

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 25, 1926.

I.—GENERAL; PLANT; MACHINERY.

Modified fusion pot. V. S. BABASINIAN and R. D. BILLINGER (Ind. Eng. Chem., 1926, 18, 340).—A nickel vessel for the fusion of organic substances with alkalis, has a thermometer inserted into the fused mass through the hollow shaft of the stirring apparatus. More efficient stirring is obtained than with the ordinary method of having a thermometer separate from the stirring shaft. By suitable modification, the vessel may be converted into an autoclave. L. M. CLARK.

Influence of segregation on the corrosion of boiler tubes and superheaters. G. R. WOODVINE and A. L. ROBERTS (Iron and Steel Inst., June, 1926, Advance proof. 4 pp.).—Superheater coils and tubes in high-pressure water-tube boilers fail in service much more quickly when made from segregated steel than from carefully made high-grade steel. A coil made from a solid-drawn tube of segregated steel and a tube of steel free from segregates arc-welded into one length was used as a superheater in a boiler of 200 lb. working pressure with an average steam temperature of 316–371°. After 12 months the unsegregated portion was quite sound, whereas the segregated portion, scale from the interior of which contained 0.19% of sulphur, was badly corroded and perforated. The failures of tubes made from segregated steel ingots are attributed to electrolytic action promoted by blowhole segregates. M. COOK.

Preparation and use of industrial special sands. W. M. WEIGEL (Trans. Amer. Inst. Min. Eng., Feb., 1926. Advance copy. 14 pp.).—A description of the methods of washing and screening American sands for special purposes, e.g., filter beds, abrasives, cements. In all cases the elimination of clay matter is essential and a low content of iron oxide is necessary if the product (e.g., sodium silicate) is required of good colour. After washing, sands are invariably dried before grading and are despatched in the dry condition. C. A. KING.

Proposed British standard table of wire screens. O. C. ELLINGTON (J.S.C.I., 1926, 45, 128–134 T).—Screens of a standard table must be capable of easy and inexpensive manufacture in large sheets by normal trade methods and must be equally suitable for use in both manufacturing and laboratory control work. They must satisfy the following requirements:—(1) A systematic basis of graded apertures and nomenclature emphasising the primary importance of accurate aperture; (2) the inclusion of screens equivalent in aperture to all those

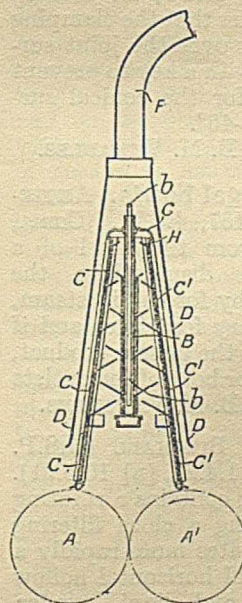
of established use in commerce; (3) the use of component wires from those quoted in the British S.W.G. table; (4) adherence to an integral and normally adopted number of meshes per linear inch; (5) the choice of wires of the same suitable relative thickness for each screen (a value of approximately 1.5 for the ratio aperture/wire diameter, and, consequently, a value of about 36% for screening area, are suggested). A table of screens possessing these characteristics is suggested and the disadvantages and restricted scope of existing tables of screens such as the I.M.M. table of laboratory screens, are discussed.

Theories of adsorption and technique of its measurement. MCBAIN.—See A., May, 467.

PATENTS.

Disintegrating apparatus. E. ROTH (E.P. [A] 242,312, 2.11.25, and [B] 247,526, 15.12.25. Conv., 1.11.24 and 16.2.25).—(A) Two cones rotating in opposite directions and having their bases opposed are provided on their interior surfaces with radial ribs. (B) Annular or circular ribs are also provided.

B. M. VENABLES.



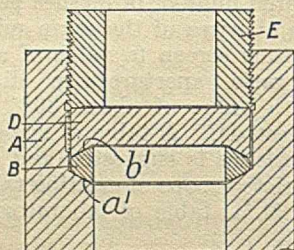
Evaporating apparatus. J. GOLDING and J. C. STEAD (E.P. 248,131, 23.12.24).—Referring to the figure, *A* and *A'* are steam-heated rolls and the vapour arising therefrom is guided by deflectors against the evaporating plates, *C*, *C'*, and the preheater, *B*, finally being drawn through the exhaust, *F*, by means of a fan. The liquid to be evaporated, e.g., milk, is fed through the pipe, *b*, to the bottom of the preheater, *B*, which is in the form of a narrow deep trough, and overflows in thin sheets over the evaporating plates, *C*, *C'*, finally being dried to a solid on the rolls, *A*, *A'*. B. M. VENABLES.

Evaporators. H. FOTHERGILL (E.P. 249,904, 1.12.24).—The liquid to be evaporated is first heated and then sprayed in the form of sheets into a vessel maintained at a pressure lower than corresponding with the b.p. of the heated liquor. The spraying apparatus consists of a tube in which longitudinal

slots are formed for the exit of liquor and which is situated under a horizontal or sloping cross baffle in the evaporating chamber. Other baffles are provided with the object of preventing entrainment, and methods of constructing the spraying tube are described.

B. M. VENABLES.

High-pressure joints. SYNTHETIC AMMONIA & NITRATES, LTD., and F. H. BRAMWELL (E.P. 249,001, 23.7.25).—In apparatus for pressures of the order of 200–400 atm., for use in the synthesis of ammonia for example, joints which may be as much as 4 ft. in



diameter are constructed as shown in the figure, where *A* is the body of the vessel to be sealed, *B* is a lens-shaped jointing ring, *D* a lid, and *E* a clamping screw. The gas-tight joint is made between the spherical surfaces of the lens ring and the conical surfaces, *a*¹, *b*¹, of the body and lid. It is essential that the lens ring be made of comparatively hard and elastic material, such as mild steel, and although it is compressed with sufficient pressure to obtain surface contact at *a*¹ and *b*¹ it must never be stressed beyond its elastic limit. The clamping screw, *E*, is used instead of a number of bolts in order that the clamping pressure may be applied directly over the joint surfaces, and the lid, *D*, is provided with a key to prevent rotation. A suitable obliquity for the conical surfaces *a*¹ and *b*¹ is stated to be 30–45°.

B. M. VENABLES.

Process of cleaning centrifugal bowls. AKTIEBOLAGET SEPARATOR (E.P. 249,457, 4.3.26. Conv., 18.3.25).—Sludge is removed from the bowl of a centrifugal separator without dismantling the apparatus but after stopping it, by forcing in steam, gas, or liquid through the ordinary feed inlet and if desired also through the outlet for the lighter product, the sludge being blown out of one or both of the outlets for separated products.

B. M. VENABLES.

Centrifugal separator. W. BIRCHALL (U.S.P. 1,572,419—20, 9.2.26. Appl., [A] 1.4.21, [B] 10.5.21).—(A) A rotating vertical cylinder contains a rotating coaxial cylindrical screen carrying a cloth filtering medium. Within this screen rotates more rapidly a hollow shaft carrying a number of horizontal hollow arms. The mixture of solid and liquid under treatment is introduced into the hollow shaft and passes through the hollow arms to the filtering screen. The liquid passes through this while the solid accumulates on the screen and is supported in place by metal flaps attached to the arms. The arms also carry scrapers which owing to the difference in the speeds of rotation of the screen and the arms

rotate on a vertical axis relatively to the screen and remove the solid material so that it falls to the bottom of the apparatus, whence it is recovered. (B) The separator is modified in that the scrapers consist of hollow cylinders fitted with slots through which the solid passes to the interior of the scraper where a vertical screw conveyor carries it to the bottom of the apparatus. Means are also provided for freeing the pores of the filtering material from solid by air under pressure. This is delivered to a compartment in the central shaft, whence it passes automatically to the outer side of those portions of the screen which are being cleared of solid by the scrapers. It passes inwards through the material thus clearing the pores.

T. S. WHEELER.

Protection of metallic surfaces against incrustation and corrosion. W. THALHOFER, ASSR. to A.-G. & CHEM. IND. IN LIECHTENSTEIN (U.S.P. 1,558,647, 27.10.25. Appl., 14.8.23).—This process is similar to that of E.P. 208,115 (B., 1925, 231) except that a "pulsating" electric current is used.

Recovered inorganic filter-aid. H. S. THATCHER, ASSR. to CELITE Co. (U.S.P. 1,571,074, 26.1.26. Appl., 24.1.23).—The spent filter-cake is dried, finely divided, and calcined at a temperature sufficiently high to remove part of the inorganic in addition to the organic impurities.

H. HOLMES.

Apparatus for the rapid filtration of liquids. A. BIGOT (F.P. 599,161, 6.6.25).—A rotating vacuum filter drum is just completely immersed in the liquid to be filtered, and at the top is in contact with a similar drum rotating around a parallel axis outside the liquid. A greater degree of suction is maintained in the upper drum, so that the solid material collected on the lower drum passes on to the upper at the point of contact and is removed from it by a scraper. As the whole of the filtering surface of the lower drum is utilised very rapid filtration is attained.

T. S. WHEELER.

Fluid [methylene chloride] for industrial purposes [fire-extinguishing etc.] E. R. BRODTON (U.S.P. 1,575,967, 9.3.26. Appl., 20.2.25).—Crude industrial methylene chloride is rectified to give a product, b.p. 38–71°, consisting chiefly of methylene chloride. It is non-explosive, does not corrode brass, copper, aluminium, or iron, and can be used as a fire extinguisher and in thermostats etc.

T. S. WHEELER.

Drying apparatus. E. C. READER (U.S.P. 1,576,547, 16.3.26. Appl., 12.2.25).—A heating chamber communicates with an external conduit and a second conduit arranged within the chamber is provided with a frusto-conical end extending into and spaced from the end of the first conduit. Means are provided for delivering material to be dried into the first conduit and for forcing a drying fluid through the second conduit.

H. HOLMES.

[Magnetic] separation of gaseous or liquid mixtures. P. E. HAYNES, ASSR. to LINDE AIR PRODUCTS Co. (U.S.P. 1,575,587, 2.3.26. Appl.,

2.9.19).—A mixture of fluids having different magnetic susceptibilities is passed through a conduit having a permeable wall and is acted upon by a transverse magnetic flux which draws the constituent of greater susceptibility through the permeable wall out of the main stream. B. M. VENABLES.

Apparatus for treating liquids with gases. W. E. GREENAWALT (U.S.P. 1,579,355, 6.4.26. Appl., 11.6.23).—The gas is passed into the liquid through a hollow rotating paddle working on a vertical shaft, the fins of the paddle being so disposed that they create upward currents in the liquid. A mixture of gas and liquid is fed into the paddle and emerges into the main body of liquid through the ends and sides of the fins. A. R. POWELL.

Furnace fired with coal dust and like pulverulent fuel. E. VOGT and L. KIRCHHOF (U.S.P. 1,580,080, 6.4.26. Appl., 26.1.26).—A furnace is provided with a combustion chamber which is completely open to the space to be heated. The powdered fuel is introduced at the top of the combustion chamber together with primary air, and a regulated amount of secondary preheated air is also admitted through the fuel inlet. B. M. VENABLES.

Apparatus for taking samples from closed vacuum or pressure vessels. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of G. HENNINGER (G.P. 423,603, 27.6.22).—An apparatus for taking samples from autoclaves or the like during working consists of a closed cylindrical container, which by rotation in a socket can be connected either to the autoclave or to the open air. Means are provided for admitting air or the like to the sampling vessel to force out the sample taken. T. S. WHEELER.

Fractional distillation and condensation of complex mixtures. SOC. ANON. D'OUGRÉE-MARIHAYE (E.P. 239,502, 20.8.25. Conv., 8.9.24).—See F.P. 598,414; B., 1926, 309.

Rotating retort. F. S. BACON, Assr. to M. A. BACON (Reissue 16,326, 20.4.26, of U.S.P. 1,556,194, 6.10.25. Appl., 22.1.26).—See B., 1925, 946.

Protecting from incrustations metallic surfaces which are in contact with water or other liquids. W. THALHOFER, Assr. to A.-G. F. CHEM. IND. IN LIECHTENSTEIN (U.S.P. 1,558,646, 27.10.25. Appl., 25.10.22. Renewed 2.2.25).—See E.P. 208,115; B., 1925, 231.

Eliminating scale in fluid containers. K. SCHNETZER, Assr. to ANTISCALE, LTD. (U.S.P. 1,581,024, 13.4.26. Appl., 25.7.24).—See E.P. 208,115; B., 1925, 231.

Apparatus for evaporating liquids indirectly. K. L. E. THUNHOLM (U.S.P. 1,562,525, 24.11.25. Appl., 9.3.21).—See E.P. 156,592; B., 1922, 163 A.

Automatic means for letting off steam from a container having in it a boiling liquid. FARBENFABR. VORM. F. BAYER & Co., Assees. of O. EINSLER (U.S.P. 1,572,272, 9.2.26. Appl., 25.11.24).—See E.P. 246,567; B., 1926, 304.

Centrifugal machine and process. L. D. JONES and A. U. AYRES (U.S.P. 1,575,061, 2.3.26. Appl., 29.5.25).—See E.P. 233,327; B., 1926, 343.

Separating device operating by centrifugal force. S. KARPINSKY and J. S. ANDERSON (U.S.P. 1,583,048, 4.5.26. Appl., 16.1.24).—See E.P. 214,577; B., 1924, 885.

Separating or recovering a gas from a mixture of gases. E. B. MILLER, Assr. to SILICA GEL CORP. (U.S.P. 1,577,534, 23.3.26. Appl., 15.6.22).—See E.P. 227,309; B., 1925, 157.

Combustion, reduction, or treatment of solid material or of pulverised minerals. X. DE SPIRLET (U.S.P. 1,581,397, 20.4.26. Appl., 2.10.24).—See E.P. 222,876; B., 1925, 871.

Straining or filtering apparatus. F. C. FULCHER (U.S.P. 1,581,998, 20.4.26. Appl., 26.11.23).—See E.P. 223,627; B., 1924, 1000.

Electrical purification of gases (U.S.P. 1,579,462 and G.P. 425,026, 425,039, and 425,273-4).—See XI.

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

Cleaning of coal. I. W. R. CHAPMAN and R. A. MOTT (Fuel, 1926, 5, 143-149).—The economy to the consumer of using clean coal is emphasised. Usually the largest lumps will contain least ash, but the actual optimum size varies from coal to coal, and practically every coal appears to produce lumps of one particular size which is the cleanest. The nature and quantity of the inorganic matter in coal, which is divided into "inherent" and "adherent" ash, is affected by the manner in which the coal-forming material was laid down and on the origin of the coal, and differs, also, in vitrain, clarain, durain, and fusain. The ash content of fusain is usually high, and, though fusain is usually present in small quantity, its great friability leads to its concentration in the fines and slurry. All cleaning operations aim at reducing the sulphur content of the coal, and the success attained will depend not so much on the total amount of sulphur present as on the relative proportions of organic sulphur, iron sulphides, and mineral sulphates. Our knowledge of the efficiency of the removal of phosphorus in different washing processes is meagre, but it appears that under the most favourable conditions the percentage removal is small. Salt can be only partially removed from most coals by washing, and it is important to avoid an accumulation of soluble salts in the washing water. The fusibility of the coal ash may be markedly influenced by washing, in so far as the relative amounts of iron, lime, magnesia, silica, and alumina are thereby altered. W. T. K. BRAUNHOLTZ.

Application of the Chance sand flotation process to washing bituminous coal. A. GREENWELL (Fuel, 1926, 5, 163-166).—A detailed description of an American plant in which 335 tons per hr.

of bituminous coal are washed in conical washers by the Chance sand-water process (cf. B., 1924, 699). The material treated is sized to pass through a screen with $4\frac{1}{2}$ in. sq. openings and over a $\frac{3}{8}$ in. screen, the washing of the smalls being considered unnecessary. The refuse is drawn off at the base of the cone, and clean coal, sand, and water overflow at the top. The sand is separated from both the coal and refuse by screening, and is returned to the washer. The ash in the washed coal is constant within about 0.75% and the sulphur within 0.2%.

W. T. K. BRAUNHOLTZ.

Rate of travel of fusion zones in coke ovens.

W. P. RYAN (Fuel, 1926, 5, 150—157).—Curves are drawn illustrating the changes in temperature at various points in an oven during carbonisation, the distribution of temperature across the oven at any moment, and the passage across the oven of two isotherms lying in the neighbourhood of the fusion point and of the point of maximum pressure. It is shown that if maximum pressure is not synchronous with fusion it follows shortly after, and that a given condition of temperature or pressure moves across the oven at a variable rate depending on the distance from the heating wall. The thermal conductivity of coal does not change in order of magnitude on fusion and conversion into semi-coke, but that of coke is much higher than that of coal. The heat of fusion and thermal decomposition is negligible in effect on the rate of heating of the coal. The rate of travel of the fusion zone is rapid nearest the heating walls, decreasing to a minimum as the fusion zone passes towards the centre of the oven, and finally increasing again as it comes near the centre, where the coal comes under the influence of heat transmitted from both sides.

W. T. K. BRAUNHOLTZ.

Thermal decomposition of cellulose and lignin

in presence of catalysts and hydrogen under pressure. A. R. BOWEN and A. W. NASH (Fuel, 1926, 5, 138—142; cf. J.S.C.I., 1925, 507 T).—When cellulose and lignin are gradually heated to about 450° with hydrogen under pressure (maximum up to about 240 atm.) in the presence of catalysts (e.g., alumina and nickel oxide) liquid and gaseous products are formed, but the oils obtained are not fully saturated, owing probably to the deposition of small quantities of coke on the catalyst. Cellulose semi-coke (prepared at 400°) can also be hydrogenated and yields an oil having a relatively low proportion of volatile constituents. The extent of conversion is greater with cellulose than with lignin or cellulose semi-coke, but phenols are formed to the greatest extent (4.8%) from lignin, which is also characterised by the formation of substances soluble in phenol. The volatile oil fractions have in each case an appreciable hydrocarbon content, the saturated portion of which, in the case of cellulose and cellulose semi-coke, has a paraffinic odour.

W. T. K. BRAUNHOLTZ.

Corrosion in gas meters. F. FIRTH (Gas J., 1926, 174, 41—45).—Experiments on the corrosion

of iron in electrolytes seemed to show that the rate and extent of corrosion are determined not so much by the concentration of dissolved salts as by the concentration of oxygen in contact with the wetted iron. To prevent corrosion it is recommended that the oxygen content of the gas be reduced by passing over easily corrodible iron turnings in purifier boxes, and that the gas be dried by refrigeration, by compression, or by the use of chemical desiccating agents.

S. PEXTON.

New forms of gas analysis apparatus. E. ORT (Gas u. Wasserf., 1926, 69, 289—295).—An apparatus for accurate gas analysis consists of a burette and compensator, absorption pipettes, and a quartz tube containing copper oxide or a capillary packed with fine platinum wire. The compensator and burette are connected by a manometer which is always adjusted before taking a reading; the confining liquid is mercury; the free spaces in the apparatus are filled with nitrogen. Carbon dioxide is determined by absorption in caustic alkali solution; unsaturated hydrocarbons in oleum (the oleum absorption pipette, it was found, is best filled with glass rods; the diminution in volume of the absorbent reduces the risk of error due to solution of the gas, etc.); oxygen in pyrogallol solution; and carbon monoxide in ammoniacal cuprous chloride. Hydrogen is burnt at 300° over copper oxide. Carbon monoxide may be estimated by this method alternatively; but in this case combustion should take place over platinum to avoid the absorption of the resulting carbon dioxide (especially if its partial pressure is high) by the copper and copper oxide. Allowance for volume changes due to the reduction of copper oxide to copper, and other intricacies in this combustion process are discussed. Methane is burnt at a bright red heat over copper oxide, or if ethane is present, over platinum, oxygen being admixed. Figures from actual analyses are given, and the method of calculating results is shown. For technical purposes a portable type of apparatus is described; it is somewhat modified and does not possess a compensator; in this form it is an elaboration of the Orsat.

R. A. A. TAYLOR.

Grosny petroleum. A. SACHANOV (Neftjanoe slancevoe Chozjajstvo, 1925, 9, 554—563; Chem. Zentr., 1926, I, 2269).—The oil from the newer Grosny oilfields contains 2.5—4.7% of paraffin wax, whilst that of the older may be divided into two types containing 2—2.5% and less than 0.4% of paraffin wax respectively. A typical oil of the former class had η^{20} 0.098 (C.G.S. units), d 0.838, f.p. + 11°, and contained 12% of resins. On distillation it gave 7.64% below 100°, 15.67% at 100—200°, 13.31% at 200—270°, and 62.75% of residue having η^{50} 3.6 (Engler), d , 0.898, f.p. 30°. Distillation of this residue with steam gave 25.4% (of the petroleum) of solar oil, 10.2% of spindle oil, 10.2% of machine oil, 12.2% of "goudron" oil, 4.5% of resin, and 0.9% of asphaltenes. An oil of the second type had η^{20} 0.075, d 0.851, f.p. —15° to —20°, and contained 15% of resins. It yielded on distillation 7.73%

below 100°, 20.06% at 100—200°, 12.54% at 200—270°, and 59% of residue having η^{50} 4.5, d 0.909, f.p. 19°, and giving on distillation with steam 23.6% of solar oil, 7.1% of spindle oil, 7.1% of machine oil, 13.9% of "goudron" oil, 6.5% of resin, and 1.5% of asphaltenes. An oil of the third type had η^{20} 0.140, d 0.879, f.p. below -20°, and contained 20% of resins. When distilled alone or with steam it gave 8.46% below 100°, 20.07% at 100—200°, 12.55% at 200—270°, 14.1% of solar oil, 9.4% of spindle oil, 9.4% of machine oil, 4.7% of cylinder oil, 11.2% of "goudron" oil, 0.2% of paraffin wax, 8% of resin, and 2% of asphaltenes. This type of paraffin-free oil is characterised by its high asphaltene content. A fourth type of product of the Grosny oilfields is natural gas, which has a high density and benzene content owing to the high temperature (up to 90°) of the majority of the boreholes. The gas comprises about 30—40% of condensable gases and 60—70% of methane, ethane, and propane. The benzene obtained from the natural gas has d 0.677, and 58% distils below 100°. W. T. K. BRAUNHOLTZ.

Abridged volume correction table for petroleum oils (U.S. Bur. Standards, Suppl. to Circ. 154, 8.10.25, 4 pp.).—The abridged table is for use in converting oil volumes at various temperatures to the volume at 60°F. (15.5°) when extreme accuracy is not required. For high accuracy the table in Circular 154 should be used. H. MOORE.

Synthesis of petroleum at atmospheric pressure from products of coal gasification. F. FISCHER and H. TROPSCH (Brennstoff-Chem., 1926, 7, 97—104; Ber., 1926, 59, 830—836, 923—925).—Petroleum products, from benzene to paraffin wax, can be synthesised at atmospheric pressure from gas mixtures containing carbon monoxide and hydrogen (e.g., water-gas) by passing the gas over a catalyst at moderate temperatures (up to about 270°). Finely-divided metals of the 8th group of the periodic system, prepared by reducing their oxides with pure hydrogen at 350°, may be used as catalysts, alone or in conjunction with metal oxides (e.g., oxides of chromium, zinc, beryllium, etc.), and with or without a carrier (e.g., pumice, asbestos). With iron alone the reaction is slow; cobalt is more active, but with nickel the main product is methane, which is also the sole product in all cases at higher temperatures. Increasing the basicity of the catalyst by addition of alkali carbonates or hydroxides increases the proportion of higher-boiling products. It is essential that the gases used should be free from sulphur compounds, which poison the catalyst, and it may be necessary from time to time to free the latter from clogging by heavy paraffins. As an example of the yields obtained, 100 g. of liquid and solid products were recovered by repeatedly circulating 1 cb. m. of water-gas over an iron-copper catalyst. The products included "gasol" (ethane, propane, butane, with only traces of olefines), benzene, "petroleum," and paraffin wax. The benzene, extracted from the gases by active charcoal, was water-white and remained so, and had d^{15} 0.6718, cal. val. (gross) 11,360 cal. per g.,

and spontaneous ignition temperature 335°. On distillation it gave 18.2% at 50°, 75.3% at 100°, 92.3% at 150°. The "petroleum" had d^{20} 0.7804, and yielded 4.4% at 180°, 14.4% at 200°, 49.2% at 250°, 70.4% at 300°, and 83.2% at 330°. The paraffin wax, recrystallised from acetone, was colourless and odourless, and had f.p. 61°. The catalyst is supposed to function by virtue of the intermediate formation of unstable metal carbides of high carbon content. W. T. K. BRAUNHOLTZ.

Action of ozone upon petroleum. I. Action of ozone on the saturated hydrocarbons of petroleum. G. KITA, R. ABE, and S. TADA (J. Soc. Chem. Ind. Japan, 1926, 29, 7—12).—By the action of ozonised oxygen containing about 5% of ozone on a fraction (b.p. 150—300°, mainly saturated hydrocarbons) of Kurokawa petroleum, the iodine and acid values, sp. gr., refractive index, and viscosity increased to an extent depending on the time of the treatment, while the rotatory power decreased. In the case of liquid paraffin (Jap. Pharm. IV.), the acid value, sp. gr., and viscosity increased slightly, while the rotatory power decreased and the iodine value and refractive index were not influenced by treatment with the ozonised oxygen. K. KASHIMA.

Methods of testing oils for transformers, switches, and turbines. G. BAUM (Z. angew. Chem., 1926, 39, 473—475).—A review of the most important methods for testing the suitability of an oil for use as an electrical insulator, pointing out the unsatisfactory results given by most methods and emphasising the need of further research to discover the reason why the insulating power of certain oils breaks down more quickly than that of others. Comparable results for the amount of tarry matter formed by oxidation of the oil are not obtained by any of the present methods—sodium peroxide, exposure to air at 120°, and bubbling air through the oil at 120°—and considerable difference of opinion exists as to the value of the method involving agitation with sulphuric acid. A. R. POWELL.

Syzran asphalts. A. PARSCHIN (Neftjanoe slancevoe Chozjajstvo, 1925, 9, 582—584; Chem. Zentr., 1926, I, 2270).—The bitumen is separated from the bituminous rock by boiling with acidulated water. A type of bituminous sandstone known as "Garj" yields no bitumen by this process and is regarded as a waste product, although it contains as much as 5.9—16.8% of bitumen that can be extracted with chloroform. The bitumen is not separated by boiling water owing to its high m.p. (96—147°). A sample having m.p. 107° contained 52.7% of asphaltenes (d 1.074), 29.0% of resins (d 1.069), and 16% of oils (d 0.910). The extraction of the bitumen with benzene or carbon disulphide could probably be applied commercially.

W. T. K. BRAUNHOLTZ.

Solubility of formolites. K. KOSTRIN (Neftjanoe slancevoe Chozjajstvo, 1925, 9, 579—581; Chem. Zentr., 1926, I, 2270—2271).—By shaking

one of the oils obtained as a by-product in the refining of machine oil with concentrated sulphuric acid and 40% formaldehyde solution, warming, diluting, and neutralising, a solid formolite was obtained. This was extracted successively with benzene, alcohol, carbon tetrachloride, chloroform, acetone, and ether. The quantity of formolite soluble in benzene and of that insoluble in all solvents increased with the b.p. of the original oil.

W. T. K. BRAUNHOLTZ.

Naphthenic acids derived from kerosene distillate of Californian petroleum. Y. TANAKA and S. NAGAI (*J. Soc. Chem. Ind. Japan*, 1926, 29, 58–63).—From the waste lye produced in the refining of kerosene distillate 0.19% (on the weight of kerosene) of crude naphthenic acids was obtained and these were purified by conversion into the methyl esters. About 73.5% of the esters distilled at 120–180°/8.9–9.0 mm. On saponification of the methyl esters a mixture of pure naphthenic acids was obtained, d_4^{20} 0.9448, n_D^{20} 1.4661, and neutralisation value 246.2. About 77% of the mixture distilled at 160–230°/8.9–9.0 mm. The main fractions were considerably lighter (d 0.95) than those (d 0.98) of the naphthenic acids obtained from a gas oil distillate (*B.*, 1926, 307). A white crystalline mass separated in the high-boiling fractions.

K. KASHIMA.

Micro-structure of coal. R. THIESSEN (*J. Soc. Arts*, 1926, 74, 535–557).

Occurrence of ulmic compounds in deteriorated fabrics and bearing of their formation on the origin of peat and coal. A. C. THAYSEN, W. E. BAKES, and H. J. BUNKER (*Fuel*, 1926, 5, 217–220).—See *B.*, 1926, 305.

Determination of olefinic, aromatic, and saturated hydrocarbons in petroleum and tar oil fractions. N. DANAILA and E. MELINESCU (*Bul. Chim., Soc. Româna Stiin.*, 1924, 27, 3–10).—See *B.*, 1925, 835.

Utilisation of aromatic hydrocarbons in petroleum fractions. I. Preparation of α -trinitrotoluene. N. DANAILA and N. POPA (*Bul. Chim., Soc. Româna Stiin.*, 1924, 27, 45–80).—See *B.*, 1925, 745.

See also A., May, 464, System ethyl alcohol-benzene-water (BARBAUDY). 467, Adsorption of ethylene and hydrogen by zinc oxide, iron oxide, nickel, and copper (LAZIER and ADKINS). 480, Combustion of well-dried carbon monoxide and oxygen mixtures (BONE and WESTON; BONE, FRASER, and NEWITT).

Silica brick from coke-oven walls. COLE.—See VIII.

PATENTS.

Coal washeries. A. ROBINSON, and SIMON-CARVES, LTD. (E.P. 249,977, 10.2.25).—The fine coal and slurry are independently separated from the coarse coal and treated for the removal of water.

The sieved fines are allowed to drain in bunkers and the water recovered can be utilised again in the washery. The slurry is jigged on a sieve. Both products can then be mixed with the coarse coal, from which the water easily drains when the slurry and fines are absent.

S. PEXTON.

Decomposition of peat. G. MEUNIER and C. DE BRISSON DE LAROCHE (F.P. 597,387, 24.4.25).—Colloidal peat is treated with alkalis to separate fibrous material and other fine constituents, and the latter are hydrolysed to recover the humus constituents.

L. A. COLES.

Process of modifying ignition temperature etc. of carbonaceous materials. I. H. DERBY and C. B. EDWARDS, Assrs. to P. C. REILLY (U.S.P. 1,576,179, 9.3.26. Appl., 11.8.23).—The ignition temperature of coke or similar substance is lowered some 200° and its rate of burning correspondingly raised by coating it with lead, lead oxide, etc., or with a thallium compound, copper oxide, barium peroxide, or generally with a compound of one of these metals which easily yields the oxide on heating or with the oxide itself. For example, 42,000 pts. of coal tar are mixed with 160 pts. of lead oxide and distilled. The coke obtained is as easily ignited as charcoal and has a lower ash content. The products described are of value as a covering for the molten lead used in the heat treatment of steel; owing to their freedom from ash and relatively high sp. gr. they are superior to charcoal, which is usually employed for this purpose.

T. S. WHEELER.

Coke ovens and batteries thereof. KOPPERS Co., Asses. of J. BECKER (E.P. 232,173, 24.12.24. Conv., 10.4.24).—The battery units have two pairs of heating walls with vertical flues and connected by a number of cross-over ducts at both the ram and quencher sides so that the gases flow in the two sides in separate paths without intermingling, the direction of flow in one pair of heating walls being the reverse of that in the other. Reversible regenerators are arranged in a row beneath the oven, the arrangement being such that the fuel gas regenerators are separated by air regenerators from the regenerators receiving waste gases from the downflow heating walls.

W. T. K. BRAUNHOLTZ.

Coking retort ovens. KOPPERS Co., Asses. of J. VAN ACKEREN (E.P. 246,500, 29.10.24. Conv., 4.1.24).—In a battery of regenerative vertical retort ovens, the long vertical flues are supplied with preheated air and fuel gases both at the top and middle. The heating flues are interconnected in pairs at the bottom by ducts which pass around the end of the retorts on the regenerator side. Half the combustion flues operate for inflow, whilst the other half operate for outflow. During the reversal periods some retorts are heated from both sides by down-flowing flame gases, but as the reversals are regular all retorts get substantially the same treatment. The regenerators are so grouped that those containing fuel gas are separated

from those containing waste gases by secondary air regenerators. S. PEXTON.

Horizontal coke oven. H. B. CARPENTER (U.S.P. 1,579,075, 30.3.26. Appl., 18.10.21).—The apparatus consists of retorts and flues arranged alternately and separated by common walls. The sidewalls of the lower halves of the retorts and of the flues are parallel. The sidewalls of the upper half of each retort slope slightly towards the central vertical plane of the retort. The sidewalls of the upper half of each flue slope away from the central vertical plane of the flue. The retorts are thus narrower at the top than at the bottom, while with the flues the reverse is the case. R. A. A. TAYLOR.

