticity. The vesicular coefficient is greater for soils than for subsoils. The percentage of vesicular water is equal to the cubical shrinkage coefficient.

G. W. ROBINSON.

Anomalous flocculation in colloidal clays and soils. F. HARDY (J. Physical Chem., 1926, 30, 254—265).—Theoretical. Flocculation phenomena in lateritic clays and soils are discussed in the light of Comber's theory (Trans. Faraday Soc., 1922, 17, 349; cf. B., 1922, 69A), which attributes the anomalous flocculation of clay by lime to the formation of insoluble colloidal complexes. Hydrous

alumina and hydrous ferric oxide are considered to be colloidal ampholytes, possessing isoelectric points not far removed from neutrality, and the mechanism of their anomalous flocculation by lime and by anions such as the silicate and phosphate ions is explained on this view. The theory is extended to hydrous silica, hydrous aluminosilicates, and ferrosilicates. These may also be regarded as ampholytes. Mutual precipitation is discussed in relation to flocculation in certain soils. L. S. THEOBALD.

Action of some antiseptics on soil amœbæ in partially sterilised soils. L. SEWERTZOV (Zentr. Bakt. Parasitenk., Abt. II., 1925, 65, 278—291; Chem. Zentr., 1926, I, 421).—The effect on soil amœbæ of a number of sterilising agents was determined. The fatal dose of easily soluble antiseptics was much smaller for organisms in culture solutions than for the same organisms in the soil. The fatal dose for amœbæ in soil is so great as to place these organisms beyond consideration in partial sterilisation phenomena. Spores of soil bacteria are more resistant to antiseptics than are amœbæ cysts but non-sporing bacteria have a lower resistance. Treatment of sterilised soils with 0.5—1.5% of calcium sulphide has little effect on the number of amœbæ, but causes a decided reduction in the number of bacteria. Treatment of sterilised soil with 5% of calcium sulphide leads to an initial decrease in the number of amœbæ, followed by an increase in the numbers of both amœbæ and bacteria. Treatment of a normal soil with 1.5% of calcium sulphide has a negligible effect on the number of amœbæ. Partial sterilisation effects cannot be attributed to reduced numbers of amœbæ. A. G. POLLARD.

Age of seedlings as a factor in the resistance of maize to sodium chloride. G. J. HARRISON and C. J. KING (J. Agric. Res., 1925, 31, 633—639).—The resistance of germinated maize seedlings to solutions of sodium chloride depends on the degree of development, the young plants with radicles 25 mm. long and no plumules being the least resistant, the old plants with two developed plumules coming next, whilst those of intermediate age show the greatest resistance. Artificial excision of the radicles increases the resistance, which is also influenced by the weather conditions.

W. O. KERMAK.

Determination of perchlorate in Chili saltpetre based on precipitation with methylene-blue. K. A. HOFMANN, F. HARTMANN, and U. HOFMANN (Ber., 1925, 58, 2748—2754).—In consequence of its toxic action towards plant life the limit of perchlorate, calculated as potassium perchlorate, in Chili saltpetre is fixed at 0.5%. Percentages of this order of magnitude are rapidly determined by dissolving 20 g. of the sample of nitrate to 100 c.c. in water and adding 10 c.c. of this solution to 5 c.c. of 0.1% aqueous methylene-blue B extra; the time requisite for the first appearance of a precipitate is noted. This with 0.6% KClO_4 in the saltpetre is 1—2 sec., with 0.5%, 10 sec., with 0.4%, 40 sec., and with 0.3% 90 sec. at atmospheric temperature.

As control, the solutions are filtered after 10 min. and the filtrates are absorbed in equal strips of white filter-paper, which are then drained and dried at 100°. The depth of colour of these papers is compared with that of standard papers prepared by use of saltpetre of known perchlorate content from 0.3 to 0.6%. With 0.6% KClO_4 the paper is almost white, with 0.5% just visibly bluish, with 0.4% distinctly and with 0.3% strongly pale blue in colour. If the perchlorate content is above 0.5% the method may be applied to a suitably diluted solution. In the region of 0.5% KClO_4 the results are accurate to $\pm 0.05\%$. If the perchlorate content lies near the limit it is advisable to compare the colours of the filtrates described above with those of methylene-blue solutions of known content and to refer to curves obtained by use of artificial mixtures of potassium perchlorate and sodium nitrate. Closer examination of the precipitate shows that it contains the nitrate in addition to the perchlorate of methylene-blue. With solutions containing up to 10% of pure sodium nitrate the precipitation of methylene-blue as nitrate from the chloride of the dye is proportional to the solubility product of the nitrate, whereas at higher concentrations salting out is also evident. For this reason methylene-blue chloride is unsuitable for the determination of very small amounts of perchlorate in Chili saltpetre, but it may be replaced by a solution of the perchlorate of the dye prepared by mixing 320 c.c. of 0.1% methylene-blue B extra solution with 250 c.c. of 0.1% potassium perchlorate solution, filtering, and washing the precipitate with 3×10 c.c. of water. 5 c.c. of a solution of 40 g. of sample made up to 100 c.c. with water are mixed with 5 c.c. of this solution and the time is taken until the first precipitate appears; with 0.2%, 0.15%, and 0.1% of potassium perchlorate in saltpetre the times are 9, 15, and 90 sec.; with 60% saltpetre solutions the times for 0.1%, 0.08%, and 0.06% KClO_4 are 20, 45, and 120 sec. respectively. Controls are effected exactly as described for methylene-blue B extra solutions. The presence of chlorate (up to 1%), iodate, periodate, or chloride in the concentrations observed with technical, crystallised Chili saltpetre has no influence on the results and the ordinary variations in atmospheric temperature need not be taken into account. H. WREN.

Petroleum insecticides. G. P. GRAY and E. R. DE ONG (Ind. Eng. Chem., 1926, 18, 175—180).—Toxic action of petroleum oils on plant life takes the form of defoliation, stunted growth, and deterioration in the quality of fruit, and in general has been found to follow the use of unrefined oils as a spray. Representative samples of petroleum oils were examined with a view to correlate their physical and chemical properties with their efficiency as insecticides. Further samples were also obtained by bulking the first samples, redistilling, and collecting fractions at 25° intervals. Tests on foliage and fruit were carried out by spraying orange, citrus, and black walnut trees with these oils in the form of soap emulsions of various strengths. In general

the heavier oils, *i.e.*, the higher-boiling fractions, are the most toxic to plant life, but this property is more clearly indicated by the amount of unsaturated hydrocarbons present. The oil most soluble in concentrated sulphuric acid is the most toxic in its action, and the worst oil in this respect was a sample recovered from the acid tar formed in the treatment of kerosene with sulphuric acid. A test is described for the determination of the solubility of the oil in 37*N*-sulphuric acid, together with a simple test for showing comparative capillarity. A table showing the characteristics and action of all the oils examined is given. S. BOWMAN.

Solid calcium cyanide. METZGER.—See VII.

PATENTS.

Calcium nitrate fertiliser. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 246,377, 22.6.25).—Concentrated calcium nitrate solutions are mixed with urea in the proportion of 1 mol. of the former to 1—4 mols. of the latter, and the heated solution is forced through a spray nozzle, *e.g.*, by compressed air, to obtain a granular product which is readily distributed.

