

B.—APPLIED CHEMISTRY

NOVEMBER 12, 1926.

I.—GENERAL; PLANT; MACHINERY.

Theory of fine grinding. II. G. MARTIN, E. A. BOWES, and J. W. CRISTELOW. III. G. MARTIN, E. A. BOWES, and F. B. TURNER (Trans. Ceram. Soc., 1926, 25, 51—62, 63—81; cf. J.S.C.I., 1926, 160 T).—II.—A method of determining accurately the surface of crushed sand particles consists in exposing the material to the solvent action of hydrofluoric acid under definite conditions of time and temperature, measuring the loss in weight, and equating this against the loss in weight of a specimen of quartz having an accurately measurable surface, such as a cube. The experiments were carried out with 5*N*-acid in considerable excess, a water thermostat being used to maintain a temperature of 25°. The acid and sand, in a bakelite container, were shaken together for one hour by means of special apparatus. The loss of weight of a known surface of quartz under the conditions of these experiments was 0.0000418 g./sq. cm. using *N*-acid. III.—To determine the relation between the work expended and the degree of fineness (surface produced) in grinding sand, a given weight of sand was ground for varying periods in a ball mill. The power consumed was accurately measured by electrical apparatus, and the surface of the crushed sand was determined by the hydrofluoric acid method (see above). The results showed that, in ordinary tube mill grinding, the surface produced is directly proportional to the work done. To avoid the "cushioning" effect of the dust in extremely fine grinding, the dust must be removed in an air stream. The work required in grinding sand to any given degree of fineness with a given grinding medium is found by the formula: $W = B(S_2 - S_1)$ ft.-lb., in which S_2 is the final surface of the powder in sq. ft., S_1 the original surface of the sand in sq. ft., and B a constant peculiar to the grinding medium and the material ground, representing the work required to increase the surface by 1 sq. ft. B varies considerably with the grinding medium used; with 1-in. steel balls in an 18 × 18-in. mill, it is 60.9 ft.-lb. F. SALT.

Determination of the relative surface areas of powdered materials. W. A. KOEHLER (J. Amer. Ceram. Soc., 1926, 9, 437—443).—Relative surface area measurements were made, on the assumption that, for a given material, the adsorption of thorium-*B* is a function of the surface, regardless of the degree of fineness of the particles. From the amount of thorium-*B* adsorbed, which is determined with an electroscope, the surface area relations were calculated. The thorium-*B* solution was dissolved either in distilled water or in ethyl alcohol, with or without the addition of ammonia. Equal weights of various powdered materials were placed in equal volumes of the radioactive solution, and, after equilibrium had been established, 10 c.c. of the clear

supernatant liquid from each sample were evaporated to dryness. The relative radioactivities of the residues were then measured with the electroscope. Since the adsorption does not vary directly with the quantity of adsorbing material present (this relation being expressed approximately by the Freundlich adsorption isotherm), it was necessary to determine adsorption curves for each material. The method is illustrated by reference to quartz sand. For silica and silicon carbide, 2 c.c. of concentrated ammonia solution per 100 c.c. of thorium-*B* solution were required to effect adsorption, whilst with kaolin the results were vitiated owing to the slaking action of aqueous solutions. The use of anhydrous alcohol instead of water still gave erratic results with this material. F. SALT.

Proposed British standard table of wire screens. II. O. C. ELLINGTON (J.S.C.I., 1926, 45, 353—354 T; cf. B., 1926, 471).—Amended proposals are made for a standard table, in which the screens are designated by whole numbers of ten-thousandths of an inch, expressing with sufficient accuracy the average aperture widths required. It has been confirmed (1) that the nomenclature proposed will present no difficulty to weavers; (2) the use of half-gauge wires is unnecessary and inadvisable; (3) no coarser screens can be included in the proposals, since such are not normally machine-woven; (4) brass and phosphor-bronze wires are to be preferred to those of copper, because of their greater strength, durability, and resistance to corrosion; (5) although the screens proposed will not meet all special and peculiar trade requirements, they are suitable for all normal screening purposes, and form a suitable basis for authoritative standardisation.

Problems in connexion with ancient Egyptian materials.—A. LUCAS (Analyst, 1926, 51, 435—450).—Some of the unsolved problems in connexion with the following ancient Egyptian materials are described:—flint implements, faience, glass, metals, mortar and plaster, oils and fats, pigments and varnish, pottery, resinous materials, bitumen, coloured mummy hair, stone, and textile fabrics. D. G. HEWER.

Thermoregulator. J. HUME (J.S.C.I., 1926, 45, 246 T).—A regulator, requiring no relay, is made from a steam-pressure gauge, the mechanism of which acts as a switch. Movement is caused by the expansion and contraction of the toluene with which the apparatus is filled.

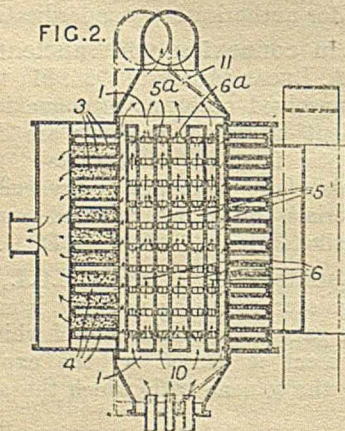
See also A., Oct., 1001, **Adsorptive power of charcoals** (SURUN); **Adsorption of gases by wood charcoal** (MAGNUS and KAHN); **Wood charcoal as adsorbent for gases** (MAGNUS).

Miscometer. HOUSTON.—See XIX.

PATENTS.

Steam or other vapour power plants utilising [A] caustic potash, [B] soda, [C] caustic soda, or like auxiliary fluid. E. KOENEMANN (E.P. 242,979—81, 6.11.25. Conv., [A—B] 14.11.24, [C] 12.11.24).—(A) The bulk of the lye is taken from a mixer at an intermediate pressure and has its pressure raised by a pump, and then passes forward through a heat interchanger to a primary evaporator which is heated from an external source. The more concentrated lye returns through the heat interchanger and a throttle valve to the mixer. The vapour from the primary evaporator does work in an engine and is then sprayed into the mixer. A smaller part of the lye is passed by a circulating pump through the heating coil of a secondary evaporator, and returned to the mixer, also in the form of spray, where its combination with the primary vapour produces heat. Low-pressure vapour from the secondary evaporator does useful work and is condensed. In (B) the mixer is in thermal contact with the secondary evaporator and the lye passing forward through the heat interchanger is raised to a higher pressure than in the primary evaporator, throttle valves being used after the lye has passed the heat interchanger in each direction. In (C) two units, each comprising a mixer and an evaporator in thermal contact, are used, and the flow of lye is from the first mixer through a pump to the second evaporator, to the first evaporator through a throttle to the second mixer, and back to the first mixer. There is also a rapid local forced circulation of lye between the bottom and top of each mixer. Vapour from a power plant enters the first mixer and produces heat by combination with the lye and the heat passes by conduction to the adjoining evaporator. Vapour from the latter does work and passes to the second mixer. Heat from the last produces vapour in the second evaporator, which is also available for work. B. M. VENABLES.

Devices for drying, roasting, or otherwise treating materials in bulk. O. DOBBELSTEIN (E.P. 250,224, 26.3.26. Conv., 4.4.25. Addn. to 226,543; cf. B., 1925, 655).—A slowly rotating drum is divided into annular



compartments used alternately for heating (4) and for containing the material (3). The annuli for heating are provided with radial guiding partitions, alternate sides of which are in communication with passages, 5, for heating gases, or 6 for exhaust gases in the central

part of the furnace. The passages 5 are closed at one end of the furnace and the passages 6 at the other.

B. M. VENABLES.

[Continuous] kiln. H. FREEBOROUGH (U.S.P. 1,590,935, 29.6.26. Appl., 7.3.22).—A multi-chamber kiln has fire boxes extending along the sides of each chamber, one wall of each fire box terminating near the top of the kiln. Channel-like members extend across each kiln near the end, and communicate with the space beneath the perforated floor. The channel of one chamber is connected by a passage with the fire box of the next. Additional channels, placed equidistantly between the end channels, also communicate with the space beneath the floor. F. SALT.

Tunnel kiln. W. E. WILSON and H. G. LYKKE (U.S.P. 1,590,462, 29.6.26. Appl., 14.6.24).—The cooling zone of a tunnel kiln has a convection space above the trucks carrying the ware. F. SALT.

Pulverising mill. STRACHAN TUBE MILL Co., Assees. of C. B. STRACHAN (E.P. 252,331, 29.3.26. Conv., 25.5.25).—See U.S.P. 1,583,324; B., 1926, 520.

Production of dry liquefied gases. J. Y. JOHNSON. From BADISCHE ANILIN & SODA FABRIK (E.P. 257,372, 22.6.25).—See F.P. 600,028; B., 1926, 392.

Centrifugal apparatus for dust separation [from gases]. J. KEITH & BLACKMAN Co., LTD., and G. KEITH (E.P. 257,301, 27.3.25).

[Centrifugal] apparatus for separating or expelling solid impurities from air, combustion gases, etc. O. STOTT (E.P. 257,321, 25.5.25).

Dust collectors or separators and the like. DAVIDSON & Co., LTD., and J. WHITMORE (E.P. 257,403, 14.8.25 and 5.12.25).

Recuperative heat exchange apparatus for air or other media. A. SANDBERG (E.P. 245,111, 18.12.25. Conv., 19.12.24).

Woven fabrics for use as air filters. HOOVER, LTD. From HOOVER Co. (E.P. 257,484, 14.1.26).

Emulsifying apparatus. J. MCGOUGAN and J. HUNTER (E.P. 257,704, 4.7.25).

Air seals for furnaces with travelling grates. L. WINTER, and KLEB & BARK G.M.B.H. (E.P. 252,201, 17.5.26).

Purification and reclaiming of lubricating oil. AKT. SEPARATOR, Assees. of F. R. C. BOYD (E.P. 240,438, 9.9.25. Conv., 26.9.24).

Evaporating and crystallising apparatus (E.P. 252,686).—See XVII.

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

Change in weight of coal during prolonged oxidation at low temperatures. D. J. W. KREULEN (Fuel, 1926, 5, 345—346).—A Scotch (Wemyss Leven) coal was ground to pass 80 meshes per cm., and 5 g. were heated at 105°; the loss in weight during intervals of time for 4000 hrs. was determined and a circular graph constructed. A sudden loss in weight of 7.92% first

occurs, due to the removal of water, and the subsequent changes, due to oxidation, take place in three distinct periods. In the first there is an increase in the weight of the coal, in the second (968—1275 hrs.) there is an equilibrium between the increase in weight by fixation of oxygen and the loss in weight due to the decomposition of the coal, and in the third period there is a loss in weight.

A. C. MONKHOUSE.

Determination of fineness of coal dust. E. F. GREIG (Safety in Mines Research Board, Paper No. 25, 1926, 31 pp.).—The size of particles and their specific surfaces (surface per unit mass of dust particle) may be determined from measurements of linear dimensions (microscopical counting, sieving), by methods based on terminal velocities (sedimentation, elutriation, electrical precipitation), or by empirical methods (bulk-density, heat of wetting, angle of repose, filtering constants). Sieving is considered insufficiently accurate; air-elutriation methods provide means of obtaining grades of dusts of definite ranges of specific surfaces for the purpose of correlating specific surface and degree of inflammability of a dust-cloud; by a combination of elutriation, sedimentation, and microscopic examination it is possible to grade the specific surfaces of dusts. Some of the empirical methods (*e.g.*, percolation through a bed of the material under test) may be useful for rapid determinations and field use.

W. T. K. BRAUNHOLTZ.

Combustion of particles of coal in air. Cenospheres. II. H. E. NEWALL and F. S. SINNATT (Fuel, 1926, 5, 335—339).—Six coal seams were examined in connexion with the formation of cenospheres (*cf.* B., 1925, 87). Cenospheres were obtained only from the vitrain and clarain of coking seams, and not from durain or non-coking seams. Ignition points were obtained for a number of coal dusts by allowing the coal to fall through a heated tube at 650° and measuring the mean distance fallen before ignition occurred. Individual screened particles from three coals were similarly examined and it was concluded that with a caking coal a cenosphere is first formed, and the films or "windows" then undergo oxidation, leaving a particle consisting of lattices, which subsequently burn. Cenospheres and particles of carbonised durain examined at 600°, 650°, and 700° were found to be more readily inflammable than the original coal particles, due in the case of the cenosphere to the larger surface per unit mass, and with durain to the tarry matter on the surface.

A. C. MONKHOUSE.

Influence of moisture on the process of combustion, especially of coke. P. OBERHOFFER and E. PRIVORSKY (Stahl u. Eisen, 1926, 46, 1311—1320).—The effect of moisture in the blast on the process of combustion of coke in an open-shaft furnace has been investigated in a series of tests in which observations have been made of the maximum temperature obtained in the furnace, the thickness of the zones of maximum temperature and of maximum carbon dioxide content, composition of the flue gas, and rate of combustion of the coke. With a blast of relatively low humidity the maximum temperature is higher; the zones of maximum temperature and maximum carbon dioxide content are lower and the carbon monoxide content of the flue gases is greater the higher the moisture content of the air.

Above a certain limit of humidity the maximum furnace temperature begins to decrease with an increase in the moisture, until when the air is nearly saturated a rapid fall in the maximum temperature takes place, accompanied by a rapid decrease in the carbon dioxide content of the gases and a corresponding increase in the carbon monoxide content of the flue gas. The introduction of the moisture in the form of spray into the tuyères instead of in the form of vapour in the blast causes a more rapid gasification of the coke and a maximum temperature 50—60° higher to be obtained. Increasing the rate of supply of blast or its temperature up to 400° improves the process of combustion in a manner similar to that obtained by the addition of a little moisture to an almost dry blast. An explanation of the beneficial effect of small quantities of moisture is given, based on a theory of surface adsorption of moisture.

A. R. POWELL.

Examination and valuation of coking coals.

R. KATTWINKEL (Fuel, 1926, 5, 347—355).—A comparison is made with coals from six Westphalian coke-oven plants of the crucible test, the tube-distillation (Bauer) test, and the caking index (Meurice) test as compared with the results obtained in large-scale practice. The Meurice coking test is modified by using a mixture of coal and sand in the proportions of 1 : 10 and a crushing strength apparatus with lead shot in order to apply a gradually increasing load. The experimental methods are found to be suitable for the classification of coals, but the results are not sufficiently comparable with those obtained in practice. The theories of Fischer (B., 1925, 233) correlating the bitumen content with the coking properties of coal are similarly deficient, being based on crucible carbonisation.

A. C. MONKHOUSE.

Thermal decomposition of coal in presence of catalysts and under hydrogen pressure. A. R. BOWEN and A. W. NASH (Fuel, 1926, 5, 361—364).—Arley coal was heated for 2—3 hrs. up to 480° with hydrogen under pressure in an autoclave (B., 1926, 474), in the presence of nickel and iron oxides, stirring being carried out by the admixture of small steel cylinders to the charge. Under comparable conditions 45.64% of oil products was obtained with nickel oxide as compared with 30.95% with iron oxide. The proportion of phenols and bases in the oils obtained with both oxides was approximately the same, and independent of the oil yield; mean values were phenols, 9.21%, bases, 6.41%. The ether-insoluble and chloroform-soluble fractions were less and the light spirit fraction was greater with increased temperature. Iodine values showed the lighter fractions to be not fully saturated.

A. C. MONKHOUSE.

Parr process of low-temperature carbonisation of coal. W. R. CHAPMAN (Fuel, 1926, 5, 355—361).—The process is primarily designed for the carbonisation of coal of high oxygen content and is carried out in two stages. The coal, ground to $\frac{1}{4}$ -in. size, is fed into a cylindrical rotating retort of 8 in. diam., horizontally mounted and heated externally by flue gases and gas burners. This preliminary carbonisation takes place slowly to 100° and then rapidly to 300°, which is 50° below the temperature of the plastic stage of the coal. The charge (400 lb.) is then transferred from the preheater to a steel vertical retort 12 ft. high and 12—14 in. diam.

maintained at 750–800° and the products of distillation are collected. In this stage, due to an exothermic reaction equivalent to 65 B.Th.U. per lb. of coal, rapid carbonisation occurs, yielding a coke of 3–4% volatile matter suitable for a domestic fuel. The total time of carbonisation is 4 hrs.

A. C. MONKHOUSE.

cyclopentanone in the distillation products of lignite. D. VORLÄNDER and W. GÖRNANDT (Z. angew. Chem. 1926, 39, 1116–1117).—The presence of *cyclopentanone* (0.1%) in the crude fraction, b.p. 110–140°, obtained in the distillation of lignite has been detected by the formation of the crystalline-liquid anisylidene-*cyclopentanone* (cf. Vorländer, A, 1921, i, 867) on treatment with anisaldehyde and sodium hydroxide. The ring ketone is more soluble in water than is generally recognised in the literature, and may be extracted by shaking the crude oil with water, and the aqueous distillate and wash liquors are rich in acetone and *cyclopentanone*. Treatment of the crude oil with sulphuric acid at ordinary temperature or washing with alkalis as employed in the purification of motor spirit, removes 90% of the *cyclopentanone*. The higher fraction of the oil (b.p. 140–180°) contains a very small quantity of *cyclopentanone*, and no *cyclohexanone* was found in either fraction.

J. W. BAKER.

Peat. I. Thermal decomposition of peat under reduced pressure. J. REILLY and G. PYNE (Sci. Proc. Roy. Dublin Soc., 1926, 18, 329–341).—In the rapid distillation of dry peat in a vacuum there are obtained less ammonia, acetic acid, methyl alcohol, and gaseous products and more tar, especially of the higher b.p. or more paraffinic fraction, than is the case in distillation at the ordinary pressure. The difference is probably due to less secondary decomposition of the more complex products. The gases evolved are similar in the two cases.

G. M. BENNETT.

Effect of anti-knock materials on spontaneous ignition temperatures of some inflammable liquids. Y. TANAKA and Y. NAGAI (Proc. Imp. Acad. Tokyo, 1926, 2, 221–223).—Small amounts of diethyl selenide, lead tetraethyl, pyridine, or aromatic amines raise the spontaneous ignition temperatures of alcohols very considerably, but have little or no effect on those of hydrocarbons or ethyl ether.