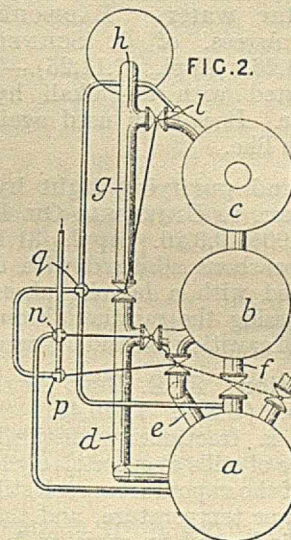
Stand-pipes for use in the manufacture of coal gas. J. G. W. ALDRIDGE, W. M. CARR, and T. J. ASHLEY (E.P. 248,557, 3.3.25).—The stand-pipes carrying the crude gases from a tier of retorts up to the foul main increase in cross-sectional area in the direction of flow of the gases, thereby reducing the gas velocity and increasing the efficiency of the cooling and scrubbing effected by a stream of liquid which is projected into the upper end of the stand-pipe, passes down counter-current to the gas, and is drawn off at a seal-box at the level of the stage floor. The connexions from the retort to the stand-pipe are provided with valves and means for deflecting the stream of liquor from the connexion. The upper portion of the stand-pipe may be further extended and enlarged above the retorts, and provided with baffles for improving the contact between the liquor and gas. A. B. MANNING.

Manufacture of hard coke. P. DVORKOVITZ (E.P. 249,901, 3.11.24).—Coal is subjected to a preliminary carbonisation at 500° prior to carbonising at 900°, with a view to increase considerably the output of the high-temperature carbonising plant. In addition, non-oxidising gases containing hydrogen are passed through the coke at 700–800°, whereby sulphur and nitrogen are eliminated from the coke. S. PEXTON.

Treatment of wood. W. A. AUDIBERT (F.P. 586,856, 4.10.24).—Wood is de-aerated in closed vessels, treated with acids or acid salts under pressure, and washed with water. The treatment results in the decomposition of the ligneous constituents, and the destruction of material capable of causing rot, and also the production or separation of resin, acetone, acetic acid, etc., which are recovered. L. A. COLES.

Manufacture of [water-]gas. HUMPHREYS AND GLASGOW, and J. C. STELFOX (E.P. 246,970, 16.12.24).—In a water-gas producer the fuel is coked by contact with water-gas and excess steam during the run with superheated steam, and by contact with water-gas obtained during a run with steam which has not passed through the recuperator, with or without part of the coal gas and water-gas mixed and superheated with fresh steam. The blow may be prolonged after heating the recuperators, and the resulting producer gas used to coke the fuel and

then conveyed to storage. The producer, *a*, is connected by pipes, *d*, *e*, *f*, with a carburettor, *b*, and pipes, *g*, *h*, lead to storage. The recuperator, *c*,



is connected to the pipe, *h*, through a valve, *l*, to enable water-gas to be re-circulated through the plant. Steam is supplied through valves, *n*, *p*, *q*, to the required parts of the plant, and the valves are coupled as shown by the full thick lines; the broken lines indicate interlocking means between the valves. S. PEXTON.

Producing a high-grade mixed gas. C. ASBECK (E.P. 249,772, 6.11.25).—The plant comprises a gas producer and an auxiliary coal distillation retort. The producer, which is an elongated water-gas generator, is fed with fresh coal which is distilled in the upper part by the sensible heat of the water-gas which streams through it during the "run." During the blow the waste gases are led off from the generator below the coal distillation zone and burnt in a chequered chamber which surrounds the auxiliary retort in which coal is carbonised and from which coke is discharged. The waste gases from the chequered flues are passed through a waste-heat boiler which is fed with water preheated by circulating through an annular space surrounding the lower part of the auxiliary retort, thereby cooling the coke. The distillation gases from the producer and the retort during the "run" are circulated through the hot chequered chamber surrounding the retort in order to crack the tarry vapours and completely fix them. S. PEXTON.

Recovery of carbon monoxide from industrial gases. A. A. L. J. DAMIENS (G.P. 425,418, 26.11.24. Conv., 12.4.24. Addn. to 422,621).—Sulphuric acid of *d* below 1.84 is used in the process described in the chief patent (cf. E.P. 230,106; B., 1925, 847), and temperatures up to or above 100°, according to the dilution of the acid, are employed, the gases being compressed during the absorption, if necessary. The cuprous compound and carbon monoxide are

recovered by heating the complex compound which is formed, with sulphuric acid of *d* below 1.84.

L. A. COLES.

Treating tar water. BADISCHE ANILIN- & SODA-FABR., Assees. of C. SCHNEIDER (U.S.P. 1,579,957, 6.4.26. Appl., 7.11.25).—The hot tar water is treated with an alkali hydroxide and exposed to air. It is then used again for cooling coal gas or the like.

C. IRWIN.

Blending and purifying light hydrocarbons. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,575,905, 9.3.26. Appl., 31.12.20).—Gasoline-like hydrocarbons admixed with uncondensable gas are blended with a heavier absorbing oil and purified by passing the mixture through a solution having a base of caustic soda and litharge. H. MOORE.

Oil distillation. J. E. BELL Assr. to SINCLAIR REFINING Co. (U.S.P. 1,576,401, 9.3.26. Appl., 7.10.21).—Oil is fractionally distilled by passing through a series of stills at successively higher temperatures. Part of the vapour from each still is condensed by oil at a lower temperature, and the oil in intermediate stills is heated by vapours from stills later in the series.

H. MOORE.

Heating system for oil stills. F. E. WELLMAN, Assr. to KANSAS CITY GASOLINE Co. (U.S.P. 1,576,563, 16.3.26. Appl., 17.12.21).—A vertical still is surrounded by an annular combustion space, the upper part of which communicates with the stack. Means are provided for producing localised combustion in the lower part of the space, and for constantly changing the point of application of the resulting flame to the still wall.

H. MOORE.

Continuous process for refining petroleum and the like. H. L. CARROLL (F.P. 598,295, 2.9.24).—Crude petroleum, or petroleum from which the lighter constituents have been removed, is circulated rapidly through a vessel in which it is heated to a temperature approximating to the boiling point of the highest-boiling fractions to be distilled off, after which it enters the lower end of a column in which the escaping vapours are subjected to fractional condensation.

L. A. COLES.

Breaking [petroleum] emulsions. L. G. GATES, Assr. to STANDARD OIL Co. (U.S.P. 1,573,389, 16.2.26. Appl., 14.5.25).—A petroleum emulsion of the water-in-oil type is brought into contact with cotton, iron, monel metal, or other suitable substances whereby the positively charged oil acquires a negative charge and the repulsion between the acquired charge and the similar charge on the water particles breaks the emulsion. The crude oil emulsion heated to about 70° is delivered to one side of a filter screen in which the filtering medium is cotton, and is withdrawn from the other side; preferably several filter units in parallel are used. To prevent clogging of the pores of the cotton the direction of flow of the oil is frequently reversed. The filtering medium is earthed to remove the positive charge which it acquires during the process. The rate of separation

of oil and water is increased if a cloth filter be fitted to the pipe discharging the mixture from the filter into the separating tank.

T. S. WHEELER.

Emulsified solid greases. M. J. HEITMANN (E.P. 232,259, 14.4.25. Conv., 10.4.24).—An emulsified solid grease, containing about 50% of water, particularly suitable for lubricating axles of trucks etc., as its consistency is maintained irrespective of temperature changes, is obtained by subjecting lubricating oil to a preliminary treatment by electric glow discharges (cf. E.P. 209,060) before adding the water. No soap or soap-forming substance is used.

H. M. LANGTON.

Making organic-acid soap [cutting oil] from mineral oils. E. M. HUGHES, Assr. to SUN OIL Co. (U.S.P., 1,577,723, 23.3.26. Appl., 2.7.18).—Lubricating oil stock from the distillation of crude petroleum is agitated with sulphuric acid and the sludge drawn off. The oil is then agitated with caustic soda. An emulsion consisting of soap, salt water, free alkali, and unsaponifiable mineral oil is removed, after which the soap is decomposed with a dilute solution of sulphuric acid. Water and salts are separated and the remaining product is saponified and mixed with a mineral oil. A cutting oil is thus formed which gives a milky emulsion when mixed with water.

R. B. CLARKE.

Separating [paraffin] wax from lubricating oil. E. PETTY, Assr. to DE LAVAL SEPARATOR Co. (U.S.P., 1,577,852, 23.3.26. Appl., 24.8.21).—Lubricating oil stock from crude petroleum is mixed with a light petroleum distillate and agitated with sulphuric acid. After removing the sludge, the oil is washed with water, neutralised by agitating with caustic soda, and again washed. Soap and alkaline water are removed in a centrifugal separator, after which the oil is clarified and decolorised by stirring with fuller's earth and filtering on a vacuum filter. Clarification is completed in a centrifugal clarifier. Wax is precipitated from the oil by chilling it, partly with brine and partly with cold oil. Oil and wax are then separated by centrifuging.

R. B. CLARKE.

Treating gilsonite. Gilsonite products. Gilsonitic product and its manufacture. (A, B) C. N. FORREST, and (c) C. N. FORREST, H. P. HAYDEN, and O. R. DOUTHETT, Assrs. to BARBER ASPHALT Co. (U.S.P. 1,573,764-5, 16.2.26, and 1,578,235, 23.3.26. Appl., [A] 18.9.19, [B] 19.9.22, [c] 28.8.19).—(A, B) Gilsonite is distilled to about 450°, care being taken as the temperature approaches 285° when an exothermal reaction occurs. The products consist of fuel gas (12.75%), consisting chiefly of methane, and carrying light oil (2%), and ammonia (0.25%), an oil distillate (55%), and coke (30%). The coke contains 5-6% of nitrogen, which is recovered as ammonia when the coke is gasified in a producer. The oil distillate is fractionated, preferably in a current of carbon dioxide, as described in U.S.P. 877,620 (cf. F.P. 379,521; B., 1907, 1233) and yields a light solvent oil, an

intermediate gas oil, and a heavy oil which gives a lubricating oil and wax. All these oils have both asphaltic and paraffinoid characteristics, contain a high proportion of nitrogen, and are highly unsaturated. (c) The middle oil is purified by treatment with sulphuric acid and is then sulphonated with mixtures of oleum and sulphuric acid containing increasing proportions of oleum. A thin clear oil with the properties of a highly refined petroleum oil remains. The acid sludges on dilution yield three layers, a semi-drying oil, a solution of water-soluble sulphonic acids, and dilute sulphuric acid. The sulphonic acid solution can be used for the hydrolysis of fats, and the salts of these acids have detergent properties.

T. S. WHEELER.

Briquetting fuels. R. KATTNER, Assr. to FRANKENSTEINER MAGNESITWERKE A.-G. (U.S.P. 1,562,564, 24.11.25. Appl., 19.9.25).—See E.P. 241,175; B., 1926, 4.

Gas producer with a revolving grate. W. BENNHOLD, Assr. to J. PINTSCH A.-G. (U.S.P. 1,581,159, 20.4.26. Appl., 18.8.20).—See E.P. 146,964; B., 1921, 803 A.

Producing hydrogen from water-gas. J. H. DE GRAER (U.S.P. 1,583,673, 4.5.26. Appl., 26.1.23).—See E.P. 212,943; B., 1925, 356.

Manufacturing active carbon. BADISCHE ANILIN- & SODA-FABR., Assees. of F. WINKLER (U.S.P. 1,582,718, 27.4.26. Appl., 29.9.23).—See E.P. 216,761; B., 1924, 664.

Oil still. J. PRIMROSE, Assr. to POWER SPECIALTY Co. (U.S.P. 1,566,921, 22.12.25. Appl., 19.6.20).—See E.P. 176,101; B., 1922, 284 A.

Fractionation of hydrocarbon oils and like liquids. R. H. CROZIER (U.S.P. 1,583,340, 4.5.26. Appl., 7.7.25).—See E.P. 240,502; B., 1925, 951.

[Bituminous] emulsion. L. KIRSCHBRAUN (Reissue 16,328, 20.4.26, of U.S.P. 1,479,042, 1.1.24. Appl., 30.12.25).—See B., 1924, 210.

Bituminous emulsion. G. S. HAY, Assr. to ASPHALT COLD MIX (1925), LTD. (U.S.P. 1,582,467, 27.4.26. Appl., 18.8.25).—See E.P. 243,398; B., 1926, 120.

Apparatus for discharging the solid products from retorts and the like. W. R. DEGENHARDT (E.P. 250,716, 23.2.25).

See also Class III., Purifying petroleum oils (E.P. 226,188); Paraffin wax from low-temperature tar (F.P. 597,919). Page 486, Making hydrocarbons and cyanides (U.S.P. 1,567,241). 487, Hydrogen (E.P. 249,925). 488, Sulphur from gases (G.P. 425,664).

III.—TAR AND TAR PRODUCTS.

PATENTS.

Purifying commercial benzene [and petroleum oils]. SCHLESISCHES KOHLENFORSCHUNGS-INST.

DER KAISER-WILHELM-GES. (E.P. 226,188, 27.11.24. Conv., 11.12.23).—Crude benzol or other distillates of coal, lignite, or petroleum are purified by heating to 100—250° under a pressure of 100 atm. or to 250—500° at atmospheric pressure. Unsaturated constituents unstable to light are polymerised and carbon disulphide and hydrogen cyanide are destroyed, but thiophen is not affected. The residues of the subsequent distillation of the treated oil are lightened in colour by the presence of copper, tin, or lead, *e.g.*, as lining to the apparatus used. (Reference is directed in pursuance of Sect. 7 (4) of the Patents and Designs Acts, 1907 and 1919, to E.P. 223,066 and 123,806.) C. IRWIN.

Recovery of paraffin wax from low-temperature tar. A. MAILHE (F.P. 597,919, 13.8.24).—The tar is treated cold with methyl alcohol, and undissolved wax is filtered off, or it is treated with the alcohol at 40—60°, allowed to cool until no more wax separates, and is then filtered. The solution is distilled to recover the methyl alcohol.

L. A. COLES.

Extracting benzols from heavy oils. E. A. BARBET (U.S.P. 1,561,899, 17.11.25. Appl., 21.3.21).—See F.P. 473,082; B., 1915, 485.

Producing viscous liquids [from tar oils]. L. LILIENFELD (U.S.P. 1,563,203, 24.11.25. Appl., 10.1.21).—See E.P. 149,317; B., 1921, 840 A.

IV.—DYESTUFFS AND INTERMEDIATES.

Colour measurement and standardisation. L. C. MARTIN (J. Oil and Colour Chem. Assoc., 1926, 9, 92—105).

Fusion pot. BABASINIAN and BILLINGER.—See I.

PATENTS.

Manufacture [purification] of reddish-yellow vat dyestuffs [flavanthron]. BRIT. DYESTUFFS CORP., J. BADDILEY, and A. SHEPHERDSON (E.P. 249,993, 5.3.25).—Crude flavanthrone is purified by fractional precipitation from its solution in concentrated sulphuric acid by dilution with water, or by treatment with sulphuric acid of such concentration that the impurities dissolve but not the flavanthrone. For example, 15 pts. of ice are added to a solution of 10 pts. of crude flavanthrone (Indanthrene Yellow G) in 100 pts. of 100% sulphuric acid at 30—40°, and, after stirring for 2 hrs., the precipitated dye is filtered off and washed successively with 80—90% sulphuric acid and water, yielding a product giving shades as pure as those obtained from Indanthrene Yellow R. A dye yielding dull shades is precipitated on further dilution of the sulphuric acid after filtration. L. A. COLES.

[Vat] dyestuffs. SOC. OF CHEM. IND. IN BASLE, Assees. of J. FRÖLICH (U.S.P. 1,575,678—9, 9.3.26. Appl., [A] 21.10.24, [B] 7.11.24).—2:5-Diaryldio-*p*-benzoquinones are treated with sulphur dichloride, if necessary in presence of a diluent such as nitrobenzene, and with addition of an alkaline compound

and a condensing agent, such as sulphuric acid, to yield vat dyestuffs which may contain both sulphur and halogen and which give fast shades on animal fibres, *e.g.*, wool, darker shades being obtained at higher temperatures. Particularly suitable are 2:5-dianilidobenzoquinones not substituted or only monosubstituted in the 3- or 6-position in the quinone nucleus, for such compounds react at ordinary temperatures, whereas 3:6-disubstituted compounds require a higher temperature. The 2:5-dianilido-compounds are obtained from two aromatic amines which may be the same or different and which may be monosubstituted at the nitrogen atom and mono- or poly-substituted in the nucleus by halogen, or alkoxy-, alkyl-, aryloxy-, nitro-, amino-groups, etc. The shades obtained vary from yellow-brown to red-brown, olive green, green-blue, and grey. For example, 14.5 pts. of 2:5-dianilidobenzoquinone are treated for a long period in nitrobenzene at 5° with 11.3 pts. of sulphur dichloride to give a dyestuff yielding blackish-brown shades on wool in an alkaline hyposulphite vat. If 17 pts. of sulphur dichloride are used the product gives full brown shades when the condensation is carried out at 5°; the products obtained at 55°, 105°, and at the boiling point of nitrobenzene, give dark brown, red-brown, and grey to black shades respectively. 90 pts. of 3:6-dichloro-2:5-dianilidobenzoquinone treated with 78 pts. of sulphur dichloride in nitrobenzene at its boiling point yield a product giving blue-grey shades. (B) If in the processes described above sulphur dichloride is replaced by sulphur monochloride, the temperature being maintained below 100°, products are obtained giving in an alkaline hyposulphite vat fast yellow-brown to red-brown, olive green, green, and grey tints on wool. For example, 7.2 pts. of 2:5-dianilidobenzoquinone are treated for a long period in nitrobenzene with 11 pts. of sulphur monochloride at 2–6° to yield a product giving fast brown shades on wool; if the reaction is carried out in presence of sodium acetate the shades are olive-brown, whilst with magnesium oxide a product giving dark brown shades is obtained. 14.5 pts. of 2:5-dianilidobenzoquinone and 34 pts. of sulphur monochloride heated in sulphuric acid at 70° yield products giving yellow-green to green shades according to the time of reaction. 40 pts. of 6-chloro-2:5-dianilidobenzoquinone treated at 5° in nitrobenzene with 50.5 pts. of sulphur monochloride yield a product which gives fast yellow-brown shades. T. S. WHEELER.

Manufacture of oxidation products of aromatic hydrocarbons [phthalic acid, anthraquinone] by air oxidation. A. G. GREEN (E.P. 249,973, 6.2.25).—In the oxidation, *e.g.*, of naphthalene to phthalic acid or of anthracene to anthraquinone, the catalyst, consisting of pumice stone coated with vanadium pentoxide, is disposed upon perforated plates in a reaction chamber, and the temperature during the reaction is regulated by introducing an atomised spray of water into the spaces between the plates. L. A. COLES.

Manufacture of vat-dyestuffs. O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 245,623, 16.2.25).—See F.P. 593,117; B., 1926, 266.

Indigoid dyestuffs. SOC. OF CHEM. IND. IN BASLE, Assecs. of B. MAYER and J. WÜRLER (U.S.P. 1,561,560, 17.11.25. Appl., 4.1.24).—See E.P. 210,413; B., 1925, 66.

Manufacture of vat [dibenzanthrone] dyestuffs. BADISCHE ANILIN- & SODA-FABR., Assecs. of H. NERESHEIMER (U.S.P. 1,562,468, 24.11.25. Appl., 11.11.24).—See E.P. 212,145; B., 1924, 369.

Black [dibenzanthrone] vat dyestuff composition. BADISCHE ANILIN- & SODA-FABR., Assecs. of R. JUST, K. WILKE, and P. NAWIASKY (U.S.P. 1,581,638, 20.4.26. Appl., 26.1.25).—See E.P. 241,437; B., 1925, 983.

Acid azo dyestuffs for wool containing chromium. SOC. OF CHEM. IND. IN BASLE, Assecs. of H. FRITZSCHE, H. GÜBLER, and F. STRAUB (U.S.P. 1,581,572, 20.4.26. Appl., 15.10.23).—See E.P. 210,890; B., 1924, 328.

Azo dyestuffs. SOC. OF CHEM. IND. IN BASLE, Assecs. of G. DE MONTMOLLIN and G. BONHÔTE (U.S.P. 1,582,029, 27.4.26. Appl., 23.3.25).—See E.P. 232,251; B., 1925, 910.

Hydrogenation of naphthalene. G. SCHROETER (U.S.P. 1,582,310, 27.4.26. Appl., 29.8.21).—See G.P. 324,861; B., 1921, 253 A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chlorination of wool. S. R. and E. R. TROTMAN (J.S.C.I., 1926, 45, 111–115 τ).—The authors have shown (J.S.C.I., 1922, 219 τ) that wool is damaged more quickly by chlorine than by hypochlorous acid, and that the structural damage caused during chlorination is due to the presence of free chlorine in the chlorinating liquors. The destruction of the epithelial scales of wool, while diminishing the felting power, does not produce unshrinkability. There is no relation between damage and shrinkage, the former being merely a secondary effect. It is impossible to produce absolute unshrinkability by means of chlorine alone. Felting and shrinking appear to be distinct phenomena depending upon the different proteins present in the epithelium and cortex respectively (cf. J.S.C.I., 1926, 20 τ). The action of chlorine on wool proceeds by both adsorption and chemical combination. When chlorine is used as hypochlorous acid, neither damage nor loss of weight is produced until over 4% of chlorine on the weight of the wool has been absorbed (cf. E.P. 239,360; B., 1925, 844). In the case of chlorine, serious damage is apparent, even when small percentages are employed and the rate of adsorption is affected by the presence of acid. It is possible that hypochlorous acid may act by liberation of nascent oxygen. Other oxidising agents investigated reduced felting, proportionately to the number of epithelial scales destroyed, but no marked decrease

in shrinkage was observed. The action of hypochlorous acid is fundamentally different from that of chlorine.

Influence of atmospheric humidity on the absorption of moisture by textile fibres. J. OBERMILLER and M. GOERTZ (*Textilber.*, 1926, 7, 71—76).—The moisture absorption of various types of fat-extracted cotton, wool, natural (not fat-extracted) and artificial cellulose silks was determined after prolonged exposure at 20° to air, the relative humidity of which was successively maintained (within 2% limit of error) at 2.5%, 35%, 55%, 75%, 92%, and 97% by contact with potassium sulphate, crystalline sodium carbonate, potassium chloride, calcium nitrate, crystalline calcium chloride, and anhydrous calcium chloride respectively. Previous treatment considerably influences the absorption of moisture by all textile materials; *e.g.*, raw silk before drying and after drying for 72 and 177 hrs. at 75—100° absorbed 15%, 13.02%, and 12.33% of moisture when exposed afterwards at 20° to air of 75% relative humidity. Previous drying at a high temperature decreases the subsequent rate of absorption of moisture. The average moisture contents of cotton (American and Indian), wool (pure and chrome-mordanted), raw silk, degummed silk, and artificial (cuprammonium and viscose) silks exposed in air of 2.5%, 35%, 55%, 75%, 92%, and 97% relative humidity, and at 20° are 1.3, 5.5, 8.0, 11.0, 16.5, and 22.0%; 1.8, 11.0, 15.0, 18.5, 25.0, and 29.0%; 2.0, 8.0, 11.5, 15.5, 23.0, and 32.0%; 1.8, 7.3, 10.0, 13.5, 21.0, and 29.0%; 2.5, 8.5, 12.5, 17.5, 28.0, and 38.0% respectively. The higher moisture absorption of raw silk indicates that silk gum (sericin) is more hygroscopic than pure silk (fibroin). Indian cotton and viscose silk have slightly greater affinities for moisture than American cotton and cuprammonium silk respectively. The moisture regains of cotton, wool, raw silk, degummed silk, and artificial silks exposed at 20° in air of 65% relative humidity are 9.5%, 16.5—17.0%, 13.5%, 11.5—12.0%, and 15.0% respectively, as compared with the international standard regains of 8.5% (cotton), 18.25% (wool), and 11.0% (natural and artificial silks). The maximum moisture contents of cotton, wool, raw silk, degummed silk, and artificial silk exposed at 20° in air of 100% relative humidity are greater than 26%, 32%, 35%, 35%, and 40% respectively, the results of Scheurer (*cf. B.*, 1921, 359 A) being thereby confirmed. The absorption of moisture by textile materials is considerably decreased by rise of temperature, but is not appreciably affected by changes of barometric pressure. Dry cotton, wool, raw and degummed silk, cuprammonium and viscose silks suffer a considerable rise in temperature during subsequent absorption of moisture. A. J. HALL.

Relationship between the [physical] structure of artificial silks and their resistance to attack by enzymes. P. KARRER (*Textilber.*, 1926, 7, 23—24; *cf. B.*, 1925, 441, 585).—Scoured cotton, the same cotton mercerised, pure filter paper, cuprammonium silk of 120 denier, and cellulose regenerated from cellulose triacetate suffer 2.1%, 10.2%, 7.1%,

30.8%, and 70.0% hydrolysis respectively, when exposed under similar conditions to the action of an enzyme extracted from the intestines of vineyard snails. Cuprammonium silk and other regenerated celluloses may be quantitatively converted into dextrose by means of snail enzymes, but raw cotton even by successive treatments with such enzymes cannot be hydrolysed more than 25%. Raw cotton apparently consists of two types of cellulose distinguished by their resistance to attack by enzymes. It is suggested that something other than structural chemical change is responsible for the increased susceptibility to attack by enzymes of regenerated or alkali-treated celluloses. "Milky" patches sometimes found in commercial viscose silk materials contain fibres which have abnormal increased susceptibility to attack by snail enzymes, but which have the same microscopic characters, the same moisture content and swelling properties as normal viscose fibres, and yield similar X-ray spectrograms. Differences in the behaviour of various types of cellulose towards snail enzymes correspond to differences in their dyeing and absorptive properties. A. J. HALL.

Thorne bleaching process [for cellulose]. J. FUNCKE (*Papier-Fabr.*, 1926, 24, 217—220).—Cellulose pulp is concentrated to 7—9% in a drying cylinder, and then to 35% in a screw press. The material emerging from the press passes into a mixer, where the requisite amounts of bleach liquor and water are added, the volume and temperature of the water being adjusted so that the mixture contains 20% of cellulose at 20°. This then passes into a bleaching tower provided with an air-blast, and at the exit at the bottom it is diluted with water to a concentration of 1%, and so washed. Bleaching may be completed either by a repetition of this process or in a hollander. This continuous process, working at a high cellulose concentration, effects economies in chlorine (30—40%), steam (50%), and time (30%), and the product is stronger, has a higher cellulose content, and does not turn yellow on keeping. A. GEAKE.

Researches on viscose. III. Preparation of alkali-cellulose. G. KITA, R. TOMISHISA, and H. ICHIKAWA (*Cellulose Ind.*, Tokyo, 1926, 2, 67—79; *cf. B.*, 1926, 45).—When the alkali-cellulose was xanthated immediately after steeping and pressing, the optimum results were obtained with a period of immersion in the mercerising liquor of 24 hrs. at 8°, the proportion of combined alkali and the viscosity of the viscose being then at a maximum. The temperature and time of ageing of the alkali-cellulose have little influence on the proportion of combined alkali, but a very strong influence on the viscosity of the viscose even when the ageing takes place in an atmosphere of hydrogen. Viscose prepared from alkali-cellulose aged at a relatively high temperature (20°) undergoes only small changes in viscosity during ripening. The proportion of combined alkali in the viscose tends to be lower the higher the concentration of sodium hydroxide in the steeping liquor. An equilibrium tends to be established between the sodium hydroxide in the alkali-

cellulose and the carbon disulphide with which it is brought in contact, so that with an excessive quantity of alkali a larger proportion of the carbon disulphide reacts with the soda and less alkali is combined as xanthate unless sufficient carbon disulphide is present to cover both requirements. The maximum proportion of combined alkali, up to 0.5 mol. per mol. of cellulose, is attained if the reaction is performed at 0°, or if alkali-cellulose containing a high concentration of sodium hydroxide is treated with a large excess of carbon disulphide. A low temperature of xanthation together with a low temperature of ripening favours the maximum combination of alkali in the form of xanthate and its preservation during a prolonged period.

J. F. BRIGGS.

Laboratory preparation of viscose. E. H. MORSE (Ind. Eng. Chem., 1926, 18, 398—400).—To prepare on the laboratory scale viscose grades comparable with commercial grades, the commercial procedure must be modified. The alkali concentration for the first stage of treatment should be 20—21% at room temperature. Pressing may be carried out in a letter-press and crumbing by means of any sharp pointed instrument. The fluffy mass of crumbs must be kept for about 24 hrs. at a temperature between 18° and 23°, after which it is mixed in a revolving container with a quantity of carbon disulphide equal to rather more than a third of the weight of α -cellulose in the original pulp; the disulphide is added in four portions at room temperature. Ripening proceeds satisfactorily at room temperature. For relatively thick films 2 drops of viscose should dissolve clear in 10 c.c. of 2.5—3% of magnesium-free sodium chloride solution, but not in a stronger solution. Filtering through several layers of fine linen, or cotton wool between fine bleached muslin, should not be carried out until the solution has been kept for at least 24 hrs., and two filtrations are necessary if the solution is to be used to spin fine filaments. For general use a viscosity such that a steel ball 3 mm. diameter will fall 100 mm. through the solution in 15 sec. is satisfactory, and may be altered by addition of small amounts of clear 15% caustic soda solution.

D. G. HEWER.

Apparent viscosity of solutions of nitro-cotton in various solvents. J. W. McBAIN, C. E. HARVEY, and L. E. SMITH (J. Physical Chem., 1926, 30, 312—352; cf. Baker., J.C.S., 1913, 103, 1653; Piest, B., 1911, 835; Mardles, J.S.C.I., 1923, 42, 129, 211 r).—The apparent viscosity of solutions of nitro-cotton depends on the rate of shear and hence is not a true viscosity. It is so great that it must be ascribed to interference between ramifying aggregates of colloidal particles in the solution. No accepted formula represents the dependence of viscosity on concentration, the more concentrated solutions being less than proportionately viscous (cf. Hatschek, Biochem. J., 1916, 10, 325; Baker, *loc. cit.*; Mardles, J.C.S., 1923, 123, 1951). Solutions which have aged for considerable periods show viscosity more nearly proportional to concentration. The temperature coefficient is very large. The period of drying the

cotton at 60° affects the viscosity of solutions made from it, the most viscous resulting from material which has been heated for less than 24 hrs.; when the heating is extended to weeks or months the viscosity is progressively less. Vigorous shaking of the solutions diminishes the viscosity at 55°, presumably by breaking down the residual structure derived from the original fibres. The greatest effect occurs at the beginning when the largest aggregates are being disintegrated, whilst at a later period the effect becomes negligible. The higher the temperature of preparation the lower is the viscosity; the difference between solutions prepared at 55° and 90° showed no change after several months.

The behaviour of all known mixtures of solvent is tabulated. In the majority of cases a mixture in suitable proportions is a better solvent than the constituents of the mixture alone, and the solvent power of a given substance is largely dependent on adventitious impurities; generally speaking, the purer the solvent the less its efficiency as such. Data for fifteen solvents show that at 55° the viscosity of one definite sample of nitro-cotton in these solvents is inversely proportional to their solvent power (cf. Duclaux and Wollman, B., 1920, 528 A); low relative viscosity is a general measure of solvent power, the best solvents giving the least viscous solutions; and to a limited extent, increase in mol. wt. of comparable compounds lowers solvent power. The formanilides appear to be a better group of solvents than the urethanes or the pure esters. Viscosity in any solvent diminishes on keeping at 55° or at 90°, and, with the possible exception of benzylformanilide, is approximately proportional to the logarithm of the time. The changes in viscosity are very large, but they are in accord with the hypothesis that the best solvents are those which most quickly and effectively resolve the colloidal aggregates into the smallest particles. The higher the temperature the quicker and the greater the amount of degradation, but no definite end value is reached. Nitration analyses show that the ageing cannot be due to the denitration of the nitro-cotton by the solvent. Nitro-cotton recovered by precipitation with water or by evaporation from solution in acetone is much more difficult to dissolve than the original nitro-cotton, and the resulting viscosity may be affected and even increased. Anhydrous alcohol is a complete solvent for propulsive soluble nitro-cotton at the temperature of a carbon dioxide-ether mixture, whilst, at the ordinary temperature, the sol sets to a stable jelly, which shows the reverse change on cooling even after a period of 18 months. Syneresis is also shown to a marked degree. The imperfect solution obtained with alcohol at 130° is due to impurities resulting from denitration and decomposition. A theory of the behaviour of solvents towards nitro-cotton, which should be applicable throughout colloid chemistry, is advanced.

L. S. THEOBALD.

See also A., May, 468, Formation of an alkali-cellulose compound in an aqueous alcoholic medium (KATZ). 502, Depolymerisation of cellulose (HEUSER and HIEMER). 547, Incrustations

of flax (EHRlich and SCHUBERT). 548, Incrustive substances of plants (HEUSER); Leaf-tissue fluids of Egyptian cottons (HARRIS and others).