A. G. POLLARD.

Insecticides, sheep dips and the like. McDougall and YALDING, LTD., and P. J. FRYER (E.P. 246,252, 12.11.24. Addn. to 229,773; cf. B., 1925, 334).—Sulphonated castor oil specified in the earlier patent may be replaced by any other sulphonated fatty oil, sulphonated sperm oil, or sulphonated wax. The active principles of derris are extracted with benzene or other solvent, and after distilling off the solvent the residue is dissolved in sulphonated oil or wax. Calcium salts of the sulphonated oils may be used in some cases. Lime-sulphur may be mixed with the insecticide without precipitation if a proportion of a colloid, *e.g.*, casein, is added. A. G. POLLARD.

Composition of matter. [Insecticide.] H. P. BASSETT and M. R. ISAACS, Assrs. to UNITED PRODUCTS CORP. OF AMERICA (U.S.P. 1,568,044, 5.1.26. Appl., 1.11.22).—Calcium diarsenate (70 pts.), calcium oxide (30 pts.), and casein (3 pts.) are mixed in a moist condition to form a product which when sprayed on plants or the like, either dry or with water, adheres for long periods owing to the adhesive action of the calcium caseinate formed. The caseinate also coats the arsenate particles, so that the insecticidal and vermicideal properties of the mixture are preserved. T. S. WHEELER.

Manure from sewage (E.P. 224,226).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Hydroxyl-ion concentration and volumetric alkalinity in beet juice clarification. A. H. W. ATEN and P. J. H. VAN GINNEKEN (Tijdschrift; Int. Sugar J., 1926, 28, 54; cf. B., 1925, 684, 1014).—Using a form of hydrogen electrode potentiometer, the p_H values were determined for juices after the

first and second carbonatations, and for the carbonated juice after sulphitation, comparison being made with an artificial juice consisting of a solution containing 12.5% of sucrose and 1% of potassium chloride to which lime had been added. At the same volumetric alkalinity the p_H values of ordinary limed juice were considerably lower than those of the artificial juice containing lime only as base. At the end of the first carbonatation, the p_H value was not very much lower than that of the limed artificial juice; after the second carbonatation the difference was greater; and after sulphitation it was greater still.

J. P. OGILVIE.

Behaviour of raffinose in the process of crystallisation of sugar. R. MEHRLE (Deuts. Zuckerind., 1925, 50, 1325—1328, 1357—1361).—Sugar crystallising out from massecuites containing raffinose may contain 40—50% of this trisaccharide, 5—10% of ash, and 45—50% of organic non-sugars reckoned on the non-sucrose substances present. Generally about one-third of the raffinose originally present crystallises out with the sugar, this amount depending on whether raw or white sugar is being made. Raffinose has a considerable influence on the yield of crystals obtained in refining raw sugars.

J. P. OGILVIE.

Decolorising carbons [for sugar refining]. A. A. BLOWSKI and J. H. BON (Ind. Eng. Chem., 1926, 18, 32—42).—A number of commercial decolorising carbons were found all to show moderate or high decolorising power and ash-adsorptive properties, but certain of them proved poor filtering media, making them unsatisfactory for practical use in the sugar refinery. Laboratory-scale tests indicated that by the use of decolorising carbon it would be possible to produce a granulated sugar which compares favourably with that made by the bone-char process. The nett cost of the two processes would, however, be about the same, and in view of the difficult practical problems which would require solution, no advantage would be gained by substituting the former for the latter and older mode of refining. A combined carbon-and-char process is considered to be an undesirable substitute for the simple bone-char process.

J. P. OGILVIE.

Determination of mixtures of sucrose and invert sugar or lactose. II. H. JESSEN-HANSEN (Compt. rend. Trav. Lab. Carlsberg, 1925, 16, [4], 1—7).—Work on this subject previously published (B., 1923, 1237 A) has been elaborated, the procedure being modified so that the action of hydrochloric acid on the lactose is reduced to a minimum, and so that the method may be applicable when sucrose is present in notable excess. Moreover, the tables have been extended.

J. P. OGILVIE.

Detecting coal tar dyes in caramel. L. VON NOËL (Pharm. Zentr., 1926, 67, 33—35).—For the detection of aniline dyestuffs in commercial caramels it is essential that very dilute solutions be used and that double dyeing be adopted, *i.e.*, that a second

thread is dyed by means of the colour on the first coloured thread. As a fixing agent tartaric acid is to be preferred to potassium sulphate.

J. P. OGILVIE.

PATENTS.

Refining of saccharine liquids. P. SINGH and S. K. S. MAJITHIA (E.P. 244,924, 6.1.25).—A process for clarifying or decolorising saccharine liquids consists in removing suspended impurities, rendering the liquid alkaline, adding aluminium salt and alkali to form aluminium hydroxide, heating to about 100°, and separating the precipitated matter. A bleaching and clarifying agent which under heat will decompose into a metal hydroxide and nascent sulphur dioxide (*e.g.*, aluminium hydroxide suspended in sulphur dioxide solution) is added to the separated liquid after it has been rendered acid, the mixture being boiled, and the precipitated matter finally separated. In the first filtration kieselguhr may be added to form easily washed cakes.

J. P. OGILVIE.

Manufacture of sugar. A. P. LEONARD (U.S.P. 1,568,512, 5.1.26. Appl., 26.7.21).—Bagasse is passed successively through a series of mills, or crushing units, and the juice is conveyed from the foremost of them to separators, where clear juice is extracted. Sugar is separated from the clear juice from one separator, and water is introduced into the residuum in another separator. The clear juice from the separators is then returned to an intermediate mill of the series, juice from these intermediate mills being passed to the foremost mill. Juice from the last mill of the series is sent to a mill in advance of that receiving the liquid from the separators, whilst that from the advance mills goes to the foremost mill.

J. P. OGILVIE.

Recovery of sugar from molasses. G. R. BLUM (F.P. 594,526, 10.5.24).—Molasses is mixed at ordinary temperature with 60—90% of a fatty acid, or an alcohol, or a hydrocarbon, the mixture kept for several hours, and the sugar then separated from the liquor above it, from which potassium salts, nitrogenous substances, and the added substances can later be recovered.

J. P. OGILVIE.

Making grape sugar. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,571,212, 2.2.26. Appl., 28.5.23; cf. U.S.P. 1,508,569; B., 1924, 1024).—Starch is converted into dextrose, and the aqueous solution is supersaturated by heating and cooled to, and maintained at, a temperature favourable to production of crystals of anhydrous dextrose. These are separated, and the mother-liquor is cooled further to induce production of hydrated crystals, which are then separated from the mother-liquor.

D. G. HEWER.

Extraction of total sugar from apples and pears. DISTILLERIES DES DEUX-SÈVRES (Soc. ANON.) (F.P. 596,201, 12.7.24).—The fruits are subjected to a single or repeated pressing and the residue is treated in a diffusion battery. The juice

obtained is worked up for sugar by the known methods.

E. H. SHARPLES.

Treatment of condensate obtained by distillation of carbohydrate solutions containing hydrochloric acid. T. GOLDSCHMIDT A.-G., Assees. of E. HÄGGLUND and E. FÄRBER (G.P. 421,829, 19.7.22).—The condensate containing hydrochloric and acetic acids is treated with a dehydrating agent, e.g., sulphuric acid, phosphoric acid, or calcium chloride, at a temperature below the boiling point and the issuing vapours are collected. The residue after expelling hydrochloric acid is heated to boiling to obtain the acetic acid.