R. CUTHILL.

Firedamp explosions: projection of flame. M. J. BURGESS (Safety in Mines Res. Bd., Paper 27, 1926, 14 pp.).—The distance of projection of the flame produced on igniting a mixture of methane and air has been determined under varying conditions. The apparatus consisted of a glass tube, 5.5 cm. in diameter in one series of experiments and 9 cm. in a second series, which was divided into two parts by a brass shutter. One part contained the combustible mixture, which was ignited by a spark between electrodes at the end of the tube remote from the shutter. The latter was opened just before ignition so that the flame was projected into the other part of the tube, the distance of projection being determined by visual observation in a darkened room. The diameter of the aperture through which the flame was projected could be varied. The projection of the flame into air in an unobstructed tube was between five and six times the length of the original column of

explosive mixture, mixtures richer in methane giving a longer projection than weak mixtures of corresponding explosive power, owing to the subsequent combustion of the excess of methane in the air. Reduction of the diameter of the aperture increased the length of the projected flame for mixtures containing an excess of methane, except for very small apertures, but decreased the length for the weak mixtures. The projection of the flame into an atmosphere of carbon dioxide was shorter than in air, being about three times the length of the original column of explosive mixture with an unobstructed tube. Reduction of the diameter of the aperture decreased the distance of projection of the flame into carbon dioxide. The use of carbon dioxide at the mouth of a stopping when sealing off a gob fire is suggested in order to minimise the distance of projection of flame should an explosion occur behind the stopping.

A. B. MANNING.

Detection of firedamp by electrical means. C. S. W. GRICE and A. G. GULLIFORD (Fuel, 1926, 5, 364–368).—To indicate the presence of methane to the user of a miner's electric lamp, a piece of platinum fuse wire 3 mm. long and 0.35 mm. diameter, is fitted into the bulb circuit of the lamp, so that, when an electric current of 2.0 volts is passed through, it will fuse if exposed to an atmosphere containing 3% of methane. The fuse wire is held in a replaceable fuse-holder which fits into two socket contacts in the base of a perforated brass cylinder, protected by wire gauzes, attached to the side of the lamp. A switch is also provided to re-light the bulb after the circuit is broken. The detector is inaccurate for voltages below 1.8, but is reliable for the normal 8-hour discharge of a 2-volt accumulator in good condition.

A. C. MONKHOUSE.

Removal of carbon dioxide from industrial gases by washing [with water] at ordinary pressure. F. FISCHER and P. DILTHEY (Brennstoff-Chem., 1926, 7, 277–282).—Water is fed in at the top of a tower, packed with coke or Raschig rings, up which the gases pass, and, having taken up carbon dioxide, flows by gravity from the bottom of the tower into the top of another, exactly similar, tower up which air is passed, and in which the carbon dioxide is again expelled from the water, the latter being returned, in closed circuit, to the first tower. For optimum results, with a gas mixture containing about 20% of carbon dioxide and at a temperature of about 20°, the ratio of gas : air : water is 1 : 10 : 2 by volume, and an effective tower space of 0.5 cub. m. is required to wash 1 cub. m. of gas per hour. Under these conditions an initial gas mixture containing 21.9% of carbon dioxide, 75.9% of carbon monoxide and hydrogen (together), 0.2% of oxygen, and 2.0% of nitrogen yields a washed gas containing 2.6% of carbon dioxide, 89.7% of carbon monoxide and hydrogen (together), 1.6% of oxygen, and 6.1% of nitrogen. Coke is a rather more efficient tower packing than Raschig rings.

W. T. K. BRAUNHOLTZ.

Determination of free sulphur in spent gas-purifying material. D. STAVORINUS (Gas- u. Wasserf., 1926, 69, 790–791).—Methods of determining sulphur in spent oxide are criticised. The removal of tar from the sulphur when extracted by carbon disulphide has

been effected at the Rotterdam gasworks by adsorption on active charcoals, the most suitable being finely powdered "medicinal norit." A piece of glass tubing drawn to a fine point is packed with a piece of cotton wool pressed gently down; on to this is placed a charge of active carbon, preferably "medicinal norit," and then a weighed sample (10 g.) of the spent oxide under test. The tube is clamped upright with its end dipping into a tared evaporating basin. Carbon disulphide is poured in to fill the tube; the tarry and oily matters are retained on the active carbon, and the sulphur solution passes into the dish, where the solvent evaporates. Further lots of carbon disulphide are poured through the tube until in all 150 c.c. have been used.

R. A. A. TAYLOR.

Cracking of tars and mineral oils at atmospheric pressure without addition of hydrogen, compared with the Bergius process. H. HERBST (Petroleum, 1926, 25, 947—949; cf. László, B., 1926, 523).—In the "berginisation" of lignite mixed with coal tar, the tar cannot be recovered as such. It may be advantageous to crack the coal tar and the tar obtained from lignite, at ordinary pressures without hydrogen. The coke can be gasified and worked up into synthetic petroleum-like products by known methods. The high temperature of berginisation favours the elimination of hydrogen. The unsaturated compounds arising from the original cracking process may be converted into saturated substances by treatment with hydrogen at a comparatively low pressure. Removal of unsaturated compounds by the Edeleanu process has the disadvantage that traces of sulphur dioxide are difficult to remove, and extraction with organic solvents is preferable. W. N. HOYTE.

See also A., Oct., 1018, **Detection and determination of carbon monoxide** (KAST and SELLE).

Refractories for gas plants. COLE.—See VIII.

PATENTS.

Manufacture of agglomerated fuel. L. LIATS (E.P. 251,605, 9.4.26. Conv., 30.4.25).—Sodium bicarbonate, either solid or in solution, is added to the pitch or tar binder used in making briquettes. The effervescence produced by the carbon dioxide generated in the binder causes it to spread more efficiently over the particles of the briquetted material, and the proportion of binder required to produce the same strength is consequently reduced.

R. A. A. TAYLOR.

Production of finely divided carbon at a low temperature. BRAUNKOHLN PRODUKTE A.-G. (G.P. 431,933, 29.5.24).—Producer tar or crude oils rich in asphalt, if necessary with the addition of charcoal or wood, are heated to 300—500° under pressure so that a part of the oil remains liquid during the reaction. The resulting carbon may be purified by washing with hot water. Alternatively the oily raw material is heated to 300—500°, and is passed through a tube which is partly throttled.

W. G. CAREY.

Method of burning pulverised fuel. A. L. MOND. From E. H. PEABODY (E.P. 256,685, 12.5.25).—The main body of air, supplied either by forced or induced draught, is put in rotation by a series of vanes and enters the furnace through a bell-mouth formed in the furnace

wall. Just behind the furnace wall the pulverised fuel suspended in air enters in the form of a thin sheet of annular form, which also rotates and enters the main stream of air from the outside at an angle to the axis of flow. Just behind the pulverised fuel entrance is a central oil burner for starting or maintaining combustion.

B. M. VENABLES.

Operating coke ovens. R. B. KERNOHAN (U.S.P. 1,596,048, 17.8.26. Appl., 12.9.24).—A horizontal coke oven is divided into two vertical chambers by a non-combustible core. Heating takes place both from the core and from the oven walls, causing vertical planes of cleavage in the coke in both chambers.

A. C. MONKHOUSE.

Coke oven. M. W. DITTO, Assr. to CHICAGO TRUST Co. (U.S.P. 1,596,408—9, 17.8.26. Appl., 14.1.22. Renewed 8.1.26).—(A) The oven is constructed with a series of U-shaped flues, with vertical arms in the two oven walls and a cross connexion in the sole structure. The combustion chambers are at the upper end of each arm, with connexions to the regenerators for the admission of air and gas and the removal of waste gas. (B) In the heating walls of the oven is arranged a mesh of interconnected flues which transfer the gases horizontally to a series of combustion chambers at each end of the wall in which are gas nozzles. A series of laterally spaced ports connect the heating flues between the combustion chambers.

A. C. MONKHOUSE.

Apparatus for extracting gas from bituminous materials. E. W. HARTMAN, Assr. to HARTMAN INTERESTS, INC. (U.S.P. 1,595,301, 10.8.26. Appl., 31.5.21).—An apparatus for gasifying solid or liquid bituminous material, such as shale or heavy petroleum, consists of a cylindrical retort with a vertical axis fixed on an annular firebox arranged to heat all the bottom plate. Mounted in the axis of the retort is a hollow vertical rotating shaft, which drives a series of rakes adapted to stir the charge on the bottom of the retort, and through which the gas formed is removed. Means are provided for feeding the solid or liquid material into the retort, and for withdrawing residue when required.

T. S. WHEELER.

Continuous carbonisation and cracking of bituminous materials under pressure. K. BUBE (E.P. 256,845, 22.12.25).—A pulp obtained by mixing bituminous materials, e.g., lignite, with liquid hydrocarbons, e.g., lignite producer tar, is pumped through vessels capable of resisting pressure at or above 300°. The process is continuous, the products being withdrawn so that the level in the reaction vessel is kept constant.

A. C. MONKHOUSE.

Gas generators. F. UMPLEBY (E.P. 257,023, 15.8.25).—The plant consists of a central cylindrical chamber, in which any suitable solid or liquid fuel may be gasified by means of air or steam, or both. The chamber is surrounded for heating purposes by surface-combustion burners made of H-shaped bricks of refractory material, arranged polygonally, which form, with the wall of the generating chamber, a channel for air, and, with the outer casing, a channel for fuel gas.

The products of combustion pass down a channel in the wall of the generating chamber, and communicate their heat to it, and they may pass into the generator.

R. A. A. TAYLOR.

Production of hydrogen from water-gas and steam. M. FRÄNKL (G.P. 431,758, 7.7.25).—A temperature of 1500–1800° is maintained continuously in a highly refractory furnace by using pure oxygen with a powdered coal, or a gas, flame. A mixture of water-gas and steam is passed through the furnace, the carbon monoxide reacting with the steam to produce hydrogen and carbon dioxide.

W. G. CAREY.

Thermal decomposition of gaseous hydrocarbons, natural gas, etc. by heat. I. SZARVASY (U.S.P. 1,592,474, 13.7.26. Appl., 23.2.24).—Gaseous hydrocarbons are passed into a chamber maintained at decomposing temperature, and provided with pervious walls. The chamber may be heated by gases passing through the walls and burning in proximity to them.

H. MOORE.

Petroleum still. L. J. HUSTED, Assr. to F. L. ROUNSEVELL (U.S.P. 1,596,660, 17.8.26. Appl., 4.3.20. Renewed 28.12.25).—A series of horizontal cylindrical stills are connected end to end, division walls being left between the adjacent stills. A group of pipes runs parallel to the axis of the stills from end to end of the series, and hot liquid is forced through the pipes. In each still petroleum is sprayed upwards against the hot pipes, the vapours are condensed separately, and the residue is drawn off.

W. N. HOYTE.

Cracking petroleum oil. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,592,560, 13.7.26. Appl. 27.11.22).—The oil is passed in a restricted stream through the coolest part of a heating zone, and then united with the reflux condensate formed in the process. The mixture is passed in a restricted stream through the hottest part of the heating zone, and thence to an enlarged reaction zone, from which no oil returns to the stream mentioned. The vapours from the enlarged zone are passed to a dephlegmator, where part is condensed to form the reflux condensate, while the vapours pass to a condenser. Pressure above atmospheric is maintained on the oil.

H. MOORE.

Cracking hydrocarbons. E. C. HERTHEL and E. W. ISOM, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,592,489, 13.7.26. Appl., 20.11.24).—Oil is circulated to and from a body of oil in a supply tank through a heater in which it is raised to cracking temperature. The oil from the heater is passed to the upper stratum of the tank, while oil is withdrawn from the lower stratum for recirculation through the heater. Free flow downwards in the tank is obstructed, causing stratification of the oil.

H. MOORE.

Cracking of hydrocarbons. SINCLAIR REFINING Co., Assees. of E. C. HERTHEL and H. L. PELZER (E.P. 246,116, 22.12.24. Conv., 17.1.25; cf. B., 1925, 907; 1926, 352).—In pressure tube or shell stills for cracking heavier oils to produce motor spirit, oxides, such as copper oxide, but not iron oxide or bauxite, are introduced into the oil, which is continuously circulated through them. The oxides are supported on a bed of asbestos, firebrick, coke,

fuller's earth, etc. on a perforated screen. They exercise a selective action, retaining asphaltic and sulphur-containing substances which tend to form deposits on the surfaces of the still. The oil containing an excess of asphaltic substances may be gradually withdrawn and replaced by fresh charging stock.

H. MOORE.

Distillation of lubricating oils from mineral oils. SIMPLEX REFINING Co., Assees. of G. A. KRAMER (E.P. 251,928, 5.8.25. Conv., 9.5.25).—The oil to be distilled, preferably one topped to 320°, is preheated and charged into a still, in which pressure is reduced to and maintained at about 5 mm.; external heat is applied and steam is blown in so that the vapours contain about 10% by weight of steam. An air condenser causes the temperature of the vapours to drop 6–30°, and so to deposit the lubricating oil, which is drawn off. Some steam and oil of low b.p. are then condensed in a water condenser, and a steam ejector raises the pressure of the vapours to about 50 mm., when the remainder of the steam and oil are condensed.

W. N. HOYTE.

Process for breaking water-in-oil [petroleum] emulsions. M. DE GROOTE, Assr. to W. S. BARNICKEL & Co. (U.S.P. 1,596,585–1,596,598, 17.8.26. Appl., 23.12.24, 6.4.25, 29.5.25, 1 and 7.10.25).—The following means are employed in breaking emulsions: passing the emulsion through a non-absorbent material, such as pure zinc, on which a demulsifying agent is held by adsorption; filtering the emulsion mixed with demulsifying agent through a bed of sand; treating emulsions with equal quantities of non-saponaceous demulsifying agent and a substance capable of forming brine-in-oil emulsions; treating emulsions with a soap obtained from a shale oil sludge or a sulphonated shale oil sludge; treatment with a lactone or a sulphonated condensation product containing a polycyclic aromatic compound and a fatty material, or with an ester of a naphthenic acid or with the condensation product of a sulphonated naphthenic acid and an aromatic hydrocarbon; treatment with an oil-soluble hydrophobe compound in which water has been combined by a hydrophobe soap; bringing the emulsion to incipient breaking point without dilution by treating with a suitable agent and then added further emulsion; treating emulsion with a highly colloidal coagulescent suspension containing a soap-forming radical.

W. N. HOYTE.

Apparatus for recovering heat from coke, slags, ashes, and other solid substances. P. BRINGHENT (U.S.P. 1,597,718, 31.8.26. Appl., 9.6.24. Conv., 18.7.23).—See E.P. 228,406; B., 1925, 232.

Complete gasification of bituminous fuel. A. BREISIG (U.S.P. 1,598,473, 31.8.26. Appl., 25.9.22. Conv., 25.5.21).—See E.P. 207,651; B., 1924, 122.

Purification of gas. HUMPHREYS & GLASGOW, LTD., Assees. of W. H. FULWEILER (E.P. 248,355, 15.2.26. Conv., 27.2.25).—See U.S.P. 1,589,749; B., 1926, 699.

Removing tar from the vapours arising from the carbonization of wood. E. A. BARBET (U.S.P. 1,598,547, 31.8.26. Appl., 20.10.21. Conv., 21.10.20).—See E.P. 170,591; B., 1923, 343 A.

Removing hydrogen sulphide from gases

W. GLUUD (U.S.P. 1,597,964, 31.8.26. Appl., 11.9.22. Conv., 20.9.21).—See E.P. 186,316; B., 1923, 887 A.

Liquid fuel burners. W. A. HEDWORTH (E.P. 257,687, 11.6.25).

Atmospheric gas and oil burners. C. M. HUNTER (E.P. 257,416, 18.9.25).

Carbon black, gases, etc. (U.S.P. 1,597,277).—See XIII.

III.—TAR AND TAR PRODUCTS.

Cracking of tars. HERBST.—See II.

PATENTS.

Tar distillation apparatus. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,596,050, 17.8.26. Appl., 6.7.21).—A tar boiler is fitted with longitudinal fire tubes joined by removable sections outside the boiler. The tubes are connected at one end to the source of gas supply and at the other to the flue. Provision is made for the expansion of the boiler during distillation. A. C. MONKHOUSE.

Decreosotising tars and tar products. K. BUBE (E.P. 257,151, 22.12.25).—The material is extracted with mixtures of alcohol and water containing more water than alcohol, at temperatures above 50°, under the ordinary or slightly raised pressure. Alcohol is recovered from the solution by distillation without the use of a dephlegmator, until the creosote constituents separate from the residual liquor. For example, lignite tar oil containing 14% of creosote is extracted at 83° with a mixture of 75 pts. of water and 25 pts. of alcohol; the residual oil then contains 3% of creosote. L. A. COLES.

Manufacture of prepared tar for road-making, etc. L. H. SENSICLE (E.P. 257,024, 21.5.25).—Tar is charged at 55–60° into a high-speed centrifugal separator, after which the moisture content is further reduced to 0.5% or less by passing a current of cold air through the tar maintained at a temperature sufficiently high to remove moisture but not to cause chemical action, e.g., at about 60°, or at a somewhat higher temperature if it is desired to remove naphthalene and light oils as well as water. Bitumen and solid material removed from the bowl of the centrifuge, as well as other materials, such as tar distillates or pitch, may be added to the tar during the air treatment. L. A. COLES.

IV.—DYESTUFFS AND INTERMEDIATES.

Action of chlorine on aniline. Formation of induline dyestuffs. I. D. G. BROWN and W. M. CUMMING (J.S.C.I., 1926, 45, 355–360 T).—The action of chlorine on aqueous aniline (3%) at ordinary temperature results in the formation of azophenine contaminated with aniline-black. At low temperature in an inert water-free solvent, such as carbon tetrachloride, an almost theoretical yield of *s*-trichloroaniline is obtained. The action of chlorine on boiling aniline produces ultimately an induline dyestuff with evolution of hydrogen chloride. This induline, which is the hydrochloride of a base, can be recrystallised from alcohol, the free base being liberated by means of alkalis. The induline in acetic acid solution gives a yellow leuco-

compound with sodium hyposulphite (hydrosulphite), which is almost immediately reoxidised in air. When the induline is heated with fuming hydrochloric acid at 300° in a sealed tube, aniline and diphenylamine (the latter by a side reaction) are formed as well as a greenish-black substance with characteristic fluorescence. The parent substance of the dyestuff is therefore very stable. From an investigation of the high-temperature chlorination it was suspected that the chloro-substituted anilines were responsible for the formation of the dyestuff. It was found ultimately that *p*-chloroaniline when heated with aniline hydrochloride to melting point, forms an induline with evolution of ammonia. The analytical figures indicate that the formula for the hydrochloride prepared by direct chlorination is $C_{36}H_{25}N_6Cl_2 \cdot 2HCl$, whilst the base from *p*-chloroaniline is $C_{36}H_{27}N_6Cl$. It is therefore believed that the parent substance contains a phenazine nucleus, and is represented by $C_{36}H_{28}N_6$, the nucleus being $C_{12}H_4N_2$. Suggested configurations for the molecule are given and discussed. Absorption spectra of the two dyestuffs indicate that the configuration is the same in each case. The dyeing properties of the hydrochloride and base are described.

Identification of naphthalenoid reduction products of azo dyes. R. B. FORSTER and T. H. HANSON (J. Soc. Dyers and Col., 1926, 42, 272–275).—Reduction is effected by acid stannous chloride or hyposulphite. The ammoniacal solutions of the azo coupling components, chiefly aminonaphtholsulphonic acids, are spotted on filter paper with hydrochloric acid, ferric chloride, vanadium chloride, potassium dichromate, nickel chloride, uranium sulphate, potassium ferrocyanide, potassium ferricyanide, copper sulphate, Schweizer's reagent, ferrous ammonium sulphate, cobalt nitrate, and silver nitrate. Many of these give well-defined colour reactions, which are tabulated, and described in detail. J. S. H. DAVIES.