PATENTS.

Reducing the inflammability of nitrocellulose threads. J. R. LAVAUD (F.P. 600,852, 17.7.25. Conv., 23.4.25).—The material is successively treated with 10% acetic acid or 5% formic acid for 30–40 min., centrifuged, treated with a 10% solution of crystalline sodium sulphide and crystalline magnesium sulphate for 2½–3 hrs. at 25–30°, steeped in 5% sodium hyposulphite solution, dipped in a 10% solution of crystalline aluminium chloride, and treated for 30 min. in a bath containing 8 pts. of ammonium sulphate, 2½ pts. of commercial ammonium carbonate, 3 pts. of boric acid, and 1½ pts. of borax, after which it is rinsed and dried.

L. A. COLES.

Producing acid liquor in sulphite pulp processes. J. D. JENSSEN, Assr. to G. D. JENSSEN Co. (U.S.P. 1,571,271, 2.2.26. Appl., 24.2.25).—The plant comprises a primary (weak liquor) tower for the preliminary step, a secondary (strong liquor) tower for further treatment, and a mixer for heating the liquor in its passage from the primary to the secondary tower.

J. F. BRIGGS.

Process of hydration in paper-making, using friction. J. A. DE CEW (U.S.P. 1,578,405, 30.3.26. Appl., 29.1.25).—The cellulose is beaten and “jordaned” (refined) in water containing cold dilute caustic alkali and the fibres are then sized by a special series of operations in which the alkalinity is neutralised.

J. F. BRIGGS.

Acetylation of cellulose. SOC. CHIM. DES USINES DU RHÔNE (E.P. 231,837, 24.1.25. Conv., 2.4.24).—See U.S.P. 1,566,398; B., 1926, 152.

Plastic mass [from cellulose ethers]. L. LILIENFELD (U.S.P. 1,563,204, 24.11.25. Appl., 11.1.21).—See E.P. 149,319; B., 1922, 95 A.

Manufacture of artificial silk and like materials from viscose solutions. W. P. DREAPER (U.S.P. 1,583,534, 4.5.26. Appl., 18.10.23).—See E.P. 215,028; B., 1924, 592.

Preparation of threads from cellulose acetate. FARBENFABR. VORM. F. BAYER & Co., Assces. of E. HUBERT, O. LEUCHS, and L. LOCK (U.S.P. 1,583,717, 4.5.26. Appl., 14.7.24).—See E.P. 219,333; B., 1925, 843.

Asbestos paper etc. N. SULZBERGER (U.S.P. 1,581,618–9, 20.4.26. Appl., 30.4 and 18.8.21).—See E.P. 186,409; B., 1922, 894 A.

Treatment of wood (F.P. 586,856).—See II.

Separating alkali hydroxides from solutions (E.P. 246,355).—See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching artificial silk with “Aktivin” [sodium *p*-toluenesulphochloramide]. R. FARBELMANN (Textilber., 1926, 7, 47–48).—Aktivin is

a satisfactory substitute for hypochlorites and the like in the bleaching of viscose silk. Raw silk is bleached to a uniform pale cream shade without deterioration by treatment with a 3–4% solution of Aktivin at 60–70°; the resulting loss of activity of the Aktivin is strictly proportional to the amount of bleaching effected, and the residual liquor may be used again after titration with potassium iodide and sodium thiosulphate and addition of a suitable amount of Aktivin. White is obtained by bleaching with a 0.3% solution of Aktivin containing 0.5% of 50% acetic or formic acid. Under these conditions the bleaching solution is slightly cloudy owing to the separation of *p*-toluenesulphochloramide, but no deterioration of the bleached silk occurs even when high temperatures are used. Viscose silk materials bleached by means of Aktivin have a more uniform affinity for dyes than those bleached by other methods.

A. J. HALL.

Determining the degree of mercerisation of cotton fabric. R. HALLER (Textilber., 1926, 7, 65–66).—Measurement of the amounts of white, black, and pure colour tone in dyed mercerised cotton fabric by means of the Ostwald method is used for the determination of the degree of mercerisation of the dyed fabric, the method being rapid but somewhat less accurate than the titanous chloride method of Knecht (B., 1905, 154). Examination of the colour contents of cotton fabrics mercerised with caustic soda of 2–30° B. (*d* 1.014–1.261) and afterwards dyed with Diamine Blue 3R, Benzo Azurine, and Congo Red showed that the black and white contents of the resulting shades remained constant from 2 to 12° B. (*d* 1.014–1.091), then distinctly increased and decreased respectively from 12° to 26° B. (*d* 1.091–1.221), and then remained constant; simultaneously the content of pure colour decreased similarly but less regularly. It is concluded that the chief mercerisation effects are produced by caustic soda of 12° to 26° B., although Knecht (B., 1908, 400) found that mercerisation distinctly commences with caustic soda of 7° B. (*d* 1.052). White cotton suffers a slight darkening during mercerisation.

A. J. HALL.

Physics and chemistry of dyeing processes.

II. Dyeing of non-denitrated nitrocellulose silk. K. H. MEYER, C. SCHUSTER, and W. BÜLOW (Textilber., 1926, 7, 29–31; cf. B., 1926, 316).—Nitrocellulose silk absorbs *o*-nitroaniline and benzamide from aqueous solutions in accordance with Henry's law, subject to correction in the case of benzamide for the molecular association which it suffers in concentrated aqueous solution. The solubilities of *o*-nitroaniline and benzamide in nitrocellulose silk are 250 and 30 times greater respectively than their corresponding solubilities in water. One gram of nitrocellulose silk is capable of absorbing (dissolving) a maximum of 1.25 g. of *o*-nitroaniline but absorbs only 0.9 g. when immersed for 1½ months in an aqueous solution maintained saturated with *o*-nitroaniline. Nitrocellulose silk behaves similarly to cellulose acetate silk in respect

of its absorption of *o*-nitroaniline from aqueous solution and its affinity for feebly basic and non-sulphonated dyes. Cotton cellulose nitrated so that it contains 12% N behaves similarly to nitro-cellulose silk.

A. J. HALL.

[Dyeing] cellulose acetate silk effects [in union fabrics]. G. RUDOLPH (Kunstseide, 1926, 8, 13—15).—Blue, red, and white effects in cotton-wool-cellulose acetate silk union fabric are obtained by dyeing the wool with a (blue) acid dye from an acid bath, the cotton with a (red) cotton dye at 50° in the presence of 3% of Katanol W (for retarding absorption of the cotton dye by the wool), and leaving the cellulose acetate silk white. Three-colour effects on similar fabric are obtained by first dyeing the cellulose acetate silk with Cellit Fast Yellow 2GN and developing a red shade by the usual processes of diazotisation and coupling (the wool being thereby stained yellow and the cotton not affected), and afterwards dyeing the wool with a (blue) acid dye, the fabric being then thoroughly washed and the cotton dyed green or olive with a direct dye in the presence of Katanol W. Four colours are obtained on cotton-wool-natural and cellulose acetate silk union fabric by first dyeing the cellulose acetate silk red as described above, then dyeing the wool and silk together with wool dyes having different affinities for wool and silk under different conditions, *e.g.*, Patent Blue and Tartrazine, *e.g.*, by dyeing for $\frac{1}{2}$ – $\frac{3}{4}$ hr. at a low temperature (the silk thereby becomes dyed to a deep peacock blue) and afterwards adding sulphuric acid to the dye liquor and heating it to about 100° (the wool thereby becomes dyed Russian Green); subsequently the cotton is dyed with a violet direct dye in the presence of Katanol W at 50°. Three colours are obtained on cotton-viscose and cellulose acetate silk or cotton-mercerised cotton-cellulose acetate silk fabrics by previously dyeing the cotton (black) when in the form of yarn and afterwards dyeing the cellulose acetate silk and mercerised cotton or viscose silk in different colours by the usual methods.

A. J. HALL.

[Use of] Katanol W [in dyeing union fabrics]. G. RUDOLPH (Dtsch. Färber - Ztg., 1925, 107, Textilber., 1926, 7, 88).—The presence of Katanol W (sulphurised phenol) in dye-baths used for dyeing half-silk and half-wool materials considerably retards absorption by the animal fibres of direct cotton dyes, the dyeing of the cotton being unaffected. This restraining influence of Katanol W is used to facilitate the production of solid shades on half-wool fabrics by the neutral single-bath process of dyeing; the wool is dyed to shade first at a high temperature, the dye-bath is then cooled to 75–80°, 3% of Katanol W added, and the cotton allowed to absorb colour until the desired shade is obtained, there being no simultaneous change of shade of the wool. Katanol W is not added at the commencement of the dyeing process since it also retards the absorption by wool of certain acid dyes which have an affinity for wool in a neutral bath. Cotton in half wool fabric containing wool dyed with an acid dye is

preferably dyed with substantive dyes at 75–80° in the presence of Katanol W since the dyed cotton is faster to rubbing, the wool is less stained and deteriorated in handle, and the dye-bath more exhausted than in the usual method of dyeing at 30° in the presence of sodium carbonate. Katanol W may be of assistance in the dyeing of half silk materials, but natural silk better resists direct dyes when pre-mordanted for 1 hr. at 80–90° with 8–10% of Katanol W and 3–4% of formic acid.

A. J. HALL.

[Dyeing] Naphthol AS colours on artificial silks. H. LINT (Textilber., 1926, 7, 46).—A satisfactory range of colours very fast to all influences are obtained on artificial silks by means of Naphthol AS dyes. In dyeing, the silk is treated for 20–30 min. at 25–30° with a solution of a suitable Naphthol AS compound, centrifuged without previous rinsing, and then treated for 15–20 min. with a cold solution containing a diazotised amine. The dyed silk is thoroughly washed, soaped for 20–30 min. at 60–80° in a solution containing 4–5 g. of soap per litre, again thoroughly washed, and then dried after brightening the shade by treatment in dilute acetic or formic acid. Naphthol AS colours dyed on crude viscose silk are not changed in shade by the usual subsequent process of desulphurisation.

A. J. HALL.

Use of Aktivin for finishing printed fabrics and window-curtain materials. R. FEIBELMANN (Textilber., 1926, 7, 144–146).—Neutral, transparent, homogeneous, highly glutinous starch pastes containing no dextrin, maltose, glucose, or other sugar substances and suitable for finishing textile fabrics are prepared by boiling aqueous suspensions of starch (maize, rice, wheat, potato, etc.) containing about 1% (calc. on the weight of starch) of Aktivin (sodium *p*-toluenesulphochloramide); the viscosity of the resulting pastes is lowered by prolonged boiling, but no formation of reducing sugars occurs. This method of preparing finishing pastes is superior to processes now used in which starch is subjected to the action of diastatic enzymes, since the conditions of preparation (concentration of Aktivin and time) may be varied widely without the danger of formation of dextrin and lower degradation products of starch which ultimately promote the yellowing of white fabrics during storage. Starch pastes prepared with Aktivin are transparent only when hot, for insoluble starch separates when they are cooled; transparency is regained when the paste is reheated. A paste suitable for finishing white (lace) curtain materials is prepared by boiling a mixture of 10 kg. of potato starch, 250–270 litres of water, and 100 g. of Aktivin for 10–15 min., and then adding 1–2 kg. of Turkey-red oil, soap, or other suitable fatty substance. A paste for finishing printed fabrics is prepared by boiling a mixture containing 10 kg. of potato starch, 80 litres of water, 100 g. of Aktivin, and 20 g. of sodium carbonate for 15 min., afterwards adding 4 litres of 10% solution of soap and 1 litre of Turkey-red oil; suitable weighting substances or hygroscopic compounds (*e.g.*, glycerin) for conferring

a linen-like handle to the finished fabric may be added.

A. J. HALL.

PATENTS.

Weighting fibres. O. BERG and M. IMHOFF (U.S.P. 1,579,628, 6.4.26. Appl., 26.12.22).—Textile material is weighted with tin by any method including the use of a fixing bath, and is subsequently treated with a soluble lead compound.

L. A. COLES.

Production of pattern effects on woven fabrics. HEBERLEIN & Co. A.-G. (E.P. 237,909, 28.7.25. Conv., 29.7.24).—Textile material containing cellulose acetate silk interwoven with, e.g., cotton threads, is printed with a reserve, and subsequently treated with a solvent, such as chloroform, acetone, pyridine, or epichlorohydrin, capable of dissolving out the artificial silk not protected by the reserve. The reserve may protect the artificial silk mechanically by forming a layer insoluble in the solvent, glue solution and alcoholic shellac solution being suitable for the purpose, or it may contain alkalis, which hydrolyse the cellulose acetate and thereby render it insoluble in the solvents. Various modifications are obtained by introducing dyeing or printing processes to obtain different colour effects.

L. A. COLES.

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

Nitrogen fixation in the high-tension arc. H. V. TARTAR and M. F. PERKINS (J. Physical Chem., 1926, 30, 595—619).—The effect of pressure on the yield of fixed nitrogen, the equilibria obtained with various mixtures of nitrogen and oxygen, and the effect of moisture on the yield when air is used have been examined in a laboratory arc furnace which can be made to give an efficiency almost equal to that obtained in commercial practice. Under constant conditions, the concentration of nitric oxide for any given mixture between the limits oxygen 1 vol., nitrogen 4 vols., and oxygen 4 vols., and nitrogen 1 vol., decreased linearly with increasing velocity, but the yield of fixed nitrogen reached a maximum at an intermediate velocity, which is dependent to some extent on the composition of the gas and its rate of flow. Very high air velocities gave a low output in respect to both yield and concentration of fixed nitrogen. A gradual decrease in concentration of nitric oxide and in the yield per kw.-hr. accompanies a reduced working pressure, and at corresponding pressures a mixture of equal volumes of nitrogen and oxygen gave the greatest yield. No marked improvement in yield by using decreased pressure could be detected. Experiments with various mixtures of nitrogen and oxygen using different amperages showed that the mass action law holds good for the equilibria obtained. Cooling from the whirling arc was very effective. A method is given for the calculation of the concentration of nitric oxide resulting from repeated exposures of a nitrogen-oxygen mixture to a high-tension arc.

The presence of water-vapour in the air supplied to the arc reduced both the yield and the concentration of the nitric oxide. With 2.6% of moisture the yield was reduced by 25%. L. S. THEOBALD.

High-test bleaching powder—calcium hypochlorite and basic calcium hypochlorite. S. URANO (Trans. Amer. Electrochem. Soc., 1926, 49, 303—311. Advance copy).—Basic calcium hypochlorite, $\text{Ca}(\text{OCl})_2 \cdot 2\text{Ca}(\text{OH})_2$, is obtained by adding pure slaked lime to a clear solution of bleaching powder (5—6% of available chlorine) and evaporating *in vacuo* at 40°. In definite conditions of concentration of the liquid phase the basic salt crystallises and may be separated, washed, and dried *in vacuo* at 80°. In this way a bleaching powder containing 40—48% of available chlorine is obtained. The salt is stable at above 115°, odourless, and not deliquescent. The rate of loss of chlorine, whether the salt is wet or dry, is less than in the case of ordinary bleaching powder. On adding water to the basic salt the calcium hypochlorite passes into solution and if the calcium hydroxide is filtered off, this solution may be concentrated *in vacuo* until normal calcium hypochlorite (90—95% of available chlorine) crystallises. This material is also relatively stable at 40—50°. Above 150° it is decomposed into calcium chloride and oxygen with violent explosions. C. IRWIN.

Pyro- and hydro-treatment of magnesite and dolomite. H. M. HENTON (Trans. Amer. Inst. Min. Eng., Feb., 1926. Advance copy, 34 pp.).—Experiments on the separation of magnesia from dolomite were conducted on a laboratory scale and later on in an extended plant situated near the quarries and utilising carbon dioxide from the calcining kilns. When calcined no carbon dioxide was evolved from dolomite at any temperature near 450°, the dissociation point of pure magnesium carbonate, and it seems evident that energy is required to break up a natural combination of the calcium and magnesium carbonates. At 500° magnesium carbonate was decomposed to the extent of 50% and complete decomposition was effected in 1½ hrs. at 600° under a very slight partial pressure of carbon dioxide. This stage of calcination, in which the calcium carbonate remains undecomposed, is the most suitable for the after-extraction process and may be effected in a rotary kiln. No advantage in calcining was obtained by reducing the size of the material below ¼ in. Carbon dioxide driven off during calcination was cooled, cleaned, and compressed for use in converting the magnesium oxide into the soluble bicarbonate. The apparatus used for carbonating a slurry of the calcined material was of the "Pachuca" type in which a vertical cylindrical vessel having a conical bottom contains a central tube which carries the liberated compressed gas and rising slurry on the same principle as the air lift. The best results were obtained by carbonating at atmospheric pressure with a gas mixture of the composition CO_2 25%, air 75%, and a number of tests showed from 87.3 to 96.4% extraction of magnesium oxide, using four stages of extraction. A

nearly clear solution of magnesium bicarbonate may be decanted, filtered, and then precipitated by heating, and although complete precipitation was effected usually below 100°, boiling seemed necessary in order to cause a quick settling of the precipitate. Long needles were observed frequently at the edges of a supersaturated solution of the bicarbonate before boiling though magnesium bicarbonate is known only in solution.

C. A. KING.

Transportation of liquefied chlorine gas. H. P. WELLS, H. M. MABEY, and J. M. ROWLAND (Trans. Amer. Electrochem. Soc., 1926, 49, 269—284. Advance copy).—Liquid chlorine is distributed in America by means of cylinders of 10—150 lb. capacity, in 13-ton tank wagons, and in multiple-unit tank wagons comprising 15 1-ton containers loaded on a suitable underframe and removable as required. Similar containers are suitable for sea transport. In all cases specifications and testing are controlled by the Inter-State Commerce Commission. The cylinders are tested to 1000 lb. hydrostatic pressure when new and to 500 lb. after five years. They are fitted with safety plugs of fusible alloy, as are also the 1-ton containers. These, which are tested to 500 lb. per sq. in., have concave ends for convenience in clamping and lifting. Both types of container are filled by weight. Tank wagons are fitted with top discharge only and are insulated with pressed cork lining covered by steel plate. Valve parts in contact with the gas are of monel metal. Chlorine can be withdrawn as either gas or liquid through different valves. If necessary air pressure or a steam coil may be used to assist discharge. Vapour pressure curves of chlorine and of chlorine plus air are given.

C. IRWIN.

See also A., May, 466, Dithionic acid and its salts (DE BAAT). 474, Thermal dissociation of sodium cyanide (GUERNSEY and SHERMAN). 475, System water-aluminium oxide (GUICHARD). 482, Metals and nitric acid (KLEMENC). 487, Action of silica on metal sulphates (MARCHAL); Oxidation of ammonia (PARTINGTON); Action of hydrogen and water on phosphorus at high temperature under pressure (IPATIEV and NIKOLAJEV). 489, Preparation of cyanogen (RICCA); Preservation of gaseous cyanogen (RICCA); Determination of perchloric acid (LOEBICH); Hydrazine sulphate as standardising reagent for iodine solutions (CATTELAÏN). 490, Electro-metric titration of oxidising acids (MALAPRADE); Determination of nitrogen, nitrate, chlorate, and perchlorate (KÜRSCHNER and SCHARRER); Colorimetric determination of hydroxylamine (PUCHER and DAY); Determination of phosphorous and hypophosphorous acids and calcium hypophosphite (ZIVY); Determination of arsenic acid (FRANCIS).

Preparation of industrial sands. WEIGEL.—See I.

Insoluble anodes for electrolysis of brine. FINK and PAN.—See XI.

PATENTS.

Manufacturing sulphuric acid. H. PETERSEN (E.P. 249,914, 29.12.24).—In the chamber process, an intimate mixture of gases and liquid is obtained by using two grades of finely divided filling material not exceeding 40 mm. size on the average. The finer material of 1—10 mm. size is used in the parts of the plant where nitrogen oxides are absorbed, whilst pieces the size of a walnut are preferably used in the denitration towers. The filling material is contained in chambers of greater width than height, the gases being forced in a vertical direction either upwards or downwards by means of a fan. One or more additional units may be placed between the denitrating and absorbing parts of the plant and rinsed with water or weak sulphuric acid, the liquid circulating in these units not being allowed to enter the other parts of the system, whilst the gases flow in a downward direction and thus become thoroughly mixed and distributed over the entire sectional area of the units.

H. ROYAL-DAWSON.

Recovering or separating caustic [alkali] hydroxides from solutions containing them. J. Y. JOHNSON. From VISCOSE Co. (E.P. 246,355, 23.10.24).—The alkali hydroxide contained in waste liquors resulting from mercerisation, the digestion of pulp, and similar operations is recovered by dialysis. The process is preferably worked on the counter-current principle, using a number of dialysing cells in series. Woven fabric, e.g., cotton sheeting, parchmensed by immersion for 2 min. in sulphuric acid, *d* 1.10, at a temperature below 30°, may be conveniently used as the membrane.

D. J. NORMAN.

Manufacturing hydrocarbons and cyanides. J. C. CLANCY (U.S.P. 1,567,241, 29.12.25. Appl., 18.4.25).—Comminuted carbonaceous material is destructively distilled in contact with molten alkali cyanide. The volatile products of the reaction, largely hydrocarbon oils, are collected and the residue is subjected to a cyanide-producing reaction, e.g., by mixing it with sodium carbonate and treating at a high temperature with nitrogen.

Method of making calcium cyanide. F. J. METZGER, Assr. to CALIFORNIA CYANIDE Co., INC. (U.S.P. 1,573,732, 16.2.26. Appl., 23.11.25; cf. B., 1926, 272).—Finely divided calcium carbide (100 pts.) is added to a mixture of 400 pts. of hydrocyanic acid containing about 1% of water; if lime be present in the carbide sufficient water is formed in the reaction. The mixture is maintained at 60° for about 2 hrs. The product is calcium cyanide of the formula $\text{Ca}(\text{CN})_2 \cdot x\text{HCN}$, where *x* is approximately 2; it is comparatively safe to handle and when exposed to air gives off hydrocyanic acid so that it is of value as an insecticide and the like.

T. S. WHEELER.

Process for making (A) heavy basic carbonate of magnesium, and (B) light basic carbonate of magnesium. B. B. GRUNWALD (U.S.P. 1,573,603-4, 16.2.26. Appl., 30.6.24).—(A) Calcined magnesite

is mixed with 10—15 pts. of water and treated with carbon dioxide so that when carbonation is finished the temperature has reached 38°. The product is much denser than basic magnesium carbonate as usually prepared and does not shrink on drying. It is of value as a cement and binder in the preparation of heat-insulating materials (cf. B), and as a filler for paper pulp and the like. The product obtained by mixing with asbestos and calcining at about 100° has great strength and good heat-insulating properties. (B) Heavy basic magnesium carbonate prepared as in (A) is agitated and heated with water to about 45° and kept for 15 hrs. when expansion is complete. Alternatively the heavy basic carbonate is heated to 60° with water until sufficient expansion has occurred, when cold water is added to stop further expansion. The product is very light, does not shrink in drying, and is of value as a heat-insulating medium. For storage purposes the light basic carbonate is prepared by heating the heavy material with water at 80° until expansion is complete. When required it is mixed with a suitable quantity of heavy basic magnesium carbonate, as when completely expanded at 80° it is too light to be of value. T. S. WHEELER.

Silicious alkaline-earth product. R. CALVERT, ASS. to CELITE Co. (U.S.P. 1,574,363, 23.2.26. Appl., 1.12.23).—Diatomaceous earth is heated with excess of water and 5—80% of lime or other alkaline-earth hydroxide at about 100° until all expansion ceases. The product is very finely divided and is hence more efficient than diatomaceous earth as a filter aid, and a decolorising agent.

T. S. WHEELER.

Production of potassium sulphite, bisulphite, and metabisulphite. E. HENE (G.P. 424,949, 2.3.24).—Potassium bisulphite solution containing a relatively high proportion of calcium ions, is prepared by treating calcium bisulphite solution, with or without the addition of solid calcium sulphite, with potassium sulphate, solid or in solution, or with syngenite. After filtration, the solution is treated with potassium sulphate so as to obtain a solution containing relatively few calcium ions, and the precipitate, which contains a high percentage of potassium, is used for treating further quantities of calcium bisulphite solution. The process may be carried out at low temperatures, or under increased pressure. Calcium sulphite used in the process is obtained by heating calcium bisulphite solution to expel sulphur dioxide. The potassium bisulphite solution obtained is purified by heating it, and filtering off precipitated calcium hydroxide and iron compounds.

L. A. COLES.

Manufacture of nitrites from nitrous gases. A. BÄHR (G.P. 424,950, 2.5.25).—Waste nitrous gases are dried and diluted with dry air free from carbon dioxide, before passing into absorption apparatus. If necessary, the nitrous gases are subjected to a reduction process before dilution with air.

L. A. COLES.

Continuous production of anhydrous sodium sulphate. W. RECHA (G.P. 424,983, 4.10.24).—Crystalline sodium sulphate, $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$, is heated to a temperature above 32.4°, e.g., in the trough of a rotating filter, and the anhydrous sodium sulphate which crystallises out is removed continuously from the liquor.

L. A. COLES.

Stabilising ammonium carbonate, bicarbonate, and carbamate. A. and L. WELTER, and A. WEINDEL (G.P. 425,335, 3.12.24).—Dry, finely powdered ammonium compounds, alone or mixed with organic or inorganic salts, and atomised inorganic salt solutions, or cellulose waste liquor, or mixtures of these, are charged simultaneously into the top of a tower in which the material is dried by contact with a counter-current of air, preferably charged with acid gases, e.g., hydrogen chloride (cf. G.P. 336,612; B., 1921, 613 A).

L. A. COLES.

Continuous purification of salts. I. G. FARBENIND. A.-G. (G.P. 425,503, 1.7.24).—The salt to be purified trickles down a fall tube through an ascending current of a saturated solution of pure salt of the same composition. Purified salt after deposition, and if necessary after dissolving in water added for that purpose, is withdrawn, whilst a further quantity of impure salt is fed to the fall tube, e.g., by means of the wash liquor used in the purification.

J. S. G. THOMAS.

Production of potash and alumina from leucite. S. I. P. SOC. ITAL. POTASSA (G.P. 425,797, 24.1.24).—Leucite is heated with lime until the mixture fuses, and the product, in the form of big lumps, is allowed to cool slowly whereby it falls to a powder.

L. A. COLES.

Manufacture of silica gel. CHEM. FABRIK AUF ACTIEN (VORM. E. SCHERING), and W. KLAPHAKE (E.P. 250,078, 20.7.25).—Silica gel previously deprived of the major part of its water content, or dried at ordinary temperature for several days, is heated for a short time to about 650° in a current of air. This has the advantage of causing any coloration of the gel to disappear, giving the product a more glass-like appearance.

H. ROYAL-DAWSON.

Manufacture of iron carbonyl. J. Y. JOHNSON. FROM BADISCHE ANILIN- & SODA-FABR. (E.P. 250,132, 25.11.25).—In the manufacture of iron carbonyl by passing carbon monoxide over metallic iron at a high pressure, the speed of reaction is increased when the temperature is raised above 140°, and under such conditions the yield of product is considerably increased by reducing the temperature of the gases before releasing the pressure. The temperature of the reaction gases is preferably reduced by utilising the heat thereof, while the gases are still under high pressure, for preheating the fresh gases introduced into the apparatus.

H. ROYAL-DAWSON.

Manufacture of hydrogen. W. P. ROGERS (E.P. 249,925, 1.1.25).—In the process in which iron oxide is reduced by water-gas and re-oxidised with

steam, smaller depths of the oxide (about 5 ft.) are used in the retorts, which are so arranged that the heating flues are heated by gas burnt in a central flue. While the oxide is reduced in one set of retorts, steam is reduced with formation of hydrogen in the adjoining set. The spent (oxidised) reducing gases from the retorts are returned to the water-gas generator where they are reduced by incandescent carbon and, after purification from hydrogen sulphide, are used again in the process. The generator is placed as near as possible to the retorts and the water-gas prior to entering the retorts passes through a heat-exchanger where it is heated by the hot gases from the oxidation retorts. H. ROYAL-DAWSON.

Recovery of sulphur from gases. F. RIEDEL (G.P. 425,664, 24.8.24).—Gases containing sulphur compounds, alone or mixed with air, are used in place of air in coal-dust or oil furnaces, contact material being added to the fuel, if necessary. Coal-dust is carried into the furnace by a blast of gas containing little or no oxygen, *e.g.*, flue gases or nitrogen. L. A. COLES.

Effecting catalytic reactions, particularly in catalytically producing hydrocyanic acid. BADISCHE ANILIN- & SODA-FABR., Assees. of A. MITTASCH and W. MICHAEL (U.S.P. 1,582,851, 27.4.26. Appl., 12.7.23).—See E.P. 220,771; B., 1924, 869.

Producing anhydrous magnesium chloride. C. ARNOLD. From DOW CHEMICAL CO. (E.P. 243,978, 27.4.25).—See U.S.P. 1,557,660; B., 1925, 991.

Purification or extraction of soluble substances [sodium nitrate from caliche]. W. BROADBRIDGE and W. G. SELLERS, Assrs. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,562,863, 24.11.25. Appl., 13.6.22).—See E.P. 182,859; B., 1922, 669 A.

Synthesis of ammonia. G. CLAUDE, Assr. to LAZOTE, INC. (U.S.P. 1,579,647, 6.4.26. Appl., 5.4.21).—See E.P. 161,195; B., 1922, 590 A.

Process of forming sulphite of lead. S. C. SMITH (U.S.P. 1,581,030, 13.4.26. Appl., 3.9.25).—See E.P. 239,558; B., 1925, 881.

Process of making lead sulphate. S. C. SMITH (U.S.P. 1,581,031, 13.4.26. Appl., 12.10.25).—See E.P. 239,559; B., 1925, 881.

Producing calcium hypochlorite compounds. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of G. PISTOR, H. S. SCHULTZE, and H. REITZ (U.S.P. 1,583,421, 4.5.26. Appl. 4.11.22).—See E.P. 188,662; B., 1923, 656 A.

Sodium compound. H. E. COCKSEGE, Assr. to SOLVAY PROCESS CO. (U.S.P. 1,583,660, 4.5.26. Appl., 30.10.22).—See E.P. 202,678; B., 1923, 1020 A.

Process of forming sodium compounds. H. E. COCKSEGE, Assr. to SOLVAY PROCESS CO. (U.S.P. 1,583,661, 4.5.26. Appl., 9.11.23).—See E.P. 206,372; B., 1924, 16.

Process of forming sodium carbonate. C. SUNDSTROM and G. N. TERZIEV, Assrs. to SOLVAY PROCESS CO. (U.S.P. 1,583,663, 4.5.26. Appl., 19.11.23).—See E.P. 223,015; B., 1924, 980.

High-pressure joints (E.P. 249,001).—See I.

Carbon monoxide from industrial gases (G.P. 425,418).—See II.

VIII.—GLASS; CERAMICS.

Chemical composition of window glass. E. P. ARTHUR (J. Amer. Ceram. Soc., 1926, 9, 203—205).—Typical analyses of sheet glass made by various processes show variations (after allowing for minor constituents) of SiO₂ 71.9 to 74.3%, CaO+MgO 11.1 to 14.2%, Na₂O 12.7 to 15.1%. Although the variations are small, the different glasses are distinct in properties, for the glass-making machines are sensitive to composition changes. The results of Morey and Bowen for the system SiO₂-CaO-Na₂O (B., 1925, 921) are confirmed in window-glass practice. For example, the devitrification temperature of the glass SiO₂ 74%, CaO 11%, Na₂O 15% is given as about 1000°, whilst in practice, although the glass will work well at 993° a variation of 10° causes difficulty. Similar confirmation is obtained from hand-working conditions. A. COUSEN.

Deflocculation of clay slips and related properties. E. W. SCRIPTURE, JUN., and E. SCHRAMM (J. Amer. Ceram. Soc., 1926, 9, 175—184).—Further experiments on a series of English china clays, ball clay, and kaolins (cf. B., 1925, 500) showed that an increase of the content of organic matter (as determined from the carbon content) coincided with an increase of toughness and plasticity. The change in the state of dispersion with variation of p_H for 20% slips was determined and proved the more plastic clays to have high dispersion with low p_H . The absorption of alkali in 20% and 40% slips was greater in the case of the more plastic clays. There appeared to be some relationship between p_H at the isoelectric point, the carbon content of the clay, absorptive power for caustic soda, and plasticity, although this could not be expressed in terms of any one variable. The viscosity- p_H curves for 40% slips were of the same general shape and location as the turbidity and settling curves. Casting slips were prepared in which the china clays and two ball clays were used in varying proportions. Ball clay was found to produce fluidity, and more viscous slips were obtained from more plastic than from less plastic china clays. A. COUSEN.