E. H. SHARPLES.

Manufacturing yeast (E.P. 246,002).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Loss of alcohol in spirits. H. LÜHRIG (Pharm. Zentr., 1926, 67, 49—55).—There may be an apparent loss of alcohol due to an increase of the specific gravity of the spirit by extraction and solution of matter from the (wooden) container. When freshly scalded containers are used a diminution of from 0.1 to 0.15% in the alcohol content may be caused by extraction of water from the pores of the wood. The contents of part-filled casks, subject to fluctuation of temperature, may not be homogeneous, the upper portions being the richer in alcohol. Differences of 4.4% and 0.4% of alcohol (by volume) respectively were found in two brandies which had been in cask for 7 months and for 2½ years. The same action may take place, on a smaller scale, in the retailer's bottle. No loss of alcohol was observed in brandy kept in bottles (for four months) the stoppers of which were removed for a minute four or five times a week. Brandy kept in an open bottle for a month lost 0.5—0.7% of alcohol. A margin of 1% of alcohol should be allowed for both by the distiller and by law, to cover possible losses of alcohol during storage and sale. The literature of the subject is reviewed.

G. T. PEARD.

See also A., Mar., 274, Specific action of α -glucosidase from yeast (HELPERICH, KLEIN, and SCHÄFER). 321, Isolation of enzymes (WILLSTÄTTER); Amylase action of emulsin preparations (JOSEPHSON); Milk-diastrase (CHRZASZCZ and GORALOWNA); Decrease of invertase in yeast (WILLSTÄTTER and LOWRY, JUN.); Invertase (WILLSTÄTTER, SCHNEIDER, and WENZEL). 322, Enzyme action and ion antagonism (HÖBER and SCHÜRMEYER); Maltase of barley malt (LEIBOWITZ); Hitherto undescribed oxydase in certain fungi (WOLFF); Cell-free fermentation of hexose-diphosphoric acid, dextrose, lævulose, sucrose, and invert-sugar (NEUBERG and KOBEL). 323, Nomenclature of fermentation enzymes and oxydases (NEUBERG and OPPENHEIMER); Dehydrogenases of yeast (VON EULER and NILSSON); Hydrolysis of polysaccharides by yeast (IWATSURU).

PATENTS.

Process of manufacturing yeast. H. W. DAHLBERG (E.P. 246,002, 20.5.25).—The waste water produced in the process of extracting sugar from beet molasses and syrups, which contains amino-nitrogen, is added to the yeast mash, so as to supply from 0.4 to 1.0 pt. of amino-nitrogen to 100 pts. of sugar in the mash. The waste water is alkaline, and is added at intervals in quantities depending on the p_H of the mash and is thus used both to supply nitrogen and to control the acidity during the growth of the yeast. C. T. GIMMINGHAM.

Preparation and application of an adsorption agent for the purification of alcohols and oils. RIERT-WERK E. RICHTER (G.P. 422,313, 14.6.21).—The product obtained by calcining a mixture of silica, aluminium hydroxide, and lime-wood charcoal is agitated with the liquid to be purified and then separated. Removal of empyreumatic substances, fusel oil, and substances producing turbidity is thus quickly effected without injury to the product.

E. H. SHARPLES.

XIX.—FOODS.

Determination of the ash-content of flour. A. GOSKE (Z. Unters. Nahr. Genussm., 1925, 50, 428—429; of B., 1925, 778).—A weighed quantity of sample is made into a paste with water and about twice its weight of powdered pumice (previously heated and weighed) in a platinum dish. The paste is dried, heated gently, and after the char has been crushed, is placed in a muffle for 15 min. at a moderate temperature, cooled, and again weighed.

J. GRANT.

Relation between the bacterial count of whole milk and that of the cream and skim milk separated from it. C. S. LEETE (J. Agric. Res., 1925, 31, 695—699).—Cream obtained by a clean centrifugal separator does not give a greatly higher bacterial count than the whole milk from which it is derived, whereas cream separated by gravity gives a much higher bacterial count than the whole milk.

W. O. KERMAK.

Alkaline milk and its detection by the bromocresol-purple test. I. F. PROCTER and A. T. R. MATTICK (J. Agric. Sci., 1926, 16, 145—148).—Directions are given for the testing of milk for alkalinity by the use of bromocresol-purple test papers. Results are given for samples of milk from a number of cows at different dates. In 92.8% of the cases examined, the reaction was between p_H 6.5 and 6.75, whilst in 3.3% of the cases the reaction was between p_H 6.9 and 7.13. G. W. ROBINSON.

Determination of palm kernel oil and butter fat in margarine. G. D. ELSDON and P. SMITH (Analyst, 1926, 51, 72—76).—In continuation of earlier work (B., 1925, 224), Reichert-Polenske-Kirschner figures have been determined for various mixtures of palm kernel oil, butter fat, and other vegetable oils, and tables of corrections are given for

deducing the true figure for each value from the observed. The Reichert values are 0.3 unit too high when no butter is present and correct when 10% is present. The Kirschner values are corrected by deducting 0.1 when the butter content is small. The Polenske corrections are irregular.

A. R. POWELL.

Apple-juice and commercial pectin products. H. ECKART (Z. Unters. Nahr. Genussm., 1925, 50, 405—415).—A number of analyses of raw and boiled apple-juices and of commercial pectin products have been carried out, the density, refractive index, acidity, total solids, viscosity, and pectin and starch contents being determined. Straight lines are obtained when these values are plotted against the concentration of the juice at different stages of evaporation. An attempt is made to assess the commercial value of a product by means of its "pectin-value," *i.e.*, pectin content \times 100/percentage extract, the pectin being determined by the calcium pectate method. The influence of concentration on the pectin-value and jelly-forming capacity is also discussed, and some additional criteria are suggested for the assessing of commercial products.

J. GRANT.

Distinguishing reconstituted dried preserved peas from preserved green peas. C. F. MUTTELET (Compt. rend., 1926, 182, 238—240).—Preserved green peas have the same chemical characteristics as fresh peas, *viz.*, ratio of starch to soluble sugars 5.1:1; starch to cellulose, 4.1:1; nitrogen content of insoluble constituents to nitrogen content of soluble constituents, 3.8:1. Reconstituted dried peas have a different composition; the ratio of starch to soluble sugars is 11.1:1; starch to cellulose, 7.6:1; insoluble to soluble nitrogen, 9.6:1. The reconstituted dried peas, therefore, have a higher proportion of insoluble constituents, due, it is suggested, to the cooking process they undergo during reconstitution.

L. F. HEWITT.

Determination of husk in cocoa. C. H. KOPPERBERG (Chem. Weekblad, 1926, 23, 64—66; cf. Silberberg, B., 1923, 26 A).—2 mg. of the cocoa are defatted by treatment successively with nitric acid and potassium hydroxide, the liquids being separated in each case by centrifuging, and the residue is examined microscopically. The husk cells are counted, and the results compared with those obtained from pure dehusked and defatted cocoas to which known proportions of husk have been added. Material which has been passed through the bolting mill loses a proportion of its husk. S. I. LEVY.