Method for distinguishing α - and β -naphthols. F. LEWISCH (Textilber., 1926, 7, 863).—A carmine-red coloration develops immediately when 10 c.c. of concentrated sulphuric acid are poured over 0.1 g. of α -naphthol in the presence of a piece of bleached cotton, the coloration changing to a permanent deep reddish-violet within 1 hr. Under similar conditions, β -naphthol yields a brown coloration which changes to bluish-green. When the coloured solutions are diluted six times, the reddish-violet and bluish-green colorations change to bright violet and green respectively. These reactions are used to detect the presence of traces of α -naphthol in β -naphthol or *vice versa*. A. J. HALL.

See also A., Oct., 1028, 2:3:4-Trinitrotoluene (GORNALL and ROBINSON). 1030, *o*-Toluidine (TANNER and LASSELLE). 1031, **Optically active dyes** (BRODE and ADAMS). 1032, **Derivatives of benzene- and toluene-azonaphthylamine** (TRÖGER and SCHAEFER). 1043, **Delphinidin chloride 3-methyl ether** (GATEWOOD and ROBINSON); **Synthesis of peonidin chloride** (NOLAN and others). 1049, **Determination of methoxyl** (NIERENSTEIN); **Determination of primary aromatic amines** (GRIGORJEV). 1050, **Determination of benzidine and its analogues** (HERZOG).

PATENTS.

Manufacture of [chromed azo] dyestuffs. SOC. CHEM. IND. IN BASLE (E.P. 247,556, 9.2.26. Conv., 14.2.25).—Azo dyes derived by coupling a nitrated *o*-hydroxydiazo compound with an α -naphthylamine substituted in the *ortho*-position to the amino-group are treated with salts, oxides, or hydroxides of trivalent chromium. For example, 48.9 pts. of the sodium salt of the azo dye obtained from picramic acid and naphthionic acid are dissolved in 900 parts of boiling water, 18 pts. of chromium oxide in the form of a 4% solution of chromium fluoride are added, the whole is boiled under reflux in presence of glass powder for 18–24 hrs., and the new dye salted out. It dyes wool a fast dark olive-green shade. A. DAVIDSON.

Manufacture of [azo] dyestuffs containing chromium. O. Y. IMRAY. FROM SOC. CHEM. IND. IN BASLE (E.P. 257,820, 4.2.26).—A solution of the azo dye from 6-nitro-2-aminophenol-4-sulphonic acid and β -naphthol is boiled under reflux with a solution of chromium fluoride in presence of glass powder. The dye, which contains chromium, is separated by evaporation and salting out. Similar dyes are obtained by using other agents yielding chromium such as chromium acetate or formate or neutral freshly precipitated chromium hydroxide. The products dye wool in reddish-black shades fast to light and fulling. (Cf. E.P. 26,460 of 1912 and 116,535, B., 1913, 905; 1918, 460 A.) A. DAVIDSON.

Manufacture of [azo] dyestuffs [from barbituric acids]. O. Y. IMRAY. FROM SOC. CHEM. IND. IN BASLE (E.P. 257,797, 15.12.25).—Barbituric acids of the general formula $R(N < \begin{smallmatrix} CO \cdot NH \\ CO \cdot CH_2 \end{smallmatrix} > CO)_n$, in which R = H, alkyl, aryl, or alkaryl, and $n = 1$ or 2, are coupled with an *o*-hydroxydiazo compound. The products are acid mordant dyes which dye wool in red to brown shades which change, when chromed, to brown and red shades fast to fulling and potting and very fast to light. The dyes themselves, by treatment with chroming agents such as oxides, hydroxides, or salts of trivalent chromium, yield acid dyes which dye wool in uniform fast shades of yellow to red and rose. For example, 233 pts. of 4-sulpho-2-aminophenol-6-carboxylic acid are diazotised and added to a mixture of 134 pts. of barbituric acid, 133 pts. of 30% sodium hydroxide solution, 170 pts. of sodium carbonate, and 1500 pts. of water. After stirring for several hours, the dye is filtered off. The preparation of a chromed dye is carried out, for example, by boiling a solution of 37.2 pts. of the above dye in 600 pts. of water with a solution of chromium formate corresponding with 15.2 pts. of Cr_2O_3 . The barbituric acids are obtained by condensing carbamides of the general formula $R(NH \cdot CO \cdot NH_2)_n$ in which R and n have the significance already mentioned, with a malonic ester. A. DAVIDSON.

Yellowish-red azo dyestuffs. H. WAGNER and A. FUNKE, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,595,269, 10.8.26. Appl., 8.10.25).—Diazotised *m*-nitro-*p*-aminophenyl alkyl ethers, especially the ethyl or methyl derivatives, are coupled with an *o*-alkyl- or *p*-alkoxy-anilide of acetoacetic acid, e.g., acetoacet-*o*-

toluidide, *p*-anisidide, or *p*-phenetidide, to give products which dye fabrics yellowish-red shades, which in the form of lakes are very fast to light.

T. S. WHEELER.

Manufacture of anthraquinone dyestuffs possessing affinity for acetyl silk. BRIT. DYESTUFFS CORP., LTD., W. H. PERKIN, and C. HOLLINS (E.P. 257,353, 4.6.25).—By the condensation of a primary anthraquinonylamine with α -aminoanthraquinone, or α -diaminoanthraquinones, or simple derivatives of these such as diaminoanthrarufin or diaminochryszazin, in presence of anhydrous calcium chloride or iodine as condensing agent, at 200–300°, new substances are obtained which are probably anthraquinonylimino-anthrone. The products dye acetyl silk from an aqueous suspension. A. DAVIDSON.

Anthracene dye [from dihalogenopyranthrones]. I. GUBELMANN, ASSR. to NEWPORT CO. (U.S.P. 1,596,528—31, 17.8.26. Appl., [A, B] 4.4.25, [C, D] 9.5.25).—Dichloro- or dibromo-pyranthrone is heated with (A) 1-aminoanthraquinone or a derivative such as 1-amino-2-methylanthraquinone or 6-chloro-1-aminoanthraquinone, (B) 2-aminoanthraquinone or a derivative, (C) carbazole, or (D) aminodibenzanthrone, when condensation occurs, one of the halogen atoms of the pyranthrone derivative being eliminated with a hydrogen atom attached to the nitrogen atom in the other compound. The products dye cotton from a hyposulphite vat violet (A, B, C) or dull bluish-violet (D) shades oxidising in air or on treatment with oxidising agents to rich brown shades fast to washing and light. Reaction is effected by heating the components at about 200° for about 15 hrs. in a suitable liquid such as nitrobenzene or naphthalene in presence of an acid-binding agent, such as anhydrous sodium carbonate or acetate, and of a catalyst such as basic copper acetate. In the reactions described under (C) and (D) the shades obtained vary slightly with the time of heating. T. S. WHEELER.

Manufacture of phenols [from benzene hydrocarbons]. W. J. HALE, ASSR. to DOW CHEMICAL CO. (U.S.P. 1,595,299, 10.8.26. Appl., 7.1.24).—An autoclave two-thirds filled with an emulsion of equal volumes of 20% aqueous caustic soda solution and benzene containing an oxide of uranium, with or without small proportions of oxides of vanadium, molybdenum, and tungsten, is heated at 320–400° for 1 hr., the internal pressure being maintained at about 200 atm. The oxygen in the air above the liquid in the autoclave is absorbed with formation of phenol, which dissolves in the alkali. Only a small proportion of the benzene is converted in each operation, but as there are no side reactions, that remaining after separation from the alkaline liquid can at once be used again.

T. S. WHEELER.

Azo colouring matter dyeing wool fast to milling. J. BADDILEY, J. HILL, and A. RILEY, ASSRS. to BRITISH DYESTUFFS CORP., LTD. (U.S.P. 1,598,109, 31.8.26. Appl., 21.9.25. Conv., 17.9.24).—See E.P. 243,115; B., 1926, 85.

Azo dyestuffs and intermediates. E. B. HIGGINS (U.S.P. 1,597,791, 31.8.26. Appl., 29.11.24. Conv., 19.12.23).—See E.P. 230,920; B., 1925, 437.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Distribution of nitrogen in the flax plant and its elimination from flax during manufacture. J. W. PORTER (J.S.C.I., 1926, 45, 335—337 T).—The nitrogen content of flax at all stages of manufacture from the dried flax straw to the bleached yarn is given. The nitrogen is located in the cortical and epidermal cells, and from the nitrogen figures deductions are made as to the completeness of the bleaching operations. Reference is also made to the potash and phosphate content of the cortical and epidermal cells.

Pectin contents of flax. W. KIND (Spär- u. Wirtsch., 1926, 17, 12; Textilber., 1926, 7, 872).—Irish water-retted, Courtrai, Russian, and Peuffaillit-retted flax contained 7.02, 4.46, 6.45, and 5.78% of pectins respectively. A brown powder containing 84.67% of pectin was obtained by extracting flax stalks with a 1% solution of ammonium oxalate. Pectins remaining in bleached linen have a considerable influence on its lustre, appearance, and tensile strength. A. J. HALL.

Load-elongation curves and lustre of artificial silk. K. ATSUKI (Cellulose Ind., Tokyo, 1926, 2, 218—226).—The load-elongation curves have been determined for three types of Japanese viscose silks, wet and dry, as compared with cotton and natural silk. For the artificial silks the tensile strength is lower, the elongation is greater, and the loss of strength on wetting is greater than for cotton. The ratio stress : strain is much lower for viscose silk than for cotton. These inferior mechanical properties are attributed to the modification of the cellulose, the heterogeneity of the cellulose particles and the irregularity of their arrangement, and to the presence of lower grade impurities such as β -cellulose. The load-elongation curves of the different samples of viscose silk vary considerably, especially under small loads, some showing a pronounced elastic limit and yield point, whilst one had a smooth curve right up to the break point without definite elastic limit or yield point. The latter had a soft damp feel considered to be inferior for weaving purposes. The form of the curve may be considerably influenced by the ageing and conditions of coagulation of the viscose. The lustre is influenced by the surface structure, the fineness of the filament, and the transparency or the arrangement and size of the cellulose particles and the existence of minute gas cavities. J. F. BRIGGS.

Valuation of artificial silks. N. U. BERCHIN (Chem.-Ztg., 1926, 50, 643).—The numbers obtained by multiplying together the elasticity (expressed as a percentage) and the wet and dry tensile strengths (expressed in g. per denier) of artificial or natural silk yarns are considered to be suitable for use in comparing their commercial value. For example, artificial silk yarn having 20% elasticity, 2.0 and 0.65 g. tensile strength dry and wet respectively ($20 \times 2.0 \times 0.65 = 26$), is considered superior to artificial silk yarn having 25% elasticity, 1.3 and 0.55 g. tensile strength dry and wet respectively ($25 \times 1.3 \times 0.55 = 17.67$). The method of valuation may be extended so as to include lustre and purity of white colour. A. J. HALL.

Action of oxygen on alkali-cellulose. W. WELT-

ZIEN and G. ZUM TOBEL (Papier-Fabr., 1926, 24, 413—414).—Cotton or artificial silk cellulose, when swollen with sodium hydroxide, absorbs oxygen freely, even at the ordinary temperature, and the reaction becomes very pronounced at 40°. Observations made with cotton impregnated with 3 times its weight of 10% sodium hydroxide showed a steady absorption of oxygen at 60°, amounting to 2 atoms of oxygen per mol. of $C_6H_{10}O_5$ after 35 days, with no indication of any break in the process after further exposure. J. F. BRIGGS.

Chemistry of the sulphite wood pulp digestion. E. HÄGGLUND (Papier-Fabr., 1926, 24, 449—450, 483—488; cf. B., 1925, 238, 586; 1926, 312).—In the first stage of the digestion there is an absorption of the bisulphite from the liquor to form an insoluble ligno-sulphonic acid or salt in the wood, which is later liberated by acid hydrolysis from its union with the carbohydrates and enters the liquid phase in the soluble form. The lignin of under-digested sulphite pulp exists in the form of the insoluble lignosulphonic acid; it possesses fluorescent properties; it cannot be extracted by washing with dilute alkali, but on boiling with 1% hydrochloric acid it is hydrolysed, rapidly at first, but later very slowly. After boiling with acid for 1 hr., 65% of the contained sulphur and methoxyl of the crude sulphite pulp had been removed, but after 8 hrs. the residue still retained 18% of the original lignosulphonic acid contents. Hence it appears that simultaneously with hydrolysis, under the prolonged action of acids, the lignosulphonic acid undergoes irreversible polymerisation. To this polymerisation, with its attendant resinification, the phenomenon of "blackened," over-digested charges may be attributed. The hydrogen-ion concentration of the bisulphite liquor may be measured by the sucrose-inversion method; it decreases in the early stage while bisulphite is being fixed by the lignin of the wood, and increases later during the hydrolysis stage. The pentosans are hydrolysed at first more rapidly than the hexosans, but later the latter are more profoundly attacked. Comparative digestions with calcium, magnesium, and ammonium bisulphites showed but slight differences in chemical mechanism. J. F. BRIGGS.

Violet fluorescence of sulphite wood pulp and waste liquors. O. GERNGROSS and K. TSOU (Papier-Fabr., 1926, 24, 497—499).—Sulphite wood liquors and unbleached sulphite pulp show a strong lilac fluorescence in ultra-violet light, which changes to bright green by the action of alkali, and is gradually destroyed during bleaching. The substance with this property has been located in the free state in the living secondary bark layers of fresh spruce wood, but it also exists in a latent and firmly combined form in the wood itself. The fluorescence is observed in the light of a Hanau analysis quartz lamp and shows up very strongly when the outer bark is lifted with a knife. Aqueous extracts of spruce bark, as used for tanning, are exceptionally rich in this fluorescent substance and show the phenomenon even when highly diluted. From acid or neutral solutions the fluorescent substance is irreversibly absorbed by cellulose fibres, both in spruce-bark extracts and in sulphite waste liquors, although in the latter the fixation properties on

cellulose are considerably weakened. Although the free, water-soluble fluorescent substance is derived only from the secondary bark layers, wood which has been carefully separated from these layers and shows none of the characteristic lilac fluorescence develops this substance when digested with calcium bisulphite at 115°. The digestion liquor acquires the same properties as ordinary sulphite waste liquor and the fibre residue is firmly "dyed" with the violet fluorescence. Simple boiling of wood with water is not sufficient to liberate the fluorescent substance, but if a piece of cotton cellulose be digested with wood and water for 8 hrs. at 110°, the cotton acquires a slight and somewhat modified violet fluorescence, which changes to bluish-green with alkali, instead of to the characteristic bright green. At the same time the wood shows no fluorescence, but the water shows the characteristics of the normal fluorescence. This proves that the fluorescence of unbleached sulphite pulp is not due to a lignosulphonic acid, but to a natural constituent of the wood.

J. F. BRIGGS.

Testing the strength of [paper] half-stuffs. H. SCHWALBE (Papier-Fabr., 1926, 24, 465—468, 481—483).—The testing of half-stuffs is carried out by the preparation of sheets of paper under carefully standardised conditions. For routine tests in the pulp mill the fibre may be conveniently prepared in the Lampen ball mill, but for the complete investigation of a new type of pulp the ball mill must be supplemented by beating trials in a properly designed hollander capable of treating stuff at the normal 5% concentration. The hollander tests must be controlled by determinations of the degree of beating by sedimentation or Schopper's apparatus. The formation of the sheets is the most difficult operation, and accurate conditions must be established. Sheets should be made at a substance of 100 g./m.² The pressure used for compacting the sheets has a great influence on the strength of the paper; a press adjusted at 35 kg./cm.² is used. Conditions of drying the sheets, preferably on a heated metal surface, must be standardised; careful adjustment of atmospheric humidity is necessary when performing the tensile tests.

J. F. BRIGGS.

Aluminium resinate in the sizing of paper. E. OMAN (Papier-Fabr., 1926, 24, 410—413, 451—455).—Aluminium resinate, freshly prepared and dried without heat, is completely soluble in ether, benzene, and carbon tetrachloride; it is insoluble in alcohol, which decomposes it, dissolving only a portion of free resin acid. It also undergoes decomposition on exposure to heat, after which ether selectively extracts a portion of free resin acid and the aluminium resinate becomes insoluble. Aluminium resinate is very sensitive to acid conditions; a hydrogen-ion concentration of p_H 3.5 equivalent to the degree of acidity in the beater liberates a considerable proportion of free resin acid. Sulphite pulp also modifies the precipitated aluminium resinate so that it is no longer completely soluble in ether even when dried at the ordinary temperature, whilst the pulp after drying at 115° only yields 40—55% of its total resin on extraction with ether. Acidification is necessary for the quantitative extraction of total resin. An ethereal solution of aluminium resinate is capable of sizing

paper; a suspension of precipitated neutral aluminium resinate also possesses sizing properties, but gives results inferior to those obtained when a certain amount of free resin is liberated by acidification either with alum or with sulphuric acid. A hydrogen-ion concentration of p_H 5.5 produced with excess of alum gave a slightly better sizing than one of 3.5 produced with sulphuric acid. The inferiority of the neutral resinate as a sizing material is ascribed partly to the coarser dimensions of its particles and partly to the absence of sintering on the drying cylinders. Under ordinary conditions of sizing with slight acidity no important part can be ascribed to the precipitation of aluminium hydroxide in the fibre. If aluminium is fixed at all in acid conditions it is in exchange for calcium ions, and it may thus confer on the fibre a positive charge whereby the negatively charged particles of free resin are attracted.

J. F. BRIGGS.

See also A., Oct., 1926, **Molecular dimensions of cellulose** (BRUNSWIG); **Constitutional formula for cellulose** (GRAY); **Esterification of alkali-cellulose** (KITA and others).

PATENTS.

Degreasing textiles [wool]. N.V. ALGEM. CHEM. PRODUKTENHANDEL, and W. A. MEYER (E.P. 257,192, 6.4.26).—Wool is continuously degreased by treatment with organic solvents so that it retains its original strength, softness, and elasticity while felting is avoided. The greasy wool, heaped on a perforated conveyor belt, is drawn through a closed chamber and between squeezing rollers, being simultaneously sprayed with a suitable solvent such as benzene. It then passes through a drying chamber maintained under reduced pressure and the solvent vapour is withdrawn, cooled to below 0°, and the solvent recovered by passage over a suitable adsorbent.

A. J. HALL.

Washing or cleaning wool, silk, soiled fabrics, and the like fibrous textile materials. E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (E.P. 234,055, 2.3.25. Conv., 13.5.24. Addn. to 221,521, B., 1925, 985).—In washing wool or the like with aqueous solutions or suspensions of detergents, purification of the solution by continuous centrifuging may be supplemented or replaced by other continuous treatments, e.g., frothing, filtration, or electrical treatment.