De-airing as corrective in drying [clay ware]. F. BRAND (J. Amer. Ceram. Soc., 1926, 9, 188—190).—An examination of current views on the improvement in drying qualities found in the case of de-aired clays. Facts do not support the theory that the drying quality resides within the clay itself. A. COUSEN.

Heat required to fire ceramic bodies. A. E. MACGEE (J. Amer. Ceram. Soc., 1926, 9, 206—247).

—Differential specific heats of ceramic materials, as determined by comparison with quartz, are firebrick about 0.43 (25—1200°), kaolin 0.5 (0—1000°), ball clay and diaspore clay 0.45 (0—1000°), flint and feldspar 0.28 (0—1050°). Endothermic reactions occur with diaspore clay at 525°, firebrick and ball clay 550°, kaolin 575°, and exothermic processes with firebrick 950°, kaolin 960°, and ball clay (slight) 975°. The efficiencies of kilns tested were about 35%.
A. COUSEN.

Properties of silica brick from coke-oven walls. S. S. COLE (J. Amer. Ceram. Soc., 1926, 9, 197—202).—Silica brick samples made from different quartzites showed like properties after long service. They were in good condition after 12 years at operating temperatures of 1370—1430°, and the only vitrification was a glazed surface on the flue face. Little change had taken place in chemical composition or porosity. The quartz had been inverted, on the flue face chiefly to tridymite, on the coke face chiefly to cristobalite.
A. COUSEN.

Use of overglazes for polychrome terra-cotta. A. L. BENNETT (J. Amer. Ceram. Soc., 1926, 9, 185—188).—Of the methods of polychroming terra-cotta, the use of overglazes to produce various colour effects in a single firing is economical and flexible. The overglaze should be insoluble in the liquid used and contain little or no clayey material. One difficulty lies in the tendency towards translucency and the influence of the character of the underlying glaze upon the colour reactions. A suggested base white overglaze is 0.621 ZnO, 0.379 CaO; 0.453 Al₂O₃; 1.324 SiO₂, 0.126 SnO₂, which may be varied in shades of colour by blending the proper stains or colouring oxides. It is made up entirely from calcined or non-plastic materials. The methods of preparation and application of the glaze are described.
A. COUSEN.

Preparation of industrial sands. WEIGEL.—See I.

Treatment of magnesite and dolomite. HENTON.—See VII.

PATENTS.

Making elongated articles of vitreous silica. BRIT. THOMSON-HOUSTON Co., Assees. of B. F. NIEDERGESASS (E.P. 241,544, 8.10.25. Conv., 20.10.24).—Particularly clear, bubble-free, and uniform elongated articles (as rods and tubes) of vitreous silica are produced by drawing directly from slugs of the vacuum-fused material, without contact with a die or solid body. A machine for performing this operation is described.
A. COUSEN.

Ovens or kilns [for ceramic ware etc.]. J. LAURENT (E.P. 233,313, 24.2.25. Conv., 30.4.24).—The oven consists of a circular brickwork heating chamber with refractory lining. Around the periphery of the chamber is a number of fireboxes, each surrounded by a duct, opening to the atmosphere at one side and leading, on the other, to a chamber lying in the upper oven wall, between layers of

refractory brick. Air enters by the ducts and is heated therein and in the chamber. During the heating process it is used for secondary combustion at the points where the furnace gases enter the chamber. In the cooling process the hot air enters the oven chamber at points in the upper part thereof and passes downwards over the charge, cooling as the charge cools.
A. COUSEN.

Manufacture of molybdenum compounds [abrasives]. NORSKE MOLYBDENPRODUKTER A.-S. (G.P. 424,990, 15.5.21).—Molybdenum-sulphur compounds are heated with boron, titanium, or zirconium, or with mixtures of these, in such quantities that molybdenum boride, titanide, or zirconide is produced. Thallium, vanadium, or the metals of the iron and chromium groups, or closely allied metals, or compounds of these metals may also be added, and the hardness of the product can be increased by the addition of small quantities of carbon, silicon, nitrogen, or phosphorus, or compounds containing them.
L. A. COLES.

Quartz working. E. R. BERRY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,581,829, 20.4.26. Appl., 29.3.21).—See E.P. 191,463; B., 1923, 226 A.

Channel oven for ceramic and chemical purposes. H. T. PADELDT (U.S.P. 1,583,739, 4.5.26. Appl., 27.6.25).—See E.P. 245,005; B., 1926, 275.

Method of treating ceramic mixtures. H. SPURRIER (E.P. 242,916, 1.7.25).—See U.S.P. 1,559,652; B., 1926, 55.

Annealing glassware. R. L. FRINK (E.P. 250,634, 13.12.24).

Burning ceramic wares. E. C. R. MARKS. From A. C. SPARK PLUG Co. (E.P. 250,732, 16.3.25).

IX.—BUILDING MATERIALS.

Preparation of industrial sands. WEIGEL.—See I.

PATENTS.

Impregnation of wood. R. DITMAR (Austr. P. 100,729, 27.11.23).—Wood, particularly that for use in the manufacture of violins, organs, and pianos, is improved by steeping in rubber latex mixed with 0.1—10% of glycerin.
L. A. COLES.

Device for burning cement, magnesite, lime, etc. H. STEHMANN (U.S.P. 1,581,522, 20.4.26. Appl., 30.8.21).—See E.P. 188,424; B., 1923, 82 A.

Cement-roasting apparatus. I. E. LANHOFFER (U.S.P. 1,582,831, 27.4.26. Appl., 24.5.22).—See E.P. 195,585; B., 1923, 834 A.

Process of making vesicular products [building materials]. C. W. BOYNTON, Assr. to Government of the United States and to the People of the United States (U.S.P. 1,583,521, 4.5.26. Appl., 20.12.18).—See E.P. 153,030; B., 1920, 821 A.

Mixing composition for use with wood aggregates and structures. J. R. GARROW,
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ASSR. to NOVOCRETE CO. OF AMERICA, INC. (U.S.P. 1,583,713, 4.5.26. Appl., 25.9.23). See E.P. 220,677; B., 1925, 12.

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

Combustibility of coke and direct reduction in the blast furnace. W. W. HOLLINGS (Iron and Steel Inst., June, 1926. Advance proof. 9 pp.).—In discussing reduction in the blast furnace from the results of other workers, the author states that so far as the iron itself is concerned, Gruner's ideal—that the whole of the reduction should take place indirectly by means of carbon monoxide—should be fulfilled in practice. Direct reduction must involve a greater demand for heat from the furnace and so should be avoided. The only lines on which it would seem possible to make any considerable saving in the heat demands of a modern blast furnace would appear to be by a considerable enrichment of the blast with oxygen, whereby the rapidity of cooling of the ascending gases would be increased and the volume of gas per unit of carbon burnt reduced; or by the substitution of pulverised quicklime blown in at the tuyères for limestone added at the top. This latter would save carbon which reacts with liberated carbon dioxide, and would conserve the heat required for this reaction and also the heat necessary for the dissociation of the limestone.

C. A. KING.

Chemical equilibrium of manganese, carbon, and phosphorus in the basic open-hearth process. C. H. HERTY, JUN. (Trans. Amer. Inst. Min. Eng., Feb., 1926. Advance copy. 26 pp.). Experimental data have been obtained from laboratory furnaces of $\frac{1}{2}$ lb. capacity and from 100-ton stationary open-hearth furnaces, the three fundamental principles taken into consideration in the work being the law of mass action, the van't Hoff isochore, and the distribution law of Nernst. Except when the slag contains more than 5% P_2O_5 , the amount of residual manganese is controlled by the total manganese charged, the amount of this element volatilised, the volume, basicity, and iron oxide content of the slag, and the temperature. High-manganese charges increase the fluidity of the slag and the speed of working and increase the ratio of manganese sulphide to ferrous sulphide in the metal, resulting in a more complete elimination of ferrous sulphide by the final manganese addition. The solubility of ferrous oxide increases 1.25 times as fast as the solubility of manganese oxide (MnO) for a given rise of temperature. The quantity of iron oxide in the metal is not controlled by the residual manganese. Temperature and the concentration of iron oxide in the slag govern the carbon content of the metal and at 1482–1538° the equilibrium constant for the reaction $C + FeO = CO + Fe$ in the metal phase is 0.36. At the same temperature the solubility of carbon monoxide in pure iron is 0.018% by weight. The amount of phosphorus remaining in the metal depends on the total phosphorus in the

charge, the temperature, and the volume, basicity, and iron oxide content of the slag. Equations are given showing the relationship between the variables controlling the phosphorus and those controlling the manganese.

M. COOK.

Catalytic action of iron ores in decomposing carbon monoxide. P. FARUP (Tidskr. Kem. Bergv., 1926, 6, 1–6, 15–17; Chem. Zentr., 1926, I., 2530–2531).—The catalytic action of various iron ores in powdered form, in lumps, and in the form of briquettes on the decomposition of carbon monoxide at 420° according to the equation $2CO \rightleftharpoons CO_2 + C$ has been investigated. The results obtained were in all cases extremely erratic; sometimes no action appeared to take place whilst at other times the reaction was strongly catalysed with the same sample of ore. In general briquetting appeared to accelerate the decomposition of carbon monoxide. The presence of much silica in the ore lowered considerably its catalytic action and addition of calcium carbonate or sodium chloride decreased the action to a slight extent, owing to the glazing of the surface.

A. R. POWELL.

Prevention of embrittlement in malleable cast-iron. L. H. MARSHALL (Trans. Amer. Inst. Min. Eng., Feb., 1926. Advance copy. 8 pp.).—Embrittlement of malleable cast-iron observed after galvanising is not due either to the pickling process or to the etching action of the molten zinc, as malleable iron itself heated to 460° and chilled becomes brittle. Higher impact values are obtained if malleable iron is quenched from 600–750°, and such heat-treated metal may be galvanised in the ordinary manner without becoming brittle. A further advantage of such heat-treatment is that malleable iron may be subjected to low temperatures (0°) without any lessening of shock resistance.

C. A. KING.

Report on the heterogeneity of steel ingots. (Iron and Steel Inst., June, 1926. Advance proof. 113 pp.).—A large number of ingots ranging from 15 cwt. to 172 tons in weight and, in general, chill cast, were examined along a section taken longitudinally through the central axis. Transverse sections were also made in certain cases. Photographs of the sections, after etching with various reagents, and sulphur prints were taken. Analyses of definitely located samples and scleroscope hardness determinations over the sections were carried out. The results so obtained indicate that sulphur, phosphorus, and carbon exhibit segregation, whilst silicon and manganese, which diffuse more readily in solid solution, do not show this phenomenon. In general there exists in an ingot an outer layer of ill-defined crystalline structure, due to the initial rapid cooling of the steel in contact with the mould. From this layer, a layer of columnar crystals grows inwards and upwards, the thickness of which is dependent on several factors. Proceeding towards the centre of the ingot, there is a region richer in segregates, which may occur in spheroids or thin strings. This zone of segregation is annular, its

inner boundary having the form of a truncated cone or pyramid, depending on the shape of the mould. Extending inwards from this zone, there is a central region, in the lower half of which the steel has a lower sulphur, phosphorus, and carbon content than in any other part of the ingot, while the composition of the upper portion approaches the average value for the steel; at the extreme top there is a region of maximum segregation. The axial portion of the ingot is always rather unsound.

L. M. CLARK.

Distribution of silicates in steel ingots.

J. H. S. DICKENSON (Iron and Steel Inst., June, 1926. Advance proof, 20 pp.).—Slaggy matter in the form of small silicate particles occurs most abundantly in the central lower part of steel ingots, in which locality carbon, sulphur, and phosphorus are reduced to a minimum by segregation. This has been found to be the case in small as well as large ingots of carbon, nickel, and nickel-chromium steels both top-cast and bottom-cast. The slaggy matter, obtained as residues from complete solution of normal samples, scraped from external slag streaks, and from pipe cavities is of similar composition, containing large amounts of silica and manganese oxide together with varying amounts of ferrous oxide and alumina. In large ingots the silicate content of the first frozen skin at the bottom end is low and similar to that of the outer skin at the upper end, but it rises upwards and inwards to a maximum at or near the central axis within the lower part of the central pyramid of free crystals. The silicate globules from the region of high silicate content are larger than those from other portions of the ingot. It is suggested as a possible explanation of the high silicate region that the purer crystallites, of which there is a continuous downward movement to the narrowing and rising solid floor, intercept and carry down the coalesced particles of slaggy matter.

M. COOK.

Hardness of carbon steels at high temperatures. I. G. SLATER and T. H. TURNER (Iron and Steel Inst., June, 1926. Advance proof. 9 pp.).—The impact hardness of a series of carbon steels ranging from 0.02% to 1.105% C was determined at 50° intervals from 650° to 1200°. Little hardness was lost up to 650°, but between 650° and 900° all the steels softened considerably, the degree of softening increasing with the carbon content. Decrease in hardness was relatively less rapid above 900° and the hardness values of all the steels became more nearly equal. For riveting purposes the higher the carbon content up to the eutectoid composition, the more energy is required to shape the rivet, and there is a distinct advantage to be gained in performing rapid work on the rivet at the highest working temperature before the steel cools. C. A. KING.

Ratio of tensile strength of steel to the Brinell hardness number. R. H. GREAVES and J. A. JONES (Iron and Steel Inst., June, 1926. Advance proof. 18 pp.).—The results of a large number of laboratory tests, under definite conditions, on several

kinds of steels have been studied by statistical methods and the results of other investigators on the tensile strength-Brinell hardness number ratio have been examined. This ratio is dependent on the hardness of the material and on its yield ratio and to some extent on the composition and heat-treatment. It decreases with increase of yield ratio for a given class of steel and with increase of hardness up to 375 to 450 Brinell. The ratios recommended are 0.21, 0.215, and 0.22 respectively for heat-treated alloy steels with a Brinell hardness of 250—400, heat-treated carbon steels and alloy steels with a hardness below 250, and medium carbon steels as rolled, normalised, or annealed. The value 0.23 is suitable for mild steels as rolled, normalised, or annealed. None of these ratios is applicable to austenitic or severely cold-drawn steels. Owing to the want of uniformity in a piece of steel and consequent variations in the Brinell figures the probable error of the ratio for a given series of steels is rarely less than ± 0.003 .

M. COOK.

Ghost lines and the banded structure of rolled and forged mild steels.

J. H. WHITELEY (Iron and Steel Inst., June, 1926. Advance proof. 6 pp.).—Only when the variation in phosphorus content between two adjacent areas exceeds 0.07% can it cause removal of carbon, between the Ar₃ and Ar₁ points, from the richer area. In most cases it is still doubtful whether this heterogeneity is the cause of ghosts, since other factors may influence the movement of carbon to an extent which outweighs the influence of phosphorus. Banded structures have been observed in steels containing phosphorus as low as 0.004%, and it is shown that in certain cases carbon may move from one region to another higher in phosphorus. Ziegler's theory that non-metallic inclusions act as nuclei for the crystallisation of ferrite and so produce a ghost formation does not adequately account for the facts which are given.

M. COOK.

Effects of arsenic on steel. A. E. CAMERON and G. B. WATERHOUSE (Iron and Steel Inst., June, 1926. Advance proof. 20 pp.).—Steels containing carbon about 0.5% and having similar compositions with regard to sulphur, phosphorus, manganese, and silicon, with arsenic contents varying from 0.20 to 0.46% in the molten state, exhibit segregation of arsenic on solidification, since the arsenic content of the billets obtained by cropping off the ends of the ingots falls in each case to approximately 0.2%. It is concluded that an equilibrium exists between the arsenic and carbon, which tends to limit the arsenic content to 0.2% in 0.5% carbon steels. Indications of red-shortness are obtained during the forging of these steels, and this is ascribed to the presence of arsenic, since no red-shortness is apparent in a control sample of steel free from arsenic. The position of the transformation points of the 0.5% carbon steel is not materially modified by arsenic (0.22%), while a slight increase in tensile strength and a small but rather greater decrease in ductility result. There is a considerable reduction

in the shock-resisting capacity of these steels as measured by the Charpy impact test, and this effect is most noticeable when the metal is in a hardened condition. No effect on the corrosibility or on the case-hardening properties is caused by the presence of arsenic up to 0.22% and the steels can be welded if a suitable welding compound be used. Microscopical examination shows that arsenic segregates, forming a banded structure which persists throughout all the heat treatments applied, and it is concluded that arsenic does not diffuse in γ -solid solution. No evidence was obtained for the existence of a brittle eutectic of iron-iron arsenide. Arsenic occurs in steel only as a solid solution of arsenic in iron.

L. M. CLARK.

Effect of mass in the heat treatment of nickel steel. W. ROSENHAIN, R. G. BATSON, and N. P. TUCKER (Iron and Steel Inst., June, 1926. Advance proof. 33 pp.).—Results of tests in which round bars of nickel steel (about 3.5% Ni) of 2, 4, 7, and 10 in. diam. were normalised at 850°, oil-hardened at 850°, and oil-hardened and quenched at temperatures between 300° and 700°, showed that the quenching effect was insufficient to effect complete hardening of the steel even in pieces 2 in. in diameter. In this size the strength after quenching was only about 60 tons per sq. in., rising to 80 tons on tempering at 300°, and then falling almost to the strength of normalised material when tempered at 650°. A bar of 4 in. diam. behaved somewhat similarly and the strength of the 7 in. and 10 in. diam. bars was not increased by tempering the hardened pieces unless the temperature exceeded 500°. The largest size was little affected at the centre by quenching. Quenched nickel steel possessed a low notched-bar figure compared with normalised steel, but tempering at high temperatures, *e.g.*, 600° and 650°, caused a considerable improvement even in the 7 in. and 10 in. pieces. The effect of heat treatment on the structure of the steel was marked in so far as the outer portions of the material were concerned but the centre of a 10 in. bar was little different in micro-structure or mechanical properties from the normalised steel.

C. A. KING.

Hardening and tempering of high-speed steel. A. R. PAGE (Iron and Steel Inst., June, 1926. Advance proof. 24 pp.).—The conditions of hardening and tempering two high-speed steels, J1 and J2, of the composition C 0.55–0.65% (J2, 0.7–0.8%) Si 0.2%, W 13–14%, Cr 3–4%, Mn 0.4–0.5%, V 0.25–0.5%, S 0.05%, P 0.06% were investigated. J1 was hardened completely by soaking for 10 min. at 1250°, or at 1300° for 2 min., though at the latter temperature there was enormous crystal growth if the time of soaking was extended. The higher carbon content of J2 reduced the temperature required for correct hardening to 1150°, but the range of temperature and time for hardening were greater than for J1. Excessive brittleness in the hardened material due to the production of an intercrystalline eutectic constituent, evidently caused by incipient fusion at the crystal boundaries, was more marked in the higher carbon steel. The

hardened steels after tempering at 600° for 2 min. appeared perfectly stable as regards hardness at any temperature up to 600°, but the hardness and appearance of the fracture in the hardened state were no criteria of the production of the red-hardness property.

C. A. KING.

Advances in high-speed alloys. E. H. SCHULZ, W. JENGE, and F. BAUERFELD (Z. Metallk., 1926, 18, 155–158).—The effect of heat treatment on the structure and magnetic properties of a cobalt magnet-steel containing 16% Co, 9% Cr, 2.5% Mo, and 1% C has been investigated. This alloy in the soft annealed state has a sorbitic structure with large white inclusions of carbide; it exhibits arrest points on heating at 600° and 820°, and on cooling at 775° and 590°, the higher points corresponding with the Acl and Arl points of steel respectively. The usual heat treatment for developing the magnetic properties comprises three steps: (i) heating for 5 min. at 1150–1200° followed by cooling in air, whereby a homogeneous austenitic structure is produced; (ii) heating to 750° followed by slow cooling to 500° in the furnace and to the ordinary temperature in air, whereby the austenite is decomposed partially with the precipitation of finely-divided carbide; (iii) heating to 1000° followed by air-cooling, whereby the carbide redissolves in the α -iron forming hardenite. The changes in hardness and magnetic properties undergone by the metal in this treatment are shown in the following table:—

	Brinell hard- ness.	Remanence.	Coerci- vity.	Electri- cal con- ductivity.
Annealed ..	341	12,600	35	2315
after (i) ..	218	1500	—	1482
„ (ii) ..	394	12,000	49	1919
„ (iii) ..	700	8000	190–205	1357

The structure of stellite consists of a ground-mass of eutectic nature containing needles of carbide. The smaller the needles the greater is the cutting power of the alloy; to obtain this structure the alloy should be cast in as thin plates as possible so that cooling is relatively rapid. In this form the alloy is suitable for making draw-rings for steel shells, having a life 250–1000 times as long as that of similar rings of cast iron, besides being non-rusting. Corrosion tests over 4 months showed that stellite alloys lose only 0.07% in weight in 20% nitric acid and 0.3% in 50% sulphuric acid and are not attacked by saturated solutions of sodium chloride or sulphide, 50% solutions of sodium hydroxide, or glacial acetic acid.

A. R. POWELL.

Determination of carbon in pig-iron, steel, and ferro-alloys. E. SCHIFFER (Stahl u. Eisen, 1926, 46, 461–468; cf. van Royen, B., 1924, 425).—The mixture of carbon dioxide and oxygen obtained by burning ferrous materials in the minimum quantity of oxygen at 1100–1200° is collected. The volume of carbon dioxide absorbed by potassium hydroxide solution enables the carbon content of the material to be determined. The method is satisfactory for steels and pig-irons containing little sulphur and having a carbon content of not more than 1%,

especially when lead oxide is used as a flux. It is not satisfactory with ferrous alloys in general. The speed with which the determination can be made renders this method valuable for controlling smelting processes. Alternatively, the gas mixture may be passed through barium or sodium hydroxide solution. Titration of the excess of alkali gives the amount of carbon dioxide formed in the combustion.

L. M. CLARK.

Determination of phosphorus in steels containing tungsten. T. E. ROONEY and L. M. CLARK (Iron and Steel Inst., June, 1926. Advance proof, 6 pp.; cf. A., 1923, ii, 251).—Confirmation is given for the observation of previous investigators that, when tungsten steels are decomposed by treatment with acid, some phosphorus is contained in the iron solution whilst the remainder is retained in the insoluble tungsten residue. Previously described methods for the determination of phosphorus in such steels are unsatisfactory. A newly-devised method gives trustworthy results. The steel is digested with *aqua regia* until the iron is dissolved and the tungsten is converted into tungstic oxide. After evaporation, the solid residue is heated to drive off nitric acid and the ferric oxide is dissolved out with hydrochloric acid. The tungstic oxide containing some phosphorus is dissolved in ammonia and a little ferric chloride is added to the solution, which is then made slightly acid with hydrochloric acid. On precipitation of the iron from this solution with ammonia, the phosphorus is carried down with the ferric hydroxide. The precipitate is dissolved in hydrochloric acid, reprecipitated with ammonia, and redissolved in hydrochloric acid. This solution is added to the main bulk of ferric chloride solution from which hydrochloric acid is expelled by evaporation with nitric acid. Phosphorus is then determined by precipitation as phosphomolybdate and titration with alkali. The method is applicable to steels containing vanadium.

L. M. CLARK.

H-ion concentration [of water] and corrosion. J. R. BAYLIS (Eng. and Cont., Water Works Issue, 1925, 64, 1279—1280).—A factor in corrosion is the effect of p_H on oxidation rates, soluble iron in water containing excess oxygen oxidising much more rapidly at p_H 9 than at p_H 6. A large number of experiments on the amount of iron which will remain in solution at different p_H values gave the following results: p_H 9.0, less than 0.1 p.p.m.; p_H 8.0, 0.1 p.p.m.; p_H 7.5, 1.0 p.p.m.; p_H 7.0, 4 p.p.m. Below p_H 7.0 the soluble iron content increased rapidly. There is a natural tendency for tubercles to form an impervious coating near the surface exposed to the water, where the soluble iron from the interior comes in contact with dissolved oxygen. If the water is slightly acid, say p_H 6.0, the rate of oxidation is low and there is a tendency for the soluble iron to diffuse into the water. If the water is alkaline, about p_H 8.5, nearly all the soluble iron will be precipitated at the surface of the tubercle and in a few months will form an impervious coating of a crystalline iron oxide. If alkalinity is due to calcium carbonate

or bicarbonate, and p_H is adjusted to a point which favours precipitation of calcium carbonate, this will also aid greatly in preventing corrosion, in addition to the natural tendency of corroding iron to protect itself in alkaline water. Pits have been found to be concentration cells in which negative ions, e.g., chloride and sulphate, are concentrated. The solution in the interior of an active pit has a p_H close to 6.0, regardless of the H-ion concentration of the surrounding solution. R. E. THOMPSON.

Factors other than dissolved oxygen influencing the corrosion of iron pipes. J. R. BAYLIS (Ind. Eng. Chem., 1926, 18, 370—380).—Dissolved oxygen is the governing factor in the corrosion of new iron water pipes by town water only until the pipe becomes coated with a film of rust. After this other factors chiefly connected with the composition of the water become of primary importance. Ferrous hydroxide is the first product of corrosion; its solubility in the water depends on the p_H , and with a p_H above 8 and in the absence of dissolved oxygen, it is almost insoluble in natural waters. Ferrous carbonate is only very slightly soluble in water saturated with calcium carbonate, but its solubility increases rapidly as the p_H falls below calcium carbonate equilibrium. In waters having a high p_H the final product of the reaction between iron and the dissolved carbonates is a very insoluble ferrous carbonate. No gaseous hydrogen is liberated from pure distilled water by iron even after several months but the presence of negative ions other than OH' ions causes hydrogen to be evolved relatively rapidly. Thus, addition of calcium sulphate or sodium chloride to air-free distilled water in which are placed some iron filings results in hydrogen evolution after a few days and an increase in the p_H of the water to about 9; similar results are obtained on the addition of calcium hydrogen carbonate, and it therefore appears that this reaction is of fundamental importance in the corrosion of iron water pipes, for, unless it takes place, the surface of the metal becomes coated with a passive film of ferrous carbonate which is only extremely slowly oxidised by the dissolved oxygen. Some of the corrosion products occurring in water pipes are very magnetic and attach themselves to the surface of the metal, forming a very porous, fibrous aggregate. These deposits do not cause pitting, as the corrosion products readily diffuse from them. When, however, the rust coating becomes so thick that diffusion of soluble iron salts from inside the coating is prevented the water just outside the deposit still retains its content of dissolved oxygen and the corrosion products are precipitated inside the coating, thus soon rendering it impervious, and a pit is formed. This will continue to increase in size as long as the p_H of the water inside the membrane of rust is about 6, i.e., as long as the water retains iron salts (sulphates or chlorides) in solution. When the p_H rises above 6 action ceases and the pit becomes dormant. In acid waters the covering membrane has a great tendency to break; when this occurs the whole process is repeated, so that after a time these

membranes become of considerable size. In alkaline waters breakage occurs only rarely and the pipes therefore have a longer life. A. R. POWELL.

[Silver-gold] parting plant at the U.S.S. Lead Refinery Inc. F. F. COLCORD (Trans. Amer. Electrochem. Soc., 1926, 49, 229—238. Advance copy).—Doré containing 95—98% Ag and 1—5% Au, with small amounts of lead, copper, bismuth, selenium, and tellurium, is refined electrolytically in horizontal cells at the U.S.S. Lead Refinery. The electrolyte contains 50 g. of silver and 60 g. of copper (as nitrates) per litre; the cathode consists of a graphite plate lying on the bottom of a stoneware cell, $52 \times 24 \times 9$ in., and the anodes of thin ingots of doré inside a wooden framework sliding on the top of the cell and covered with cotton duck outside and muslin inside. A current density of 40 amp. per sq. ft. of anode surface at 3 volts is used in the electrolysis and from time to time the silver crystals are scooped out from the bottom of the cell with a perforated aluminium shovel. The gold slime from the muslin linings is periodically collected, washed to remove soluble salts, and boiled with sulphuric acid in an "ironac" pan. The resulting gold sponge when melted into bars is 995 fine. The silver sulphate solution is treated with copper to precipitate the silver, which is melted in a Faber du Faur furnace. Photographs and plans of the plant showing working details are given. A. R. POWELL.

Conductivity of electrolytes used in the electrolytic separation of silver and gold. F. F. COLCORD, E. F. KERN, and J. J. MULLIGAN (Trans. Amer. Inst. Min. Eng., Feb., 1926. Advance copy. 9 pp.).—The conductivity of the electrolyte increases with the copper and silver content; copper is more effective than silver, one unit raising the conductivity to the same extent as $1\frac{1}{2}$ units of silver in electrolytes containing 30—60 g. per litre each of silver and copper. Ammonium nitrate increases the conductivity, the maximum effect being shown when it is present to the extent of 165 g. (=35 g. of ammonia) per litre. The deposited silver is of a fine crystalline nature when the silver and copper content of the electrolyte is low (40 g. per litre) and with very low silver content (35 g. per litre), the crystals are either finer or they assume a needle-like shape. In the presence of copper the crystals become coarser as the silver increases up to 60 g. per litre, and increasing copper to 80 g. per litre results in a coarse and more compact deposit. Ammonium nitrate causes the formation of a more compact deposit from electrolytes containing between 40 and 60 g. per litre each of silver and copper. M. COOK.

Electrodeposition of zinc from electrolytes containing gelatin and aluminium sulphate. P. K. FRÖLICH (Trans. Amer. Electrochem. Soc., 1926, 49, 285—302. Advance copy).—The electrodeposition of zinc from chloride and sulphate solutions of varying degrees of acidity with and without the addition of gelatin and aluminium sulphate has been investigated. Gelatin invariably causes a dark, rough and nodular deposit to be

obtained, whilst the cathode polarisation and to a smaller extent the anode polarisation increase with increasing acidity. From this and other data it appears that there is a thin film of electrolyte on the surface of the cathode having a p_H above 5, while the p_H of the main solution is less than 5; hence the gelatin is positively charged and basic in character in the body of the electrolyte and negatively charged and acidic in character on the inner surface of the film owing to its inversion in passing through the isoelectric point, p_H 4.7. The action of aluminium sulphate is similar to that of gelatin in causing poor deposits of zinc, probably owing to precipitation of colloidal aluminium hydroxide in the cathode film. This would explain the difference in the action of gelatin and aluminium sulphate in the electrodeposition of copper and of zinc. A. R. POWELL.

Properties of sand-cast alloys of aluminium containing silicon and magnesium. S. DANIELS (Ind. Eng. Chem., 1926, 18, 393—398; cf. B., 1925, 73, 506).—Tensile, hardness, and density determinations were carried out on aluminium alloys containing 0.5—0.6% Mg, and silicon in amounts increasing from 0.27 to 4.67%. For sand-cast specimens containing up to 1% Si, the ultimate strength is about 16,000 lb./sq. in., and rises to 20,000 at 3—4.7% Si. The Brinell hardness increases similarly, but the elongation and ductility are markedly diminished. The properties are greatly improved by heat treatment for 96 hrs. at 550°, quenching from this temperature, followed by ageing for 8 hrs. at 150°, after which alloys with more than 0.3% Si showed a uniform ultimate strength of about 30,000 lb./sq. in., whilst the ductility was increased compared with the sand-cast state. The Brinell hardness also increased, but the density was not affected by heat treatment. Alloys containing 0.27% Si were little affected by heat treatment because the silicon content is less than that required to convert all the magnesium into the compound Mg_2Si ; the quantity required for this purpose is not that given by the formula but is modified by the presence of iron or other impurities. The microstructure of the alloys is described. W. HUME-ROTHERY.

Electrochemical and X-ray studies of lead deposits. P. K. FRÖLICH, G. L. CLARK, and R. H. ABORN (Trans. Amer. Electrochem. Soc. 1926, 49, 239—260. Advance copy).—Lead electro-deposits from acetate, nitrate, perchlorate, fluoborate, and fluosilicate electrolytes, in the presence and absence of free acid and of gelatin, have been investigated by X-ray analysis and potential measurements. The best deposits are given from solutions of perchlorate with excess of both acid and gelatin, when maximum cathodic polarisation, minimum grain size, absence of dendrites, but evidence of preferred orientation were observed. Small grain size is favoured by high acidity and current density and by the presence of gelatin. The effect of the base metal of an iron cathode is small because no penetration takes place, but distinct differences were observed between waxed and unwaxed platinum cathodes where the lead

penetrates the platinum surface. In contrast to other metals, increased current density favours preferred orientation of the crystal grains, and the data indicate that the (211) planes are parallel to the electrode surface. Preferred orientation is favoured by small grain size and hence by the addition of gelatin. Grain size increases with the thickness of the deposit, and hence the outer layers show less preferred orientation than the innermost. The different electrolytes in the presence of free acid but with no gelatin give increasing grain size in the order, fluosilicate, perchlorate, and fluoborate. The polarisation curves indicate that the resistance met with in the cathodic deposition of lead crystals is small. The equilibrium potential of lead in *N*-nitrate solution was determined as -0.158 volt at $24-25^{\circ}$. A Laue diagram of a lead tree or dendrite shows that it is not one single crystal, but is built up in orderly fashion, with a certain amount of internal strain.