Study of the process of making stack silage. H. E. WOODMAN and F. HANLEY (J. Agric. Sci., 1926, 16, 24—50; cf. Amos and Woodman, B., 1925, 1008).—An account, with temperature readings and chemical data, of experiments on stack ensilage. During the ensilage two distinct temperature maxima occur. The significance of the second maximum is not certain, but the evidence points to anaerobic cellulose bacteria as being responsible. The losses

of dry matter in the "sweet" silage layer compare favourably with those obtained in tower ensilage and are appreciably smaller than those involved in the production of "sour" silage in the stack. The initial high temperatures largely inhibit the formation of amino-acids from proteins owing to the destructive effect on the plant enzymes. Ether-soluble material decreases during ensilage. The decrease in digestibility of the protein is largely due to changes similar to humification; a similar explanation may serve for the decreased protein digestibility of heated brown hay. The destruction of carbohydrates cannot be correlated with the temperatures attained during fermentation. G. W. ROBINSON.

Sunflower as a silage crop—composition and yield at different stages of maturity. W. L. GAINES and W. B. NEVENS (Univ. Illinois Agric. Exp. Sta. Bull., 1925, [268], 407—455; Chem. Abstr., 1926, 20, 78).—Sunflowers produce greater yields of dry matter per acre than maize; they contain more ash, crude fibre, and crude fat, but less nitrogen-free extract. A sunflower crop removes about 900 lb. of ash from 1 acre of soil. The relative proportions of the constituents of the ash are approximately constant, but not that of the ash and the organic matter. A. A. ELDRIDGE.

Dry matter of swedes. I. F. W. SANSOME (J. Agric. Sci., 1926, 16, 51—58).—Mainly an investigation of methods of sampling swedes for analysis. There is a well marked inverse correlation between dry matter content and size of root.

G. W. ROBINSON.

Modern views on vitamins. J. C. DRUMMOND (J. Soc. Arts, 1926, 74, 369—388).

Vitamin potency of cod-liver oils. HOLMES and PIGOTT.—See XII.

PATENTS.

Drying and congelation of egg albumins. E. E. LIBERSA (E.P. 229,669, 18.2.25. Conv., 22.2.24).—Certain alkaline-earth hydroxides or basic salts, *e.g.*, calcium hydroxide or calcium saccharate, are added in the proportion of about 5 pts. per 1000 to liquid white of eggs before drying or congelation. The albumin may then be dried directly, without preliminary fermentation and addition of ammonia; and all the properties of fresh egg albumin are retained. C. T. GIMMINGHAM.

Puffed [food] products. ANDERSON PUFFED RICE Co., Assees. of A. P. ANDERSON (E.P. 232,543, 21.7.24. Conv., 21.4.24).—The material, *e.g.*, cereal food, containing moisture and/or matter which expands greatly on reducing the pressure, *e.g.*, a gas, is reduced to such a form that it flows under pressure. From the pressure vessel, after impregnating or not with carbon dioxide, it is passed under a pressure of 500 lb. through a heater, which is a steam jacket containing a number of tubes leading to an orifice or orifices, the total cross-sectional area of the orifices being less than that of the tubes. The material is then passed through a drying chamber in which the

heat preferably decreases in intensity in the direction of travel. A puffed product made thus consists of a strip of cellular formation, the walls of the cells containing disrupted starch granules. The outer cells are smaller and have thicker walls than the inner, and the centre of the strip is hollow.

D. G. HEWER.

Spray drying apparatus for manufacturing milk or like powder. MILKAL, LTD., and J. M. SIERRA (E.P. 246,239, 3.11.24).—Milk or the like is concentrated and then fed under pressure to the atomising nozzles, by a pump provided with a by-pass containing a loaded relief valve capable of adjustment so that return of pressure liquid to the suction side of the pump may take place when a definite pressure is attained. With this arrangement the milk may be pre-concentrated to a higher degree than is usual; or the use of additional heat to reduce the viscosity of the concentrated milk may be avoided.

D. G. HEWER.

Filter, especially applicable to milk and other potable liquids. R. SELIGMAN (U.S.P. 1,568,001, 29.12.25. Appl., 13.5.25).—The filter comprises a fixed median plate fitted on both sides with cover plates so constructed that filter chambers are formed between them and the median plate, the chambers being divided by means of filtering media into spaces for filtered and unfiltered liquid, respectively. Supply of liquid through one or other of the filter chambers is controlled by means of a multi-way valve on the median frame, the valve being connected with the filter chambers through the median plate and through projections on the cover plates.

L. A. COLES.

Vitamin [malt] preparation. SOC. OF CHEM. IND. IN BASLE, ASSEES. OF O. STINER, A. HAUSWIRTH and A. GAMS (U.S.P. 1,568,196, 5.1.26. Appl., 18.1.19).—Amylaceous material rich in vitamins is mashed and saccharified with ground, fresh, green malt at temperatures below 60°, and sufficient of a non-toxic polybasic organic acid is added to make the mash feebly acid to Congo-red. The filtered mash is evaporated to the desired consistency. The preparation is in a durable form and contains both heat-stable and heat-labile vitamins.

G. T. PEARD.

Production of caffeine-free coffee. F. KÜNDIG, JUN., & CO. (F.P. 597,392, 28.4.25. Conv., 6.5.24).—The raw, whole coffee-beans are treated in an autoclave under 22 atm. pressure, with hydrocarbons, ketones, or alcohols, and alkalis or organic acids.

J. GRANT.

Production of caffeine-free coffee. SCHWEIZER UND CO. (Swiss P. 100,167, 4.12.22).—The raw coffee-beans are treated with 1% ammonia solution *in vacuo*, and extracted with acetone.

J. GRANT.

Baking powders. C. H. BOEHRINGER SOHN, CHEM. FABR., ASSEES. OF F. RAHN (G.P. 418,341, 20.7.22).—The rising-power of baking powders prepared from lactic acid and calcium lactate is increased by the addition of comparatively small quantities of

mono- or di-basic phosphates so that the total acidity of the powder is not reduced by more than one-half. Such baking powders yield results similar to those obtained by the use of yeast.

A. J. HALL.

Electrical apparatus for preserving sap-containing fodder. SIEMENS-SCHUCKERTWERKE G.m.b.H., ASSEES. OF O. KAUFMANN and E. CRAMER (G.P. 418,481, 10.11.21).—The consumption of electrical energy in the treatment of green fodder is considerably reduced by using a number of electrodes to which the current is supplied separately so that on introducing fresh fodder, the current can be cut off from that part of the container containing the fodder which has already been subjected to treatment.

A. J. HALL.

Manufacture of preparations which evolve carbon dioxide when heated or dissolved in water. J. D. RIEDEL A.-G. (G.P. 418,485, 15.1.21).—Effervescent preparations which yield refreshing beverages having a pleasing flavour are prepared from adipic acid or its salts and ammonium or alkali carbonates; they are not hygroscopic and are thus superior to the usual preparations which contain tartaric acid or its salts.

A. J. HALL.

Manufacture of a foodstuff [from yeast]. C. ENGEL (G.P. 421,707, 18.9.23. Addn. to 413,414).—By raising the temperature to 180–220°, yeast treated as described in the previous patent (B., 1925, 735) is converted into a paste suitable for spreading on bread.