A. GEAKE.

Manufacture of artificial silk. M. P. LANFRY and J. E. BRANDENBERGER (E.P. 255,527, 22.4.25 and 16.2.26).—Glossy filaments with a hollow core are spun from viscose containing alkali carbonate and of any degree of ripening, the ratio of alkalinity (as NaOH) to cellulose before adding the alkali carbonate being less than 6:7.3. The viscose is squirted into a coagulating bath containing sulphuric acid and sodium sulphate in proportions depending on the degree of ripeness of the viscose and on the alkalinity. Example: Viscose containing 7.3% of cellulose and 4% of total alkalinity (as NaOH) is mixed with sodium carbonate to bring the alkalinity to 7%. The viscose is ripened at 13° for 73 hrs. from the beginning of the ripening stage, and is spun into a bath containing 145 g. of sulphuric acid and 228 g. of sodium sulphate per litre, at a speed of 45 m.

per min., filament denier 6, temperature 45°, length of immersion 35 cm. With a time of ripening of 110 hrs. at 13°, the concentration of the coagulating bath for the same viscose would be 125 g. of sulphuric acid and 40 g. of sodium sulphate per litre. J. F. BRIGGS.

Artificial silk, thread, films, ribbons, etc. from viscose. O. FAUST and A. KÄMPF (G.P. 431,846. 16.11.22).—Sulphonic acids of condensation products of phenolsulphonic acids with aldehydes, aldoses, ketoses, complex carbohydrates, or wood are added to viscose, or the free acids or their salts are added partly to the viscose and partly to the spinning bath. Unripe viscose may be used. A. GEAKE.

Method of moth-proofing. J. M. ROSS and M. J. ROSS, Assrs. to I. B. ROSS (U.S.P. 1,594,632, 3.8.26. Appl., 3.7.25).—The fibres of the textile are softened and expanded by means of steam impregnated with volatilised benzaldehyde and phenol. J. F. BRIGGS.

Preparation of fur for shrinking and felting. J. H. MARTIN (U.S.P. 1,597,992, 31.8.26. Appl., 3.3.25).—Fur is treated with a solution of an alkali orthoborate. A. GEAKE.

Obtaining pure cellulose fibre from impure material containing incrusting substances. H. SILBERMANN (G.P. 432,052, 15.11.23. Addn. to 407,500, B., 1925, 203).—Saturated or superheated steam may be added to the gaseous chlorine with which the material is treated, and the vessel cooled after removing the steam. The material may be cooled at -10° to 0° before treatment. For the alkaline treatment, a dilute solution of a salt of a weak acid is used, and heated under a pressure of at least 1 atm. A. GEAKE.

Bleaching process [for paper pulp]. W. D. GREGOR, W. M. OSBORNE, and A. J. KEMZURA (U.S.P. 1,597,880, 31.8.26. Appl., 14.7.24).—Wet paper pulp is treated with insufficient bleaching agent at 21° , and, when the latter is exhausted, the pulp is washed and sufficient bleaching agent added to bleach to the required degree at 29° . A. GEAKE.

[Filler and coating for] paper. H. R. RAFSKY, now H. R. RAFTON (U.S.P. 1,595,416, 10.8.26. Appl., 3.4.22).—A mixture of finely-divided calcium carbonate and basic magnesium carbonate forms a cheap filling or coating material for paper, and gives a uniform opaque product with enhanced whiteness. It is obtained by slaking magnesian lime, and treating the product with carbon dioxide in presence of water until calcium hydrogen carbonate begins to be formed. Alternatively a soluble carbonate is added to a solution of a calcium and a magnesium salt. T. S. WHEELER.

Manufacture of artificial products from viscose. D. VAN DER WANT and M. P. A. BOUMAN, Assrs. to N.V. NEDERLANDSCHE KUNSTZIJDEFABR. (U.S.P. 1,596,906, 24.8.26. Appl., 4.3.26. Conv., 5.3.25).—See E.P. 248,750; B., 1926, 871.

Paper pulp and paper. B. S. SUMMERS (U.S.P. 1,597,840—1, 31.8.26. Appl., 9.7.24).—See Can. P. 246,537; B., 1926, 153.

Reclaiming used paper. G. HAMMOND (E.P. 257,774, 5.11.25).—See U.S.P. 1,572,478; B., 1926, 316.

Apparatus for treating artificial silk and other threads. S. WALKER & SONS, LTD., and G. E. WALKER (E.P. 257,706, 7.7.25).

De-waxing, de-greasing, or de-oiling textile fabrics. D. MCKELLAR and J. MACGREGOR (E.P. 257,491, 9.2.26).

Means for removing moisture from pulp, paper, straw boards, etc. E. E. TASKER (E.P. 257,449, 4.11.25).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Wetting-out agents [for textile materials]. W. SECK and H. LACHMANN (Textilber., 1926, 7, 851—854).—Determination of the efficiency of wetting-out agents by methods dependent on the rate of sinking of yarn or fabric in an aqueous solution (cf. Auerbach; B., 1926, 705), or the spread of a solution along a horizontally suspended thread (cf. Herbig and Seyferth; B., 1926, 312) are considered unsatisfactory because they are subject to the disturbing influences of air bubbles occluded in the textile material and the humidity of the surrounding atmosphere respectively. The relative wetting-out powers of various proprietary substances were determined by comparison of the amounts of solution absorbed by similar pieces of fabric (5×6 cm.) when immersed for 10 sec. in 0.2, 0.4, and 0.6% solutions of the wetting-out agents, and then allowed to drain for 50 sec. after withdrawal. The wetting-out powers of Oranit pdr., Nekal, Avivan, Avirol KM, Diffusil N, Koloran K, Marseilles soap, Monopole soap, Turkey-red oil (50% fat), and Tetracarnite, decreased in the order named. The first three agents named are said to be alkyl derivatives of naphthalenesulphonic acids. A. J. HALL.

Influence of additions to the indigo vat. H. POMERANZ (Textilber., 1926, 7, 862—863).—The orange colour of an indigo vat containing nitrobenzene as described by Haller (B., 1926, 891) is probably due to oxidation of the indigo by the nitrobenzene whereby dehydroindigo (cf. Kalbe; B., 1909, 1192) is formed. A. J. HALL.

Use of Aktivin in volumetric analysis [of mordants etc.]. E. JANGMIGHL and J. HACKL (Textilber., 1926, 7, 850—851).—Crystalline Aktivin, $C_6H_4Me \cdot SO_2NCiNa \cdot 3H_2O$, is a satisfactory oxidising agent for use in the volumetric analysis of tartar emetic and antimony lactates as used for the mordanting of cotton, and of sodium formaldehyde-hyposulphite. During oxidation, Aktivin is decomposed thus: $C_6H_4Me \cdot SO_2NCiNa + H_2O = C_6H_4Me \cdot SO_2NH_2 + NaCl + O$. In the analysis of tartar emetic, a mixture of 25 c.c. a 1.6% solution of the sample of tartar emetic and 20 c.c. of a 2% solution of sodium bicarbonate is titrated with a 0.1N-solution of Aktivin (14.1 g. per litre) using starch-potassium iodide as an indicator. In the analysis of sodium formaldehyde-hyposulphite, a 0.4% solution of the sample is titrated directly with a 0.1N-solution of Aktivin, using starch-iodide as an indicator. The results obtained by means of Aktivin agree very closely with those obtained by the usual methods using iodine, titanous chloride, or hydrogen sulphide. Aqueous solutions of Aktivin do not deteriorate when protected from sunlight during storage; appreciable deterioration is

produced by sunlight. Commercial Aktivin is sufficiently pure for volumetric analysis after filtration of its aqueous solution, but it may be readily purified by crystallisation from its aqueous solution.
A. J. HALL.

PATENTS.

Use of hyposulphites or sulfoxylates in the removal of dyes from textile fibres. C. S. HOLLANDER, Assr. to ROHM & HAAS Co. (U.S.P. 1,594,670, 3.8.26. Appl., 9.6.25).—Dyed goods are immersed in a mixture of a substantially insoluble formaldehyde-sulfoxylate and a readily soluble substantially neutral salt of a substance having a strongly alkaline hydroxide.

J. F. BRIGGS.

Finishing cotton yarn. J. G. A. RHODIN, and E. B. WESTMAN, LTD. (E.P. 257,097, 20.8.25).—Cotton yarn under tension is impregnated or coated with a solution of cellulose (*e.g.*, cellulose acetate), then drawn through sapphire dies or between fluted rollers whereby excess of the impregnating solution is removed and the tension on the yarn is controlled, dried, and wound on bobbins. The treatment may involve simultaneous mercerisation of the cotton yarn.

A. J. HALL.

Dyeing or coloration of cellulose acetate. BRITISH CELANESE, LTD., and G. H. ELLIS (E.P. 255,962, 2.5.25).—Oxidation shades from fawn to full black are produced on cellulose acetate, without impairing handle or lustre, by first applying a free amino-base solubilised or dispersed in water, then preferably rinsing and impregnating the goods with a solution of suitable oxidising and catalytic agents, drying without rinsing, and "ageing" by exposure to warm moist air or steam. Alternatively, the amino-base may be applied in the form of its water-soluble salts. Example: To produce a full black, 0.75 lb. of *p*-aminodiphenylamine is intimately mixed with 6 lb. of Turkey-red oil by heating at 80° and the mixture is dispersed in 25 gals. of soft water at 80°; 10 lb. of cellulose acetate yarn are worked in the liquor as for dyeing for about 2 hrs. and then rinsed off. The yarn is then impregnated with about its own weight of a solution containing 2 pts. of gum tragacanth, 8 of chromium chloride, 4 of aluminium chloride, 3 of cupric chloride, and 60 of sodium chlorate, each separately dissolved, and made up with water to a total of 1000 pts. The impregnated goods are preferably dried at a moderate temperature and "aged" for 5 min. in a steam ager, then washed off in hot water or very dilute formic acid solution.

J. F. BRIGGS.

Apparatus for acidulating textile and other material. E. MOLINGHEN and J. GAYE (E.P. 245,466, 2.1.26. Conv., 2.1.25).

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

Preparation of sulphuryl chloride. T. H. DURRANS (J.S.C.I., 1926, 45, 347—350 T).—The rate of formation of sulphuryl chloride by the interaction of sulphur dioxide and chlorine is greatly accelerated by certain organic substances. The most potent catalysts belong to that class of esters which are compounds of saturated alcohols or of monohydric phenols and monobasic aliphatic acids containing carbon, hydrogen, and

oxygen only; certain other esters, ethers, and ketones have also been found to act catalytically.

Effect of time and temperature of burning on the properties of lime. R. T. HASLAM and E. C. HERMANN (Ind. Eng. Chem., 1926, 18, 960—963).—The limestones investigated were calcined in an electric resistance furnace for 2—6 hours at temperatures from 980° to 1320° and from the quicklime, hydrate was formed containing 50% excess of water. Plasticity of the hydrate when mixed with water was determined in an improved form of the Emley plasticimeter, whilst the rate of slaking was found by noting the time taken for the lime when mixed with water to reach a maximum temperature. Rate of settling of the hydrate in water, the volume of putty formed, and the rate of reaction with hydrochloric acid were also noted. The results showed that the temperature and the time of burning are important, hydrates with the maximum plasticity being produced when the limestones examined were burnt at 1090° for 4 hrs. Fineness of the hydrate particles, as shown by greater rate of reaction with acid and slower rate of settling, is also an important factor in producing plasticity.

W. G. CAREY.

Separation of potassium nitrate and recovery of other salts from crude Indian saltpetre. M. A. HAMID (J.S.C.I., 1926, 45, 315—320 T).—The author discusses the application of modern phase-rule methods to the refining of crude Indian saltpetre. The requisite processes are based on a study of the isotherms at 25° and 90° of the ternary systems $H_2O-K_2SO_4-KNO_3$, $H_2O-NaNO_3-KNO_3$, $H_2O-Na_2SO_4-NaNO_3$, and the quaternary systems $NaNO_3-KCl-KNO_3-NaCl-H_2O$ and $Na_2SO_4-KNO_3-K_2SO_4-NaNO_3-H_2O$.

See also A., Oct., 1000, **Partial pressures of nitric and hydrochloric acids** (KLEMENC and NAGEL). 1011, **Evolution of hydrochloric acid from chloride solutions** (BODNÁR and ROTH). 1015, **Reaction between sodium phosphates and calcium hydroxide** (HAYASHI and MATSUI). 1016, **Behaviour of sulphurous acid towards thiosulphuric acid** (FOERSTER and VOGEL). 1017, **Determination of halides** (BERG); **Determination of halogen compounds** (KÖNIG). 1018, **Electrometric titration of phosphates** (BODFORSS); **Titration of hypophosphorous acid and its salts** (KOLTHOFF). 1019, **Determination of ferro- and ferri-cyanides** (CUMMING). 1021, **Potash analysis apparatus** (MÜLLER).

PATENTS.

System of concentrating sulphuric acid. A. A. VINEYARD (U.S.P. 1,596,907, 24.8.26. Appl., 9.9.25).—The acid flows from the heater into a chamber fitted with an agitator, by which means the organic matter is broken up into small particles and the released vapours and gases pass through a condenser. The acid from the chamber outlet then flows through a second heater, and through a series of concentrating stills, each having an exit leading to the condenser. H. ROYAL-DAWSON.

Chemical analysis apparatus and process [for oleum]. H. HOWARD, Assr. to GRASSELLI CHEMICAL Co. (U.S.P. 1,594,264, 27.7.26. Appl., 1.3.24).—Standard acid and samples of oleum are passed through a temperature equaliser and then mixed in equal quantities

in the analysis chamber (cf. J.S.C.I., 1910, 3). The resulting change in temperature actuates a heat-sensitive device, *e.g.*, an electrical resistance thermometer, placed in the analysis chamber, and the effect is recorded on a suitable scale as percentage of free sulphur trioxide. The heat-sensitive device can also control the absorbing acid supply valve.

E. S. KREIS.

Concentrating and purifying phosphoric acid.

H. E. LA BOUR (U.S.P. 1,597,984, 31.8.26. Appl., 13.9.23).—The vapour evolved from phosphoric acid at a temperature below its b.p. is blown away, carrying the fluorine with it.

H. ROYAL-DAWSON.

Manufacture of hydrofluoric acid. M. BUCHNER (E.P. 255,464, 2.6.25. Conv., 2.6.24. Addn. to 234,852, B., 1925, 670).—A volatile fluoride, such as boron fluoride, is added to a mixture of hydrofluoric and hydrofluosilicic acids, and the latter, together with the complex hydrofluoric acid produced, are caused to react with a metal fluoride, whereby hydrofluoric acid and the metal salts of hydrofluosilicic acid and the complex hydrofluoric acid are obtained. These, when heated, yield the metal fluoride, silicon fluoride, and the volatile fluoride first added.

H. ROYAL-DAWSON.

Apparatus for drying and neutralising ammonium sulphate etc. T. B. SMITH (E.P. 256,385, 13.7.25).—The apparatus consists of a shallow steam-heated pan, in which are one or more sets of stirring blades mounted on a radially disposed moving arm operating from a main shaft, and provided with a counterbalance lever worked by hand or trip mechanism. In action, one set of blades is put in operation to convey the material from the periphery of the pan to the centre, followed by the second set in conjunction with the first to convey material from the centre to the periphery, the first set being finally thrown out of action to allow the second set to convey the material to the delivery passage.

H. ROYAL-DAWSON.

Catalytic oxidation of ammonia. I. G. FARBENIND. A.-G., Assees. of O. BALZ and F. REUSCHER (G.P. 431,506, 23.12.24).—Oxygen, or gases rich in oxygen, and ammonia are circulated through one or more catalyst chambers, ammonia being added as necessary to avoid an explosive mixture. A portion of the reaction gas is drawn off, continuously or intermittently, for absorption.

W. G. CAREY.

Production of alkali carbonates from alkali sulphates. W. H. FRIEDRICH (G.P. 431,509, 17.9.24).—An alkali carbonate is produced by the interaction of a freshly precipitated alkaline-earth carbonate, particularly barium carbonate, and a solution of an alkali sulphate with the addition of a small amount of an ammonium salt.

W. G. CAREY.

Preparation of stable alkaline-earth polysulphide solutions. E. GLÜCKSMANN (G.P. 431,642, 17.4.25).—The solutions are stabilised in a similar manner to alkali polysulphide solutions (see G.P. 419,910; B., 1926, 237).

W. G. CAREY.

Manufacture of alkali hydrosulphites [hyposulphites]. FARBENFABR. VORM. F. BAYER & Co. (E.P. 247,524, 12.12.25. Conv., 14.2.25).—In the manufacture of hyposulphites from alkali bisulphites and alkali amalgam, the alkali sulphite in the reaction liquid

is maintained at not more than 45 g. per litre by withdrawing the mother liquor and replacing it by water and salt.

H. ROYAL-DAWSON.

Production of barium oxide of high porosity and purity. H. SCHULZE (G.P. 431,617, 11.6.24).—A mixture of barium carbonate and charcoal is heated indirectly, by electrical means, in such a manner that the mass is heated from two or more sides. The ordinary or slightly increased pressure may be used.

W. G. CAREY.

Separation of barium chloride from solution.

RHENANIA VEREIN CHEM. FABR. A.-G., Assees. of F. RÜSBERG (G.P. 431,643, 2.10.24; see G.P. 429,716; B., 1926, 778).—Rock salt is used in place of common salt, and is mixed by degrees with definite portions of the solution, the impurities being removed by filtration, after each introduction of rock salt. The rock salt may partly be replaced by pure salt.

W. G. CAREY.

Production of magnesium carbonate with simultaneous recovery of ammonium salts. B. WAESER (G.P. 431,618, 24.2.23).—Solutions of magnesium salts, or crude potash salts containing magnesium, are treated with not more than 10% of ammonia, and the mixture is cooled with the addition of carbon dioxide so that the formation of bicarbonate is avoided. The precipitate of magnesium carbonate is separated by filtration from the solution of ammonium salts with any potassium salts.

W. G. CAREY.

Process for producing stannous chloride. W. T. LITTLE, Assr. to METAL & THERMIT CORP. (U.S.P. 1,597,653, 24.8.26. Appl., 12.1.25).—A solution of stannic chloride is reduced in the cathode compartment of a divided cell under conditions preventing the deposition of tin.

H. ROYAL-DAWSON.

Production of crystallised sulphates of iron, copper, zinc, and nickel. G. AGDE (G.P. 431,581, 14.1.25).—Sufficient sulphuric acid is added to the solution so that it may be used again as dissolving acid, the saturated sulphate solution is cooled, and such conditions of temperature and acid and salt concentration are maintained as to prevent the contamination of the crystals by simultaneous formation of crystals with less water.