W. HUME-ROTHERY.

Reaction $R_mS_n + 2nSO_2 = R_m(SO_4)_n + nS_2$.
Chemistry of the roasting of some ores. J. MILBAUER and J. TUCEK (Chem.-Ztg., 1926, 50, 323—325).—With the exception of mercuric sulphide which volatilises unchanged, the natural and artificial sulphides of the metals react at high temperatures with sulphur dioxide according to the equation $R_mS_n + 2nSO_2 = R_m(SO_4)_n + nS_2$. With sulphides of the alkalis and alkaline earths sulphates are the sole products but with other metal sulphides various side-reactions take place; with the sulphides of copper, lead, bismuth, and antimony large amounts of metal are formed by the interaction of unchanged sulphide with the sulphate first formed. The reaction in the case of the sulphides of aluminium, chromium, zinc, manganese, iron, cobalt, nickel, tin, and cadmium begins only at temperatures at which the sulphate is unstable, so that the final product is the oxide.

A. R. POWELL.

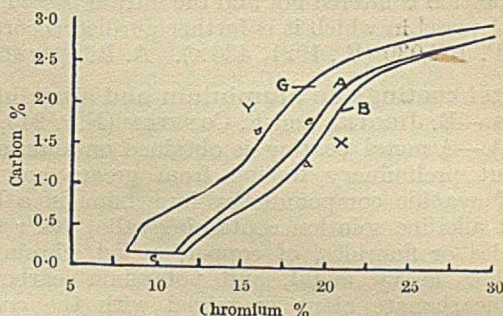
See also A., May, 459, **Determination of orientation of crystallites in metallic conglomerates** (TAMMANN and MÜLLER); **X-Ray investigations of structure of some alloys** (HOLGERSSON). 460, **Crystal structure of Heusler alloys** (HARANG). 461, **Temperature coefficient of magnetic permeability of sheet steel** (SPONER); **Sublimation and crystallisation of metals** (PECZALSKI). 462, **Polymorphism of iron** (BREDEMEIER). 463, **Free energy and heat of formation of iron carbide between 650° and 700°** (MAXWELL and HAYES). 466, **Relation of ferrous metals** (FLETCHER). 474, **Amalgams of gold** (BRITTON and MCBAIN). 475, **System iron-tin** (WEVER and REINECKEN). 482, **Speed of dissolution of copper in aqueous ferric chloride** (BEKIER and TRZECIAK); **Metals and nitric acid** (KLEMENC).

Influence of segregation on corrosion of boiler tubes and superheaters. WOODVINE and ROBERTS.—See I.

PATENTS.

Article [chromium steel] resistant to food acids and process of making it. P. A. E.

ARMSTRONG, Assr. to LUDLUM STEEL Co. (U.S.P. 1,554,615, 22.9.25. Appl., 1.12.22).—Chromium steels containing chromium and carbon corresponding



to the co-ordinates of any point within an area A. bounded by lines, *a, b, c, d* on the graph are hardened and rendered immune to food acids by heating them to $24-300^{\circ}$ above their upper transformation point and cooling rapidly.

C. A. KING.

Protection of metals [iron] against corrosion. I. T. BENNETT, Assr. to T. E. MURRAY (U.S.P. 1,578,254, 30.3.26. Appl., 26.6.24).—The surface of iron or steel is coated first with chromium and then with another protective coating, *e.g.*, nickel.

C. A. KING.

Treatment of ores or other materials with liquids. A. RAMÉN (E.P. 237,571, 18.6.25. Conv., 23.7.24).—For the moistening of roasted ore previous to extraction, the material is introduced in a thin layer on to the bottom of a closed container from a feeding hopper, the outlet of which extends close to the bottom of the container. The mouthpiece of the hopper is capable of rotation so as to distribute the material in a circle on the bottom of the container, whence, after wetting, it is discharged by a scraper through an outlet. The liquor pipe may be fitted inside the feeding hopper and extends nearly to the bottom of the container. In another form the container is provided with a raised conical middle part which is surrounded by a circular gutter. Ore fed in spreads into the gutter in which it is wetted and then is removed by a scraper.

C. A. KING.

Electrolytic production of zinc from ores. S. FIELD, F. PETERSSON, W. E. HARRIS, and METALS EXTRACTION CORP., LTD. (E.P. [A] 249,764 and [B] 249,609, 29.12.24).—(A) Zinc sulphide ores containing iron and manganese are roasted at 650° until the sulphur content is reduced to a point at which steam does not interfere with the oxidation of the sulphur (about 9% S) and the roasting is continued in an atmosphere of steam and air to prevent the formation of ferrites and manganites and to convert the maximum amount of iron into the magnetic oxide. (B) The roasted ore is crushed and introduced, a little at a time, into dilute sulphuric acid at $85-90^{\circ}$ until the acidity, after stirring, is reduced to 0.2%. The hot solution is then completely neutralised with a small amount of high-grade zinc residues and agitated with a small amount of calcium carbonate which flocculates the colloidal silica. To oxidise any

small amount of ferrous sulphate present a quantity of anode mud (chiefly lead dioxide) is then added and agitation continued until all the iron is precipitated. The solution is filtered hot and the filtrate passed to a second vessel in which it is further purified according to E.P. 162,030 (B., 1921, 436 A). A. R. POWELL.

Metal coatings on aluminium and aluminium alloys.—B. JIROTKA and F. CONSTEN (E.P. 249,971, 5.2.25).—A metal coating is obtained on aluminium without preliminary freeing from grease and the use of cyanide compounds by immersion in a bath of hot alkaline solution containing salts, other than cyanides or fluorides, of copper, nickel, chromium, or other heavy metal, and potassium carbonate and bicarbonate alone or mixed with the corresponding sodium salts. Compounds which liberate oxygen, *e.g.*, soluble chromates, may be added to the bath. The addition of 10–20% of glycerin to the bath produces brighter coatings. A suitable bath contains 25 g. of crystalline copper sulphate, 25 g. of potassium carbonate, 25 g. of sodium bicarbonate, and 10 g. of potassium dichromate in 2.5 litres of water. M. COOK.

Light aluminium alloy.—V. E. HYBINETTE (U.S.P. 1,579,481, 6.4.26. Appl., 22.1.25).—An alloy containing more than 90% Al, 0.50 to 3.0% Ni, and from 0.25 to 1.50% of metals of the chromium group is claimed. M. COOK.

Bearing material. H. M. WILLIAMS, Assr. to GEN. MOTORS RESEARCH CORP. (U.S.P. 1,556,658, 13.10.25. Appl., 24.3.22).—A mixture of finely-divided copper 90 pts. and tin 20 pts., together with 6% of graphite, is compressed under a pressure of 40,000–100,000 lb. per sq. in., then packed in lampblack and heated in a sealed container for 5–7 hrs. at 590–760°. The finished bearing metal will absorb up to 8% of oil.

Metallic composition [lead amalgam]. A. MILLER (U.S.P. 1,570,763, 26.1.26. Appl., 19.7.24).—A metallic composition containing 90% Pb and 10% Hg is claimed. It is specially suitable for storage battery plates. It has a greater porosity than lead, is rigid, practically non-corrosive, and has a high electrical conductivity. M. COOK.

Melting metals. Y. A. DYER (U.S.P. 1,578,648, 30.3.26. Appl., 5.5.25).—Carbonaceous material is burned in a combustion chamber in the presence of air which is preheated to 315° but not exceeding 1040° by passing it through pipes which form the dome of the chamber. The resulting hot gases, after mixing with additional air, are introduced at a pressure of $\frac{1}{4}$ to $1\frac{1}{2}$ lb. per sq. in. into a second chamber and through the charge which consists of the metal to be melted and carbonaceous matter in alternate layers. M. COOK.

Metallurgical process [for recovering copper from ores]. W. E. GREENAWALT (U.S.P. 1,579,356, 6.4.26. Appl., 27.1.25).—Roasted copper ore is agitated with an acid solution to extract a portion of the copper, and the rich liquor is separated from

the insoluble residue, which is then treated to separate the slimes from the sand. The copper solution is reduced with sulphur dioxide, treated with copper sulphide precipitated from weak solutions, and finally electrolysed for the deposition of copper and regeneration of acid. This acid liquor is then used for extracting the remainder of the copper from the sands by percolation. A. R. POWELL.

Separation of copper and bismuth from molybdenite. E. POKORNY (G.P. 425,364, 16.6.23).—Molybdenite is treated with chlorine in the presence of water, preferably at a raised temperature, until the copper and bismuth are completely dissolved, or, alternatively, the ore is treated with chlorine until the copper and bismuth particles have been attacked superficially, and is then subjected to a flotation process. L. A. COLES.

Protecting metallic articles against oxidation. BRIT. THOMSON-HOUSTON CO., LTD., Asses. of G. H. HOWE (E.P. 224,534, 5.11.24. Conv., 6.11.23).—See U.S.P. 1,555,578; B., 1925, 996.

Producing an adherent coating of an oxygenous magnesium compound [on magnesium or its alloys]. C. B. BACKER (Reissue 16,340, 4.5.26, of U.S.P. 1,451,755, 17.4.23. Appl., 29.1.25).—See B., 1923, 507 A.

Treating metals to inhibit excessive grain growth. G. L. KELLEY, Assr. to E. G. BUDD MANUF. Co. (U.S.P. 1,581,269, 20.4.26. Appl., 23.4.24).—See E.P. 248,801; B., 1926, 411.

Treating impure molten metals. H. HARRIS (U.S.P. 1,582,037—8, 27.4.26. Appl., 26.4.24).—See E.P. 217,391; B., 1924, 679.

Sulphating process for ores and concentrates. J. B. READ and M. F. COOLBAUGH, Assrs. to COMPLEX ORES RECOVERIES Co. (U.S.P. 1,582,347, 27.4.26. Appl., 26.11.19).—See E.P. 200,852; B., 1923, 1077 A.

Magnetic alloy. W. S. SMITH, N. POPPLEFORD, and H. J. GARNETT (U.S.P. 1,582,353, 27.4.26. Appl., 2.5.25).—See E.P. 224,972; B., 1925, 76.

Method of coating vessels [with an alloy]. M. DREIFUSS (U.S.P. 1,582,668, 27.4.26. Appl., 14.11.23).—See E.P. 220,791; B., 1924, 875.

Method of treating aluminium-silicon alloys. A. G. C. GWYER and H. W. L. PHILLIPS, Assrs. to BRITISH ALUMINIUM Co., LTD. (U.S.P. 1,583,549, 4.5.26. Appl., 9.6.25).—See E.P. 219,346; B., 1924, 793.

Modifying ignition temperature of carbonaceous materials (U.S.P. 1,576,179).—See II.

High-frequency electric furnaces (E.P. 245,414).—See XI.

Arc welding electrodes (U.S.P. 246,140).—See XI.

XI.—ELECTROTECHNICS.

Insoluble anodes for the electrolyses of brine.

C. G. FINK and L. C. PAN (Trans. Amer. Electrochem. Soc., 1926, 49, 183—228. Advance copy).—Graphite, platinum, magnetite, carborundum, ferrosilicon, lead peroxide, manganese dioxide, and natural anthracite were found to be not entirely satisfactory in use. Although the rate of anodic corrosion of pure silver, lead, or mercury is very high, the rate and with it the cell voltage drop instantly upon the addition of a small percentage of silver to lead or *vice versa*. A silver-lead anode containing 61% Ag best fulfils the requirements of an anode material for the electrolysis of brine. The sharp reduction in anodic corrosion is due to the formation of a protective film over the surface of the anode. With a properly formed film, the anode does not appreciably corrode or lose weight in the anolyte, nor is it attacked by chlorine providing the cell is not on open circuit. The silver-lead alloy has a low electrode potential and low chlorine overvoltage, is easily cast and machined, is mechanically strong, ductile, malleable, and has a low melting point. The anodes even when dissolved, do not contaminate the products of the brine cell. They have a high specific electrical conductivity compared with graphite and can be "burned" or welded so as to eliminate a contact resistance entirely. Their initial cost is higher than that of graphite anodes but their maintenance cost is lower than for either the graphite or platinum anodes used in some types of cells. A small amount of mercury or manganese in silver-lead anodes increases the corrodibility of the anode in brine solutions. An alkali-chlorine cell using a silver-lead anode is described. J. S. G. THOMAS.

Simple method of measuring polarisation and resistance [of electrolytes].

H. E. HARING (Trans. Amer. Electrochem. Soc., 1926, 49, 31—46. Advance copy).—The polarisation at an electrode during electrodeposition may be measured by determining the potential difference between the electrode and an auxiliary electrode of the same metal in the same electrolyte. The auxiliary-electrode vessel consists of a glass tube with a capillary extension which is bent at right angles near one end. The capillary tip is either kept in contact with the main electrode or else kept at a constant distance; in each case a correction for the potential drop is necessary, though in the former arrangement its magnitude is uncertain. A rod of the metal dips into the tube; replacement of the rod by a gauze spiral or the introduction of a high resistance in the auxiliary circuit eliminates any small polarisation which may be produced at the auxiliary electrode. A permanent cell for polarisation and resistance measurements is described. The cathode and anode are placed at the ends of a rectangular vessel which is divided into three equal compartments by means of rectangular partitions of coarse gauze. The latter are shown to act as auxiliary unpolarised electrodes and the above considerations apply. A measurement of the poten-

tial drop between the partitions (with a potentiometer or a high-resistance voltmeter, the maximum reading always being taken) enables the resistance of the electrolyte to be calculated, the current being known. The method was tested with three typical plating solutions; the accuracy approached that of the usual methods. The results of the tests are represented graphically. A device by means of which a calomel electrode can be used as the auxiliary electrode is described. S. K. TWEEDY.

Secondary reaction in the lead accumulator.

C. FÉRY with C. CHÉNEVEAU (Bull. Soc. chim., 1926, [iv], 39, 603—612).—See B., 1925, 997; 1926, 412.

Influence of alternating currents on electrolytic corrosion of iron. A. J. ALLMAND and R. H. D. BARKLIE (Trans. Faraday Soc., 1926, 22, 34—45).—See B., 1926, 277.

See also A., May, 479, Theory of overvoltage (KNOBEL); Electrolysis of acid solutions of copper sulphate (BURT-GERRANS), 483, Electrolytic separation of metals (BROWN); Electrolysis of electrolytes containing cuprous chloride (HÄNSEL). 509, Electrochemical oxidation of aromatic hydrocarbons containing nuclear chlorine (FICHTER and ADLER).

See also pages 475, Oils for transformers etc. (BAUM). 485, Nitrogen fixation (TARTAR and PERKINS). 494, Conductivity of electrolytes (COLCORD and others); Silver-gold parting (COLCORD); Electrodeposition of zinc (FRÖLICH). 503, Dielectric constant etc. of rubber and guttapercha (CURTIS and MCPHERSON).

PATENTS.

High-frequency electric furnaces. WESTINGHOUSE LAMP CO., Asses. of H. C. RENTSCHLER and J. W. MARDEN (E.P. 245,414, 3.1.25. Conv., 5.1.25).—In a high-frequency electric induction furnace for heating rare metals, a support consisting of a crucible or disc for the material to be heated is located within an induction coil, the support being composed of a rare metal compound, *e.g.* thoria, which does not dissociate at high temperatures and which will not contaminate the metal being heated.

J. S. G. THOMAS.

High-frequency dielectric and magnetic furnace.

C. T. ALLCUTT, Assr. to WESTINGHOUSE ELECTRIC AND MANUF. CO. (U.S.P. 1,572,873, 16.2.26. Appl., 29.12.23).—The furnace consists of a crucible, surrounded by a coil connected to a source of high-frequency alternating current, and placed between two electrodes also connected to a source of high-frequency alternating current. It is used to heat substances, such as metal oxides, which have a relatively high negative temperature coefficient of resistivity. The substance is first heated by the action of the alternating electric field set up by current applied to the electrodes, and when sufficiently hot to have its initial resistivity greatly reduced it is rapidly heated by the alternating electromagnetic field generated by passing an alternating current through the coil. If the substance to be heated is

a conductor even when cold, the crucible is made of an imperfect dielectric, such as glass or quartz, and is initially heated by the electric field. The apparatus is also of value in heating articles such as vacuum bulbs which comprise a metallic conductor and an imperfect dielectric such as glass. The former is heated by the electromagnetic field and the latter by the electric field. T. S. WHEELER.

Electric rotating resistance furnace. F. ANDERSEN, Assr. to TROLLHÄTTANS ELEKTROTHERMISKA AKTIEBOLAG (U.S.P. 1,576,621, 16.3.26. Appl., 22.3.23).—In a resistor for electric furnaces, the specific electrical resistance of the material constituting the current-supplying parts is lower than that of the material constituting the intermediate part of the resistor, in the proportion of at least 1 : 2. M. E. NOTTAGE.

Arc-welding electrodes. BRITISH THOMSON-HOUSTON Co., Assees. of J. M. WEED (E.P. 246,140, 12.1.26. Conv., 13.1.25).—For use with an iron electrode for arc welding, a flux containing sodium titanate and finely-divided metallic zinc is claimed. The method of compounding the electrode may vary, e.g., the iron core may be galvanised and then coated with a flux containing sodium titanate, or an iron core may be inserted in a galvanised iron sheath of higher m.p. than the core, the intervening space being occupied by the flux. C. A. KING.

Separating light materials from gases. H. A. WINTERMUTE, Assr. to RESEARCH CORP. (U.S.P. 1,579,462, 6.4.26. Appl., 11.2.25).—A gas stream is subjected to the action of an electric field, the velocity of the stream being such that masses of material deposited from the gas by the field are detached from the walls of the conduit in agglomerated form and carried forward by the stream. J. S. G. THOMAS.

Electrical purification of gases by means of alternating current. H. ROHMANN, and ELEKTRISCHE GASREINIGUNGS-GES.M.B.H. (G.P. 425,026, 30.10.21).—In plant for the electrical purification of gases by means of alternating current, in which dust particles carried by the gas stream are first electrically charged by passage between high-tension discharge electrodes and subsequently precipitated on electrodes at lower potential, a phase displacement of such magnitude as to produce the maximum efficiency of precipitation is introduced between the respective potentials employed. J. S. G. THOMAS.

Electrical purification of gases. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 425,039, 15.10.22).—Gas is caused to flow helically and turbulently through a tube wherein it is subjected to the high-tension discharge from an upright conductor arranged along the axis of the tube. The direction of rotation of the gas in the tube is chosen to correspond with the direction of electric lines of force surrounding the conductor. Each period of gas purification is followed by a period during which dust particles clinging to the

sides of the tube are removed by means of a sand blast blown into the tube so as to circulate in the same direction as the gas stream. If desired, dried sand or similar material may be added to the gas during the process. J. S. G. THOMAS.

Discharge electrode for electrical gas purification. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 425,273, 29.8.23).—A discharge electrode for use in the electrical purification of gases containing corrosive material is composed of a fused mass of glass, porcelain, quartz, white ware, or similar material together with electrically conducting bodies, e.g. metals. Alternatively a non-conducting core may be coated with this fused mass. J. S. G. THOMAS.

Plant for electrical precipitation. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 425,274, 15.9.23).—Discharge electrodes of U-cross-section are arranged above the upper ends of tubular precipitating electrodes which are inclined to the horizontal, and are so shaped that the discharge is effective only in the direction of apertures provided in the upper sides of the tubes, whilst a pure electrostatic field is produced in the opposite direction. The precipitating electrodes are provided with valves for discharging dust from the tubes to a collecting chamber containing gas for the combustion of the precipitated material. J. S. G. THOMAS.

Electrode for use in the electrolytic evolution of gases. E. HEINZE, Assr. to FARBENFABR. VORM. F. BAYER & Co. (U.S.P. 1,575,627, 9.3.26. Appl., 10.12.24).—An electrolytic cell for the production of gases contains a pair of electrodes in gas-tight spaced relation, one of which has within it a gas-collecting chamber and passages leading thereto from the space between the electrodes. M. E. NOTTAGE.

Purification of filter diaphragms in electrolytic processes. SIEMENS & HALSKE, Assees. of A. WEYL (G.P. 425,275, 3.12.24).—At intervals of time a cleansing solution comprising a solution of an electrolyte and an acid or alkaline solution is introduced in the neighbourhood of the diaphragm, the current strength being suitably reduced and the strengths of the acid or alkaline solution added so adjusted that the alkali or acid concentrations resulting from electrolysis at the cathode or anode are neutralised, or the solutions in these regions remain slightly acid or alkaline. J. S. G. THOMAS.

Filament [for electric lamps] and method of manufacture thereof. G. R. FONDA, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,565,724, 15.12.25. Appl., 29.7.20).—See E.P. 182,699; B., 1922, 673 A.

Coating [electric] incandescent lamp bulbs. J. B. WHITMORE and J. E. FERGUSON, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,581,766, 20.4.26. Appl., 19.3.21).—See E.P. 230,643; B., 1925, 402.

Quartz [mercury vapour] lamp. K. MENSING (U.S.P. 1,582,849, 27.4.26. Appl., 24.8.21).—See E.P. 165,081; B., 1922, 742 A.

Production of compressed gases by electrolysis. P. HAUSMEISTER (U.S.P. 1,581,944, 20.4.26. Appl., 17.11.23).—See E.P. 228,231; B., 1925, 249.

[Self-baking] electrodes for electric furnaces. B. E. D. KILBURN. From DET NORSKE AKTIESELSKAB FOR ELEKTROKEM. IND. (E.P. 235,894, 5.1.25).

Emulsified solid greases (E.P. 232,259).—See II.

Lead amalgam (U.S.P. 1,570,763).—See X.

XII.—FATS; OILS; WAXES.

Identification of rapeseed oil by isolation of erucic acid. A. W. THOMAS and M. MATTIKOW (J. Amer. Chem. Soc., 1926, 48, 968—981).—The oil (10 g.) is hydrolysed by a mixture of 50 c.c. of alcoholic 5% potassium hydroxide and 50 c.c. of 95% alcohol, the warm soap solution made neutral to phenolphthalein, and alcoholic potassium hydroxide added to restore a permanent pink colour. 25 c.c. of alcoholic magnesium acetate solution (50 g. dissolved in 100 c.c. of water and the solution diluted with 3 vols. of 95% alcohol) are added, the mixture is boiled, then kept at 10° overnight. The insoluble soaps are filtered off and washed with 50 c.c. of 95% alcohol, then washed back into the original flask with hot 5*N*-hydrochloric acid, decomposed by boiling for 10 min., and cooled. The solidified acids are filtered off, washed free from chloride and magnesium, transferred to a 150 c.c. beaker by means of warm 90% alcohol, and kept at 10° overnight. Crystals of saturated acid are then filtered off, and the filtrate is collected in a weighed 150 c.c. beaker. The solvent is slowly evaporated at 60—70°, drying being completed at 60° under reduced pressure. The iodine value, m.p., and mol. wt. of the acid are then determined. Pure rapeseed oil gives in this way about 44% of erucic acid, m.p. about 26°, iodine value about 73, mol. wt. 336. A confirmatory test consists in dissolving the acid in 80—100 c.c. of 90% alcohol, adding 2 c.c. of 1% palladous chloride solution and 0.5 c.c. of 1% gum arabic solution, and bubbling hydrogen through the mixture for about 3 hrs. The mixture is kept at 25° overnight, the precipitate collected, washed with cold 90% alcohol, and dissolved in hot 95% alcohol into a weighed beaker, in which the solvent is evaporated and the product dried at 80°. Pure rapeseed oil gives in this way about 33% of behenic acid, m.p. 77—79° after two recrystallisations. No "erucic acid product" is obtained by the above method from pure olive, cottonseed, linseed, soya bean, perilla, maize, wild mustard, or peanut (*arachis*) oils. The amount of erucic acid obtained from mixtures of rapeseed and olive oils gives a rough indication of the proportions of the two oils in the sample. Results obtained with mixtures of linseed and rapeseed oils indicate that the presence of large proportions of unsaturated acids interferes with the precipitation of magnesium erucate. Since erucic acid is a solid acid of low m.p., the "solid acids" of an oil will, in presence of rapeseed oil, show lower m.p., and data are given for the m.p. of the solid acids obtained

from mixtures of olive and cottonseed oils with rapeseed oil. The above method does not distinguish rapeseed from mustard seed oil, as the latter yields a large proportion of erucic acid. F. G. WILLSON.

Method of Bertram, Bos, and Verhagen for the determination of coconut oil and milk fat. C. BAUMANN, J. KUHLMANN, and J. GROSSFELD (Z. Unters. Lebensm., 1926, 51, 27—31; cf. B., 1926, 165).—The "A" and "B" numbers of some commercial milk-products and mixtures of coconut oil, milk fat, and cacao butter have been determined. While the method is less simple than the Reichert-Meissl or Polenske methods, it affords a clearer insight into the composition of the fats and is especially valuable for the determination of coconut oil in cacao butter. In the examination of milk products it is useful in detecting substitution of skim-milk powder for whole-milk powder or cream. The effects of variation in the conditions of experiment are discussed and the original method has been considerably simplified. E. H. SHARPLES.

Distillation of coconut oil at very low pressures. H. I. WATERMAN and H. J. RIJKS (Z. Deuts. Oel- u. Fett-Ind., 1926, 46, 177—178).—The distillation is carried out in a glass flask, high vacuum being maintained by a Langmuir mercury pump as an auxiliary to an ordinary pump. Between the two pumps were two flasks containing charcoal cooled in liquid air. Between the ordinary receiver and the pump was an auxiliary receiver cooled in liquid air, followed by a vessel containing phosphorus pentoxide. Distillation temperatures were measured by thermo-elements. A vacuum of 0.0023—0.0034 mm. was maintained, as recorded by a MacLeod manometer. During distillation no ordinary ebullition was observed. Four fractions were collected, viz., 208—218°, 218—223°, 223—232°, and 232—259°, and amounted to 96.5% of the total. The refractive index, acidity, saponification value, and iodine value of the original material, calculated from that of the fractions, agreed closely with the actual observed figures, indicating absence of decomposition. It is suggested that by such methods of distillation it may be possible to separate the individual glycerides in fats. A. RAYNER.

Determination of the bromine value of fats. E. RUPP and W. BRACHMANN (Z. anal. Chem., 1926, 68, 155—160).—To reduce the loss of bromine by evaporation in Winkler's bromide-bromate method of determining the bromine value of fats (B., 1924, 755) a large excess of potassium bromide should be present in the solution. A suitable standard solution consists of 5.5674 g. of potassium bromate dissolved in 1 litre of 20% potassium bromide solution. The excess of bromine is preferably determined by adding 0.3—0.5 g. of potassium iodide and titrating the liberated iodine with thiosulphate. Substitution of a 0.1*N*-solution of bromine in carbon tetrachloride or chloroform containing hydrogen chloride for the aqueous bromide-bromate mixture yields low results, probably because water acts as a catalyst in the latter case. A. R. POWELL.

Theory of the practice of steam deodorisation of saponifiable oils. W. BRASH (J.S.C.I., 1926, 45, 73—75 τ).—The object of steam deodorisation of saponifiable oils is the removal of those substances giving rise to taste and odour; they are neutral compounds and in coconut and palm kernel oils have been found to consist largely of the higher aliphatic ketones. The theory of the process is developed and, with certain assumptions, it is shown that the concentration, x , of the odoriferous matter in the oil after deodorisation for a time, t , is given by the equation $x = a e^{-Kkt}$ where a is the original concentration of the odoriferous matter, and K and k are constants. Formulæ are given for the design of suitable apparatus and for calculating the pressure and temperature of the entering steam.

Bleaching of wool grease. I. LIFSCHÜTZ (Chem.-Ztg., 1926, 50, 245—246).—The bleaching of wool grease on exposure to air and light is due to oxidation of the products of hydrolysis (alcohols and fatty acids) of the esters, the undecomposed esters not being bleached. This principle has been applied on a large scale in conjunction with the process described in G.P. 324,667 (B., 1920, 790 A) for bleaching wool grease. The sodium soap of the completely saponified grease, pressed into thin flakes, was exposed to sunlight in glass boxes. After 3—4 days the dark brown soap flakes had become bleached to a pale yellow. If the grease was only partly saponified with lime the results were much inferior. The process is stated to be applicable on a large scale to all grades of wool grease, and the sodium soaps may be used for many industrial purposes. They are particularly suitable as additions to toilet soaps, as in virtue of the nature of the unsaponifiable matter present the lathering and detergent properties of the soap are improved. A. RAYNER.

Polymerisation of fatty oils. V. J. MARCUSON (Z. angew. Chem., 1926, 476—478; cf. B., 1925, 889).—Linseed oil films contain about 55% of solid polymerised and oxidised gel and 40% of polymerised and oxidised sol with 5% of unchanged oil. Poppy-seed oil films contain 51% of insoluble gel (mol. wt. 707), 31% of easily gelatinised sol containing 40% of unpolymerised fatty acids and a variable amount of acids of mol. wt. 420, and 18% of unchanged oil. The acetone-insoluble gel of tung oil films (83%) contains a unimolecular, oily product soluble in furfuraldehyde and having a mol. wt. of over 100 by Rast's camphor method. During the drying of tung oil, isomerisation due to the action of light first takes place, followed by polymerisation and oxidation. The halogens and sulphur have a similar action to light in that at 10° they precipitate β -elæostearin and at room temperature a solid polymerisation product. For the detection of tung oil in mixtures 5 g. of the oil are shaken with 5 c.c. of a cold saturated solution of iodine in chloroform; with high percentages of tung oil a gelatinous mass is obtained but with 15—20% of tung oil the mixture must be warmed on the water-bath. No other fatty oil behaves similarly. A rough quantitative test may be made by extracting the reaction product

with chloroform in Graefe's apparatus, removing the iodine by shaking with thiosulphate, distilling off the chloroform, drying, and weighing.

A. R. POWELL.

See also A., May, 471, **Viscosity and elasticity of soap solutions** (FREUNDLICH and JORES). 481, **Partial saponification of mixed glycerides** (TREUB). 498, **Chinese wood (tung) oil** (NAGEL and GRÜSS).

Vitamin-A in oleo oil and oleo stearin. HOAGLAND and SNIDER.—See XIX.

PATENTS.

Simultaneous purification and deacidification of oils and fats. KUNEROLWERKE E. KHUNER & SOHN A.-G. (Austr. P. 96,831, 13.11.22).—The oil or fat is treated with oxidising agents having a neutralising action, especially sodium peroxide, which can be used in solution. S. S. WOOLF.

Emulsified solid greases (E.P. 232,259).—See II.

Gilsonitic products (U.S.P. 1,573,764—5 and 1,578,235).—See II.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Chrome yellow problems. H. WAGNER and E. KEIDEL (Farben-Ztg., 1926, 31, 1567—1573).—Many precipitations of pure lead chromate and of various mixtures of lead sulphate and chromate were carried out with variations in the temperature, concentration, agitation, precipitants used, etc. By sedimentation experiments and microscopical examination the state of dispersion of the product was shown to be dependent on its solubility in the mother liquor. This in turn is controlled by the salts used and the practical conditions in general, concentration being the most important variable. In the case of chrome yellow containing lead sulphate the existence of mixed crystals was recognised and their bearing on the tone, opacity, etc. of the pigment is discussed. The optimum conditions deduced from this investigation agree with those empirically found to be practical for any desired type of chrome yellow. S. S. WOOLF.

Photometric method for measuring the hiding power of [light-coloured] paints. H. D. BRUCE (U.S. Bur. Standards Tech. Papers, 1926, 20, [306], 173—190).—The paint under test is poured on to a rotating circular glass disc—half black and half white—and allowed to dry, producing a dried film tapering from a slight peak in the centre to a minimum at the outer edges. The relative brightness ("contrast ratio") of the two halves of the plate for different thicknesses of the covering film is measured by a Martens photometer (in which the two fields are brought to equal brightness by means of a Nicol prism; cf. Physikal. Z., 1900, 1, 299). Film thickness is obtained by the use of an Ames dial, a reading being taken on the film, which is then removed with a penknife and a second reading taken

on the bare plate. The minimum thickness of film (x) that will hide "completely" (except for personal equation) is computed from the formula $x = 7a\sqrt{1/b-1}$, where a is the measured film thickness and b the measured contrast ratio. This formula is a simplified form of the functional relationship developed in the paper. In addition, the hiding power may readily be expressed in square feet per gallon. S. S. WOOLF.

Adsorption of soluble salts by paint films. H. WOLFF and G. ZEIDLER (*Korrosion u. Metallschutz*, 1925, 1, 211—213; *Chem. Zentr.*, 1926, I., 2746).—Sodium chloride can readily be dissolved out of films of oil paints containing, in addition to the salt, zinc oxide, lithopone, and red oxide of iron respectively, but corresponding films containing white lead or iron-mica retain the salt to a considerable extent. The interfacial tension relationships in the system pigment, salt, oil, and water must be such as to permit adsorption in the last two cases. If concentrated sodium chloride solution be stirred into the paint, instead of incorporating the dry salt, the washing out proceeds more rapidly owing to water retained in the film assisting diffusion. Rain cannot completely wash salt out of iron-mica films. S. S. WOOLF.