J. GRANT.

Production of high-grade flour from maize. J. HÄUSLER (G.P. 421,870, 6.10.18).—Dry coarse-ground maize is freed from embryos by washing in water, and steeped in an acid solution at 70–90°. After neutralisation and drying, the meal is separated by any convenient means and finely ground.

J. GRANT.

Production of high-grade flour from maize and other cereals. J. HÄUSLER (G.P. 421,871, 21.12.18).—The material is converted into a paste by means of an acid solution, preferably a solution of hydrochloric acid, and is then atomised and carried by a current of dry air through a chamber in which the flour-, gluten-, and bran-ingredients separate and are deposited in different parts of the chamber according to their densities.

J. GRANT.

Conversion of hard cheese into durable soft cheese. M. CLAASS (Austr. P. 100,222, 5.3.24).—Hard cheese is heated for a short time at a temperature not exceeding 100° with neutral alkali phosphates.

A. J. HALL.

Manufacture of margarine and edible fats. E. V. SCHOU (U.S.P. 1,570,529, 19.1.26. Appl., 14.10.21).—See E.P. 187,299; B., 1922, 994 A.

Manufacture of albumins and fatty matters. M. KAHN, ASSR. TO SOC. FRANÇ. DES PROD. ALIMEN-TAIRES AZOTÉS (U.S.P. 1,572,933, 16.2.26. Appl., 14.4.25).—See E.P. 236,158; B., 1925, 939.

Fats (G.P. 422,687).—See XII.

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

Determination of atropine. P. BOURCET (Bull. Sci. Pharmacol., 1925, 32, 585—588; Chem. Zentr., 1926, I, 1465).—A mixture of 100 g. of the powdered drug, ground to pass through an 80-mesh sieve, with 20 g. of finely powdered calcium oxide, is extracted with ether in a Soxhlet apparatus. The ether extract is shaken with 2% hydrochloric acid, and the aqueous layer is removed, filtered, rendered alkaline with sodium hydroxide, and extracted with chloroform. After filtering the chloroform solution through a layer of anhydrous sodium carbonate, the solvent is evaporated off, and the residue is dried at 100° and weighed, giving the total weight of non-volatile alkaloids. After heating to 120° for 5 hrs. in order to convert hyoscyamine into its isomeride, the alkaloid mixture is dissolved in absolute alcohol, and the atropine is precipitated as oxalate by the addition of an ethereal oxalic acid solution, the precipitate being removed, washed with ether, dried at 100°, and weighed.

L. A. COLES.

Use of hexylresorcinol and its homologues as internal antiseptics. V. LEONARD (Science, 1925, 62, 408—412; Chem. Zentr., 1926, I, 1231).—The lower alkylresorcinols possess strong bactericidal properties, and are relatively non-poisonous, the antiseptic properties increasing and the poisonous action decreasing in proportion to the sum of the atomic weights of the alkyl radicals. The maximum bactericidal action is reached in hexylresorcinol, which has a phenol coefficient of 46—56.3. This compound, and other higher members of the series, have been prepared as described in E.P. 219,922, 223,190, and 224,913 (cf. B., 1925, 378, 568). No poisonous symptoms developed when hexylresorcinol was given to dogs in repeated per-oral doses of 0.5 g. for 21 days, or in single doses of 1 g. per kg. The compound causes no irritation of the urinary ducts, develops its bactericidal action when strongly diluted with urine of suitable reaction, and partially separates in the kidneys in the form of a non-reactive conjugation product, enough remaining uncombined, however, to exert bactericidal action in the urine. It is suitable for use in treating cases of infection of the urinary ducts, such as pyelitis and cystitis.

L. A. COLES.

Volatility of salicylic acid. A. F. LERRIGO (Analyst, 1926, 51, 79).—The salicylic acid content of an ethereal solution obtained by extracting medicinal substances with methylated ether may be determined directly by allowing the ether to evaporate spontaneously and drying the residue at room temperature for 48 hrs., or preferably for 2 hrs. in an oven at 35°. Salicylic acid does not volatilise appreciably below 40°, but between 50° and 60° volatilisation is comparatively rapid.

A. R. POWELL.

Preparation of aspartic acid from asparagine. F. PACHLOPNIK (Z. Zuckerind. Czechoslov., 1925, 50, 139—141).—Asparagine is hydrolysed by dilute

nitric acid, excess of acid neutralised by ammonia, the liquid evaporated to dryness, and ammonium nitrate extracted from the residue by hot alcohol.

J. P. OGLIVIE.

Pine oil and the products of its autoxidation. P. EPPLE and S. RUHEMANN (Ber., 1926, 59, 294—301).—The methods which have led to the isolation of azulene from the neutral oil of lignite tar (Herzenberg and Ruhemann, B., 1926, 42) have been applied to pine oil. The pine oil, which had been preserved for about ten years, was subjected to a preliminary treatment with dilute sulphuric acid and alkali and fractionally distilled under diminished pressure. The separate fractions were treated with hydroferrocyanic acid and the autoxidation products separated from the precipitates by extracting the latter with ether; the residual precipitate was treated with sodium carbonate at atmospheric temperature and the separated oil was isolated, after which the aqueous solution was boiled and the hydrolytically separated alcohols were extracted; the alkaline solution which remained contained the organic acids which, however, could not be separated from admixed hydroferrocyanic acid. The presence of camphor, borneol, cineole, terpin hydrate, and *p*-tolylidimethylcarbinol was thus established. These substances are not present in the oil previous to autoxidation. Terpin hydrate does not exist in the oil, its production being due to the action of hydroferrocyanic acid on terpineol.

H. WREN.

Anise oil and star-anise oil. W. ZIMMERMANN (Apoth.-Ztg., 1925, 40, 1344—1345; Chem. Zentr., 1926, I, 1310).—The hydrochloric acid test with pure anise oil, star-anise oil, and mixtures of the two does not give trustworthy results and the following is suggested. Five drops of a vanillin solution (0.4 g. of vanillin in 5 g. of dilute alcohol) are mixed with 2—3 drops of the oil, and fuming hydrochloric acid is added to make 1 c.c. The colour is first observed in the cold, then in a water bath at 50°, which is slowly heated to boiling. Freshly distilled anise oil becomes pale red on warming and finally brownish-red, which remains on cooling. Star-anise oil on warming gradually becomes pale green, then grass-green, and on boiling brownish-green; on cooling, olive green. With a mixture of anise oil and 10% of star-anise oil a dirty green colour is obtained, and with 30% of star-anise oil the colour produced by the anise oil is entirely masked.

E. H. SHARPLES.

Manufacture of alcohols from hydrocarbons with particular reference to petroleum as a raw material. B. T. BROOKS (Chem. Reviews, 1926, 2, 369—394).

Poisoning by chloronitrobenzene and acetanilide. RENSCHAW and ASHCROFT.—See IV.