W. G. CAREY.

Treating vanadium ores and solutions. W. E. STOKES, Assr. to U.S. PROCESSES Co. INC. (U.S.P. 1,597,216, 24.8.26. Appl., 19.1.21).—Lead vanadate, precipitated by the addition of a lead salt to a leach solution containing vanadium compounds, is separated and decomposed by excess of acid in a salt solution in which the resulting vanadium oxide is soluble. The insoluble lead salt is filtered off and vanadium oxide is precipitated by reducing the acidity of the solution.

S. S. WOOLF.

Production of pure sulphides. GES. F. CHEM. & HÜTTENWESEN (G.P. 431,253, 3.4.24).—A solution of an alkaline-earth sulphide is caused to react with an alkali carbonate solution under pressure, the sulphide liquor being concentrated *in vacuo*, in stages.

W. G. CAREY.

Production of sodium aurothiosulphate. I. G. FARBENIND. A.-G., Assees. of O. RIPKE (G.P. 431,580,

6.5.25).—A solution of a gold salt and sodium thiosulphate is evaporated under reduced pressure in a neutral atmosphere.
W. G. CAREY.

Production of silicon carbide from silicic acid and charcoal. A. REITZ (G.P. 431,759, 13.3.25).—Crude fluorine compounds are added as catalysts, the volatilisation of which is prevented by surrounding the furnace by a casing.
W. G. CAREY.

Extracting iodine and bromine from solutions. G. VELLARDI, and A.P.I.C.E. SOC. AN PROD. ITALIANI CHIMICI ESTRATTIVI (E.P. 257,134, 18.11.25).—Solutions or water containing the halogens in the natural state, or in residual mother-liquors, are admitted under pressure together with a reagent to liberate the halogen and a solvent of iodine and bromine (*e.g.* petroleum) into an emulsifier, the liquid mass then passing into centrifugal machines or decanting tanks to separate the solvent. The latter is then passed into a second emulsifier, where it meets a solution of a substance capable of converting the halogens into soluble salts, and the resulting solution is finally separated from the solvent by decantation.

H. ROYAL-DAWSON.

Production of colloidal sulphur. I.G. FARBEIND. A.-G., Assees. of F. WINKLER and F. GILLER (G.P. 431,505, 6.9.25).—Polysulphide solutions, with the addition of protective colloids, are sprayed into vapour or gas, with or without the addition of an electrolyte.

W. G. CAREY.

Manufacture of graphite. A.-G. F. ANILIN-FABR. (E.P. 252,662, 12.12.25. Conv., 26.5.25).—Carbon is heated (*e.g.*, in the electric arc) in the presence of air or other suitable gas at or above 3900°, and the escaping vapours are condensed on a highly heated surface in the immediate neighbourhood of the heated material, having a temperature of at least 1500°.

H. ROYAL-DAWSON.

Purification of hydrogen [for making synthetic ammonia]. L'AIR LIQUIDE, SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (E.P. 250,520, 26.5.25. Conv., 9.4.25. Addn. to 238,175, cf. B., 1925, 872).—In purifying hydrogen for the manufacture of synthetic ammonia, liquid air (in place of liquid nitrogen) is introduced in small quantities into the expanded hydrogen, and the oxygen is removed prior to the actual synthesis by reaction with the hydrogen in the mixture in presence of a catalyst.

H. ROYAL-DAWSON.

Catalytic preparation of hydrogen and phosphoric acid. I. G. FARBEIND. A.-G., Assees. of A. MITTASCH and G. WIETZEL (G.P. 431,504, 23.11.24).—Water vapour, with or without oxygen or gases containing oxygen, acts upon phosphorus vapour, charcoal being employed as catalyst.

W. G. CAREY.

Separation of helium from gas mixtures. F. PANETH and K. PETERS (G.P. 431,507, 2.12.25).—Gases containing helium are made to impinge upon a heated, gastight diaphragm, *e.g.*, of glass. At a high temperature the helium diffuses through more quickly than the heavier gases. The diaphragm may be heated by an electrical resistance or by hot gases, and the gas pressure on both sides may be varied at will.

W. G. CAREY.

Electrolytic decomposition of chlorides. E. SCHLUMBERGER, Assf. to KOHOLY A.-G. (U.S.P. 1,598,018, 31.8.26. Appl., 17.9.25. Conv., 28.7.24).—See E.P. 254,163; B., 1926, 757.

See also pages 908, **Hydrogen** (G.P. 431,758). 922, **Alkali metals** (U.S.P. 1,597,231). 926, **Calcium nitrate** (E.P. 242,990). 934, **Chlorine water** (G.P. 429,037).

VIII.—GLASS; CERAMICS.

[Glass] tank block corrosion by shelving. F. C. FLINT and A. R. PAYNE (J. Amer. Ceram. Soc., 1926, 9, 613—617).—Part of the rapid dissolution of tank blocks by glass is due to attack in an upward direction from cracks in the surface of the block. The upward penetration is probably due to the formation of a ledge at the crack upon which the more saturated and viscous glass rests. For this reason the ledge is attacked less rapidly than the roof of the crack, which is exposed to fresh glass. Density measurements of glass against the wall of the tank, as well as analysis of small clay-glass melts, proves the increase of sp. gr. with increasing solution of block.
A. COUSEN.

Temperature of pyrex and porcelain in sunlight. J. T. LITTLETON, JUN., and W. W. SHAVER (J. Amer. Ceram. Soc., 1926, 9, 618—625).—Calculation shows the maximum surface temperature in sunlight (with shade temperature 40°) of porcelain to be 93°, of pyrex glass 46.5°. With a shade temperature of 22°, small plates of porcelain increase in sunlight 19.7° in $\frac{1}{2}$ hr., pyrex plates remaining unchanged. When placed at the focus of metal reflectors in intensified sunlight the temperature increase of the porcelain is 3.5 times that of the pyrex.
A. COUSEN.

Requirements of refractories for manufactured gas plants. S. S. COLE (J. Amer. Ceram. Soc., 1926, 9, 462—473).—The requisite qualities, and the service expected, of the refractories used in four types of installations, viz., gas producers or water-gas plants, horizontal and vertical retorts, and gas ovens, are discussed in broad outline.
F. SALT.

Effect of repeated burning on the structure and properties of lime-bonded silica bricks. W. HUGILL and W. J. REES (Trans. Ceram. Soc., 1926, 25, 82—93).—A number of lime-bonded bricks made from Sheffield ganister were subjected to 11 reburnings in an industrial kiln, samples being withdrawn for examination after each reburn. The bricks were tested for powder density, volume changes, mechanical strength, and changes in structure. The powder density tests showed that the major part of the quartz inversion was accomplished in the first burn, but the proportion of unchanged quartz decreased steadily until the 7th burn. Very little change in volume occurred after the first burn. Porosity decreased steadily down to the 7th burn, after which it remained fairly constant. A marked increase in crushing strength up to the 7th burn was noticed, and there was a steady increase in the transverse strength up to the 5th burn. The results tend to emphasise the importance of the "soaking" period during firing. No change in chemical composition took place as the

result of reburning. Changes in structure are illustrated by photomicrographs. F. SALT.

Fusion points of firebrick-coal ash mixtures. L. C. HEWITT (J. Amer. Ceram. Soc., 1926, 9, 575—582).—Cone fusion points of mixtures of samples of six firebricks with each of five samples of coal ash over the range 10—40% of brick indicated the presence of eutectics in the mixtures. The effect of reducing conditions upon the results was very marked. Coal ashes rich in iron did not give the most pronounced effects. No definite grouping of the firebricks could be made from the results. A. COUSEN.

Comparison of the uniformity of strength and texture of firebrick made by different processes. A. E. R. WESTMAN and W. H. PFEIFFER (J. Amer. Ceram. Soc., 1926, 9, 626—632).—The following order of decreasing uniformity of transverse strength of firebricks was found:—(1) Semi-dry press, (2) stiff mud repress, (3) dry press, (4) hand-moulded. The order of decreasing uniformity of texture was 1, 3, 4, 2. A. COUSEN.

Some successful cone 10 saggar bodies. E. H. FRITZ (J. Amer. Ceram. Soc., 1926, 9, 606—612).—A successful saggar body for firing at cone 10 must have a dry modulus of rupture not less than 80 lb./sq. inch, an absorption at cone 10 of not less than 8% nor more than 11½%, and a shrinkage of not more than 7%. Mixtures of clays giving the best results were (a) Maryland No. 1 10%, N.J. No. 3 15%, N.J. No. 4 25%, grog 50%, and (b) Ohio No. 2 38%, Penn. No. 2 17%, grog 45%, with the grog size 75% of 34—12-mesh and 25% 2—3-mesh. A. COUSEN.

Thermal expansion of saggar clays. R. F. GELLER and R. A. HEINDL (J. Amer. Ceram. Soc., 1926, 9, 556—574).—The thermal expansion of 49 clays was determined up to 1000°, by heating bars (50% raw clay, 50% grog) previously fired at 1230° in a vertical electric tube furnace. Expansions were transmitted to a gauge by a quartz rod. In addition, samples were air-quenched at successively increasing temperatures until fracture occurred. Two types of clay were distinguished, one expanding fairly uniformly to 500°, the second giving a decided increase in the rate between 100° and 200°. In the quenching test practically all clays of the first group exceeded 500° before cracking, the majority of the second group failed below 500°. Repeated firings tended to decrease the expansion between 500° and 600°, and increase that between 100° and 200°. Artificial corundum increased the total average expansion of clays with which it was used, and tended to eliminate irregular expansion due to inversion of free silica. A. COUSEN.

Use of Georgia and North Carolina kaolins in a semi-porcelain body. S. E. HEMSTEGER and W. C. STIEF (J. Amer. Ceram. Soc., 1926, 9, 444—445).—A comparative study with these clays and two English china clays when used in a semi-porcelain body for dinnerware indicated that the American clays were adapted to this purpose, provided that the necessary readjustments were made in substituting for the English clays. F. SALT.

Enamel clays. V. W. BOEKER (J. Amer. Ceram. Soc., 1926, 9, 399—411).—Twelve typical enamel clays were examined for water of plasticity, drying shrinkage, modulus of rupture, and behaviour on firing to 1000°. Enamel slips were prepared by mixing 10 pts. of clay with 100 pts. of ordinary commercial frits. The viscosity, the ability to hold the frit in suspension, and the hydrogen-ion concentration of the slips were determined. The fineness of the clays was tested on a 300-mesh sieve, and they were subjected to petrographic and chemical analysis. Finally, the clays were tested in a series of cover coats on sheet steel, the enamels being made up of 100 pts. of frit, 7 pts. of clay, 4 pts. of tin oxide, 3 pts. of "leukonin," and about 40 pts. of water. The fired test pieces were examined by the impact test and the thermal shock test. The suspension test did not give reliable data, and the results did not agree with the hydrogen-ion concentration. All clays having p_H values greater than 6.0 proved to be good suspension agents for the cover coat enamels. Impurities in the clays affected the results more by their nature than their quantity. Small amounts of carbonaceous matter, other than coal or lignite, had no effect on the enamel, but lime, if present in excess of 1%, caused blistering. The degree of maturity and the lustre were inversely proportional to the porosities of the clays at 1000°. The resistance of the fired enamels to impact and thermal shock was influenced greatly by the clay used, but no direct relation was found between these factors. F. SALT.

Practical chemical control of [enamel] pickling room solutions. B. T. SWEELY (J. Amer. Ceram. Soc., 1926, 9, 590—592).—The acid solution used for pickling in the enamel industry is titrated by adding to 10 c.c. in a special cylinder sufficient $N/2$ caustic soda (containing methyl-orange) to neutralise, and reading the strength of acid from graduations on the cylinder. Similar methods are used for the cleaning and neutralising solutions. A. COUSEN.

Determination of relative surface areas of powders. KOEHLER.—See I.

PATENTS.

Process and apparatus for purifying clay etc. F. PARENTANI (E.P. 255,300, 2.12.25).—Colouring impurities are removed from clays and similar materials by treating them in the dry state with hydrogen sulphide at atmospheric temperature. The material may first be subjected to the action of a vacuum. It is finally treated with an acid solution (hydrochloric acid) to dissolve the sulphides formed, the hydrogen sulphide produced being used to treat further material. F. SALT.

Removal of iron from clays, bauxites, etc. H. FLEISSNER (Austrian P. 102,553, 7.8.23).—Before being treated with dilute acids, clays are subjected to the action of hydrogen sulphide to remove the iron. The iron sulphide formed is best decomposed with sulphur dioxide or with gases containing it. F. SALT.

Manufacture of refractory bricks. PFÄLZISCHE CHAMOTTE- U. THONWERKE (SCHIFFER & KIRCHER) A.-G., Assees. of G. WAGAPOFF (E.P. 248,369, 22.2.26. Conv., 25.2.25).—The refractory clay is worked up without

the addition of grog, and with a moisture content not exceeding 20% by weight. It is disintegrated without previous drying or heating or subsequent moistening to a grain size varying from a fine powder to 10 mm. The material is then intimately mixed, moulded under pressure, and fired in the usual way. F. SALT.

Manufacture of durable earthenware. H. OEXMANN (E.P. 231,469, 14.3.25).—The body mixture, or the roughly-shaped article, is subjected to high pressure (300 atm.) through a gaseous, liquid, or powdered medium. The pressed article may be turned, and is then fired. F. SALT.

Muffle kilns [for ceramics etc.]. S. CROSBIE and C. H. F. COLLARD (E.P. 253,959, 21.9.25).—An "endless" series of separate, continuous muffle kilns connected by flues is arranged polygonally around a central space. Each muffle in turn becomes partly heated by waste gases from the kiln immediately preceding it. A combustion chamber below the floor of each kiln is connected with a number of flues formed in the surrounding kiln walls and with a single crown flue leading to down flues in the walls. F. SALT.

[Tunnel] kilns or ovens for baking pottery etc. R. M. D'ARLEUX and E. VIOLETTE (E.P. 254,023, 28.3.25).—A mixture of gaseous fuel and air is introduced into the tunnel through tuyères constructed with a thermosiphonic circulatory cooling system. The size and number of the tuyères, the pressure of the gaseous fuel, and the capacity of the hearth are so adjusted that the pressure in the kiln is greater than atmospheric. Sloping baffles are fitted at the mouths of the tuyères to spread the flames. F. SALT.

[Brick or pottery] kilns. G. V. EVERS (E.P. 254,438, 24.4.25).—In a multi-chamber kiln, fire boxes are provided at each of the four corners of each chamber except the end chambers, in which the furnaces are arranged in the end walls. A low baffle wall is built near the inner end of each furnace to give a short vertical passage for the hot gases. Passages are provided at the base of the partitions between the chambers to give inter-communication. A central outlet in the floor of each chamber leads to the chimney flue. F. SALT.

[Down-draught] kilns for the manufacture of bricks and pipes. E. EVANS (E.P. 255,188, 18.5.25).—The hot gases are drawn through the perforated floor of the kiln chamber and pass through hooded pipes covering holes in an intermediate floor spaced from the kiln base. The holes near the peripheral wall in the intermediate floor are of larger cross-section than those near the centre. A portion of the gases passes from the space beneath the intermediate floor direct to the chimney, the remainder being drawn through closed passages beneath the kiln base, which communicate directly with the space beneath the larger apertures in the second floor. F. SALT.

Enamelling metal articles. M. AUGER (E.P. 254,452, 5.5.25).—To produce a surface to which vitreous enamels will adhere on brass and similar yellow metal alloys, without the application of a copper coating, the alloy is treated with an acid (nitric acid, or a mixture of nitric and sulphuric acids, and/or a pickle (sodium

chloride) to dissolve the metals, with the exception of the copper, from the surface. A practically pure copper surface is thus produced. F. SALT.

Artificial stone for abrasive and other purposes. H. WADE. From CARBORUNDUM Co. (E.P. 257,391, 23.7.25).—See U.S.P. 1,553,105; B., 1925, 882.

Machinery for manufacturing glass-ware. P. C. RUSHEN. From HARTFORD-EMPIRE Co. (E.P. 257,637, 7.3.25).

Kilns (U.S.P. 1,590,462 and 1,590,935).—See I.

IX.—BUILDING MATERIALS.

See A., Oct., 1012, **Activation of inert varieties of calcium sulphate** (BUDNIKOV).

PATENTS.

Rotary furnaces for burning cement and the like. A. SCHMID (E.P. 253,819, 4.1.26).—The discharge head of a rotary furnace is constructed at its lower end as a cooling chamber, to which air is admitted under pressure. The air passes from the cooling chamber through the material and is then utilised for combustion purposes. In an alternative construction, the head widens out at the lower end to form a hopper-shaped cooler, within which is a cone having a louvre-like upper part. The burnt material falls in a thin layer over the cone. Air under pressure is introduced into the cone and passes through the louvre opening over the material. F. SALT.

Production of hydraulic cement from oil shale and limestone. REKORD CEMENT IND. G.M.B.H., Assees. of O. TETENS (G.P. 429,651, 10.12.22).—The burnt material (cf. U.S.P. 1,536,165; B., 1925, 549) is quenched in a closed vessel with the ammoniacal liquor obtained in the process. W. G. CAREY.

Cement and process of making it. E. C. ECKEL (U.S.P. 1,591,662, 6.7.26. Appl., 31.3.26).—A mixture containing alumina, iron, silica, and lime is fused in the presence of sufficient free carbon to form ferrosilicon. F. SALT.

Mixture for use as a cement cold-glaze. J. LANGBEIN (E.P. 247,947, 11.2.26. Conv., 18.2.25).—A mixture to be added to the usual cement mixture for cold-glazes contains 250 pts. by weight of polymerised linseed oil, 625 pts. of ammonia water, 6000 pts. of lime water, 1000 pts. of infusorial earth, which may be admixed with asbestos, 625 pts. of barium or strontium oxide, or 1500 pts. of a chromate. F. SALT.

Process of impregnating wood. K. BUBLA (U.S.P. 1,597,010, 24.8.26. Appl., 20.6.24).—Wood is impregnated under reduced pressure with a dilute solution of a suitable substance, followed by one of higher strength on again reducing the pressure. H. ROYAL-DAWSON.

Manufacture of aluminous cements. E. MARTIN (E.P. 251,618, 20.4.26. Conv., 30.4.25).—See F.P. 597,978; B., 1926, 409.

Paving and surfacing material. C. E. RAMSDEN (U.S.P. 1,598,505, 31.8.26. Appl., 19.8.25. Conv., 1.8.24).—See E.P. 243,418; B., 1926, 129.

Prepared tar for road-making (E.P. 257,024).—See III.

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

Influence of oxidation reactions on the blast-furnace process. F. WÜST (Stahl u. Eisen, 46, 1213—1221).—A comparison is made between iron furnaces for ore-reduction and refining furnaces. In both, the solid phase has a reducing action, whilst the gas phase acts as a reducing agent in the former and as an oxidising agent in the latter. The predominating influence of the gas phase is emphasised. In the blast furnace, an oxidising zone is present in front of the tuyères, causing secondary reactions which impair the quality of the product and reduce the economic efficiency of the process. The proportions of carbon monoxide and dioxide and of oxygen at different distances from the tuyères are shown, and from these data, the extent of the oxidising zone with variation in hearth diameter is determined. The effect of hearth diameter on the quantity of blast required, coke consumption, and yield of iron is shown. Tuyères entering the furnace tangentially are advocated. L. M. CLARK.