Natural and artificial resins. J. SCHEIBER (*Farbe u. Lack*, 1926, 76—78, 87—89, 99—101, 113—114).—An account of the published work on various types of resins, natural and synthetic, in use in the paint and varnish as well as other industries, mainly from theoretical and constitutional standpoints. Under the heading natural resins, Tschirch's general classification, rosin, copals, resin-oil interactions, shellac, and Japanese lacquer receive attention, while under artificial products a short account of resin formation and "resinophore groups" is followed by discussion of polymerides of coumarone, indene, and aldehydes, and condensation products of phenol or carbamide with formaldehyde and the many similar patented products. S. S. WOOLF.

Frosting of tung oil films. The tung oil phenomenon. L. AUER (*Farben-Ztg.*, 1926, 31, 1625—1627).—At least three theories have been advanced to explain the characteristic frosted appearance of tung oil films dried under ordinary atmospheric conditions. According to Marcusson and others, the separation under the influence of light of crystals of β -elæostearin, the solid isomeride of α -elæostearin, which comprises most of the original film, is responsible for the opacity of the dried film, but macro- and microscopical examinations (as well as analyses by recent workers) show the absence of these crystals and establish wrinkling as the cause. When the puckers are sufficiently fine, diffraction and iridescence occur. Wolff, Schmidt, and others attribute this wrinkling to internal strain of the colloid system, set up by swelling and shrinking with absorption and disengaging of moisture, as evidenced by the possibility of obtaining clear films in dry atmospheres. The author now shows that non-frosted films may be obtained in desiccators

where water exchanges are proved to be proceeding, and affirms that ultra-violet light is the deciding, if not the only, cause of the frosting. The glass of which the desiccators used are made absorbs ultra-violet light almost completely, but clear films taken out of the desiccator at a not too advanced stage in the drying and exposed to sunlight, rapidly develop the usual markings. It is denied that frosted films can be obtained in the complete absence of light as has been claimed by some workers. S. S. WOOLF.

Ester gum [rosin glyceride] and the chemical reactions in rosinat varnish preparation. H. WOLFF (*Farben-Ztg.*, 1926, 31, 1573—1574).—The slight sediment in a 50% amyl acetate solution of commercial ester gum after keeping for 3 months, was found on recrystallisation etc. to be pure glyceryl triabietate. By using these crystals to seed an ester gum solution in ethyl acetate (33%) further quantities were obtained. This separation throws light on the exchanges between fatty acid glycerides and metal rosinates in varnishes, as a sample of triabietin, identical with the above, was isolated from a linseed oil solution of lead rosinat, thus confirming the earlier views of Wolff and Dorn (cf. B., 1921, 780 A, 857 A). The saponification value and molecular weight etc. of a secondary product obtained from the triabietin mother liquor agree with the values for an impure abietic anhydride. This, it is suggested, is derived from the lead rosinat by the splitting off of litharge which then by saponifying the oil glycerides frees glycerol residues to form triabietin with the abietic anhydride. S. S. WOOLF.

U.S. Government master specification for shellac varnish. (U.S. Bur. Standards Circ. 303, Feb. 9, 1926, 7 pp.).—Two types each of orange and bleached shellac varnish are specified, and each type may be provided at light, medium, or heavy body. The requirements for the non-volatile matter in the varnishes are as follows:—Maximum iodine value for orange, type I, 18.0, type II, 24.5, bleached, types I and II, 10.0. Maximum percentage of insoluble matter in hot 95% alcohol, orange type I, 1.75, type II, 3.00, bleached type I, 1.00, type II, 0.10. Maximum percentage of wax, orange types I and II and bleached type I, 5.50, bleached type II, 0.20. Maximum ash, orange types I and II and bleached type I, 1.00, bleached type II, 0.30. The colour should be no darker than that of samples mutually agreed on by buyer and seller. The body of the varnishes is controlled by minimum specified percentages of non-volatile matter, viz: for orange types I and II, light 33.4, medium 36.4, and heavy 39.1. For bleached types I and II, light 32.3, medium 35.3, and heavy 37.9. Details of reagents and manner of testing are given. S. S. WOOLF.

Polymerisation of fatty oils. MARCUSSON.—See XII.

PATENTS.

Manufacture of a substance for impregnating materials. NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB (E.P. 239,500, 15.8.25. Copy.



2.9.24).—The impregnating substance made by incorporating oil and sulphur with pitch, according to E.P. 154,570 (B., 1921, 885 A) is improved if bitumen produced from raw petroleum with an asphalt basis is the particular pitch substance used. A typical product is obtained by heating 100 pts. of castor oil with 30 pts. of sulphur for $\frac{1}{2}$ hr. at 200°, and after adding 150 pts. of the petroleum pitch, maintaining the whole at 170° for 1 hr., stirring throughout.
S. S. WOOLF.

Fluxed resinous composition. C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,579,195, 30.3.26. Appl., 17.12.24).—Hexamethylenetetramine is added to resinous condensation products of the type ordinarily hardened by hexamethylenetetramine in the hot press to delay their too rapid setting and hardening in the mould, thus improving the finish of moulded articles.
S. S. WOOLF.

Production of resinous products from crude anthracene and phenanthrene. BAKELITE G.M.B.H., and M. FLORENZ (G.P. 420,443, 30.12.22).—Anthracene and phenanthrene or their mixtures are converted into addition products and the latter resinified by heating, if necessary, with aldehydes, in the presence of condensing agents (sodium hydroxide, ammonia, ferric or aluminium chloride, etc.). The addition products are obtained by the action of halogens, picric acid, nitric acid or substances yielding nitrous fumes, such as nitrosyl chloride. For example, crude anthracene is suspended in carbon disulphide or benzene and saturated with chlorine, the medium evaporated at 100°, and the residue heated to melting above 100° till no more anthraquinone distils and the mass solidifies to a lustrous black resin. These resins, m.p. 80–90°, are soluble in benzene and halogenated hydrocarbons, and are used in the lacquer industry, for impregnating wood, and for insulation etc.

B. FULLMAN.

Preparation of artificial resins. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of A. VOSS (G.P. 422,910, 30.7.21).—Natural resins are fused with synthetic resinous products containing free carboxyl groups. If oxides, hydroxides, or carbonates of the alkaline-earths are included in the melt, the products have much higher softening points than those obtained by fusion of natural resins or their salts with phenol-aldehyde condensation products. German spruce resin, on fusion with the resinous product from salicylic acid and formaldehyde, first at 100–120°, and then at 190–200°, gives a resin having a softening point at least 30° above that of spruce resin. Colophony and the condensation product of phenoxyacetic acid and formaldehyde yield a product suitable for lacquers and for electrotechnical uses.
B. FULLMAN.

Distillation of natural resins and oleoresins. J. M. A. CHEVALIER, P. BOURCET, and H. REGNAULT (U.S.P. 1,572,766, 9.2.26. Appl., 17.5.22).—The resin or oleoresin is mixed with 1–5% of a tribasic acid, e.g., phosphoric acid, and distilled to 310°. About 50% of the resin distils and is obtained in

the form of a light oil useful as a substitute for turpentine and as a carburetting agent for alcohol. It contains a small proportion of phosphoric acid and water which is readily separated. The residue in the still consists of an odourless neutral resin oil of low viscosity.
T. S. WHEELER.

Manufacture of [resinous] phenolic condensation products. H. WADE. From S. KARPEN AND BROS. (E.P. 245,703, 23.10.25).—See U.S.P. 1,566,817; B., 1926, 202.

Polyhydric alcohol-organic acid resinous condensation products. J. G. E. WRIGHT, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,581,902, 20.4.26. Appl., 5.7.24).—See E.P. 236,591; B., 1925, 770.

Manufacture of resinous products of condensation from phenol and formaldehyde. C. KULAS and C. PAULING (U.S.P. 1,582,056, 27.4.26. Appl., 13.8.21).—See E.P. 191,417; B., 1923, 235 A.

Production of hard resinous bodies. E. SCHAAL (U.S.P. 1,583,014, 4.5.26. Appl., 25.8.24).—See E.P. 243,556; B., 1926, 137.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Physical properties of rubber. I. Influence of high temperature on the stress-strain curve of vulcanised rubber. A. VAN ROSSEM and H. VAN DER MEYDEN (J.S.C.I., 1926, 45, 67–72 T).—Tensile tests at high temperatures were carried out with the dynamometer of Schopper by heating the pulleys supporting the rings in an oven at the temperature required and quickly mounting them. Subsequently the ring, which is heated under mercury at the temperature required, is quickly mounted and the tensile test is immediately carried out. Systematic experiments with vulcanised rubber (rubber 92½ pts., sulphur 7½ pts.) showed that with rising temperature the tensile curves shift towards the elongation axis. When the rings are heated for a longer time under mercury the rubber becomes brittle but the brittleness disappears after cooling at ordinary temperature. The tensile curve of the rubber after heating and subsequent cooling coincides with the original stress-strain curve of the rubber. The time of heating necessary for the appearance of brittleness depends largely on the vulcanisation coefficient and the temperature of heating. The change in tensile properties during vulcanisation has been studied by vulcanising rings in special moulds and immediately testing them on pulleys heated at 147°. The tensile properties of the rubber during vulcanisation are entirely different from those after cooling at ordinary temperature, the tensile strength being very low during vulcanisation and the rubber soon becoming brittle.

Commoner mineral ingredients for rubber. D. F. TWISS and E. A. MURPHY (J. S. C. I., 1926, 45, 121–123 T).—Progressive vulcanisation of mixtures of rubber and sulphur with various compounding ingredients shows that, apart from reinforcing effect, these may exert a distinctly divergent effect on the rate of vulcanisation.

Factice-like product obtained by the action of the silent discharge on oil ("Volfactice"). L. HOCK (Kautschuk, March, 1926, 65—67).—In the conversion of mobile oils such as rape oil into more viscous "volf oils" by submission to the silent electric discharge in an atmosphere of hydrogen or other inert gas an insoluble deposit of "volfactice" is also obtained. This is a yellowish spongy mass of fatty odour, resembling white factice (oil rubber-substitute) in appearance. Its mixtures with rubber are inferior to similar mixtures with dark or light rubber substitute both in cold and hot vulcanisation, particularly the latter. D. F. TWISS.

Effect of accelerated ageing upon some physical properties of hard rubber compounds [vulcanites]. E. O. DIETERICH and H. GRAY (Ind. Eng. Chem., 1926, 18, 428—430).—Of the three physical characteristics, softening temperature, transverse strength, and impact strength, the last is the most promising as an index of the changes in vulcanite on ageing. At 70° the physical properties undergo little change even in 14 days, but at 150° deterioration is rapid. The use of vulcanisation accelerators or age-retarders has little influence, but under-vulcanisation favours deterioration. Heavily loaded stocks show a lower percentage alteration than simple vulcanites prepared from rubber, sulphur, and an accelerator. D. F. TWISS.

Chemical unsaturation of rubber under the action of heat, trichloroacetic acid, ultra-violet light, and mastication. H. L. FISHER and A. E. GRAY (Ind. Eng. Chem., 1926, 18, 414—416; cf. Staudinger, B., 1926, 282).—At 340° rubber yields a powdery product of a much lower degree of unsaturation; at 141°, however, even after 8 hrs. there is no decrease in the unsaturation towards Wijs' solution. Rubber, in which 10% of trichloroacetic acid has been incorporated, shows a fall in its unsaturation value after 7 days. Ultra-violet light does not cause any alteration in the unsaturation of rubber solution in an atmosphere of carbon dioxide. Mastication of rubber lowers the degree of unsaturation probably on account of oxidation; with exclusion of air the unsaturation is not affected. D. F. TWISS.

Dielectric constant, power factor and resistivity of rubber and gutta-percha. H. L. CURTIS and A. T. MCPHERSON (U.S. Bur. Standards Tech. Papers, 1925, 19, [299], 699—722).—Well-washed rubber has a dielectric constant (2.35) lower than that of the purest gutta-percha examined (2.56), and the values are more variable for gutta-percha. Vulcanisation increases the dielectric constant and the power factor of rubber, but the use of accelerators results in lower values than are obtained on vulcanisation with sulphur alone; the resistivity is little affected by vulcanisation. Softeners such as ozokerite, vaseline, beeswax, palm oil, and stearic acid have little influence on the dielectric constant of rubber, but coumarone resin and large proportions of "mineral rubber" cause a slight increase; with the exception of ozokerite, they all increase the power factor considerably. Mineral fillers generally in-

crease the dielectric constant of rubber, the increase, however, being relatively slow with increasing proportions of quartz powder, which is also exceptional in actually reducing the power factor; the resistivity is not greatly influenced by moderate proportions of most fillers. Carbon black, however, is exceptional in the degree of its effect in increasing the dielectric constant and power factor and reducing the resistivity. Absorption of water increases the dielectric constant of rubber (and gutta-percha) and the power loss and decreases the resistivity; latex-sprayed rubber, on account of its content of soluble matter, exhibits a much higher water absorption than pale crêpe rubber. D. F. TWISS.

PATENT.

Impregnation of wood (Austr. P., 100,729).—See IX.

XV.—LEATHER; GLUE.

Possible explanation of the antagonistic action of neutral salts upon hide substance. K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1926, 21, 206—216).—The explanation is based on the secondary valency concept. Where the destructive action of neutral salts upon hide substance prevails, there is a tendency to the formation of neutral salt compounds similar to the glycine-calcium chloride compounds described by Pfeiffer ("Org. Molekülverbindungen, Stuttgart," 106—124). The extent of this peptisation depends upon the magnitude and stability of the secondary valency between the salt and protein micelles. A weakening and partial breaking up of the intermolecular forces occur by this combination and part of the hide is transformed into micelles or complexes of molecular disperse order. D. WOODROFFE.

Action of chromium salts on permutit. Indirect proof of the chemical nature of one-bath chrome tanning. K. H. GUSTAVSON (Collegium, 1926, 97—124).—The behaviour of permutit with different chromium salt solutions is shown to resemble that of collagen, *e.g.*, in regard to effect of temperature, of concentration, and of basicity. The base exchange with chromium chloride solutions is, however, not the same for both permutit and hide substance. The action of chromium chloride solutions of all degrees of basicity on permutit indicates the presence of a trivalent cation. The basic chromium sulphates react with permutit as if there were complex chromium cations of less than three valencies. The maximum base exchange takes place in liquors of high basicity. Diminution of basicity or dilution results in a gradual increase in the valency. Basic chromium sulphates differ from the chlorides in showing greater tendencies to polymerisation and condensation apparently owing to the residual affinities of the hydroxyl groups. The diffusibility of the basic chromium sulphates depends on the basicity, whereas that of the basic chromium chlorides is independent of the basicity. The basicity of the cation in chromium chloride is

about 90%. This agrees with its physico-chemical nature, the basicity at which this salt precipitates, and its inferior tanning properties. Addition of up to 2 mols. of sodium chloride to basic chromium chloride solutions of 54.8% basicity caused an increase in the amount of chromium fixed by the permutit and collagen and a lowering of the p_H figure and of the basicity at which precipitation commenced. The neutral salt effect cannot be explained by the hydration hypothesis alone; there are constitutional changes also. The hydrion concentration is not the only determining factor in the absorption of chromium, which is affected also by the nature of the cation, the number of valencies, the properties of the acid and basic groups, and also the degree of aggregation. Very small additions of sodium sulphate lower the precipitation figure and the hydrion concentration. The absorption of chromium compounds from such solutions by collagen and permutit agrees with the view that the tendency to form chromium complexes is greater with sulphates than with chlorides. The valency of the cations is diminished and polymerisation and condensation processes are initiated. Increasing amounts of sodium sulphate cause increased formation of negatively charged chromium complexes, which can combine with collagen but not with permutit. The addition of small quantities of sodium sulphate to chromium chloride solutions causes an appreciable increase in their tanning power. Chrome tanning is due partly to the ordinary valencies and partly to residual valencies of the chromium compounds.

D. WOODROFFE.

Quinone tannage. A. W. THOMAS and M. W. KELLY (Ind. Eng. Chem., 1926, 18, 383—385; cf. B., 1924, 918).—The addition of sodium chloride retards the rate of quinone tanning in alkaline solutions. In neutral solutions, sodium sulphate aids the tanning and sodium chloride retards it. The amounts of "tannin" removed by alcohol

"tannin" is removed, something else is fixed by the leather. It is suggested that this is acetaldehyde. The same phenomenon has been observed with leathers tanned with hemlock bark and gambier. Very little gallotannic acid is fixed by quinone-tanned leather. The aqueous extraction of quinone-tanned leather at different p_H values resulted in a gradual loss in weight of the leather in alkaline solutions. Quinone tannage appears to take place at the same groups of the collagen molecule as vegetable tannage.

D. WOODROFFE.

Properties of shoe leather. I. Micro-structure J. A. WILSON and G. DAUB. **II. Chemical composition.** J. A. WILSON and S. O. LINEAR (J. Amer. Leather Chem. Assoc., 1926, 21, 193—198, 198—206).

—I. Photomicrographs of sections of representative samples of 18 varieties of shoe leather are reproduced. Sections of vegetable-tanned and chrome-tanned calf-skins respectively show that in the vegetable tannage the fibres are built up to a much greater extent than in the chrome tannage, which makes the vegetable-tanned leather relatively tight and full compared to the looser and more open structure of the chrome leather. The sections of the various kinds of leather show characteristic differences of structure. II. The complete chemical analyses of 18 leathers mentioned above are given in the following table, viz.: (1) coloured vegetable-tanned calf, (2) coloured chrome-tanned calf, (3) black chrome-tanned glazed kid, (4) black chrome-tanned kangaroo, (5) vegetable-tanned horse bull (cordovan), (6) chrome-tanned buffed and split cow hide, (7) chrome-tanned split cow hide, (8) black chrome-tanned "slink" calf (suede), (9) russet lining calf, (10) russet lining sheep, (11) vegetable-tanned shark, (12) patent chrome split cow hide, (13) patent chrome-tanned kid, (14) patent chrome colt, (15) heavy chrome-tanned cow hide, (16) chrome re-tan army upper leather, (17) vegetable-tanned sole eather, (18) chrome sole leather.

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Water	13.6	16.3	13.7	12.0	10.0	14.1	16.3	12.7	11.9	10.9	12.2	10.1	11.8	12.0	14.4	15.1	14.6	16.3
Skin protein (N × 5.62) ..	41.0	62.6	65.3	82.7	40.1	69.6	66.8	55.1	46.0	50.0	45.4	50.5	54.0	60.4	57.0	44.6	29.7	29.4
Fat	12.0	4.6	6.6	11.3	18.6	2.1	5.8	7.1	7.6	6.1	6.9	10.0	6.6	5.1	14.2	20.4	3.2	25.4
H ₂ SO ₄	0.3	3.4	1.0	1.8	0.6	3.2	3.6	0.8	0.1	1.7	1.5	1.8	2.1	2.3	4.4	1.1	0.8	5.9
Al ₂ O ₃	0.4	1.2	0.2	0.1	—	1.0	0.2	1.0	0.1	0.1	—	0.1	0.2	0.1	—	0.3	—	2.6
Fe ₂ O ₃	0.1	0.3	0.3	0.1	0.1	0.6	0.2	1.2	—	0.1	0.1	0.4	0.6	0.3	0.7	0.2	0.7	0.5
Na ₂ SO ₄	—	0.4	0.9	0.2	—	0.3	1.0	0.4	—	—	—	0.6	0.3	0.5	0.4	0.3	—	12.3
HCl	—	0.3	0.5	0.3	—	—	—	0.2	—	0.6	—	0.1	—	0.1	0.1	—	—	0.8
NaCl	—	0.5	0.2	0.1	—	—	0.3	0.1	—	—	—	—	0.1	0.4	0.4	—	—	0.9
Cr ₂ O ₃	—	5.4	4.5	6.0	—	5.3	3.6	5.4	—	—	—	2.9	3.6	3.6	5.5	2.4	—	1.7
CaO	—	—	0.2	0.3	0.1	0.2	0.1	0.2	0.2	0.1	0.1	0.1	0.2	0.3	—	—	—	—
MgSO ₄	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	0.8
CaSO ₄	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	2.3
Collodion	—	—	—	—	—	—	—	—	—	—	—	9.0	8.4	6.1	—	—	—	—
Soluble organic matter ..	9.1	—	—	—	8.7	—	—	—	12.3	13.0	5.4	—	—	—	—	—	—	35.6
Combined tannin (by diff.) ..	23.5	—	—	—	21.8	—	—	—	21.8	17.4	23.4	—	—	—	—	—	—	14.6
Other organic matter (by diff.) ..	—	5.0	.6	5.16	—	3.6	2.11	5.8	—	—	—	14.4	12.2	9.1	2.9	15.2	—	1.4

were compared with the p_H values of the tanning solutions and it was found that alkaline-tanned leathers were more resistant to alcohol treatment the higher the p_H value of the tanning solution. The percentages of material extracted by alcohol determined from the weight of extract and from loss in weight of the leather showed that as fixed

The fat was extracted with chloroform. After extraction with chloroform patent leathers were extracted further with a mixture of ethyl acetate and acetone for several days. The residue from this further extract was called collodion. In the chrome leather analyses, H₂SO₄ represents the combined acid sulphate determined according to the official

methods of the American Leather Chemists Association. Water-soluble matter was determined by extracting the fat-free leather with water in a Wilson-Kern extractor until the wash water gave no coloration with ferric chloride solution. D. WOODROFFE.

PATENTS.

Depilation of hides and skins. F. ULLMAN, Assec. of H. BENFEY (E.P. 246,114, 17.12.25. Conv., 13.1.25).—Solutions for the depilation of hides and skins contain lime and less than 1% of sodium hydro-sulphide. L. A. COLES.

Tanning composition. BADISCHE ANILIN- & SODA-FABR., Assecs. of C. IMMERHEISER and H. WOLFF (U.S.P. 1,557,844, 20.10.25. Appl., 9.7.20).—See E.P. 146,427 and 169,943; B., 1921, 819 A, 820 A.

Tanning and the manufacture of tanning materials. J. A. S. MORRISON (U.S.P. 1,581,224, 20.4.26. Appl., 23.1.24).—See E.P. 219,347; B., 1924, 841.

XVI.—AGRICULTURE.

Spontaneous multiplication of [nitrogen] fixing organisms [in soil]. S. WINOGRADSKY (Compt. rend., 1926, 182, 999—1001).—Addition of 0.5—1% of dextrose or mannitol to a fertile soil kept damp at 30°, results in an enormous increase in the number of *Azotobacter* present. Experiments in Petri dishes on a starch medium give almost completely pure colonies. These facts may be utilised in classifying soils according to their nitrogen-fixing capacity. Other substances which increase the C : N ratio in the soil, e.g., other carbohydrates, alcohols, and organic acids, have a similar, but generally less effect. Increase of nitric acid or ammonia in the soil, on the other hand, results in a decrease in the number of *Azotobacter* and an increase of bacteria which are harmful to the soil. When 0.005% of nitric acid was added to a soil 96% of the *Azotobacter* disappeared; when 0.01% of nitric acid was added multiplication of *Azotobacter* ceased and the soil was invaded by other organisms.

L. F. HEWITT.

Effect of basic slag on the lime status of soils. R. WILLIAMS (J. Agric. Sci., 1926, 16, 196—204).—In laboratory experiments, addition of 0.1% of basic slag to soils with a low percentage of exchangeable calcium and a low degree of saturation causes an increase in the exchangeable calcium, the degree of saturation, and the calcium soluble in an aqueous solution of carbon dioxide, similar to that obtained on adding equivalent amounts of calcium oxide or carbonate. The effect on p_H is less marked. The soil of field plots treated with basic slag also showed an increase in exchangeable calcium which was still evident 8 years after the application of the slag. C. T. GIMINGHAM.

Rôle of phosphorus in agriculture. E. VAN-STONE (J.S.C.I., 1926, 45, 78—80T).—The importance of an adequate supply of phosphorus and lime in maintaining soil fertility is discussed and illus-

trated by results of experiments at home and abroad. A brief account of some of the author's work on soil phosphates and phosphatic fertilisers is given with special reference to methods of determining the availability of the phosphorus.

Standard methods of analysis of fertilisers. Edited by J. SEN (Bull. Agric. Res. Inst. Pusa, 1926, [164], 14 pp.).—Details are published of standard procedures for the analysis of fertilisers, largely based on the methods of the Association of Official Agricultural Chemists of the U.S.A. These include determinations of moisture, total, water-soluble, and citrate-soluble P_2O_5 , total, nitric, and ammoniacal nitrogen, and K_2O by both the platinum and perchlorate methods. C. T. GIMINGHAM.

Detection of leather in fertilisers made from waste products. G. JORET and E. RADET (Ann. Falsif., 1926, 19, 148—152).—If leather has been used in the manufacture of the fertiliser tannin can usually be detected in the boiling water or oxalic acid extracts. As a rule the darker and more acid the sample the more readily is tannin to be detected, and the larger the proportion of calcium phosphate added to the fertiliser the more difficult does the detection become. If a negative result is obtained with the oxalic acid extract the product should be warmed with successive small portions of hydrochloric acid till an excess of acid is present; a little warm water is then added, the material filtered, and the insoluble residue washed several times, and then treated with boiling oxalic acid when, if leather was originally present, a tannin reaction will be obtained. D. G. HEWER.

Determination of ammoniacal nitrogen in fertilisers. F. CHASTELLAIN (Helv. Chim. Acta, 1926, 9, 205—216).—The Ronchèse method (A., 1907, ii, 651) of determining ammonia by treatment with formaldehyde and titration of the acid liberated can be applied to fertilisers, using phenolphthalein as indicator. Where hydrolytic interference occurs in the presence of compounds such as dicyanodiamine salts, phenolphthalein is replaced by rosolic acid but phosphoric acid must be absent. For fertilisers of the superphosphate type, an aqueous extract is treated with milk of lime and sodium carbonate and the ammonia in an aliquot portion of the filtrate is determined by the Ronchèse method using rosolic acid. Ammonia is removed completely from solutions of its salts by boiling with calcined magnesia and since little decomposition of urea and dicyanodiamine is caused by this process, it can be applied to the determination of ammonia in fertilisers. When ammonia-free air is bubbled through two cold solutions containing known amounts of ammonium salts and sodium hydroxide, the ratio of the ammonia removed from each at any given time is constant and is the same as the ratio of the ammonia in the solutions. Since sodium hydroxide solutions containing up to 24% of alkali have little action on urea and dicyanodiamine, the ammonia in fertilisers can be determined by passing a stream of air through a flask containing a standard amount

of ammonium sulphate, into which excess of sodium hydroxide is run, the ammonia carried over being collected in a known volume of dilute sulphuric acid; the air stream is then passed through a solution of the fertiliser and alkali and finally through another standard sulphuric acid solution. At the end of two hours, the amounts of ammonia carried over are determined. If m be the standard weight of ammonia, m_1 and m_2 the weights of ammonia carried over, then the weight of ammonia in the fertiliser is $m \times m_2/m_1$. This method is recommended for general use in determination of ammonia in calcium cyanamide fertilisers. M. CLARK.

Salt requirements of *Lupinus albus*. C. H. ARNDT (Soil Sci., 1926, 21, 1—6).—Seedlings of *Lupinus albus* were grown in culture solutions containing varying ratios of nutrient salts. The optimum salt ratios were 5:3:4 for potassium, calcium, and magnesium, and 5:9:4 for nitrate, phosphate, and sulphate. A favourable total salt concentration was found to be 0.0084*N*. Excess of phosphate in the culture solutions tended to produce chlorosis. No evidence was obtained to justify the term "calciphobe" as applied by geologists to *Lupinus albus*. A. G. POLLARD.

Remarkable correlation between [yields of] grain and straw. W. A. MACKENZIE (J. Agric. Sci., 1926, 16, 275—279).—A set of 46 unmanured plots (each 0.098 acre) was sown with wheat to test the uniformity of the soil of a field at Rothamsted. The soil was found to be very variable in fertility, but an exceedingly close association between the yields of grain and straw on the plots was evident. The correlation coefficient calculated from the 46 pairs of values was +0.990.

C. T. GIMMINGHAM.

Nutritive value of pasture. I. Seasonal variations in the productivity, botanical and chemical composition, and nutritive value of medium pasturage on light sandy soil. H. E. WOODMAN, D. L. BLUNT, and J. STEWART (J. Agric. Sci., 1926, 16, 205—274).—A detailed study of the nutritive value throughout the season of the herbage of a pasture on light soil under grazing conditions. The influence of meteorological conditions on the yield is discussed; there was a marked correlation between yield and rainfall during a period of low rainfall in June and July. The total yield per acre from hay plots was more than twice as great as that from the pasture, but, in comparison with the hay, there was a very high percentage of protein and a low percentage of fibre in the pasture grass. The digestion trials showed that well grazed pasture possesses a much higher nutritive value than has hitherto been thought, and compares with concentrates like linseed cake. The high degree of digestibility of the fibre is specially notable. The digestibility of the grass varied during the season, but, at its lowest, was superior to that of the best meadow hay. The pasture plot produced considerably more digestible protein than the hay plot.

Seasonal changes in the lime and phosphate content of the pasture grass and the utilisation of these constituents by the sheep were also followed and are considered in detail. The lime content of the grass increased to a maximum in the dry period and declined later. The phosphate behaved in the opposite manner. The system of frequent cutting favoured the spread of wild white clover on the plot to a remarkable extent. Suggestions are made with regard to the management of pasture and meadow, based on the results obtained. C. T. GIMMINGHAM.

Rôle of calcium hydroxide in hydrated lime-acid lead arsenate sprays. F. L. CAMPBELL (J. Agric. Res., 1926, 32, 77—82).—The success of hydrated lime in reducing the injury to foliage caused by acid lead arsenate alone may be due to the fact that soluble calcium arsenate is less toxic than soluble arsenic acid derived by hydrolysis of the acid lead arsenate. In humid conditions, however, acid calcium arsenate may be formed in sufficient concentration to cause foliage injury.

H. J. CHANNON.

Fungicidal properties of certain spray fluids. IV. W. GOODWIN, H. MARTIN, and E. S. SALMON (J. Agric. Sci., 1926, 16, 302—317; cf. Goodwin and Martin, B., 1925, 780).—The toxicity of various substances to the conical stage of the hop mildew (*Sphaerotheca humuli*) has been compared under standardised conditions. Solutions of potassium, sodium, and calcium polysulphides proved fungicidal at approximately the same concentration of polysulphide sulphur (0.092 to 0.12%). The fungicidal concentration of dicalcium and disodium arsenates is 0.024% As_2O_5 ; of lead thioarsenate, 0.05%; and of lead arsenate, between 0.1 and 0.2%. A mixture of calcium polysulphide and lead arsenate is superior in fungicidal value to either of its components; solutions at concentrations below fungicidal strength, when mixed, proved to be fungicidal. This is probably accounted for by the formation of soluble arsenates and thioarsenates.

C. T. GIMMINGHAM.

Alternate extraction and exposure method for the study of arsenicals. S. LOMANITZ (J. Agric. Res., 1926, 32, 499—500).—In order to determine water-soluble arsenic in arsenical insecticides under conditions approximating to those occurring in field practice, a method involving several successive extractions and exposures of the residue to the air is proposed. The results thus obtained give more reliable information as to the risk of foliage injury from the use of arsenicals than a single determination.

C. T. GIMMINGHAM.

Practicability of quantitative toxicological investigations on mandibulate insects. F. L. CAMPBELL (J. Agric. Res., 1926, 32, 359—366).—A method for the quantitative study of the toxicity to insects of soluble poisons, taken through the mouth, is based on the observation that certain caterpillars will drink completely drops of liquid placed in their feeding path. The results obtained by the method are expressed by the relation between

dose of poison and the reciprocal of the survival period or speed of toxic action. Tervalent arsenic was found to be more toxic than quinquevalent arsenic. The minimum toxic dose of arsenic for the two species studied is about 0.02 mg. of arsenic per gram of insect. C. T. GIMINGHAM.

PATENTS.

Production of available phosphate. H. H. MEYERS, Assr. to ARMOUR FERTILIZER WORKS (U.S.P. 1,578,339, 30.3.26. Appl., 20.10.22).—A mixture of ground phosphate rock and an alkali salt is sintered, thereby rendering part of the phosphate available; the sintered mixture is broken into lumps and calcined at a suitable temperature, and for a suitable time to complete the conversion of the phosphate into available phosphate.

C. T. GIMINGHAM.

Treatment of raw phosphates. M. PIERRON (F.P. 600,734, 11.10.24).—Raw phosphates rich in or mixed with calcium carbonate are roasted and slaked with water. The resulting product is easily distributed and assimilated. J. S. G. THOMAS.