See also A., Mar., 235, Partial formaldehyde vapour pressure of aqueous formaldehyde (LEDBURY and BLAIR). 236, Separation of mixed liquids by action of salts (SANDONNINI and GEROSA). 238, Quinine hydrochloride and

antipyrine (OLIVERI-MANDALÀ and CARLI); Acetanilide-antipyrine and acetanilide-pyramidone (OLIVERI-MANDALÀ and FORNI). 250, Catalytic activity of titania in decomposition of alcohols (RUDISILL and ENGELDER). 286, Organic peroxides (GELISSEN and HERMANS); Benzoyl hydroperoxide (LEVY and LAGRAVE). 288, New benzyl compound [betilon] (HINTZELMANN, JOACHIMOGLU, and OHLE). 292, Oxidation of anethole (HORIUCHI and UYEDA). 298, Isomeric terpineols; Active terpineols from active pinenes; Isomeric carvomenthols from carvacrol (PAOLINI); Caryophyllene series (HENDERSON, ROBERTSON, and KERR). 299, Oxidation of sesquiterpenes (GIBSON, ROBERTSON, and SWORD); Azulene (RUZICKA and RUDOLPH).

PATENTS.

Manufacture of styrol and its homologues. NAUGATUCK CHEMICAL CO., ASSEES. OF I. OSTROMILENSKY and M. G. SHEPARD (E.P. 232,909, 15.9.24. Conv., 7.5.24).—See U.S.P. 1,541,175; B., 1925, 692. The following temperatures are specified for the hydrocarbons named: *o*-methylethylbenzene 580–620°, *p*-methylethylbenzene 640–650°, 1:3:5-dimethylethylbenzene 640–650°, *p*-cymene 650°. The hydrocarbon vapour is passed through a heated conduit under such conditions that if R be the rate of flow in g. per min., T the temperature, and V the volume of the conduit in c.c., then the value of $K = (0.064V/R + T)/633$ falls within the limits 0.8 to 1.2.

Producing lead compounds [lead tetraethyl]. B. MEAD, ASSR. TO GENERAL MOTORS CORP. (U.S.P. 1,567,159, 29.12.25. Appl., 14.6.23).—An electrolytic cell for producing lead tetraethyl comprises a porous pot surrounding an annular lead cathode, the anodes being a number of graphite rods outside the pot. The catholyte is an emulsion of 10 pts. of ethyl iodide in 100 pts. of 10% sodium hydroxide, containing 1 pt. of casein. The anolyte consists of 10% sodium hydroxide. The porous pot is provided with a reflux condenser and an agitator and the cell is heated to 55°. On passing a current of 1.15 amp. per sq. dm. lead diethyl is formed in the porous pot and decomposes into lead tetraethyl and lead. The lead tetraethyl is separated and distilled in steam. The process gives good yields and can be applied to the preparation of other lead alkyls.

T. S. WHEELER.

Obtaining absolute propyl alcohol. J. A. STEFFENS, ASSR. TO U.S. INDUSTRIAL ALCOHOL CO. (U.S.P. 1,568,349, 5.1.26. Appl., 5.4.21).—Aqueous *isopropyl* alcohol containing 12% of water is mixed with a quantity of benzene equal to the alcohol and is passed to a steam-heated rectifying column from the bottom of which anhydrous *isopropyl* alcohol is recovered whilst a ternary mixture containing 19% of *isopropyl* alcohol, 8% of water, and 73% of benzene passes to the top of the column, whence a portion is returned to form a reflux, whilst the remainder is condensed; when cool it forms two layers, the upper containing benzene 79%, water

3%, and *isopropyl* alcohol 18%, and the lower water 73.5%, benzene 0.5%, and *isopropyl* alcohol 26%. The upper layer rich in benzene is passed to the column and the lower layer is led to a second column where it is rectified so that vapours containing 80% of *isopropyl* alcohol pass from the top of the column and are condensed and returned to the first column, whilst water flows from the bottom of the second column. The process can be applied to *n*-propyl alcohol.

T. S. WHEELER.

Preparation of solutions of mercurated hydroxybenzenesulphonic acids and their homologues. CHEMOSAN A.-G. (G.P. 419,609, 30.9.24. Conv., 23.9.24. Addn. to 399,394).—Solutions of therapeutic value, containing the mercury derivatives in molecular dispersion, are produced by neutralising a gel of the mercurated compound, using phenolphthalein or an indicator having the same range of colour change; or an accurately known weight of the gel is brought into solution, and two equivalents of sodium hydroxide solution are added. The process renders unnecessary the addition of neutral salts, as in the process described in the chief patent (cf. B. 1924, 927). For example, mercurated 2-hydroxy-5-sulphobenzene-1-carboxylic acid gel is heated below 70–75°, with sufficient water to yield a 1.5% solution. After addition of two equivalents of sodium hydroxide, the solution can be evaporated until it contains 5–6% Hg.

L. A. COLES.

Preparation of a Peru balsam substitute. O. SCHMATOLLA (G.P. 419,732, 7.10.24).—A product, similar to Peru balsam but possessing superior antiseptic powers and ease of manipulation, is obtained by dissolving condensation products of benzoic and cresotic acid derivatives in benzyl benzoate or benzyl alcohol. Thus, powdered sodium cresotate is slowly added to a cold mixture of benzoyl chloride, benzene, and alcohol, the temperature being maintained at 35°. Calcium carbonate is added and after keeping for 24 hrs., the mixture is warmed to 30°, with addition of alcohol and benzene, and filtered. The solvents are slowly distilled off, and by further heating of the residue for some hours at 60° a soft resin is obtained which is only slightly volatile in the cold and is soluble in benzyl benzoate and benzyl alcohol.

E. H. SHARPLES.

Preparation of colloidal metal solutions. A. BINZ (G.P. 417,973, 14.2.23).—The solutions, which are of use in chemotherapeutics and as plant sprays, are obtained by treating metal salts with reducing agents in the presence of condensation products obtained by the action of formaldehydesulphoxylates upon arsenobenzene derivatives substituted with amino-groups, such as 3:3'-diamino-4:4'-dihydroxyarsenobenzene, or upon arylarsinic acids, such as 3-amino-4-hydroxybenzene-1-arsinic acid. For example, a solution of sodium hyposulphite in 0.1N-sodium hydroxide is added to a mixture of aqueous solutions of 3:3'-diamino-4:4'-dihydroxyarsenobenzenemethylenesulphoxylate and copper sulphate, yielding after a short time a deep red solution of colloidal copper from which a reversible copper

colloid is precipitated by the addition of acetone. Solutions containing colloidal silver or mercury can be prepared by the same method, using sodium hyposulphite or hydrazine hydrate as reducing agent.

L. A. COLES.

Solid protected colloids in soluble form. H. VOGEL (G.P. 422,150, 12.10.22).—The colloid is precipitated by a second colloid with which it will form a peptisable insoluble mass which can be purified by washing etc. As an example, silver protected by gluten can be precipitated with tannic acid and peptised, after kneading with water to remove electrolytes, by addition of a small quantity of a base.

E. S. KREIS.

Separating butyl alcohol and water. A. STEVENS (Reissue 16,267, 16.2.26, of U.S.P. 1,394,232, 18.10.21. Appl., 9.10.24).—See B., 1921, 877 A.

Purifying alcohols and oils (G.P. 422,313).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Influence of desensitisers on the coloured tints of photographic papers. J. MILBAUER and J. LAUSCHMANN (Chem. Listy, 1926, 20, 12—17).—Addition of small quantities of Phenosafranine and Pinakryptol Green to the usual developers prevents the formation of warm tones, such as light brown or blood-red, during the development of silver chloride papers. The resulting positives are black or brownish-black. The rate of development increases appreciably with only 0.001% of desensitiser. With very dilute solutions of the developer coloured tones may be obtained even in the presence of the desensitiser, except in the case of amidol. Spent developing solutions or those diluted so that they produce only light brown tones will produce satisfactory black to brownish-black images, if the above desensitisers are added in suitable proportion (cf. B., 1925, 693).