[Formation of] graphite scum in cast iron. B. OSANN (Stahl u. Eisen, 1926, 46, 1320—1324).—The formation of a scummy porous mass of iron, very rich in graphite, during the casting of iron, is due to the precipitation of graphite in the liquid iron during cooling, but before solidification. This scum rises to the top of the casting and prevents proper filling of the mould. The presence of large quantities of sulphur or silicon, which tend to reduce the solubility of carbon in molten iron, favours the formation of this scum. Examples from practice are given with details of the melting conditions which gave rise to this behaviour in each case. To avoid the separation of graphite from the liquid mass the carbon content of the metal should not rise too high, and preheated moulds should be used for casting, so as to mitigate the bad effects of rapid cooling. In regulating the carbon content of the charge for the cupola, attention must be paid to the undoubted effect of the nature and quantity of the slag on the composition of the metal. The theories put forward to account for the separation of graphite from liquid iron are briefly discussed. A. R. POWELL.

Recrystallisation temperatures of cold-rolled electrolytic iron and open-hearth steel strip. J. R. FREEMAN (Trans. Amer. Soc. Steel Treat., 1926, 10, 67—86).—Two samples of open-hearth steel strip and one sample of electrolytic iron, 0.065 in. thick, were annealed and then cold-rolled, in one experiment with a 90% reduction in six passes, and in a second experiment with a 73% reduction in two passes. Specimens were then annealed at various increasing temperatures. There was a marked decrease in strength and increase in elongation between 500° and 600° in the case of the steel strip, and between 400° and 500° for the electrolytic iron. In the latter case the tensile strength at 500° was lower the greater the preliminary amount of cold working. Photomicrographs show that cold-rolling distorted the grain structure but caused no recrystallisation. On annealing, the degree of grain refinement increased with the amount of cold work. Recrystallisation commenced between 500° and 600° for the steel strip and between 450° and

500° for the electrolytic iron, the lower recrystallisation temperature being due to its relatively higher purity. T. H. BURNHAM.

Influence of nitrogen on the solubility of ferrous materials in hydrochloric acid. H. H. GRAY (J.S.C.I., 1926, 45, 365—366 T).—A sample of mild steel is rendered almost insoluble in hydrochloric acid of varying strengths, up to 20% by weight, by being heated in nitrogen for about 6 hrs. at 1000°. The loss of weight by the nitrogenised steel due to attack by hydrochloric acid is 0.68%, as compared with a loss of 46% suffered by a duplicate sample not treated with nitrogen.

Macroscopical examination of iron and steel. F. P. GILLIGAN and J. J. CURRAN (Trans. Amer. Soc. Steel Treat., 1926, 10, 9—30).—The progressive nature of the solidification of steel and the influence of the diffusible and less diffusible elements and of sonims on the macro-structure are described. Of the various macro-tests the hot acid etch is considered the most productive of information. A solution of hydrochloric acid or of hydrochloric and sulphuric acids is preferred. Photographs are given illustrating the use of hot acid etching in demonstrating the following phenomena in steel:—segregation, laps, ingot structure in castings, the persistence of dendritic structure in forgings, the flow of metal in forgings, decarbonised surface layers in tool steel, incipient fatigue cracks, and localisation of hardening stresses by deep stamping. The process may be used to determine the method by which steel balls are forged. The indications of hot acid etching are corroborated by photomicrographs and analytical data. T. H. BURNHAM.

Dendritic structure and crystal formation [in steel]. B. STOUGHTON and F. J. G. DUCK (Trans. Amer. Soc. Steel Treat., 1926, 10, 31—52).—Overheated 1.28% carbon steel showed an intercrystalline fracture under shock. It is suggested that this was due to large crystals and consequent large surfaces of intercrystalline cement. The same reason is advanced for intercrystalline fracture at high temperatures. Photomicrographs show that although the crystals of pearlite were large, the individual crystals of ferrite and cementite were the same size as in 1.16% C file steel. Small crystals and interlamellar crystals throughout the eutectoid area were formed by pressure due to the austenite-pearlite expansion. At a magnification of 4800 the cementite crystals were seen to be distorted and to tend to thicken and fold back on themselves. The Brinell hardness was lower the larger the crystals. In the discussion it was brought out that the intercrystalline fracture might be due to large spines of cementite along the grain boundaries, or to high concentration of impurities along the boundaries of the crystals, rather than connected with the amorphous intercrystalline layer, also that the large crystals formed at high temperature are different from the large primary crystals under discussion. T. H. BURNHAM.

Influence of heat treatment and carbon content on the structure of pure iron-carbon alloys. W. L. FINK and E. D. CAMPBELL (Trans. Amer. Soc. Steel Treat., 1926, 9, 717—754).—X-Ray studies were made on two series of carbon steels prepared from Armco iron

using the Bohlin and wedge methods. The first series, containing up to 1.30% C, was oil-hardened, and the second, containing up to 1.50% C, was water-hardened and tempered. In the oil-quenched specimens, the lattice was much the same as for pure α -iron, but there were indications that some parts of the lattice slightly dilated with increasing carbon content. Weak γ -lines did not appear until 0.97% C, the lattice constant a for γ -iron being 3.56 Å. In the water-quenched specimens up to 0.21% C the α -lattice was not deformed, but at higher contents a part was dilated until at 0.43% C very little of the lattice retained its original dimensions. γ -Iron lines appeared at 0.66% C, and at a somewhat higher content a new crystal structure, viz., a body-centred tetragonal lattice, made its appearance, the amount present increasing with the carbon content and attaining uniform dimensions at 1.50% C ($a = 2.85$, $c = 3.02$ Å). At 0.9% C for the γ -iron a was 3.59 Å, and for higher carbon contents it dilated to 3.61 Å. On tempering, the changes were in the reverse sense. At 100° the body-centred tetragonal lattice disappeared, and at 203° the γ -iron lines disappeared. The α -lattice assumed the dimensions for pure iron as the temperature increased. Iron carbides did not give any carbide lines, although precipitated in sufficiently large particles to be visible at 500 magnifications. The results from the oil-hardened specimens were the same as for water-quenched and tempered a little below 200°. T. H. BURNHAM.

Effect of reheating on cold-drawn [steel] bars. S. C. SPALDING (Trans. Amer. Soc. Steel Treat., 1926, 9, 685—716).—Rods of three types of steel, containing respectively about 3% Ni, 0.77—1.44% Mn, and 0.96% Cr and 0.18% V, were cold-drawn to a finished size of 0.45 in. with 42.5, 28.1, and 18.9% reductions, and then reheated to various increasing temperatures. After reheating to 315° the steels gained considerably in yield point with only slight reduction in tensile strength, ductility and impact values. Above 600° for the manganese and nickel steels, and 650° for the chromium-vanadium steel the yield point and tensile strength fell rapidly and the ductility and impact test increased. The response of hot-rolled and cold-rolled bars to reheating was practically identical. The Izod figures for cold-drawn bars were higher than for oil-hardened bars at low reheating temperatures, but above 315° the values for the heat-treated bars rose rapidly; for cold-drawn bars the rise did not occur till 600—650°. The chromium-vanadium steel had the highest impact value for a yield point under 50 tons per sq. in.; above this value it was equalled by the nickel steel. The minimum Izod value was found on unquenched cold-rolled steel at about 315° reheating temperature. This indicated that the same phenomenon found in quenched and tempered samples was not due to an austenite-martensite transformation. The electrical conductivity of the manganese and chromium-vanadium steels decreased slowly to 425°, and then fell rapidly; that of the nickel steel decreased more uniformly. The effect of cold work on the conductivity was practically nil.

T. H. BURNHAM.

Distribution of hardness in quenched carbon steels, and quenching cracks. T. KASÉ (Sci. Rep.

Tôhoku, 1926, 15, 371—386).—Cubes of Swedish carbon steel were heated in an electric furnace to a desired temperature and quenched after 10 min. by immersing in an oil- or water-bath. After polishing, the hardness of different points of the surface was measured by the Shore scleroscope. Quenching causes the hardness to increase to about three times that of the annealed specimens, and in severe quenching the periphery is always less hard than the interior, the difference increasing with rise of quenching temperature. The reverse is true in the case of soft quenching. This is explained by the presence of retained austenite in martensite. The change of the retained austenite into martensite explains the considerable increase in hardness by cooling in liquid air, and this change also causes the hardness of a specimen to increase with age. Specimens quenched in water from above 900° always cracked, the lines of fracture cutting the equi-hardness curves orthogonally. Samples containing less than 0.9% of carbon did not crack. The cracking is not caused by a thermal stress, but is due to the difference between the specific volumes of austenite and martensite. E. S. HEDGES.

Specific heat of carbon steels. S. UMINO (Sci. Rep. Tôhoku, 1926, 15, 331—369).—The specific heats of steels with carbon contents 0.09—2.84% were determined by the method of mixtures over the temperature range 23—1250°. The specific heat increases with rise of temperature up to the A3 transformation point, above which it is constant. The mean specific heat of cementite increases from 0.149 at 150° to 0.220 at 850°. For the dissolution of 1 g. of carbon in iron 1760 cal. are required, and 16.1 cal. for the dissolution of 1 g. of pearlite in iron. The heat changes for the dissolution of pearlite in iron, the transformation of martensite to pearlite and of austenite to martensite, were found to be proportional to the carbon content. The heat of transformation from austenite to martensite for a eutectoid steel is 5.9 cal. The A1 transformation is a function of temperature and time, but the A2 transformation is a function of temperature only.

E. S. HEDGES.

Optimum temperature for the extrusion of (α + β) brass. W. SCHREITER (Z. Metallk., 1926, 18, 285—287).—Extrusion experiments on brasses containing 52—66% Cu show that the optimum temperature for the extrusion varies with the composition of the alloy, but is, in all cases, just slightly above the temperature at which the whole of the metal is in the β form. Thus, brass containing 61.5% Cu consists entirely of β above 758°; if extruded into tubes at 740° it has a tensile strength of 40 kg./mm.² with an elongation of 32%, at 750° these values are 42.7 and 33.7, and at 760° 43 and 41 respectively. The microstructure of the metal extruded at 740° consists of long parallel lines of alternating α and β , whereas at 760° it consists entirely of the characteristic coarse-grained crystal structure usually obtained after pressure. Annealing at 760° of this brass extruded below 758° produces the satisfactory β -structure. Extrusion of the brass at 770—780° causes, however, a much more coarsely crystalline structure to develop, and the metal becomes very weak, often fracturing during extrusion or on subsequent cooling. Similar characteristics hold

for other brasses containing 52—66% Cu, so that in all cases the metal in the press should be maintained as near as possible to the temperature at which the $\alpha + \beta \rightarrow \beta$ change is completed, allowance being made for the rise in temperature caused by the pressure required for the extrusion.

A. R. POWELL.

Determination of tin in cassiterite. A. PIRLOT (Bull. Féd. Ind. chim. Belg., 1926, 5, 281—284).—The finely ground sample is digested with hydrochloric acid to remove iron and the residue is collected, washed, dried, ignited, and finally reduced to metal by heating for 2 hrs. at 740° in a current of hydrogen. The loss in weight during the last-named treatment is a measure of the tin content of the ore. As a check the metal is dissolved in hydrochloric acid, the insoluble material separated, again reduced in hydrogen and any further metal extracted as before, and the combined stannous chloride filtrates are neutralised with ammonia, oxidised with iodine, and boiled with sodium sulphate or ammonium nitrate. The precipitated metastannic acid is collected, washed with ammonium nitrate solution, and ignited to stannic oxide. For a good sample of cassiterite the tin content calculated from the loss on reduction agrees very closely with that obtained by weighing the tin oxide.

A. R. POWELL.

Oxidising salt test and the intercrystalline corrosion of aluminium and its alloys. H. BIEGLER (Z. Metallk., 1926, 18, 288—289).—The action of the oxidising salt solution of Mylius (B., 1925, 552) on aluminium takes place chiefly along the boundaries of the crystal grains and, if carried out at ordinary temperatures over a period of several days (renewing the salt solution every day), valuable information on the intercrystalline corrosion of aluminium and its alloys is obtained. In the case of technical aluminium (98—99% Al), the loss of metal per unit area increases slowly but steadily with the time provided that the original surface of the metal produced by rolling remains untouched; if this skin is removed by etching, rapid attack of the metal takes place during the first two days, after which a new protective skin is formed and the rate of corrosion falls to the normal. In the case of aluminium alloys the protective action of the natural skin is more marked, but in both the etched and unetched alloys corrosion takes place much more readily than in the case of aluminium itself, the rate rising with the time to a maximum and then decreasing rapidly as a protective film forms.

A. R. POWELL.

Stress-strain curves and physical properties of metal, with particular reference to hardness. H. P. HOLLNAGEL (Trans. Amer. Soc. Steel Treat., 1926, 10, 87—108).—The stress-strain curve is interpreted in terms of atomic restitutional forces and slip whether in mass or of an atomic nature. Deformation may be either elastic, hysteretic, or permanent, or all combined in a random aggregate of crystals under stress. Various physical properties of metals, e.g., brittleness, ductility, resilience, toughness, elasticity, and rigidity, are interpreted in terms of the characteristic points of the stress-strain curve. Hardness is considered an average property which includes the effects of all other characteristics of the stress-strain curve; it may be defined as the work

done per unit volume per unit strain, and has the physical dimensions of pressure. The lack of correlation between indentation, scratch, and rebound hardness determinations is examined. Theoretically the new definition makes possible the unification of all hardness measurements. Indentation hardness depends on surface hardness (proportional to the elastic limit) and the mechanical properties which determine cold work.

T. H. BURNHAM.

See also A., Oct., 997, **Distortion of iron crystals** (TAYLOR and ELAM); **Pure zinc** (FREEMAN and others); **Behaviour of single crystals of aluminium under stress** (GOUGH and others); **Deformation of tungsten crystals** (SMITHELLS and others). 999, **Density and expansion of white and grey pig-iron** (SAUERWALD and WIDAWSKI). 1001, **Thermal anomalies in solid solutions** (CHEVENARD). 1016, **Precipitation of metals from non-aqueous solutions** (MÜLLER and others).

Refractory articles from tungsten powder. HARDÉN.—See XI.

PATENTS.

Electrolytic production of an adherent burnish on rustless iron or steel. W. A. F. PFANHAUSER, and LANGBEIN-PFANHAUSER-WERKE A.-G. (E.P. 257,140, 30.11.25).—A superficial layer of material is removed from the rustless alloy to allow subsequent deposition of a coating of iron. The process is carried out by making the alloy the anode in a bath of neutral iron salts with some neutral conducting salts, followed by reversal of the current. The deposit of iron is oxidised to produce the burnish.

L. M. CLARK.

Removal of arsenic from tin. H. HARRIS (E.P. 257,023, 20.5.25).—Molten arsenical tin is treated with a mixture of sodium hydroxide and sodium chloride to which small quantities of sodium nitrate are added from time to time. The slag, which contains the arsenic as sodium arsenate, is skimmed off the metal and boiled with water, and the clear solution is evaporated for the recovery of the arsenate. Eventually the mother-liquor is evaporated to dryness and the residue used again in the process.

A. R. POWELL.

Treating arsenic and antimony compounds and minerals. M. F. COOLBAUGH and J. B. READ, Assrs. to COMPLEX ORES RECOVERIES Co. (U.S.P. 1,597,018, 24.8.26. Appl., 4.3.22).—Arsenic and antimony ores containing sulphur are roasted in a slightly inclined, rotating, cylindrical furnace to convert the metals into arsenates, antimonates, and sulphates, which are subsequently extracted by a leaching process.

A. R. POWELL.

Casting iron in permanent molds at a regulated cooling rate. D. H. MELOCHE, Assr. to E. HOLLEY (U.S.P. 1,597,861, 31.8.26. Appl., 3.3.23).—See E.P. 209,043; B., 1924, 751.

Electrochemical treatment of copper ores. H. S. MACKAY (U.S.P. 1,598,296, 31.8.26. Appl., 19.5.25. Conv., 29.5.24).—See E.P. 238,962; B., 1925, 854.

Separating nickel and copper from copper-nickel mattes or other material. INTERNATIONAL NICKEL Co., Asses. of R. C. STANLEY (E.P. 240,789,

28.5.25. Conv., 30.9.24).—See U.S.P. 1,553,197; B., 1925, 888.

Electrothermic production of zinc. F. THARALDSEN (U.S.P. 1,598,176, 31.8.26. Appl., 25.5.22. Conv., 2.3.18).—See G.P. 395,105; B., 1924, 752.

Vanadium ores (U.S.P. 1,597,216).—See VII.

Enamelling metal (E.P. 254,452).—See VIII.

Tin (U.S.P. 1,598,295).—See XI.

Alkali metals (U.S.P. 1,597,231).—See XI.

XI.—ELECTROTECHNICS.

Refractory articles [for electric furnaces] from tungsten powder. J. HÄRDÉN (Chem. Met. Eng., 1926, 33, 543—544).—Crucibles and cylindrical collars suitable for use as the heating medium in Northrup induction furnaces may be made by ramming a plastic mass of tungsten powder bonded with a 25% solution of dextrose into suitable steel moulds. After heating the moulded masses to 600° in a non-reducing atmosphere, they may be drilled, filed, or turned in a lathe. They are rendered hard and durable by heating at 1600° in a carbon tube resistance furnace, whereby the tungsten is converted into the carbide containing about 6% C. The fired articles have d about 12.2—12.8, whilst the electrical resistance varies from 21 to 43 microhms per c.c., according to the temperature and duration of firing. A temperature of over 2000° is readily obtained in the high-frequency induction furnace, and metals and alloys of high m.p. may be melted in magnesia crucibles placed inside the tungsten shells. A. R. POWELL.

See also A., Oct., 1013, **Electrolytic separations by graded potentials** (LASSIEUR).

PATENTS.

Preventing the formation of explosive gas mixtures or similar dangerous mixture in electrical apparatus immersed in oil. M. BUCHHOLZ (E.P. 241,228, 12.10.25. Conv., 11.10.24).—The formation of explosive gas mixtures etc. in electrical apparatus immersed in oil, e.g., in oil switches, is prevented by causing a continuous and uninterrupted current of air drawn from the atmosphere to pass through the space above the level of the oil, whereby any fumes present are either discharged into the atmosphere or may be absorbed. J. S. G. THOMAS.

Electric furnace. C. E. TAYLOR (U.S.P. 1,596,902, 24.8.26. Appl., 20.11.20).—A complementary electrode is adjustable with regard to an electrode fixed at the bottom of a vertical casing. Material to be treated is fed from the top of the casing so as to surround the lower end of the adjustable electrode, and material fed to a point above the fixed electrode is confined to the region immediately surrounding the adjustable electrode. J. S. G. THOMAS.