Insecticide-fungicide. E. C. HOLTON, Assr. to SHERWIN-WILLIAMS Co. (U.S.P. 1,578,520-3, 30.3.26. Appl., [A, B] 8.3.24, [C, D] 12.1.25. [A] Renewed 5.2.26).—(A) An aqueous solution of barium polysulphide is treated with hydrofluoric acid, in presence of a substance to prevent crystallisation. The precipitate of amorphous barium fluoride and finely-divided sulphur is dried and powdered. (B) An alkaline-earth polysulphide is treated with a suitable acid substance to produce hydrogen sulphide, and a mixed precipitate of finely-divided sulphur and a substantially insoluble toxic substance. Additional sulphur is formed by acting on the hydrogen sulphide with sulphur dioxide. The product is dried and powdered (C, D) Calcium (or barium) polysulphide is treated with hydrofluosilicic acid to give a mixed precipitate of finely-divided sulphur and calcium (or barium) silicofluoride, which is dried and powdered. C. T. GIMINGHAM.

Means for combating plant pests. W. ILISCH (Swiss P. 109,831, 26.3.24. Conv., 4.4.23).—Juices or extracts of such plants as tomato, rhubarb, tobacco, poppy, digitalis, delphinium, etc. are supplied through the roots to plants to be protected. Suitable chemicals may be added to the liquids.

C. T. GIMINGHAM.

Manufacture of meal for fertilising and other purposes, and for recovery of oil from fish or vegetable refuse and the like. S. HILLER (E.P. 215,765, 7.5.24).—See U.S.P. 1,489,940; B., 1924, 479

Arsenate of lead [insecticide]. H. B. GOODWIN, Assr. to LATIMER CHEMICAL Co. (Reissue 16,331, 20.4.26, of U.S.P. 1,322,008, 18.11.19. Appl., 8.11.21).—See B., 1920, 40 A.

Insecticide, sheep dip, and the like. P. J. FRYER, Assr. to McDougall and Yalding, Ltd. (U.S.P. 1,583,681, 4.5.26. Appl., 29.11.24).—See E.P. 246,252; B., 1926, 293.

XVII.—SUGARS; STARCHES; GUMS.

Defecation of diffusion juice with dolomitic lime, and with the scums obtained by saturating the intermediate juice. W. KOHN (Z. Zuckerind. Czechoslov., 1926, 50, 209—215, 217—221).—Dolomitic lime used for the defecation of diffusion juice gave satisfactory results provided the amount was less than 0.4% of the volume of the liquid, the mud obtained settling out in 30 min. to about 20% of the total height. When larger quantities were used, practically no subsidence of the precipitate occurred. Saturation scums from the carbonation of the intermediate juice failed to produce a sufficient defecation of the diffusion juice. J. P. OGILVIE.

Increase of colour of first-product massecuites during boiling. F. HOFFMANN (Z. Ver. Deuts. Zucker-Ind., 1926, 91—98).—Measured by the extinction coefficient in a polarisation photometer with double blue filter, the total increase of colour from thick-juice (evaporator syrup) to first massecuite in a white beet sugar factory was found to average 47%, or from thick-juice to molasses, about 255%.

J. P. OGILVIE.

Basic dyes as flocculating agents for approximate quantitative determination of colloids in sugar-house liquors. M. S. BADOLLET and H. S. PAINE (Int. Sugar J., 1926, 28, 23—28, 97—103, 137—140).—On testing the colloids separated from sugar-house liquors in a cataphoresis apparatus, they were found to be electro-negative, and a method has been devised for their approximate determination on the principle of electrical neutralisation, using night-blue as flocculating agent, and maintaining the p_H of all the products examined constantly at 6.0. Tests on cane juice which had been submitted to different methods of clarification showed the isoelectric ratio (ratio of weight of dye in mg. to weight of solids in mg., the colloid content of which is electrically neutralised by the dye) thus obtained to vary in a generally parallel manner with the amount of colloids obtained directly by ultra-filtration. Caramel, and the water-reversible fraction of the colloids separated from various sugar-house liquors by ultra-filtration, showed only a slight tendency to flocculate when electrically neutralised under the authors' experimental conditions. J. P. OGILVIE.

Determination of reducing sugars volumetrically in the presence of excess sucrose. N. SCHOORL (Archief Suikerind. Nederl-Indië, 1925, 33, 273—278; cf. B., 1925, 329, 518).—Using a 300-c.c. flask, a mixture of 25 c.c. of modified Luff's reagent (Benedict, B., 1909, 385) and the same volume of sugar solution is heated to boiling in 3 min., and maintained in ebullition for exactly 5 min., the flask having been connected with a reflux condenser. After cooling to room temperature, 3 g. of potassium iodide and immediately afterwards 25 c.c. of 25% sulphuric acid are added, and the liquid is titrated with 0.1N-thiosulphate. A control determination using the same amount of sucrose as was present in the 25 c.c. of assay solution was made under

exactly the same conditions, it being found that 10 g. gave a reduction corresponding to 0.33 c.c. of thiosulphate, that is as much as 1 mg. of invert sugar. In ordinary good refined sugar the content of reducing sugars by this method was found to be about 0.002%. This method is much more sensitive than any using Fehling's solution, and is well suited for accurate and rapid work in the refinery or sugar factory.
J. P. OGILVIE.

Determination of reducing sugars volumetrically in presence of an excess of sucrose. N. SCHOORL (Int. Sugar J., 1926, 28, 276—277).—See B., 1925, 518.

See also A., May, 502, **Mechanical liquefaction of starch** (PETIT and RICHARD). 535, **Iodometric determination of aldehyde sugars** (PAUCHARD).

Preservation of syrups. SABALITSCHKA and BÖHM.—See XX.

XVIII.—FERMENTATION INDUSTRIES.

Manufacture of Yoghurt bacteria in tablet form. SCHEERMESSE (Pharm. Ztg., 1926, 71, 499).—Yoghurt tablets, almost free from carbohydrates and albumin, may be thus prepared:—Triply centrifuged skim milk (fat content less than 0.1%) is treated with rennet (thereby destroying half the casein), the enzyme destroyed by heating, and the flocculent whey-casein mixture hydrolysed with trypsin, in alkaline medium, at 40—50°. The hydrolysis is interrupted before completion, and there should then be present only a small proportion of Siegfried's peptone, hardly any kynine, and still less mono- and diamino-compounds. The trypsin is destroyed by neutralisation of the liquid with *N*-hydrochloric acid till Congo-paper of litmus-amphoteric reaction is turned slightly blue. The liquid is heated for some time at 80—90°, filtered, sterilised at 2-day intervals with steam, cooled to 40°, and inoculated with pure Yoghurt culture. Multiplication of the *Bacillus bulgaricus* is allowed to proceed in a thermostat, and may be hastened by the addition of certain salts. The addition of slight traces of certain oxides (acting catalytically) has a marked influence on the virulence of *B. bulgaricus*. The liquid is treated with 10% of a 10% calcium chloride solution, and then with an equivalent quantity of sodium pyrophosphate (both sterilised), when, on prolonged shaking, the precipitated calcium phosphate adsorbs the Yoghurt bacteria. The supernatant liquid is decanted and the residue centrifuged, powdered, and dried at 40°. The dry substance gives tablets containing only 1.3% of organic matter; 0.5 g. is sufficient to prepare 1 litre of Yoghurt milk.
B. FULLMAN.

Volatile acidity of wines. C. LAGNEAU (Ann. Falsif., 1926, 19, 152—159).—Duclaux's method of determining volatile acidity by titration of 50 c.c. of the distillate obtained from 20 c.c. of wine and 35 c.c. of water with 0.05*N*-sodium hydroxide solution was found satisfactory, but the use of the formula $0.00245 \times n \times 50 \times 100/85$ is advised. Free

sulphurous acid passes over completely into the distillate and must therefore be deducted from the total, whilst of the combined sulphurous acid a portion only, varying according to the conditions, distils over.
D. G. HEWER.

Impurities in wines. J. VILAR (Anal. Asoc. Quim. Argentina, 1925, 13, 337—347).—Analyses are given of a number of samples of wine collected in Argentina, some of which were sophisticated. Methyl alcohol is a normal impurity of wines which have been prepared by fermentation of grape juice in the presence of the skins and is formed at the end of the period of fermentation.

G. W. ROBINSON.

Production of alcohol from rice straw. N. TAKETOMI (J. Soc. Chem. Ind. Japan, 1926, 29, 43—47).—Rice straw was heated with 1% sulphuric acid at about 134° for 30 min. in an autoclave. The liquid obtained was neutralised with calcium carbonate, concentrated, and fermented with the distillery yeast, Race XII. The yield of sugar was about 15% of the dry matter used and of alcohol about 5.4%.
K. KASHIMA.

Concentration of the alcohol content of alcohol-water vapour by separation of the aqueous distillate [phlegms]. J. DEHNICKE (Z. Spiritusind., 1926, 49, 50—51, 71—72, 86, 101, 110).—If an alcohol-water vapour mixture is condensed by cooling, the phlegm has a lower alcohol content than the dephlegmated vapour. The difference between the amounts of alcohol contained in the phlegm and the dephlegmated distillate is greater, the smaller is the separated phlegm. The smaller the aqueous distillate, the lower is its alcohol content, and the greater the amount of aqueous distillate, the higher becomes the alcohol content of the dephlegmated vapour. The increase in the alcohol content of the dephlegmated vapour corresponding to increased phlegm formation tends to disappear with increasing alcohol content of the original vapour mixture. Slight increases have even been noticed with a 96% by volume vapour. Confirmation is given of the observation of Pampé that the use of the dephlegmator rarely brings about an increase of the end product with high-percentage alcohol vapours approximating to 96% by volume. Support is also given to the view of Hausbrand and Donitz that, by the condensation of vapour containing alcohol, there is obtained a vapour of higher and a phlegm of lower alcohol content than that of the original vapour. A flow of vapour through the dephlegmator so rapid that re-evaporation cannot be effected together with a slow current of cooling water produce less efficient concentration, since less phlegm is formed with accompanying poorer concentration of the vapour. The smaller production of phlegm is followed by a reduced re-evaporation. If the cooling surface is curtailed, the temperatures of the cooling water and cooling surface are low and the action of the dephlegmator is restricted. In this case, although the predominating factor is phlegm formation, its effect is insignificant. Modern

dephlegmators work as completely as possible on the principle of flow and counterflow of phlegm and vapour and are provided with a cooling surface which ensures phlegm formation without increasing the consumption of vapour over normal limits, and which are so regulated that the phlegm travels from cooler to warmer places, thus favouring re-evaporation. C. RANKEN.

Influence of sugar on determination of ammonia in grape musts. J. VENTRE and E. BOUFFARD (Ann. Falsif., 1926, 19, 226—230).—See B., 1926, 417.

Souring of potatoes. GLAUBITZ.—See XIX.

PATENTS.

Preparation of fruit wines resembling grape wines in aroma and flavour. M. HAMBURG (Austr. P. 97,133, 5.7.23).—By evaporation under reduced pressure, fruit and berry juices are freed almost completely from their volatile aromatic substances and are subsequently fermented with wine yeast. C. RANKEN.

Acetone and similar compounds produced by fermentation. N. MOSKOVITS (Austr. P. 100,444, 6.3.22. Conv., 11.3.21).—Reducing agents such as hyposulphites, thiosulphates, or sulphites are added to the mash together with the micro-organisms, e.g., *Bacillus macerans*, which bring about the formation of acetone and similar substances. The favourable influence of the addition of sulphites is most marked. C. RANKEN.

Production of fermentable worts. W. H. CAMPBELL (U.S.P. 1,581,918, 20.4.26. Appl., 18.7.25).—See E.P. 237,321; B., 1925, 822.

XIX.—FOODS.

Adam's method of fat determination in milk. CHAVASTELON and ELOUARD (Ann. Falsif., 1926, 19, 159—160).—The line of demarcation of the ethereal solution of the fat as obtained in Adam's method is more readily defined if four drops of an alcoholic solution (saturated when hot) of Sudan III. Red are added. The chromolipoid of the tomato or pumpkin may also be used. The use of the colouring matter resulted in an increase of 0.3—1.3 g. of fat per litre of milk over that obtained without its use. D. G. HEWER.

Determination of free acid and fat in technical casein. W. HÖPFNER and K. JAUDAS (Chem.-Ztg., 1926, 50, 325—326; cf. B., 1925, 420).—Polemical. The methods of determining free acid and fat in casein described by Ulex (B., 1925, 734) give too high results in each case, and Ulex's criticism of the authors' method is shown to be unsound. The proteins of casein absorb varying amounts of alkali according to the strength of solution used, so that erratic results are obtained by any method involving treatment of the whole with alkali. Treatment of the hydrochloric acid solution of casein with ether extracts not only fat, but also some lactic acid

and certain degradation products of casein. A more satisfactory method for determining fat in technical casein consists in dissolving 2 g. in 10 c.c. of ammonia, shaking with 20 c.c. of 96% alcohol, then, after keeping for 20 min., with 10 c.c. of light petroleum and 20 c.c. of ether. An aliquot part of the ethereal layer is evaporated after 2 hrs., and the fat determined as usual. A. R. POWELL.

Vitamin-A in oleo oil and oleo stearin. R. HOAGLAND and G. G. SNIDER (J. Agric. Res., 1926, 32, 397—416).—The content of vitamin-A of a number of samples of oleo oil and oleo stearin was compared by feeding tests with young rats, the oil or stearin providing the only source of vitamin-A in an otherwise adequate ration. Various grades of oleo oil differed considerably in their content of this vitamin, the yellow oil being the richest. The samples of stearin also differed greatly, yellow stearin being richer than the other grades. Oleo oil is poorer in vitamin-A than butter, but richer than lard and vegetable fats and oils. C. T. GIMMINGHAM.

Drying of vegetables. A. W. KNAPP (J.S.C.I., 1926, 45, 123—128 T).—It is important to commence drying in an atmosphere of moderately high humidity to prevent case-hardening. The air should leave the plant at the highest temperature which is permissible without injury to the vegetable. The most efficient drying machine available during the war was the Tomlinson-Haas. It had the advantage that the air was heated between successive chambers, and also between successive batches of trays, so that it never became too humid. The properties of various vegetables and their preparation before drying are discussed. The yield is indicated by the specific gravity. Precautions must be taken to avoid the blackening of potatoes due to peroxydase. The loss on washing is from 0.3 to 0.5%, and on steaming 1%. White carrots are richer in sugar, but poorer in flavour than the red. The green colour of cabbage can be preserved by the use of ammonia. Onions should not be allowed to come in contact with copper. The moisture content of raw and dried vegetables, and of dried vegetables after exposure to the atmosphere is discussed in relation to yield and keeping properties. The yields of dry vegetables obtained from 100 pts. of the raw material were: potatoes 16 pts., carrots 10, onions 10, swedes 8, turnips 6, and cabbage 5. Dried vegetables have excellent keeping properties; they are best preserved in the dark and *in vacuo*.

Chemical, microscopical, and bacteriological study of infant foods. H. KUFFERATH (Chim. et Ind., 1926, 15, 331—348).—An extensive analytical examination of typical infant foods is recorded, and is made the basis of a critical review of the feeding values of these preparations. The foods are classified according to the type of products used in their manufacture, and a number of fraudulent and deceptive practices adopted by manufacturers are discussed. A. G. POLLARD.

Autolytic and bacterial transformation of fish muscle proteins. G. B. REED (Pub. Health J.,

Canada, 1925, 16, 568—577).—Preliminary experiments showed that there was a slight increase in non-coagulable and ammonia nitrogen in macerated haddock muscle kept under toluol at 25° for 24 hrs., and a conspicuous increase in 48 hrs. Haddock fillets were found to be heavily infected with bacteria similar to those of the gut and slime of the fish. When these bacteria were permitted to develop in fish muscle at 25° there were only slightly greater increases in non-coagulable and ammonia nitrogen during the first 24 hrs. than produced by autolysis alone, but from the twenty-fourth to the forty-eighth hour of incubation the bacteria produced very much greater protein transformation than was produced by autolysis alone. R. E. THOMPSON.

Legumin of sweet almonds. M. A. RAKUSIN and B. E. MASCHKILEISSON (Z. Unters. Lebensm., 1926, 51, 45—47).—The comminuted, shell-free almonds, after removal of oil, sugar, and protein cleavage products by extraction with petroleum ether, alcohol, and water respectively, yielded, on extraction with sodium hydroxide solution, 4.8% of legumin having $[\alpha]_D^{25}$ —42.90° (in pepsin-hydrochloric acid solution). The legumin gave colour reactions identical with those given by legumin from legumes (cf. preceding abstract). The optical rotations of the alkali salts of almond legumin are; *ammonium*, $[\alpha]_D^{25}$ —64°; *lithium*, $[\alpha]_D^{25}$ —39°; *sodium*, $[\alpha]_D^{25}$ —42.53°; *potassium*, $[\alpha]_D^{25}$ —34°. These resemble very closely those of the alkali salts of legumin from other sources (cf. B., 1923, 421). E. H. SHARPLES.

Souring of potatoes. M. GLAUBITZ (Z. Spiritus-ind., 1926, 49, 53).—Potatoes intended for use as fodder are preserved by a souring process effected by means of a fermentation by lactic acid bacteria. The raw potatoes, after washing to remove undesired acetic and butyric acid bacteria, are sliced and packed as closely as possible into pits, preferably lined with cement. A liquid preparation of lactic acid bacteria is sprayed over the potatoes and the whole covered by boards or tiles to exclude air and rain which encourage mould formation. Frosted potatoes, owing to their sugar content, are specially suitable for preserving by this treatment. The potatoes may be steamed before addition of the bacterial preparation. The lactic acid bacteria preparation is made by inoculating, with a pure lactic acid bacteria culture, the pulp produced by boiling potatoes in a mixture of water and skimmed milk, and cooling to 30—35°. The author confirms the observations of Völtz on the relative values of raw, steamed, soured-raw, and soured-steamed potatoes as fodder for ruminants, and adds that soured-steamed potatoes can be safely given to horses and even to animals with young. C. RANKEN.

Identification of dried apple marc in foods. C. BRIOUX (Ann. Falsif., 1926, 19, 142—144).—The epicarp or skin, endocarp or the hard parchment-like membrane bordering the spaces around the pips, and the testa or skin of the pips, have characteristic microscopical features (shown in plates).

D. G. HEWER.

Black currant juice and reactions of orchil. M. FRANÇOIS and L. SEGUIN (Ann. Falsif., 1926, 19, 230—235).—See B., 1926, 382.

Nutritive value of pasture. WOODMAN, BLUNT, and STEWART.—See XVI.

Yoghurt bacteria in tablet form. SCHEER-MESSER.—See XVIII.

PATENTS.

Production of coffee free from caffeine. H. FAIRBROTHER. From F. KUNDIG, JUN., & Co., CHEM. FABR. (E.P. 247,039, 22.4.25).—See F.P. 597,392; B., 1926, 297.

Evaporating apparatus (E.P. 248,131).—See I.

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

Determination of santonin in *Artemisia* sp. and in resins. H. VOGTHERR (Arch. Pharm., 1926, 264, 324—327).—The method of Fromme (Jahresber. Caesar u. Loretz, 1912, 40) is suitably modified. For whole *Artemisia* plants 50 g. (finely powdered) are extracted with 500 c.c. of chloroform and the determination then completed as described by Fromme. For resinous substances further modification is necessary: the chloroform extract must be washed with dilute sodium hydroxide solution to remove resinous substances. With the quantities and strengths recommended the santonin removed at this stage is negligible. The finely powdered drug (10 g.) is stirred at 80—100° with water (50 c.c.) and hydrochloric acid (25%; 10 c.c. or more). The cake obtained is again ground and the whole mixture washed with water (40 c.c.) into a separating funnel, where it is extracted with chloroform (150 c.c. in three portions). The total chloroform extract is washed with sodium hydroxide solution (1.5%; 50 c.c.), then with water (50 c.c.), and after filtering over sodium sulphate, if necessary, is evaporated to small bulk (10 c.c.). The solution is covered with dilute barium hydroxide solution (5%; 100 c.c.) and the remaining chloroform boiled off. The liquor is filtered from resinous matter, acidified with hydrochloric acid (25%; 5 c.c.), and again extracted with chloroform (60 c.c., in 3 portions). The extract is filtered into a tared 100 c.c. flask, the chloroform is distilled off, the residue is dissolved in alcohol (7.5 g. exactly), and reprecipitated by adding hot water (42.5 g. exactly). After 24 hrs. the crystalline santonin is collected on a tared filter paper, washed twice with aqueous alcohol (3 pts. of alcohol to 17 of water; 10 c.c.), dried, and weighed. That remaining in the flask is also weighed. To the weight found 0.034 g. should be added. If in the final crystallisation there is a separation of resin the product must be washed with more aqueous alcohol and the weight suitably corrected. W. A. SILVESTER.

Commercial papain; its purification. R. FROSSARD (Bull. Soc. Chim. biol., 1926, 8, 288—

293).—Comparison has been made of the yields, activity, and suitability for therapeutic use of the products of precipitation of the crude juice by alcohol, and by alcohol followed by dialysis and reprecipitation by alcohol. The single precipitation by alcohol yields a suitable active product.

R. K. CANNAN.

Preservation of [pharmaceutical] syrups.

T. SABALITSCHKA and E. BÖHM (Pharm. Ztg., 1926, 71, 496–498).—The action of methyl *p*-hydroxybenzoate (“Nipagin” or “Solbrol”) and various other agents against the attack of *Aspergillus* and *Penicillium* and the like on various syrups has been tested. *Syrupus simplex* (60% of sugar), *S. mannae* (55% of sugar), *S. althaeae* (63% of sugar), and *S. rhei* (60% of sugar), all prepared according to D. A.-B. V., were treated with the various preservatives, which in the efficacy of their action against moulds could be arranged as follows:—Thymol = methyl *p*-hydroxybenzoate > benzoic acid = salicylic acid > Mikrobin (sodium *p*-chlorobenzoate) = sodium benzoate. To inhibit completely growth of mould required 0.05% of thymol, or 0.025–0.075% of methyl *p*-hydroxybenzoate, or 0.1–0.15% of benzoic acid. Methyl *p*-hydroxybenzoate is approximately equal to menthol in efficacy, whilst it has the advantage of no disturbing taste; it is superior to benzoic acid in being much less susceptible to the reaction of the medium, and, being practically neutral, it has no action on the substance preserved. It may also be used for the preservation of many other preparations (for internal and external use) against moulds and bacteria.

B. FULLMAN.

Preparation of acetylsalicylic acid, acetanilide, and acet-*p*-phenetidine. P. MANICKE and P. GRIGEL (Arch. Pharm., 1926, 264, 322–324).—Directions are given for quickly preparing the above compounds in the laboratory. Salicylic acid, aniline, or *p*-phenetidine (10 g.) is mixed with acetic anhydride (20, 15, or 10 g. respectively) and to the mixture, which becomes hot, a few drops of concentrated sulphuric acid are added. The interaction being complete, the pasty mass is stirred with water (200 c.c.). The product so obtained is either filtered off directly, or the suspension is boiled to cause the compound to dissolve; it crystallises out again on cooling. Almost the theoretical yield is obtained.

W. A. SILVESTER.

Mechanism of formation of *o*- and *m*-hydroxybenzaldehydes from the nitration product of benzaldehyde. H. H. HODGSON and H. G. BEARD (J.S.C.I., 1926, 45, 91–93 τ).—Brady and Harris's statement (J.C.S., 1923, 123, 484) that not much more than a 65% yield of pure *m*-nitrobenzaldehyde can be obtained from the mixed nitration product of benzaldehyde is confirmed. It follows that the optimum yield of *m*-hydroxybenzaldehyde, as well as the economy of any process adapted for its large-scale preparation, will depend on the separation of the reaction products given by the 20% of *o*-nitrobenzaldehyde present in the raw material. The best results were obtained by reducing the mixed *o*- and

m-nitrobenzaldehydes to the amino-compounds, and converting the latter into the hydroxy-compounds by diazotisation and subsequent decomposition in a current of steam. The *o*-hydroxybenzaldehyde is volatile in steam and hence is continuously separated from the *m*-hydroxybenzaldehyde. In a mixture of *o*- and *m*-nitrobenzaldehydes sodium hyposulphite reduces the latter to *m*-aminobenzaldehyde and the former to *o*-aminobenzaldehyde, possibly via anthranil. Addition of hyposulphite in more than one batch considerably decreases the amount of anthranil ultimately formed. The amines, however, must be immediately diazotised after formation to diminish the loss entailed by their rapid decomposition, which appears to commence at formation, and the sulphuric acid present must be ample to prevent oxime or hydrazine and triazole formation.

Formaldehyde-sodium bisulphite and formaldehyde-sulphurous acid. Determination and properties. W. LEDBURY and R. TAYLOR (J.S.C.I., 1926, 45, 85–89 τ).—The hypiodite method of Romijn for the determination of formaldehyde (Analyst, 1897, 22, 221) gives satisfactory results when applied to the determination of formaldehyde-sodium bisulphite, and since formaldehyde-sodium bisulphite is not acted upon by iodine in acid or neutral solution any uncombined sodium bisulphite present can be determined by direct titration with iodine. Complete combination between formaldehyde and sodium bisulphite in solution does not occur at ordinary temperatures, even on prolonged keeping, but is attained in a few minutes by warming at 70°. Formaldehyde-sulphurous acid can be determined in solution in the presence of uncombined formaldehyde by a direct determination of the acidity, and from the total iodine value of the solution it is possible to determine uncombined formaldehyde. When formaldehyde-sulphurous acid occurs in solution in the presence of sulphurous acid, its concentration can be derived from the acidity of the solution if allowance is made for sulphurous acid, which can be determined iodometrically. Distillations of solutions containing formaldehyde-sulphurous acid with either formaldehyde or sulphurous acid in excess, give rise to distillates and residues containing formaldehyde-sulphurous acid and uncombined formaldehyde, but no sulphurous acid.

Essential oil of manuka (*Leptospermum scoparium*). W. F. SHORT (J.S.C.I., 1926, 45, 96–98 τ; cf. Gardner, J.S.C.I., 1924, 43, 34 τ; 1925, 44, 528 τ; Inder, Thesis New Zealand Univ., Nov., 1923).—1064 lb. of fresh leaves from Birkenhead, Auckland, N.Z., gave on steam-distillation a 0.37% yield of a greenish-brown oil: d_4^{25} 0.9179, n_D^{25} 1.4952, $[\alpha]_D^{25}$ –16.8°; soluble in 10 vols. of 90% alcohol and in 40 vols. of 80% alcohol. The percentage composition of the oil is as follows: *α*-pinene (12%), eudesmene and a second sesquiterpene (60%), citronellol and geraniol both free and as their isovaleric, cinnamic, and acetic esters (6%), citronellal and citral (1%), cineole (1%), leptospermol 8%, azulene (traces), isovaleric, cinnamic, and acetic acids, free and combined (4%). Leptospermol

does not occur in the sodium bicarbonate extract, and Gardner's statement (*loc. cit.*) that it is an acid and not a phenol is probably incorrect. It is probable that the sesquiterpene "manukene" of Gardner, giving a liquid monohydrochloride, is impure eudesmene, since if the crude eudesmene hydrochloride is distilled at pressures above 4 mm., the distillate has approximately the composition $C_{15}H_{25}Cl$.

See also A., May, 468, Influence of sodium hydroxide on adsorption of arsenious acid by "saccharated" iron (HERBOTH). 490, Determination of arsenic in organic compounds (TER MEULEN). 492, Determination of mercury in organic and inorganic compounds (TER MEULEN). 497, Basis for physiological activity of certain onium compounds. Sulphur analogue of choline (RENSHAW, BACON, and ROBLYER). 499, Equilibrium between lactic acid and anhydride in commercial lactic acid (EDER and KUTTER). 500, Selective reduction of citral (ADAMS and GARVEY). 516, Synthesis of hydroxydivarinol (MAUTHNER). 517, α -Naphthylcarbimide as a reagent of alcohols (BICKEL and FRENCH); Preparation of ethers from aromatic alcohols (SENDERENS). 518, Hydrolysis of saccharin (TAUFEL and NATON). 520, Catalytic hydrogenation of the carbonyl group in aromatic compounds (KUBOTA and HAYASHI). 522, α - and β -Amyrin (DISCHENDORFER and RENDI); Saponins and related substances. \mathcal{A} escigenin (VAN DER HAAR).

PATENTS.

Removing nicotine from tobacco. T. SCHLOESING (E.P. 238,517 and 242,225, 29.5.25, Conv., [A] 18.8.24, and [B] 31.10.24. Addns. to 234,845).—(A) In the process described in the chief patent (B., 1926, 465), a substantially constant ammoniacal pressure was permanently maintained. It is now shown that if the ammonia pressure be maintained at a pressure of a certain order, varying, e.g., between 25 g. or less, and 160 g. or more of gaseous ammonia per kg. of steam, then the elimination of nicotine proceeds rapidly and depends only on the quantity of steam passing. The proportion of ammonia need not be carefully regulated.

(B) The kerosene or other solvent used to recover the nicotine extracted as described in the chief patent dissolves some ammonia, which hinders the recovery of nicotine. The ammonia may be removed by passing the solvent through water (if necessary, saturated with a salt such as sodium chloride, which checks dissolution of nicotine); or by bubbling a gas such as air through the solvent, or by passing a current of the gas over the solvent, which is finely divided by causing it to fall in thin sheets or drops, or to flow over solid materials such as pebbles.

B. FULLMAN.

Preparation of salts of *N*-halogen-substituted arylsulphonamides. CHEM. FABR. VON HEYDEN A.-G. (G.P. 422,076, 24.5.24).—Solutions of alkali or alkaline-earth salts of *N*-halogen-substituted arylsulphonamides (cf. G.P. 390,658; B., 1924, 811) are treated with soluble magnesium salts. For

example on stirring an aqueous solution of calcium *p*-toluene sulphochloramide with an aqueous solution of magnesium chloride, magnesium *p*-toluenesulphochloramide separates in crystals containing 23.6% of active chlorine. It is stable and is sufficiently soluble in water to be used for preparing disinfecting solutions. Similarly the magnesium salt of β -naphthalenesulphochloramide is obtained from sodium β -naphthalenesulphochloramide and magnesium chloride, and the magnesium salt of tetrahydronaphthalenesulphochloramide from the corresponding sodium salt (cf. G.P. 377,367; B., 1923, 1149 A) and magnesium sulphate. The products are used as disinfectants and for other purposes. A. COULTHARD.

Production of heterocyclic compounds containing arsenic or antimony. A. BINZ and C. RÄTH (E.P. 250,287, 30.10.24).—Heterocyclic compounds containing arsenic or antimony are prepared by the interaction of diazotised amino-derivatives of pyridine, quinoline, or isoquinoline with arsenites or antimonites or the corresponding free acids. The parent materials may contain substituents such as the hydroxy-, amino-, carboxy-, and phenyl radicals or halogens. The salts of these arsenic or antimony compounds are markedly non-poisonous. It is best to use 2–3 mols. of the arsenite or antimonite to each mol. of the original heterocyclic compound. Reduction of the heterocyclic arsenic or antimonite compounds with, for example, hyposulphites, hypophosphorous acid, or bisulphites converts them into arsenoxides of the type, RA_sO , and further reduction into arseno-compounds of the type, RA_sAsR . These with oxidising agents such as hydrogen peroxide, yield the original arsenic or antimonite acids in a purer form. For example, a diazotised solution of α -hydroxy- β' -aminopyridine is added, drop by drop, to a solution of sodium arsenite. The mixture is made feebly alkaline and kept for some time to complete the reaction. The α -hydroxy- β' -pyridine-arsinic acid obtained from the solution as colourless crystals decomposes at 215°. When heated moderately with hypophosphorous acid, preferably out of contact with air, the arsenic acid gives $\alpha\alpha'\beta\beta'$ -dihydroxydiarsenopyridine, a pale yellow powder. This substance on treatment with cold 3% hydrogen peroxide gives the original acid but free from impurities. A. COULTHARD.

Synthesising higher molecular organic compounds containing oxygen. BADISCHE ANILIN- & SODA-FABR., Assees. of G. AND R. WIETZEL (U.S.P. 1,562,480, 24.11.25. Appl., 25.2.25).—By passing a vaporised aliphatic alcohol mixed with carbon monoxide at elevated temperatures and pressures over a catalyst containing hydrogenating constituents such as copper, silver, gold, tin, lead, or the like, and hydrating constituents such as an oxide of titanium, zirconium, thorium, vanadium, manganese, etc., products containing mixtures of higher alcohols, aldehydes, and esters are obtained. It is of advantage to add an alkali or alkaline-earth metal compound to the catalyst, and if ammonia be added to the carbon monoxide, compounds containing nitrogen are also produced.

The molecular weights and boiling points of the products vary with the time of contact, with the catalyst, and with the temperature. For example, a mixture of methyl alcohol and carbon monoxide is passed at 420° and 200 atm. over a catalyst containing an alkali, chromium, and molybdenum, when a mixture of higher alcohols, aldehydes, and esters derived from valeric and butyric acids and the like is obtained. Ethyl alcohol passed with carbon monoxide free from iron carbonyl at 400° and 200 atm. over a catalyst consisting of zinc oxide, chromium and manganese oxides contained in a tube lined with copper yields products which boil for the most part between 100° and 250°. T. S. WHEELER.