A. R. POWELL.

Photochemical oxidation of leuco-bases. CARROLL.—See A., Mar., 253.

PATENTS.

Photographic gelatin emulsion surfaces. J. LEYDE, Assee. of E. LEYDE (E.P. 241,152, 19.2.25. Conv., 9.10.24).—Gelatin emulsion surfaces are coloured with a light-filtering dye by treatment with an alcoholic solution of a water-soluble dye (e.g., for the orange-red, a mixture of Acid Aniline Red and Tartrazine, and for the green, a mixture of Tartrazine and Patent Blue, Höchst) to which has been added a quantity of water such that, by the time complete drying has occurred, the dye will have penetrated into the gelatin for a small distance which is essentially less than the thickness of the surface changed by the action of light. The method may be applied to the production of a film band provided with alternately differently coloured screen-sections by pressing absorbent rollers dipping into

the dye solution against the emulsion surface which is covered with a movable stencil so that alternate bands of the film surface may be exposed.

A. W. HOTHERSALL.

Films for instantaneous X-ray exposures. P. SPECKLIN (F.P. 591,629, 8.1.25).—The body of the film contains some substance which, under the influence of X-rays, emits radiations which intensify the action of the X-rays. Suitable substances are calcium tungstate, barium platinocyanide, zinc sulphide, quinine sulphate, Eosin, and metallic zinc, copper, nickel, iron, etc. The exposure time is shortened considerably.

W. CLARK.

Positive blue-print paper. DÜRENER FABR. PRÄPARIERTER PAPIERE RENKER UND CO. (G.P. 419,177, 28.3.25. Addn. to 403,902; cf. B., 1925, 229).—Salts of heavy metals, suitable ones being those of copper, molybdenum, and uranium, which are reduced by light are used, and subsequently treated with ferricyanide or ferrocyanide to give a coloured compound.

W. CLARK.

Photographic developer for development in bright light. F. HILDEBRANDT (G.P. 419,179, 23.11.24).—To a concentrated developer solution is added a concentrated solution of desensitiser, together with a colloid, e.g., dextrin, gum arabic, or, especially, water-glass, to prevent precipitation of the desensitiser.

W. CLARK.

Selenium toning bath for photographic developing and printing papers containing silver. LEONAR-WERKE ARNDT UND LÖWENGARD (G.P. 419,428, 17.7.24).—A selenium toning bath appreciably stronger than other known baths is prepared by dissolving selenium by boiling in an aqueous solution of sodium carbonate with addition of hyposulphite.

W. CLARK.

Development of images from diazo-compounds and components capable of coupling. KALLE UND CO. A.-G. (G.P. 419,987, 4.12.23).—Development is effected in an enclosed space in which ammonia gas is evolved from substances yielding it. The process is simple and cheap.

W. CLARK.

Toning silver images. K. PETERS (G.P. 421,011, 6.11.24).—In addition to salts of light and heavy metals, the toning bath contains compounds formed by the action of nitrites on hyposulphites in feebly acid solution. Under these conditions salts of molybdenum, tungsten, nickel, and cobalt act like gold salts.

W. CLARK.

Desilvering old [photographic] films. W. TRAXL (Austr. P. 100,855, 8.4.24).—The desilvering bath consists of 3—6% nitric acid containing about 1% of manganous sulphate, 1% of copper sulphate, or, preferably, 0.1—0.2% of potassium dichromate. The silver is then precipitated from this solution as chloride.

W. CLARK.

Photographic media and process. M. C. BEEBE and A. MURRAY, Assrs. to WADSWORTH

WATCH CASE CO. (U.S.P. 1,574,357, 23.2.26. Appl., 31.8.22).—See E.P. 203,285; B., 1924, 493.

Infra-red sensitisers (U.S.P. 1,568,667).—See IV.

XXIII.—SANITATION; WATER PURIFICATION.

Fundamentals in the conditioning of boiler water. R. E. HALL (Proc. Eng. Soc. W. Pa., 1925, 41, 347—390).—The conditioning of boiler water requires more than the softening of the water, since the questions of corrosion and scale formation on the heating and evaporating surfaces, and the entrainment of moisture and sludges and the presence of non-condensable gases in the steam must be considered. The scale in boilers is due to the deposition of crystals *in situ* from a saturated solution; the adherent deposits from natural waters consist chiefly of calcium sulphate and hydrous magnesium silicate, whilst a softer porous scale of calcium carbonate is often formed in the feed water lines or the preheater. The addition of soda-ash to the feed water will prevent the formation of adherent scale, the calcium being precipitated as carbonate and the magnesium as hydroxide, which can be removed easily. At high pressures or high ratings, however, soda-ash reacts with the water with the evolution of carbon dioxide; under these conditions sodium phosphate is used. The sulphate/carbonate or sulphate/phosphate ratio of the water must be very carefully controlled and depends on the nature of the water and the operating pressure of the boiler. Corrosion in the feed-water lines or the boiler is prevented by de-aeration of the water to remove dissolved oxygen, and by controlling the hydrogen-ion concentration by the addition of caustic soda. The amount of solid matter or sludge in suspension in the boiler will depend on whether the treatment of the water is external or internal and the proportion of condensed water from turbines, process work, etc. used to feed the boiler. The steam pipes must be protected by mechanical means from moisture and suspended solids entrained by the steam, so that the liability of corrosion and erosion by the condensing steam is minimised. It is very difficult to deal with the non-condensable gases in the steam (oxygen dissolved in the feed water, hydrogen by interaction of steam and the metal of the boiler, and carbon dioxide from the decomposition of soda-ash). If the presence of carbon dioxide in the steam is objectionable, sodium phosphate must be used for conditioning the water in place of soda-ash. In the discussion, the difference between water softening and boiler water conditioning was emphasised. It was pointed out that the removal of the sludge in the boiler is a mechanical question, involving external filtration and periodical blow-downs. The prevention of scale deposition generally leads to a tendency to form wet steam.

B. W. CLARKE.

Presence of air in pure and in alkaline water. J. PORTER (J. Roy. Tech. Coll. Glasgow, 1925, [2], 19—25).—When pure water is heated to 100° it

still retains about 11 c.c. of air per litre and this is removed only by prolonged boiling. Addition of 4 g. of sodium hydroxide per litre increases the rate of evolution of air at temperatures above 60° and the air retained at 100° is only 4.8 c.c. per litre. The solubility of air in 4% sodium hydroxide solution at 17° is 8 c.c. per litre, compared with 20.4 c.c. in pure water. Very little air is evolved on heating water until a temperature above 80° is reached, and experiments are described which indicate that the air which is not evolved as it ought to be below 80° forms a layer of no appreciable volume on the sides of the vessel and is not retained in supersaturated solution. If the water is maintained for a prolonged period at a temperature of 60°, however, all the excess air over the normal amount that will remain in solution at that temperature, is slowly liberated. Addition of a slight amount of oil to water during heating causes a more regular evolution of the dissolved air.