Electric furnace for treating comminuted material. J. J. NAUGLE (U.S.P. 1,597,208, 24.8.26. Appl., 21.4.23. Renewed 7.11.25).—A substantially continuous cylindrical heating chamber has an inlet, an outlet, and a number of heating zones which can be separately regulated and controlled at different tempera-

tures. Electrodes rotate within the chamber and carry means for forcing material through the chamber. J. S. G. THOMAS.

Electromagnetic induction heating. C. R. BURCH, N. R. DAVIS, and METROPOLITAN-VICKERS ELECTRICAL CO., LTD. (E.P. 257,021, 20.5.25).—A material of low electrical resistivity is placed in a crucible of electrically conducting material which is subjected to an alternating electro-magnetic field of frequency f cycles per sec., such that the expression $\pi D\sqrt{2f/r} \times 10^9$ is greater than unity, D denoting the outside diameter of the crucible measured in cm., and r the resistivity of the material of the crucible in ohm-cm. Thereby eddy currents are induced in, and are substantially confined to, the crucible. J. S. G. THOMAS.

[Plates for] storage batteries. G. F. DOWNER (E.P. 257,019, 20.5.25).—Positive and negative lead plates of storage batteries are pasted with a substantially inactive paste of lead oxide and water, and then sulphated by soaking in a solution of sulphuric acid, loosening of the paste during sulphation being prevented by having the surface pores of the plates full of water on immersion in the acid. The plates are subsequently desulphated by electrical treatment in a small amount of substantially pure water, whereby the acid concentration of the electrolyte is raised approximately to normal strength. J. S. G. THOMAS.

Electrolytic production of alkali metals. P. E. HAYNES (U.S.P. 1,597,231, 24.8.26. Appl., 23.3.22).—A molten compound of the metal in a suitable bath is electrolysed, and the products are withdrawn separately as gas or vapour under less than atmospheric pressure. D. F. TWISS.

Electrodeposition of tin. H. R. McILHENNEY, ASSR. to VULCAN DETINNING Co. (U.S.P. 1,598,295, 31.8.26).—During the electrodeposition of tin the strength of the electrolyte is maintained by adding to it a tin compound that is insoluble in water but soluble in the products of the electrolysis as they are formed. A. R. POWELL.

Filaments for electron discharge devices. WESTERN ELECTRIC Co. From BELL TELEPHONE LABORATORIES, INC. (E.P. 257,125, 27.10.25).—A refractory metal, e.g., nickel, is mixed with compounds of the alkaline earths, e.g., barium and strontium carbonates, and heated to about 1200° in air or oxygen. The resulting product is crushed, mixed with a second refractory metal, e.g., platinum powder, pressed, and heated to about 1600° *in vacuo*. The resulting product contains alkaline-earth oxides uniformly distributed throughout the platinum-nickel rod. J. S. G. THOMAS.

Decoration of electric incandescence lamps and bulbs. Z. HADNAGY and V. G. DE CHRISTIAN (E.P. 257,067, 16.7.25).—A diffusing coating for electric incandescence lamps consists of a cold varnish or enamel made of a solution of an alkali silicate, e.g., sodium silicate, the concentration of which is 20—30%, according to the quantity and coarseness of grain of the pigment (if any) present in the coating. The addition of pigment, e.g., finely powdered fuchsine, to the stronger solutions of silicate prevents the formation of bubbles and swellings in the coating. J. S. G. THOMAS.

Producing stannous chloride (U.S.P. 1,597,653).—See VII.

Production of burnish on iron or steel (E.P. 257,140).—See X.

XII.—FATS; OILS; WAXES.

Application of the counter-current principle to the steam deodorisation of saponifiable oils. W. BRASH (J.S.C.I., 1926, 45, 331—333 T).—A description of the counter-current process for the deodorisation of oils is given. The theory of the process is discussed, and it is shown that towards the bottom of the deodorising tower a thinning out of the oil layers occurs, and that the cross-sectional area of the tower should diminish; the conditions necessary for the most economical use of the steam supply, and the variation of the height of the tower, with the thickness of the oil layers, the temperature and the speeds of efflux of the oil and steam, are derived. The advantages claimed over the unit deodoriser are economy of steam and short time of deodorising.

Oxidation of linseed and cottonseed oils. N. G. CHATTERJI and G. I. FINCH (J.S.C.I., 1926, 45, 333—335 T).—The oxygen absorption of linseed and cottonseed oils has been ascertained by direct measurement of the volume of oxygen absorbed when air or oxygen was bubbled through the oil in a closed circulation apparatus (J.C.S., 1925, 127, 2464) at 20°. No catalyst was used, and carbon dioxide and water were absorbed. The absorption of oxygen, after an induction period, reached a maximum identical with that calculated from the iodine value of the oils, assuming $2I = O$.

Recovered fatty matter. C. DABOUST (Bull. Soc. d'Encour., 1926, 125, 365—383).—The author discusses the various methods of fat recovery by boiling, autoclave treatment, acid treatment, and the action of solvents, particularly petrol. The treatment of bones for the extraction of bone grease, the recovery of skin grease in tannery operations, of grease from oily cleaning waste and from the refuse of slaughter houses, and of wool grease is described, with illustrations of washing machines, centrifuges, etc. A. RAYNER.

Unreliability of the Tortelli-Jaffé reaction [for marine-animal oils]. M. AUERBACH (Chem. Umschau, 1926, 33, 163—164).—This reaction (cf. B., 1914, 1061), claimed to be characteristic of marine-animal oils before and after hydrogenation, is now found to be given by hardened vegetable oils and also by tallows from animals which have been fed on fish meal, whilst a definite reaction is not always obtained with marine-animal oils, the colour produced sometimes being violet. Hardened bean oil, from bean oil which did not give a positive result, gives a definite reaction, but in many other samples of hardened bean oil the result was negative. It is suggested that the chromogenic substance may be formed as a result of a particular method of hydrogenation. A. RAYNER.

Oils extracted from the head of a dolphin. H. MARCELET (Bull. Soc. chim., 1926, [iv], 39, 1265—1275).—A more detailed account of work already published (B., 1926, 677).

Acetin or dichromate method [for glycerin analysis]. W. PRAGER (Z. deuts. Oel- u. Fett-Ind., 1926, 46, 577—578).—An influential committee has been formed in Germany for the investigation of the relative efficiency of the two methods. At the formation of this committee it was stated that results by the acetin method were commonly 2% lower than with the dichromate method. Such statements are strongly repudiated by the author, on theoretical and practical grounds, and the relative advantages and possible accuracy of the two methods are discussed.

A. RAYNER.

Free alkali in soap. W. ISMAILSKY (Z. Deuts. Oel- u. Fett-Ind., 46, 545—546, 562—564).—The alcohol extraction method and the baryta method for determining free alkali in soap are untrustworthy, whilst the modification of the baryta method by Bosshard and Huggenberg yields satisfactory results only with soaps containing up to 10% of fatty acids, the errors being due to the absorption of alkali by the baryta soap, the difficulty of titration in presence of a bulky and sometimes coloured precipitate, and the presence of silicates. In an improved method 10 g. of soap are dissolved in 200 c.c. of freshly distilled water in a 400 c.c. flask, 20 c.c. of 30% barium chloride solution are added, the flask is loosely corked, and the contents are boiled and rotated until the precipitate agglomerates into a ball or a granular precipitate. The solution is cooled, the flask being corked meanwhile, and is filtered rapidly, and the precipitate washed with 100 c.c. of cold distilled water. The filtrate is titrated with 0.1N-acid, using phenolphthalein as indicator, and the result calculated to sodium hydroxide. W. G. CAREY.

Oxidation of drying oils. TARADOIRE.—See XIII.

Nutritive value of synthetic fats from fatty acids with an odd number of carbon atoms. OYAKI.—See XIX.

PATENTS.

Apparatus for extraction of oils, fats, waxes, resins, and greases from substances containing them. J. MACGREGOR, and E. SCOTT & Co., LTD. (E.P. 256,019, 25.6.25).—The material is treated with solvent in one or more percolators or extractors mounted above a steam-heated solvent recovery vessel with which the percolators are in communication by means of passages controlled by valves. When extraction has been carried to the desired extent the supply of solvent is cut off from the extractors, the solvent and oil are drawn off, and the treated material is caused to fall into the solvent recovery vessel, where it is subjected to the heat of the steam in the surrounding jacket or, where desired, subjected to open steam. The recovery vessel is vacuum-operated and when removal of solvent is completed, the treated contents are discharged. When continuous operation is desired the extracted material from the extractors, instead of going to the recovery vessel direct, is passed into a hopper, whence, by means of a worm-feed device consisting of externally heated tubes, the discharged material is ultimately delivered into a vacuum vessel, and then it passes through a discharge orifice controlled by a valve into a second vessel.

From this lowermost vessel the material can be discharged without breaking the vacuum in the remainder of the apparatus.

H. M. LANGTON.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Rapid oxidation of drying oils; effect of anti-oxidising agents. F. TARADOIRE (Compt. rend., 1926, 183, 507—508).—The spontaneous ignition of cotton waste soaked in a mixture of a drying oil, turpentine, and metallic drier is retarded or inhibited by anti-oxidising agents (cf. B., 1926, 135). Of a further series of organic compounds examined for this property, nitroso-derivatives of diphenylamine prevented ignition, whilst α - and β -naphthylamine, benzidine, pyridine, and quinoline retarded it for several hours. S. S. WOOLF.

Accelerated paint testing. M. SCHULZ (Farben-Ztg., 1926, 31, 2879—2882).—From general considerations of atmospheric disintegrating influences on paints, the following scheme for rapidly reproducing the results of a lengthy period of weathering is arrived at. The paints under test are applied to iron plates and allowed to dry for at least three days, then "matured" for 24 hrs. at 80°. Subsequent treatment consists of six repetitions of the following cycle: hanging in distilled water at 20°, exposure to ultra-violet light while moist and again when dried, re-immersion in distilled water, enclosure in a damp atmosphere containing carbon dioxide, further exposure to ultra-violet light under damp conditions, then at 50°, enclosure in an atmosphere containing sulphur dioxide, exposure to damp at 35—40° with interpolated periods at —5°, and renewed action of ultra-violet rays. The results of some tests carried out by this method on zinc oxide-lithopone paints show agreement with a 2-year exposure to the atmosphere, in that the iron plates show increasing rusting with the increase in proportion of lithopone to zinc oxide in the protecting paint. The author stresses the value of these "physical paint tests" as a complement to chemical analysis.

S. S. WOOLF.

Traffic paint. H. A. NELSON and S. WERTHAN (Ind. Eng. Chem., 1926, 18, 965—970).—The composition and properties desirable for paints suitable for marking out directional lines on roads are studied by a series of tests on consistency, opacity, visibility by day (normal illumination) and night (oblique illumination), and resistance to combined weathering and abrasion. Most satisfactory results were obtained with lithopone (of non-greying quality), titanox, or zinc oxide as pigment, asbestine (talc) as inert filler (30% of total "pigment"), and a medium of high volatile content, the non-volatile portion consisting of tung oil-ester gum or tung oil-lime-hardened rosin, with or without the addition of linseed oil varnish. Appreciable quantities of lead pigments are not desirable owing to tendency to discoloration. White is the most suitable colour for this type of paint, as yellow and orange commercial traffic paints tested had lower visibility.

S. S. WOOLF.

See also A., Oct., 1926, 993, **Phosphorescent sulphides of zinc** (GUNTZ).

§PATENTS.

Manufacture of carbon black, unsaturated gases, and hydrogen. J. J. JAKOWSKY (U.S.P. 1,597,277,

24.8.26. Appl., 10.11.22).—The products of thermal dissociation of a hydrocarbon are passed through a cooling liquid.

S. S. WOOLF.

Extracting turpentine, pine oil, and rosin [colophony]. E. P. STEVENSON, Assr. to A. D. LITTLE CO. INC. (U.S.P. 1,597,215, 24.8.26. Appl., 11.5.20).—Coniferous wood is treated with a coal tar hydrocarbon solvent of b.p. below that of turpentine. The extract is treated with hydrogen chloride to precipitate certain dark-coloured substances, and the solvent, turpentine, pine oil, and rosin are separated by distillation.

S. S. WOOLF.

Obtaining phenol-aldehyde condensation products in a purified form. BAKELITE GES.M.B.H. (E.P. 246,833, 22.1.26. Conv., 27.1.25).—Phenol-aldehyde resins in alkaline solutions are precipitated by carbonic or other acid in the presence of hydrotropic salts, e.g., soluble salts of aromatic or the higher fatty acids, especially sodium salicylate or alkali soaps.

S. S. WOOLF.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Theory of needle-shaped rubber molecule. E. LINDMAYER (Gummi-Ztg., 1926, 40, 2805—2807; cf. B., 1926, 714).—Unworked rubber has a formula $(C_5H_8)_{12}$ but when masticated undergoes fission into shorter $(C_5H_8)_6$ and $(C_5H_8)_3$ molecules; these shorter needle-shaped particles are in Brownian movement, and their rotation gives the optical effect of a round particle. The separation of "diffusion rubber" by dialysis from masticated rubber is explained by the presence of these molecules of various magnitude. At 75° the $(C_5H_8)_{12}$ molecule undergoes disaggregation into $(C_5H_8)_6$, the change being easily reversible, whereas the $(C_5H_8)_6$ and $(C_5H_8)_3$ molecules formed by mastication re-associate themselves much more slowly on account of their more drastic separation from one another. Calender grain is caused by orientation of the immobile $(C_5H_8)_{12}$ molecules; freezing of rubber at 14° is explained by the terminal coupling of two such molecules, the change being assisted by stretching. The gradual character of the softening on warming is due to the resins present, resin-poor rubbers showing a sharper change. Vulcanisation normally gives rise to $(C_5H_8)_6 \cdot S \cdot (C_5H_8)_6$ molecules, but overworked rubber needs a higher proportion of sulphur due to the formation of some $(C_5H_8)_3 \cdot S \cdot (C_5H_8)_3$ molecules; to vulcanite is ascribed the formula $(C_5H_8S)_6 \cdot S \cdot (C_5H_8S)_6$. The possibility of reclaiming rubber is dependant on the change $(C_5H_8)_6 \cdot S \cdot (C_5H_8)_6 \rightarrow (C_5H_8)_3 \cdot S \cdot (C_5H_8)_3 + 2(C_5H_8)_3$ with subsequent gradual polymerisation of the latter product to $(C_5H_8)_6$ and $(C_5H_8)_{12}$ molecules.

D. F. TWISS.

Experiments with unmasticated rubber. W. C. DAVEY (J.S.C.I., 1926, 45, 364—365 T).—The incorporation of varying amounts of glue produces effects in raw unmasticated rubber which are in some respects similar to those produced by vulcanisation. Comparative results of tests on vulcanised rubber prepared from unmasticated and masticated mixings are also given, showing the former to produce higher tensile strengths over a wider range.

Theory and practice of the acceleration of vulcanisation. F. EMDEN (Kautschuk, 1926, 1, 91—94, 110—112, 137—138, 158—161, 180).—A review of the theories as to the mode of action of accelerators, and of the present knowledge as to individual examples or groups of such agents, their activation by metallic oxides, their advantages and disadvantages. D. F. TWISS.

Antioxidants and their retarding action in the deterioration of rubber. L. E. WEBER (Ind. Eng. Chem., 1926, 18, 963—964).—Other factors than mere oxidation must be involved in the deterioration of vulcanised rubber, and further chemical investigation of this deterioration is needed. Some organic accelerators exert an antioxidant effect; certain condensation products of aldehydes with an aromatic amine, e.g., of aldol and α -naphthylamine, are very feeble accelerators of vulcanisation, but have a marked preservative action on vulcanised rubber. It is possible that these antioxidants do not act catalytically, but protect the rubber by undergoing preferential oxidation themselves. D. F. TWISS.

See also A., Oct., 1003, **Consistency of solutions of rubber in benzene** (HERSCHEL and BULKLEY).

XV.—LEATHER; GLUE.

Chrome tanning at the isoelectric point of collagen. R. M. COBB and F. S. HUNT (J. Amer. Leather Chem. Assoc., 1926, 21, 454—464).—Four series of chrome tanning liquors were prepared containing 1% of salt, 1.5% Cr_2O_3 and sodium formate or acetate, or 0.75% Cr_2O_3 and sodium formate or acetate respectively. The basicity was 33%. The p_{H} value was adjusted by adding acid or alkali, and pieces of pelt were tanned in each series at different p_{H} values for a definite period, after which the chromium content was determined. It increased as the p_{H} value increased. There was no break at the isoelectric point, but a steady unbroken increase from p_{H} 3.0 to 6.0, hence chrome tanning cannot be a chemical reaction between free ions, but the chromium unites with the collagen by means of secondary valencies. The degree to which pelt absorbs acid from the pickle must greatly affect the extent of tannage. D. WOODROFFE.

South Indian tanning materials. K. S. CHOUDARY and E. YOGANANDAM (J. Soc. Leather Trades Chem., 1926, 10, 222—228).—The analyses of eight common South Indian tanning materials are: konnan (*Cassia fistula*) tans 11.2%, non-tans 12.1%; avaram (*C. auriculata*), tans 16.8%, non-tans 10.1%; wattle (*Acacia decurrens*), tans 35.8%, non-tans 14.4%; divi divi (*Caesalpinia coriaria*), tans 33.6%, non-tans 26.4%; sumach (*Anogasis latifolia*), tans 13.0%, non-tans 9.1%; babool (*Acacia arabica*), tans 7.6%, non-tans 4.6%; gothar (*Zizyphus xylopra*), tans 12.8%, non-tans 17.7%; myrobalans (*Terminalia chebula*), tans 27.1%, non-tans 10.5%. Gothar and myrobalans contain 19.2% and 16.8% of sugar respectively, calculated on the tannin. Myrobalans and wattle have the most favourable proportion of tans to non-tans. Divi divi, myrobalans, and sumach lost most tannin, and wattle and avaram the least, when infusions were kept. The p_{H} value

of all the liquors except those of divi divi (3.5) and myrobalans (3.4) was near 4.7. Divi divi, myrobalans, sumach, and babool are the liquors in which the non-tannins diffuse more rapidly. The materials that lose most on keeping diffuse most freely. The colour of the liquor has been shown to depend on whether the material has been extracted hot or cold. Avaram bark yields a less reddish coloured infusion when extracted hot, whereas divi divi gives a more reddish colour when extracted hot. D. WOODROFFE.

[Ultrafiltration of vegetable tanning solutions.] R. J. BROWNE (J. Soc. Leather Trades Chem., 1926, 10, 235—237).—A reply to criticisms by Thomas and Kelly (B., 1926, 290). The author's ultrafiltration experiments (B., 1923, 989A) were purely mechanical and there was no chemical action. Thomas and Kelly did not test the permeability of their membranes, and did not use high pressures in every case. The author prepared various types of collodion membranes, all of which retained tannin if their impermeability was suitable. D. WOODROFFE.