Manufacture of acetic anhydride and acetaldehyde from ethylidene diacetate. M. J. MARSHALL and G. S. SHAW, Assrs. to CANADIAN ELECTRO PRODUCTS CO., LTD. (U.S.P. 1,578,454, 30.3.26. Appl., 26.1.20).—If 1.5% of phosphoric acid be added slowly to boiling ethylidene diacetate decomposition to acetic anhydride and acetaldehyde according to the reaction $\text{CHMe}(\text{OAc})_2 = \text{Ac}_2\text{O} + \text{Me}\cdot\text{CHO}$ proceeds smoothly with but little formation of acetic acid. 1000 g. of ethylidene diacetate treated in this manner yield 238 g. of acetaldehyde, 555 g. of acetic anhydride, 71 g. of acetic acid, and 126 g. of unchanged ethylidene diacetate. T. S. WHEELER.

Producing dialkyl selenides and tellurides. C. A. HOCHWALT, Assr. to GEN. MOTORS CORP. (U.S.P. 1,578,731, 30.3.26. Appl., 3.10.23).—An alkali metal alloy of selenium or tellurium is treated with an alkyl sulphate to obtain the corresponding dialkyl selenide or telluride. For example, 960 pts. of the alloy Na_2Se are finely divided and treated at 100° with 1000 c.c. of ethyl sulphate, water is added, and the diethyl selenide formed is recovered in satisfactory yield by distillation in steam.

T. S. WHEELER.

Preparation of esters of formic acid. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of O. ERNST and K. SPONSEL (G.P. 422,500, 9.3.23).—Carbon monoxide, in the presence of water, is allowed to act on alcohols at raised temperatures with or without increased pressure and catalysts. Methyl formate is formed below 200° under ordinary pressure on leading carbon monoxide, methyl alcohol vapour, and steam over a variety of contact substance including such as favour the taking up and splitting off of water, but the reaction is greatly accelerated by increased pressure. Excess of alcohol improves the yields. For example, a mixture of carbon monoxide and methyl alcohol vapour in the proportion 1:2 with 0.1 vol. of steam when once led over titanium or thallium oxide, under ordinary pressure, at 175°, gives 0.1% of methyl formate. Using zinc and manganese as contact substances with highly activated carbon as carrier, the yield is 1.0% at 260°. With activated carbon at 200° and 200 atm. pressure, the yield of methyl formate rises to 12%. Similar results are obtained with other alcohols. A. COULTHARD.

Manufacture of carbamide. BADISCHE ANILIN- & SODA-FABR., Assees. of W. MEISER (G.P. 422,525,

26.4.22).—Ammonia is caused to react with carbon monoxide under pressure in apparatus lined with lead. The outside wall of the reaction vessel is rigid and has one, or preferably several, small openings, through which any air between the walls, which would otherwise cause folds in the lead on warming, can escape. Leakages through the lead itself can be detected by the hissing and smell of the gases. With these arrangements it is not possible for the melt, should the lead lining be destroyed, to corrode the outer wall and cause an explosion without warning. For example, in iron tube of 48 mm. inner diameter, provided with several holes less than 1 mm. diameter, has a lead tube of 32 mm. inner diameter introduced and pressed on to it. The simplest way of obtaining the openings is to screw rather loosely-fitting plugs into larger holes. A. COULTHARD.

Preparation of a Bz-tetrahydrohydroxyquinoline. J. D. RIEDEL A.-G. (G.P. 423,026, 10.8.23).—6-Hydroxy-2:4-dimethylquinoline is treated with molecular hydrogen in the presence of a catalyst containing nickel. For example, hydrogen under pressure is allowed to act on 6-hydroxy-2:4-dimethylquinoline dissolved in decahydronaphthalene at 180–190° in the presence of nickel. The mixture is diluted with alcohol, filtered, and the solvents distilled off, leaving a crystalline substance insoluble in alkali. 5:6:7:8-Tetrahydro-6-hydroxy-2:4-dimethylquinoline, $\text{C}_{11}\text{H}_{15}\text{ON}$, has m.p. 162°, b.p. 181–183°/15 mm., gives no reaction with nitrous acid; the hydrochloride has m.p. 134°, picrate, m.p. 150°, methiodide, $\text{C}_{12}\text{H}_{18}\text{ONI}$, m.p. 140°. The O-benzoyl compound, m.p. 199–200°, made by warming the base in chloroform solution with benzoyl chloride, is an effective anæsthetic. A. COULTHARD.

Preparation of dicyclic bases. J. D. RIEDEL A.-G. (G.P. 423,027, 14.3.24).—Tetrahydrogenated dicyclic bases having more than three carbon atoms in the hydrogenated heterocyclic nucleus, are prepared by condensing β -phenylethylamine or γ -phenylpropylamine, or their derivatives, with α -halogen-carboxylic acids or their esters, and with acid halides, treating the products with condensing agents, such as aluminium chloride or zinc chloride, to cause ring formation with splitting off of carbon monoxide and hydrogen chloride, yielding *N*-acyl derivatives of dicyclic bases, from which the acyl residue is split off by hydrolysis. For example, ethyl phenylethylglycine, b.p. 157–159°/12 mm., obtained by the action of ethyl bromoacetate on β -phenylethylamine, on evaporation with hydrochloric acid yields phenylethylglycine, $\text{CH}_2\text{Ph}\cdot\text{CH}_2\cdot\text{NH}\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, m.p. 244°; this compound on treatment with benzenesulphochloride yields *N*-benzenesulpho-phenylethylglycine, m.p. 122°, which on heating with phosphorus pentachloride and aluminium chloride in the presence of nitrobenzene as solvent, yields *N*-benzenesulphotetrahydroisoquinoline, m.p. 154°, and this on hydrolysis yields tetrahydroisoquinoline. 6-Methyltetrahydroisoquinoline, m.p. 255–256°, is obtained by treating the *N*-benzenesulpho-derivative of *m*-tolylethylglycine hydrochloride, m.p. 212–214° (obtained from *m*-methylphenylethylamine and

chloroacetic acid) with phosphorus pentachloride and aluminium chloride, and hydrolysis of the product, and homotetrahydroisoquinoline is obtained from γ -phenylpropylglycine, m.p. 201°, by a similar process.
L. A. COLES.

Manufacture of poly-iodine substituted isatins. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 244,443, 23.11.25. Conv., 10.12.24).—A mixture of 5:7-di-iodoisatin, decomp. 234°, and 4:5:6:7-tetraiodoisatin, decomp. 190°, is formed when 2 mols. of iodine monochloride react with 1 mol. of isatin in concentrated mineral acid (e.g., hydrochloric acid) solution. After 1 hr. the liquid is poured into ice water. After 1 day the precipitate is filtered off, steam distilled, the residue dissolved in excess of 0.5% sodium hydroxide, and the mixed iodo-compounds are precipitated by addition of sulphurous acid. Separation is effected by crystallisation from alcohol. The substances are of value for the preparation of pharmaceutical compounds.
B. FULLMAN.

Manufacture of a new iodine substituted oxindole. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 244,444, 23.11.25. Conv., 10.12.24).—*Iodo-oxindole*, decomp. 168°, containing iodine in the nucleus, is thus prepared: a solution of 4.5 g. of oxindole in 100 c.c. of concentrated hydrochloric acid is added to 10 g. of iodine monochloride in 50 c.c. of concentrated hydrochloric acid. After 2 hrs. the mixture is poured into 3 litres of ice water. After 1 day the precipitate is filtered off, steam distilled, the residue dissolved in very dilute sodium hydroxide, and the iodo-oxindole precipitated by addition of sulphurous acid, and crystallised from alcohol. It is used in the preparation of pharmaceutical substances.
B. FULLMAN.

Manufacture of aromatic stibinic acids. CHEM. FABR. VON HEYDEN A.-G. (E.P. 244,746, 4.12.25. Conv., 18.12.24).—The preparation of aromatic stibinic acids (e.g., phenyl-, *p*-chlorophenyl-, and *m*-chloro-*p*-acetamidophenyl-stibinic acids) by the action of antimony oxide on the appropriate diazotised amino-compound is greatly facilitated by the presence of glycerol or other polyhydric alcohols, the formation of by-products being largely inhibited, while the acid formed is easily isolated.
B. FULLMAN.

Manufacturing barbituric acid derivatives. L. THORP, ASSR. to LAMBERT THORP Co. (U.S.P. 1,576,014, 9.3.26. Appl., 20.3.25).—5:5-Dialkylbarbituric acids are prepared by condensing dialkylmalonic esters with carbamide in an absolute alcoholic solution of sodium ethoxide and distilling off the alcohol without preliminary refluxing. Satisfactory yields are thus obtained without the use of elevated temperatures and pressures such as have hitherto been considered necessary. For example, ethyl ethylisopropylmalonate is mixed with carbamide in an ethyl alcoholic solution of sodium ethoxide, the alcohol distilled off at 107°, and the sodium salt of 5:5-ethylisopropylbarbituric acid which remains is treated with hydrochloric acid to yield the free

5:5-ethylisopropylbarbituric acid, m.p. 200–201° corr., of therapeutic value as a sedative.

T. S. WHEELER.

Preparation of aminoketones. C. MANNICH (G.P. 422,098, 30.11.19).—Aminoketones are prepared by the action of mono- or di-alkylamines and formaldehyde, or condensation products of alkylamines and formaldehyde, upon cycloketones. For example, an aminoketone, $\text{MeC}_6\text{H}_4\text{O.CH}_2\text{NMe}_2$, a colourless oil, b.p. 104°/20 mm. (cf. Mannich and Braun, A., 1920, i., 850), is obtained by heating 4-methylcyclohexanone with dimethylamine hydrochloride and 30% formaldehyde for several hours. The ketones and aromatic acid esters of their reduction products are of therapeutic value as anaesthetics.
L. A. COLES.

Preparation of organic derivatives of tervalent vanadium. FARBENFABR. VORM. F. BAYER & Co. (Austr. P. 101,685, 28.7.24).—The compounds, which are of therapeutic value, are obtained by the action of vanadium trioxide or its salts on salts of polybasic organic acids, of hydroxy-acids, or of hydroxy-thioacids, or, alternatively, by treating the free acids with vanadium trioxide or its salts, and subsequently neutralising the products with alkali or alkaline-earth hydroxides or carbonates. Examples are given of the preparation of complex sodium-vanadium compounds of nitrotri-acetic acid (cf. U.S.P. 1,517,003; B., 1925, 115), of tartaric acid, of thioglycollic acid, of salicylic acid, and of mandelic acid.
L. A. COLES.

Separation of hexamethylenetetramine from ammonium chloride. H. WADE. From S. KARPEN & BROS. (E.P. 246,415, 23.10.25).—See U.S.P. 1,566,821; B., 1926, 217.

Production and separation of hexamethylenetetramine and ammonium chloride. H. WADE. From S. KARPEN & BROS. (E.P. 246,416, 23.10.25).—See U.S.P. 1,566,822; B., 1926, 217.

Arsenical albumin compound. O. BALLY, ASSR. to HACO-GES. A.-G. BERN (U.S.P. 1,581,826, 20.4.26. Appl., 23.11.23).—See E.P. 208,143; B., 1925, 189.

Injectable solutions of drugs insoluble in water. SOC. OF CHEM. IND. IN BASLE, Assees. of E. S. FAUST (U.S.P. 1,582,456, 27.4.26. Appl., 15.6.23).—See E.P. 211,771; B., 1924, 400.

Manufacture of glutamic acid and salts thereof. K. IKEDA (U.S.P. 1,582,472, 27.4.26. Appl., 5.1.25).—See E.P. 248,453; B., 1926, 420.

Production of formamide. BADISCHE ANILIN- & SODA-FABR., Assees. of R. FICK (U.S.P. 1,582,675, 27.4.26. Appl., 3.4.25).—See E.P. 237,528; B., 1925, 827.

Preparation of hydroxy aliphatic arsonic acids. C. OECHSLIN, ASSR. to ETABL. POULENC FRÈRES (U.S.P. 1,582,775, 27.4.26. Appl., 25.10.23).—See E.P. 206,152; B., 1925, 83.

Preparation of substituted aliphatic arsonic acids. C. OECHSLIN, Assr. to ETABL. POULENC FRÈRES (U.S.P. 1,582,776, 27.4.26. Appl., 25.10.23).—See E.P. 206,143; B., 1924, 768.

Pharmaceutical product. [Compound of phenylethylbarbituric acid and 4-dimethyl-amino-1-phenyl-2:3-dimethyl-5-pyrazolone.] CHEM. FABRIK AUF ACTIEN (VORM. E. SCHERING), Asses. of A. THIELE (U.S.P. 1,582,802, 27.4.26. Appl., 18.5.23).—See E.P. 198,379; B., 1924, 114.

Silver-protein preparation. L. TAUB, H. JANSSEN, and G. WESENBERG, Assrs. to WINTHROP CHEMICAL Co., INC. (U.S.P. 1,582,940, 4.5.26. Appl., 11.7.22).—See G.P. 392,038; B., 1924, 578.

Fluid for industrial purposes (U.S.P. 1,575,967).—See I.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Conditions governing the behaviour of silver bromide grain during development. II. L. F. DAVIDSON (Phot. J., 1926, 66, 230—242; cf. B., 1925, 151).—Sensitometric data, showing the variations due to the use of different developing formulæ, are given for plates of fast, "special rapid," and ordinary contrasty types, using as developers quinol-caustic soda, pyro-metol, pyro-soda, borax metol-quinol, and *p*-aminophenol. The course of the γ -time of development curves varies markedly for a given plate with the developers used, and the γ_{∞} values vary greatly. Using single-layer plates, the form of the developed grain was studied. Pyro-soda, pyro-metol, and quinol produce the same form of developed grain, with sharply-defined edges, and the original shape is retained to a certain extent. Borax M.Q. gives irregular, feathery grains with thin, diffuse edges, and some grains only partially reduced. *p*-Aminophenol gives a more irregular, spongy grain, all sign of original shape being lost, and some grains are only partially developed; large numbers of single grains remain undeveloped, Borax M.Q. and *p*-aminophenol both give low γ values. Previous results concerning the induction period, and that grains of fast plates show a bigger percentage size increase on development than those of slower plates, are confirmed. The measurements were repeated, plotting the size-frequency curves of the same grains before and after development. Certain size-frequency curves showed a secondary hump after the maximum, on the side of increasing grain size. The curves for the developed grains show the same general characteristics as those for the undeveloped, but there is an increase in range, and a shift in position varying in amount with the developer and plate used, and a new hump at the small grain size end of certain curves, in addition to the two humps in the original curves. The causes of density variation with different developers are discussed.

W. CLARK.

Photochemical studies. VII. Physical development of the latent image. A. REYCHLER (Bull.

Soc. chim. Belg., 1926, 35, 74—80; cf. B., 1925, 265).—Physical development of a bleached and fixed negative and of the true latent image is considered. Ten plates, 9 by 12 cm., exposed and fixed preparatory to physical development by Lumière's method, were stripped and ashed, and an appreciable amount of silver, representing the latent image, was determined. The average number of silver atoms of the latent image per unit area of plate can be determined in this way. With a plate exposed, bromided (with chromic acid-bromide), and washed, and then fixed, a good image can be obtained by physical development with the mercury formula. If the plate is bromided after fixation, only very poor images can be physically developed. These facts indicate that in any grain there are numerous latent image nuclei distributed throughout the body of the grain, but only those on the surface are attacked by the bromiding mixture before fixing. Light acts not only on the surface but in the interior of the grains, and on those molecules of silver bromide of which decomposition is facilitated by a neighbouring gelatin molecule or group of molecules which act as bromine acceptors. The emulsion is a diphasic system in which the gelatin contains colloiddally dispersed silver bromide, and the grains have occluded gelatin; in both cases the substances are intimately mixed and may be chemically united

W. CLARK.

Photochemical studies, VIII. Chemical development of the latent image. A. REYCHLER (Bull. Soc. chim. Belg., 1926, 35, 80—82; cf. preceding abstract).—Results with chromic acid solutions indicate that in chemical development only the latent image nuclei on the grain surfaces are effective. Treatment of a plate in 2% chromic acid is stated not to alter its photographic properties to any great extent. Albert's process for direct positives is discussed. The reason that a latent image nucleus, destroyed by chromic acid, is not formed again at the same point on subsequent exposure, is ascribed to the fact that the silver bromide molecules which give rise to the first nucleus are in the neighbourhood of some part of a gelatin molecule (*e.g.*, CO-NH₂ or CH-NH₂) which readily takes up bromine. When these groups are saturated with bromine, they cannot function in a subsequent exposure. Physical development can be prevented by prolonged treatment of the exposed grains with chromic acid, or by a brief action of chromic acid after fixing out the silver bromide.

W. CLARK.

Photographic spectrophotometry. KELLNER.—See A., May, 483.

PATENTS.

Kinematograph multi-colour films. J. E. THORNTON (E.P. 244,571, 18.5.23).—Raw film stock of the screen-filter type, upon which the component images of a built-up positive film are printed, or upon which the negatives used in printing these images are produced, is made in the form of strips, each of which comprises a celluloid base having a two-colour filter layer formed thereon in

dot, line, or mosaic pattern, and a panchromatic gelatino-silver emulsion coated upon the filter layer, the colours corresponding each to one component of the four- or three-colour picture. The negative film stock is of standard thickness (0.006 in.), and the positive film stock is of half standard thickness. The screen layers are produced by coating the base with an adhesive substratum on which grains of starch, gelatin, resin, etc., dyed in one colour are scattered and rolled in, and dyeing the inter-spaces with the second colour; by coating the base with a layer of gelatin or albumin dyed in one colour and sensitised with dichromate, printing under a dot, line, etc. screen, washing to remove dye from the unprinted parts, and dyeing the interspaces with the other colour; or by a similar process but using a film sensitised with silver salts.

Kinematograph and other colour films. J. E. THORNTON (E.P. 246,257, 17.11.24. Addn. to 224,569-70, 230,965, 233,990, and 231,030; cf. E.P. 213,866 and 224,571, B., 1924, 538, and preceding abstract).—In a multicolour kinematograph positive film of the type described in the previous patents, the two thin films are cemented with the image-face of one to the bare celluloid or unprinted bare gelatin back of the other. This enables all the positive images to be printed from negatives in which all the component images face the same way, none being reversed.

W. CLARK.

Multicolour kinematograph or other films. J. E. THORNTON (E.P. 246,266, 25.11.24. Addn. to 224,569).—Double-width film of half standard thickness carrying a sensitised colloid is printed with two side-by-side rows of colour component positive images from a double-width negative, finished, and dried. The printed surface is then re-coated with a sensitiser and two further rows of images are printed, superimposed on the first. The film is then severed longitudinally and the two strips are superimposed in register and cemented. The first pair of prints may be made by a silver sensitising process and the superimposed second pair by a dichromate sensitising process, or by using salts of iron, uranium, etc. Alternatively, the first pair of prints may be made by a dichromate method and the superimposed pair by a sensitising process using other metal salts, such as those of silver, iron, uranium, etc.; or both first and second pairs of prints may be made by the same process, and in relief and/or non-relief, or the first pair of prints may be made by a sensitised colloid process, and superimposed by a second pair produced photomechanically without light action on an insensitive surface.

W. CLARK.

Kinematograph and other positive films. J. E. THORNTON (E.P. 246,282, 19.12.24).—For preparing a scratchless film of the enclosed-image type, the two printed colloid faces of the separate thin films are treated (in some cases after partially softening the colloid faces) with exceedingly thin layers of cellulose cement which is allowed partially to penetrate. The two moistened surfaces are

then united by pressure. The printed faces of both films may be roughened or toothed, then treated with dilute cellulose cement, and finally pressed together. The colloid faces may alternatively be both roughened and softened before cement is applied. The cellulosic cement consists of a cellulose compound such as celluloid, cellulose acetate, etc., a suitable solvent, *e.g.*, amyl acetate, acetone, etc., and a solvent or partial solvent of the gelatin or other printed colloid used, *e.g.*, glacial acetic acid. To this cement may be added for greater flexibility a colloid such as gelatin, glue, or fish-glue in a solvent such as glacial acetic acid, and a substance such as glycerin, syrup, or sugar, for increasing the flexibility.

W. CLARK.

Kinematograph and other colour positives. J. E. THORNTON (E.P. 246,283, 19.12.24).—The material for preparing multicolour transparent positives consists of two different kinds of image-layers for producing two different kinds of images. One layer is sensitised with a panchromatic gelatino-silver bromide emulsion for giving temporary negative black images; the other layer is unsensitised and consists of interspersed fine lines, dots, etc., of coloured colloid for giving multicoloured permanent positive images. The two layers are on opposite sides of the same double-width support, or else superimposed on the same side, the silver layer being over the colloid layer. The silver layer covers the whole double-width film, while the colloid layer forms two parallel stripes each containing two colours, those of one being different from the colours of the other stripe. After treatment the printed material is divided and the two parts are superimposed and cemented face-to-face, back-to-back, or back-to-face. In printing, the silver emulsion layer is exposed through a pair of positives of the double-filter-screen side-by-side type, each filter consisting of two colours; the images are developed, fixed, washed, and dried; the multicoloured colloid layer is sensitised with dichromate, dried, exposed to light through the previously-formed silver images, and developed in hot water, and the black silver image is then dissolved away.

W. CLARK.

[Non-inflammable] cellulose acetate composition [photographic film]. L. J. MALONE and S. J. CARROLL, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,575,778, 9.3.26. Appl. 2.8.20).—Flexible transparent films, free from inflammable nitrocellulose, are obtainable from compositions prepared by mixing a solution of cellulose acetate in acetone with castor oil which has been previously incorporated with a substance such as acetylene tetrachloride, methyl alcohol, methyl salicylate, or tricresyl phosphate, having the property of inducing transparency in the film.

T. S. WHEELER.

Manufacture of photographic films, papers and the like. KODAK, LTD., W. G. BENT, and R. E. CROWTHER (E.P. 250,770, 5.5.25).—A medium, *e.g.*, an aqueous solution containing 0.25% of citric acid and 0.1% of Phenosafranine, which reduces the photo sensitiveness of the emulsion, and/or

prevents the subsequent development of any latent image immediately below or above the medium, is applied to the surface of the support of a photographic film, etc., or to the sensitised coating thereof. The method is applicable more especially to films, etc., upon which rulings intended to indicate the scale of the photograph are to be marked. J. S. G. THOMAS.

Light-sensitive medium and process of producing it. M. C. BEEBE, Assr. to WADSWORTH WATCH CASE CO. (U.S.P. 1,574 356, 23.2.26. Appl., 8.3.22).—The light-sensitive varnish-like medium prepared as described in E.P. 203,285 (B., 1924, 493) can be used as a resist for etching purposes and renders practicable the direct reproduction of a photographic image on metal. T. S. WHEELER.

Photographic print-out paper. J. A. JOHNSON (U.S.P. 1,582,050, 27.4.26. Appl., 25.4.21; cf. B., 1923, 1103A).—An emulsion of a silver compound other than silver iodide is treated with an iodide solution to form a silver iodide emulsion, and then with a sensitiser to make the silver iodide sensitive to light. W. CLARK.

Treatment of waste cinematographic films. D. B. MACDONALD (U.S.P. 1,582,847, 27.4.26. Appl., 6.5.22).—See E.P. 191,091; B., 1923, 203 A.

XXII.—EXPLOSIVES; MATCHES.

See A., May, 480, Comparison between explosion temperatures calculated from specific heats and from explosion pressures (MURAOUR), 503, Nitration of cellulose (CARRIÈRE).

PATENT.

Drying smokeless powders. J. C. VICKERY and M. H. DAVIS (U.S.P. 1,579,262, 6.4.26. Appl., 29.2.24).—A gaseous medium, previously partly saturated with the solvent by means of a spray, is heated, circulated over the powder, restored to its condition of partial saturation, and recirculated through the system. This cycle of operations is repeated until the powder has reached the desired degree of dryness. D. J. NORMAN.

XXIII.—SANITATION; WATER PURIFICATION.

Detection and determination of free chlorine in drinking water. I. M. KOLTHOFF (Chem. Weekblad, 1926, 23, 203—204).—To 100 c.c. of the water acidified with acetic acid are added sodium acetate solution and a few drops of a 0.1% solution of dimethyl-*p*-phenylenediamine in alcohol, and the mixture is kept for 10—15 min. A red coloration develops, the intensity being proportional to the chlorine content; a standard iodine solution treated in the same way is used for a comparison test if quantitative results are required. The test will detect 0.03 mg. of chlorine in 1 litre. S. I. LEVY.

Natural and artificial filtration [of water] in connexion with goitre. J. DON (Kolloid-Z.,

1926, 38, 334—335).—A discussion of the connexion of goitre with the iodine content of Dutch waters.

N. H. HARTSHORNE.

Experiments on the purification of sewage water on aero-filters in 1923 [in Moscow]. (Miss) N. A. BAZIAKINA (Moscow Communal Management: 5th Rept. of the Committee for the Purification of Sewage (Russ.), 1925, 1, 127—136).—The results are given of a series of experiments on the purification of Moscow sewage waters by means of two aero-filters, each 3.5 m. in height and charged with slag of 10—25 mm. size in one case and 2—10 mm. in the other. The water, which first passed through a decantation tank, was free from nitrates and dissolved oxygen, had an oxidisability number of 65.0 (by the Kubel method; acid solution, 10 min. at 100°), and contained 109.6 mg. of chlorine and 69.0 mg. of ammoniacal nitrogen per litre. Under the conditions employed, the output per day of the aero-filters was normally 15 cub. m. per day per sq. m. and rose as high as 20 cub. m.; this output greatly exceeds that of percolation beds. The volume of air necessary for the normal functioning of the purification is from 4 to 6 times that of the water treated, the pressure in the air-pipes being from 4 to 20 mm. of water. With the finer slag the duration of the purification is about 25 min., and with the coarser slag about 12 min.

T. H. POPE.

Velocity of dissolution of oxygen as one of the factors of biological [sewage] purification. (Miss) N. A. BAZIAKINA (Moscow Communal Management: 5th Report of the Committee for the Purification of Sewage (Russ.), 1925, 1, 139—159).—Since biological oxidation of sewage water is effected at the expense of the dissolved oxygen, increase in the rate at which the oxygen dissolves, which in aeration tanks and bacteria beds is slow, would accelerate the purification. The rate at which oxygen is dissolved is proportional to its deficiency, so that $dx/dt = k(b-x)$, where b is the amount of oxygen in solution saturated with air, x is the amount actually present at any time, and k is a constant. The value of $(b-x)$ depends on the speed with which the activated sludge transmits the oxygen for the oxidation, and k depends on the intensity of the aeration, the method of distributing the air, the temperature, etc. With percolation beds the ventilation is defective and the partial pressure of the oxygen consequently low. In aeration tanks, with distribution of the air through perforated pipes, the surface of contact between the air and the liquid is considerably increased, whilst in aero-filters there is perfect combination of the activated sludge, the sewage water, and the air, resulting in maximum velocity of dissolution of the oxygen and hence of the purification process. Aeration tanks, on the other hand, allow of greater variation in the duration of the purification, and are hence better adapted to yield effluents of any desired quality. T. H. POPE.

Present state of the treatment of sewage by means of activated sludge. S. N. STROGANOV

(Moscow Communal Management: 5th Report of the Committee for the Purification of Sewage (Russ.), 1925, 1, 177—304).—A historical and critical survey of this question.
T. H. POPE.

Bactericidal action of nitroso-compounds. E. A. COOPER and G. E. FORSTNER (J.S.C.I., 1926, 45, 94—96 T).—The group of organisms comprising *Bacillus pyocyaneus*, *B. fluorescens liquefaciens* and *non-liquefaciens* are more susceptible than *B. coli* to those disinfectants, e.g., phenol, the bactericidal action of which appears to be due to their interference with the colloidal condition of the bacterial protoplasm, and which are therefore termed "physico-chemical" germicides. On the other hand, those disinfectants which react chemically with the protoplasmic constituents selectively attack *B. coli*, and the *B. pyocyaneus* group are extremely resistant to their action. From bacteriological results it is concluded that nitroso-compounds belong to the second class of bactericidal substances. Chemical evidence for the foregoing biological differentiation has been obtained by a study of the action of nitroso-compounds on proteins. It would appear that these compounds react chemically with the cell-globulins and proteoses, and this reactivity may account for their high germicidal power. An iodometric method for determining nitrosophenol is described.

Bactericidal action of the cyclotelluropentanediones and their derivatives. G. T. MORGAN, E. A. COOPER, and A. E. RAWSON (J.S.C.I., 1926, 45, 107—109 T).—The cyclotelluropentanediones are much more active germicides than various other tellurium derivatives that have been examined, and bactericidal power in general bears no relation to the percentage of the metalloid present in the substance. The cyclotelluropentanediones are also considerably more efficacious than the corresponding oximes. The outstanding bactericidal power of the foregoing germicides disappears in the presence of serum, and this collapse in activity has been shown to be due to the presence of small amounts of fats and fatty acids, which apparently adsorb the tellurium compounds from dilute solution and thus render them unavailable for disinfection.

Effectiveness of certain paraffin derivatives in attracting flies. W. C. COOK (J. Agric. Res., 1926, 32, 347—358).—A number of aliphatic alcohols and esters have been tested in regard to their attraction for flies over a uniform period of exposure of 24 hrs. The most attractive concentration bears a close relation to the boiling point of the compound, decreasing rapidly with a rise in boiling point. Further, the relative attractiveness of these compounds decreases with rise of boiling point. The addition of a CH₂ group to the acid radical reduces the attractiveness much more than addition of the same group to the alcohol radical. *iso*-Compounds are relatively more attractive than their normal isomerides.

C. T. GIMINGHAM.

Corrosion of iron pipes. BAYLIS.—See X.

Fungicidal properties of spray fluids. GOODWIN, MARTIN, and SALMON.—See XVI.

Toxicological investigations on mandibulate insects. CAMPBELL.—See XVI.

PATENTS.

Purifying of boiler sludge and the heating and supply of boiler feed water. NECKAR WATERREINIGER MAATSCHAPPIJ, and J. HERINGA (E.P. 250,061, 16.6.25).—The invention comprises a combination of a heater, purifier, and tank for feed water and an expansion tank for blow-down water from a boiler. The sludgy water from a boiler is blown off into the expansion tank, the steam from which is mixed with and heats the purified feed water and at the same time provides an atmosphere of steam in the tank, preventing solution of gases from the atmosphere. The separated sludgy water, reduced to a temperature of about 100°, if pure (except for suspended matter) or containing useful excess purifying agents from a previous treatment, passes to the feed water purifier where it mingles with raw feed water. If, on the other hand, the water contains dissolved harmful salts such as sodium chloride or sulphate, part or all of it passes through an indirect heater for the feed water and is then discarded. B. M. VENABLES.

Softening water. Water-softening material. R. G. TELLER, Assr. to PERMUTIT Co. (U.S.P. 1,571,891-2, 2.2.26. Appl., [A] 27.5.16, [B] 6.9.21).—Hard water is passed through a granular bed of base-exchanging material prepared from a natural clay. Clay containing a substantial proportion of hydrated silicate of a zeolitic nature is cut into small pieces, immersed in a hot solution of sodium chloride, dried at ordinary temperatures, baked at about 110°, granulated, then (A) heated below 700° for $\frac{2}{3}$ —1½ hrs. and finally treated with a hot solution of sodium chloride, or (B) heated to temperatures above 700° and re-hydrated by immersion in a hot solution of caustic soda. W. T. LOCKETT.

Water purifier. A. L. GRANT (U.S.P. 1,574,477, 23.2.26. Appl., 13.12.23).—The permanent hardness of water is removed by treatment with finely divided barium silicate, preferably in the presence of magnesium carbonate (contained in the water or added thereto), sufficient in quantity to satisfy the equation, $\text{CaSO}_4 + \text{MgCO}_3 + \text{BaSiO}_3 = \text{MgSiO}_3 + \text{CaCO}_3 + \text{BaSO}_4$. W. T. LOCKETT.

Base-exchange water softener. O. R. SWEENEY (E.P. 244,303, 7.3.25).—See U.S.P. 1,557,117; B., 1926, 110.

Obtaining boiler feed water by distillation. INTERNAT. GEN. ELECTRIC Co., INC., Asses. of ALLGEM. ELEKTRICITÄTS-GES. (E.P. 247,567, 10.2.26. Conv., 10.2.25).

Preparation of sterilising agents, germicides, and the like. C. H. H. HAROLD, Assr. to UNITED WATER SOFTENERS, LTD. (U.S.P. 1,581,115, 20.4.26. Appl., 3.10.25).—See E.P. 245,820; B., 1926, 302.