A. R. POWELL.

Chlorophenol tastes in Milwaukee's water supply. R. W. CUNLIFFE (J. Amer. Water Works Assoc., 1925, 14, 423—433).—The chlorinated water supply of Milwaukee, taken from Lake Michigan, on occasions in 1915 developed a taste which appeared to be the same or very similar to the taste of a dilute solution of chlorine. In 1917 and 1918, the taste occurred more frequently and was intense; whereas previously it had occurred during winter months, and was attributable to retarded absorption and dissipation of the chlorine on account of low temperatures, in 1918 the tastes occurred in summer and were persistent and marked. The taste was not affected by boiling, and residual chlorine was present in less amount during periods of taste than normally. Experimental filtration of various types, aeration, carbonation, and treatment with thiosulphate and other chemicals failed to have any effect. Samples of liquid trade waste from the different industries in the city were collected, chlorinated, boiled to remove traces of free chlorine, cooled, and tasted. Only wastes from gas plants and industries the effluents from which contained products associated with the destructive distillation of coal were found to possess taste, and of the products from this source only phenol and cresol in great dilution produced taste of stability and flavour comparable with those which had been experienced. Phenol in dilutions greater than 1 to 200,000 pts. of water could not be detected by taste, but after addition of a fraction of 1 p.p.m. of chlorine could be detected in dilutions of 1 pt. of phenol in 250—500 million pts. of water. That the tastes complained of were due to chlorophenols was confirmed by keeping the sources of pollution of the lake water by phenolic liquors under observation. The principal source of the trouble was waste liquor, discharged direct into the lake, derived from a phenol manufacturing plant; in some cases effluents from Semet-Solvay coke-oven and coal gas plants were responsible. Wastes from these sources, with one exception, are now disposed of by mixing with sanitary sewage.

W. T. LOCKETT.

Differential media for detection of *B. coli* in water. H. G. DUNHAM, M. H. McCRADY, and H. E. JORDAN (J. Amer. Water Works' Assoc., 1925, 14, 535—549).—A statement of the progress made to date in the work of the American Standardisation Council Committee (Standard Methods of Water Analysis), having reference to the present Amer. Publ. Health Assoc. standard method for determining the presence of the colon group of lactose-fermenting aerobes. Investigations include a study from a cultural standpoint of the dyes of American manufacture available for use in eosin-methylene-blue-agar, brilliant-green-bile medium, and gentian-violet-lactose broth; a comparative study of brilliant-green-lactose-bile (ox gall) and standard lactose broth; and an enquiry as to the value of brilliant-green-lactose-bile (ox gall) as a confirmatory medium. The results obtained by a large number of independent workers indicate that brilliant-green-lactose-bile is not entirely satisfactory for use as a medium for the *B. coli* presumptive test, a lack of sensitiveness as compared with standard lactose broth being noticed with some waters. The medium, however, apparently eliminates practically all spore-bearing lactose fermenters. Investigations carried out at Indianapolis regarding the value of brilliant-green-lactose-bile as a confirmatory medium, indicate that the interpolation of this medium, following growth in lactose broth, would result in the elimination of a large number of Endo-plates. Apparently, when used in this manner, brilliant-green-lactose-bile does not inhibit any organisms of the colon group, but the concentrations of brilliant-green (1:10,000) and bile (5%) used do not seem to be such as to be absolutely specific for colon group organisms. Gentian-violet-lactose-bile as confirmatory medium behaved similarly to brilliant-green-lactose-bile (cf. Howard and Thompson, B., 1925, 735).

W. T. LOCKETT.

Prophylaxis in industrial lead poisoning. D. MCKAIL (J. Ind. Hyg., 1926, 8, 74—77). A prescription is given for a mouth wash and drink so designed as to cause precipitation of lead as sulphate.

H. J. CHANNON.

Cause of "gulf" disease. P. BIGINELLI (Giorn. Chim. Ind. Appl., 1925, 7, 568—571).—This disease, which has appeared on the shores of the Gulf of Dantzig, is caused by toxic gases formed by putrefaction of waste liquors from (1) cellulose factories in which the sulphurous acid used is prepared from arsenical pyrites and (2) the treatment of arsenical hides.

T. H. POPE.

See also pages 260, Hazards from use of "ethyl-gasoline" (FLINN). 264, Effect of dyes used in paper-making on animal life in water (HAEMPEL). 265, Poisoning by chloronitrobenzene and acetanilide (RENSHAW and ASHCROFT). 272, Solid calcium cyanide (Metzger). 281, Volatilisation of lead during lead burning (ENGEL and FROBOESE).

PATENTS.

Softening of water. F. SCHMIDT (E.P. 227,429, 18.12.24. Conv., 12.1.24).—Caustic alkalis and carbonates, borates, phosphates, and like salts of alkali

metals are used in conjunction with suitable alkali compounds of acids insoluble in water (silicates, aluminates, aluminosilicates) to form a water-softener. The softening medium may be made into a solid or plastic mass by means of clay, mucilaginous substances, casein, and the like.

D. G. HEWER.

Manure from sewage and other substances. F. STÖTZEL (E.P. 224,226, 27.10.24. Conv., 3.11.23).—Sewage is filtered through a bed of peat, peat litter, or the like, and the resulting mass of sewage solids and filtering material is mixed with unburnt gypsum and/or kainite in a finely-ground condition to form a dry mass. Industrial refuse, animal waste substances, and/or chemical substances may then be added, and, if required, the mass, with or without further addition of nitrogenous substances, is pulverised.

W. T. LOCKETT.

Sewage purification plant. M. PRÜSS (E.P. 235,872, 12.6.25. Conv., 19.6.24).—The plant consists of a combined settling basin and digestion chamber separated by thin metal walls. The settling basin has a narrow and deep channel at the bottom, which communicates by means of a drag-pipe with an outer gutter at the top end of the digestion chamber. The levels of the water in the settling and digestion chambers are so arranged that the sludge from the bottom of the former can be conveyed up the pipe into the gutter by a siphon action and can be distributed as required to different parts of the digestion chamber.

C. T. GIMMINGHAM.

Preparation of sterilising agents, germicides, and the like. C. H. H. HAROLD (E.P. 245,820, 7.10 and 15.12.24).—A sterilising liquid is prepared, in which the active agent is mono- or di-chloroamine or a mixture of the two, by adding the correct proportion of chlorine in concentrated solution or as gas to an aqueous solution of ammonia containing not more than 0.0125 g. per litre. Under these conditions, the yield of chloroamines is almost quantitative. If the chlorine is allowed to diffuse through a porous diaphragm, and the ammonia solution is caused to circulate, thus avoiding local rise of temperature due to the heat of the reaction, the strength of the ammonia solution may be increased up to 0.75 g. per litre.

C. T. GIMMINGHAM.

Preheating and purifying [deaerating] boiler feed-water. C. HÜLSMEYER (E.P. 245,588, 14.7.24).

Degasifying or deaerating liquids [boiler feed water]. W. S. ELLIOTT (E.P. 247,342, 18.12.24).

Bacterial filters for purification of sewage and other liquids. C. R. B. BROWN (E.P. 247,769, 27.3.25).

Hydrogen-ion concentration of waters (G.P. 399,410 and 400,273).—See XVI.

Mixtures containing finely-divided copper compounds (G.P. 419,460).—See XVI.

Insecticides etc. (E.P. 246,252).—See XVI.

Insecticide (U.S.P. 1,568,044).—See XVI.