Action of sodium sulphate in synthetic tanning materials. E. WOLESENSKY (U.S. Bur. Standards, Tech. Papers, 1926, 20, 529—544).—Wet chromed hide powder removes sulphuric acid from weak solutions, and from a 0.5% solution of sodium sulphate acidified with 0.5% of acetic acid. Prolonged washing did not remove the sulphuric acid. Experiments showed that sulphuric acid in combination with hide substance cannot be completely displaced by a synthetic tannin whereas combined synthetic tannin can be removed from hide substance by treatment with sulphuric acid, or with a mixture of sodium sulphate and acetic acid solutions. It follows that if a hide is treated with a synthetic tannin containing a soluble sulphate, both sulphuric acid and synthetic tannin will combine with the hide. The neutralisation of the excess sulphuric acid in the manufacture of synthetic tannins by means of sodium hydroxide or any other reagent which yields a soluble sulphate, does not prevent the sulphuric acid from reacting with the hide during the tanning process. It is possible that the sulphuric acid combined with the hide is not injurious to the latter, but this remains to be determined. The behaviour of sodium sulphate in synthetic tannins must cause an appreciable error in the non-tans determination unless the free sulphuric acid and soluble sulphates are precipitated by means of an equivalent amount of barium acetate. The synthetic tannins used included phenolsulphonic and cresolsulphonic acids condensed with formaldehyde. D. WOODROFFE.

Fermentation of divi divi liquor. II. Acidity of divi divi liquor. K. S. CHOUDARY and E. YOGANANDAM (J. Soc. Leather Trades Chem., 1926, 10, 237—239; cf. B., 1925, 218, 412).—Divi divi liquors (d 1.015) extracted in the hot and in the cold were kept and analysed periodically for total acidity and p_{H} value. The total acidity increased to a maximum about the 9th day, then diminished and rose again to a maximum on the 74th day, the maximum on the latter day being 30% more. The p_{H} value was 3.7, and changed very little. It was 3.3 at the end of the period. The total

acidity was greater in the liquors in which the pods were allowed to remain. D. WOODROFFE.

Direct measurement of plumping power of tan liquors. [Report of committee of the American Leather Chemists' Association.] R. E. PORTER (J. Amer. Leather Chem. Assoc., 1926, 21, 425—435; cf. B., 1925, 603).—Dry sifted hide powder (1.5 g.) is weighed into each of two cylinders 230 mm. long and 26 mm. inside diam., covered with 50 c.c. of water, and kept for 3 hrs. with 5 stirrings. Measuring plungers made from the bulb and upper stem of a 15 c.c. pipette and fitted at the lower end with a perforated brass disc, are allowed to drop slowly on the hide powder in the cylinders. After 5 min. the height of the hide powder is read on the scale to the nearest mm. The plungers are removed and the excess soak water siphoned off to a line giving a capacity of 26.66 c.c. The compressed hide powder is well stirred up in the 25 c.c. of water, 75 c.c. of the tan liquor are pipetted into the cylinder, which is kept for 24 hrs. with occasional stirring, then the plunger is introduced as before and the new reading taken. The final reading divided by the initial reading gives the plumping ratio of the tan liquor. This method shows higher sensitivity and involves the use of hide powder and simple apparatus. The calf pieces in the Wilson and Gallun method are difficult to obtain uniform. D. WOODROFFE.

Determination of moisture in leather. [Report of a committee of the American Leather Chemists' Association.] F. P. VEITCH and T. D. JARRELL (J. Amer. Leather Chem. Assoc., 1926, 21, 435—445; cf. B., 1925, 683).—It is recommended that the toluene distillation method, Bidwell—Sterling modification (cf. B., 1926, 839), be made official for the determination of moisture in leather. D. WOODROFFE.

Determination of fat in leather. D. WOODROFFE (J. Soc. Leather Trades Chem., 1926, 10, 219—221).—Samples of chrome and vegetable tanned leathers, fat-liquored either with cod oil or with degreas fat liquors, have each been extracted with light petroleum in three ways, from the air-dry sample, from the dried leather, and from the leather which had been dried at 105° and then exposed to a humid atmosphere. The extract from the dried leathers was less than that from the air-dry sample and the difference could not be accounted for by the extraction of water-soluble matter (cf. Hey, B., 1923, 65 A). There was no evidence to show that oxidation of the oils took place when the above leathers were dried by heating at 105°. The greater amount of extract from air-dry leather is probably due to moisture. D. WOODROFFE.

PATENT.

Finishing and colouring of leather. M. C. LAMB, and P. SPENCE & SONS, LTD (E.P. 254,350, 31.12.24).—In finishing and colouring leather a previously prepared paint or dope is applied, made by adding to titanium tannate natural or artificial organic dyestuffs or mineral pigments. The dope may be mixed with mucilage prepared from gums, Irish moss, linseed, etc., and applied by brushing or spraying. H. MOORE.

XVI.—AGRICULTURE.

Chemical methods for determining whether soils need nitrogenous or potassium fertilisers. A. NEMEC (Compt. rend., 1926, 183, 483—485; cf. B., 1926, 763).—Nitrates are determined in a water extract of soil (100 c.c. water to 30 g. soil for 1 hr.) after allowing nitrification to proceed in the moist soil for 14 days. Comparison of the figures obtained with the results of field experiments with sodium nitrate indicate that, for sugar beet, soils containing less than 25—30 mg. of nitric nitrogen per kg. of air-dry soil may be expected to respond to nitrogenous fertilisers. Corresponding figures for barley are 22—28 mg. and for oats, 20 mg. of nitrogen. Potassium is determined in a water extract, cleared by adding calcium carbonate and heating on the water-bath, by Cameron and Faillyer's method (cf. B., 1903, 1259). The figures above which potassium fertilisers are not likely to be effective are, for sugar beet 37 mg. K₂O, for barley 10—20 mg., for oats 13—19 mg., for potatoes about 25 mg. per kg. of air-dry soil.

C. T. GIMMINGHAM.

Influence of the application of superphosphate and sodium nitrate on the chemical composition of the stem and leaf of pasture cuts of cocksfoot. T. W. FAGAN and R. E. EVANS (Welsh J. Agric., 1926, 2, 113—116).—The ratio of true to crude protein was higher in the leaves, which were richer in these substances and in ash than the stems. The stems contained more iron and chlorine, and the leaves more calcium. Application of superphosphate increased the fibre and decreased the chlorine content; sodium nitrate decreased the phosphate and calcium content but increased the protein content. A. A. ELDRIDGE.

Insecticidal value of certain war chemicals as tested on the tent caterpillar. F. G. BRINLEY (J. Agric. Res., 1926, 33, 177—182).—A number of compounds used during the war (chiefly organic arsenic compounds and metal stearates and resinates) have been tested, both as stomach poisons and contact insecticides, on eastern tent caterpillars (*Malacosoma americana*). Most of the compounds that proved toxic to the insects were also so toxic to plants as to have no practical value. Iminodiphenylenearsenious oxide was an exception. It was equal in toxicity to lead arsenate and was not injurious to bean plants. It also showed promise as a contact insecticide. The compound is, however, highly irritant, and great caution is required in handling it. Copper stearate and resinate were definitely repellent to the insects, though comparatively non-toxic; they have good spreading and adhering qualities.

C. T. GIMMINGHAM.

See also A., Oct., 1018, **Electrometric titration of phosphates** (BODFORSS).

PATENTS.

Preparation of calcium nitrate capable of being strewn. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 242,990, 11.11.25. Conv., 11.11.24).—Substantially anhydrous calcium nitrate, containing up to 17% of nitrogen, is mixed with a small proportion (about 0.1%) of solid hydrate of calcium nitrate at a temperature below the m.p. of the hydrate. This removes the

tendency of anhydrous calcium nitrate to effloresce, and maintains the product in a condition fit for distribution in the field for a considerable time. C. T. GIMINGHAM.

Manufacture of fungicidal and insecticidal media. W. CARPMAEL. From *FARBENFABR. VORM. F. BAYER & Co.* (E.P. 257,171, 19.2.26).—Copper sulphate is heated with an alkaline-earth carbonate in the presence of water. For example, 40 kg. of ground anhydrous copper sulphate are mixed with 60 kg. of dry finely divided chalk; 20 litres of water are stirred in to form a paste, which is heated to about 100°. The mass is dried and powdered; other insecticides or fungicides may be added. The powder thus prepared is used for dusting and is distinguished by great adhesiveness.

C. T. GIMINGHAM.

XVII.—SUGARS; STARCHES; GUMS.

Precipitation of calcium phosphate under various conditions, with special reference to sugar factories. R. G. W. FARNELL (J.S.C.I., 1926, 45, 343—347 T).—The formation and precipitation of calcium phosphate was studied in aqueous solution, in sucrose solution, and in a solution containing cane colloids. The methods employed were similar to those described in the precipitation of calcium sulphite (cf. B., 1926, 72), and the following factors were varied: (1) time of precipitation (5—60 min.); (2) temperature of precipitation (20—100°); (3) extent of neutralisation of phosphoric acid (p_H 2.2—10.6); (4) initial concentration of phosphoric acid (0.0070—0.0934 g. P_2O_5 per 100 c.c.). The experimental results are represented graphically. Whilst sucrose had no effect in delaying the precipitation of tricalcium phosphate or altering the nature of the precipitate, the precipitation was much affected by the salts and colloids present in cane syrups and sugars. The greater the amount of P_2O_5 naturally occurring in the juice, the greater is the precipitation of calcium phosphate on adding lime, and the more efficient is the clarification. If the clarified juice, after liming, heating, and settling for 1 hr., leaves the clarifiers at p_H 7.0 practically all the phosphate will have been precipitated. Phosphoric acid and phosphate generally constitute the principal buffer substances of the cane juice. The drop in p_H noticed in sugar factories on heating a limed juice appears to be partly due to the precipitation of calcium phosphate and its subsequent hydrolysis to free phosphoric acid and the more basic hydroxyapatite, which is the only phosphate of lime which is not decomposed by water.

Sucrose as an acid. [New theory of the formation of molasses.] J. DEDEK and P. TERECHOV (*Z. Zuckerind. Czechoslov.*, 1926, 50, 349—355).—Hydrogen-ion concentration determinations and mol. wt. measurements lead the authors to announce a theory of the formation of molasses based on the supposition that sucrose plays the part of a monobasic acid in dilute solution, the valency rising in higher concentrations, and being dependent on the ratio of sucrose to alkali. In general, the stronger the concentration of the salts present, and the weaker their acids, then the greater the amount of sucrose entering into the reactions. In the case of any molasses the molar concentration of

the sucrose practically agrees with the equivalent concentration of the potassium and sodium ions.

J. P. OGILVIE.

Surface tension of sugar factory juices. V. ŠÁZAVSKÝ (*Z. Zuckerind. Czechoslov.*, 1926, 50, 378—380, 423—426).—Using Traube's stalagmometer (cf. B., 1922, 121 A), the surface tension of saccharine solutions can be determined with sufficient accuracy provided the density is not more than 30° Brix. Above that degree the results are less certain, and such solutions require to be diluted and kept for a time. Lindfors' results obtained by using the Du Nouÿ apparatus (B., 1926, 72) are not considered accurate within 10%. Surface tension determinations depend largely on the p_H of the solution examined, and addition of alkali causes a marked alteration of the result.

J. P. OGILVIE.

Practical methods of p_H measurement for the control of cane juice defecation. R. T. BALCH and H. S. PAINE (*Intern. Sugar J.*, 1926, 28, 425—429).—Apparatus consisting of a recording potentiometer, a saturated potassium chloride-calomel electrode, a salt bridge, a continuous-flow electrode chamber, a tungsten or similar type of electrode, and a small centrifugal pump has been adapted for the control both of intermittent and of continuous liming. At present in continuous liming the recording potentiometer may be made to record from one to a number of circuits simultaneously, to operate such signals as warning lights or bells when the reaction does not fall within the desired limits, and, in proportion to the change in reading, to operate an electro-magnetic valve controlling the rate of lime addition.

J. P. OGILVIE.

Simple method of measuring hydrogen-ion concentration and its significance in the beet-sugar industry. F. TÖDT (*Z. Ver. deut. Zuckerind.*, 1926, 494—508).—A colorimetric spotting plate procedure is recommended, comparisons being made with a colour chart (printed in the original). Indicators are used in the form of 0.04% alcoholic solutions, and molasses is diluted to 10° Brix and other products to 20—40° Brix. Test-papers are recommended only for approximate results; any universal indicator containing bromothymol-blue as the "alkaline" constituent is condemned so far as sugar factory products are concerned. First carbonatation juice contains strong bases and salts of very weak acids and very weak bases; in the second carbonatation the strong bases have for the greater part been eliminated, whilst in the thick-juice the very weak bases are no longer to be found, having disappeared during the evaporation. Juice from damaged roots shows a low p_H value, as it contains stronger acids and weaker bases, as compared with normal juice. (Cf. B., 1925, 416.)

J. P. OGILVIE.

Applications of [electrical] ash analysis in [sugar] factory control. H. LUNDÉN (*Z. Ver. deut. Zuckerind.*, 1926, 510—516).—Yield formulæ which are based on values for the mineral matter of sugar factory and refinery products are more accurate and simpler, and are rapidly calculated when the ash is determined by the electrical conductivity method (B., 1925, 1003). This method enables the quality of

white sugars to be gauged according to their ash content, the finest refined having a value of 0.002, average refined, 0.002 to 0.003; and "melis," 0.025%. The amount of ash in a sugar is normally $\frac{1}{80}$ th of that of the massecuite from which it is crystallised, and the colour about $\frac{1}{25}$ th. In the case of persistent impurities this effect may be only $\frac{1}{8}$ th or even only $\frac{1}{2}$.

J. P. OGILVIE.

Defects of Fehling's solution. R. OFNER (Z. Zuckerind. Czechoslov., 1926, 50, 355—356, 360—364, 370—372).—Fehling's solution as a reagent for the determination of invert sugar even in first-product raw beet sugars is insufficiently reliable for the following reasons: considerable oxidation of an inconstant degree takes place in the presence of sucrose; it is too sensitive, results depending on deviations of procedure which are not realised; reduction depends too much on the regularity of ebullition; there is great inclination to delay in boiling; and certain impurities have an important influence on the separation of cuprous oxide.

J. P. OGILVIE.

PATENTS.

Extraction of sugar from sugar cane. F. KESSLER (E.P. 250,889, 12.6.25. Conv., 18.4.25).—The crushed raw cane is fed on to a sliding plate, where it is sprayed with the solvent, and then slightly compressed by means of a spring-journalled compressing roller. It is also treated from above or below with compressed air or steam from a blowing box, and finally passes to the extracting pressing rollers for the removal of the concentrated extract.

F. R. ENNOS.

Producing a reaction of lime and sucrose in the manufacture of sugar from beets. R. W. SHAFOR, E. MORRISON, R. J. BROWN, L. A. STENGER, and A. R. NEES, Assrs. to GREAT WESTERN SUGAR Co. (U.S.P. 1,593,782, 27.7.26. Appl., 15.8.24).—The apparatus comprises a bank of horizontal flues, interconnected for the passage of a cooling liquid along a zig-zag course, the flues extending transversely across a space constituting the circulation chamber, in which the reaction between the lime and sucrose is accomplished. The chamber is divided by vertical partitions to provide a zig-zag path for the material, whilst the outer compartments of the circulatory chamber connect at their lower ends with circumferential egress ports of rotary centrifugal pump impellers in order to promote movement of the liquid.

J. P. OGILVIE.

Processes for obtaining tricalcium saccharate. C. STEFFEN, JUN. (E.P. 255,175, 22.7.26).—Molasses or sugar solution is subjected on the way to the main collecting receiver immediately after the addition of calcium oxide (in powder) to a vigorous mixing, after which the liquid is passed on to the main collecting receiver and repeatedly circulated through the whole apparatus until the desired amount of tricalcium saccharate has been formed. This procedure results in economy in the use of lime.

J. P. OGILVIE.

Apparatus for evaporating and crystallising sugar and other solutions. F. LAFEUILLE (E.P. 252,686, 15.4.26. Conv., 20.5.25).—A cylindrical vessel capable of rotation about its axis and provided with a

set of circulation tubes, has a vacuum chamber of larger diameter as an extension. By the passage of steam or cold water through the tubes, the apparatus may be applied alternatively as an evaporator, under the vacuum, or as a crystalliser.

F. R. ENNOS.

Molasses-product and method of producing the same. S. D. WILKINS, H. C. REINER, and E. C. GOULD (E.P. 257,691, 15.6.25).—Molasses is heated to about 90° to lower its viscosity, mixed with a suitable colloid, e.g., casein or skimmed milk solids, and evaporated to dryness in a dehydrating apparatus. The resulting product is a non-hygroscopic, fluffy powder which, while it may be applied to substantially all the present-day uses of liquid molasses, is more readily and economically handled.

F. R. ENNOS.

XVIII.—FERMENTATION INDUSTRIES.

Resinification of the α -bitter acid of hops (humulone) by molecular oxygen. W. WINDISCH, P. KOLBACH, and J. YOFÉ (Woch. Brau., 1926, 43, 349—353, 359—363, 369—372, 379—383).—During the boiling of wort with hops the humulone, which is the most valuable of the bitter principles of hops, is partially oxidised and converted into resinous substances. Similar changes occur during the storage of hops, and cause deterioration in their brewing value. The humulone is transformed first into α -soft resins (soluble in light petroleum), and finally into insoluble hard resins. For these changes both oxygen and water are necessary. The first step is peroxide formation at a double linking, and by uptake of water the linking is broken and an acid is formed as one of the products. In the pure dry state, humulone is very stable. Exposed to the atmosphere it is oxidised to the extent of 30—40% in 2 months at the ordinary temperature and in a few hours when heated near its m.p. It is very stable when dissolved in strong alcohol, but on addition of water it becomes very readily oxidisable as the limit of solubility is approached. Considerable oxidation occurs also during the evaporation of solutions in light petroleum, and for this reason, in the analysis of hops, light petroleum extracts which have been evaporated to dryness to determine their content of dry substance should not afterwards be used for the determination of humulone. In presence of certain readily oxidisable substances humulone oxidises much more rapidly than when pure; amongst such accelerating agents are hop oil and certain soft resins formed by the oxidation of humulone itself. On the other hand, the hard resin formed from humulone appears to protect it from oxidation, for under most conditions humulone which has undergone oxidation to the extent of 30—40% oxidises further at a very much slower rate. Heating fresh hops probably tends to preserve the humulone during subsequent storage, by driving off volatile matters, such as hop oil, which would accelerate its oxidation, and by melting the humulone and thereby reducing its surface. Heating of hops which have already been stored may be harmful, since substances present which have already taken up oxygen and formed peroxides, may transfer it to the humulone. Siller's method for determining humulone by precipitation with lead acetate

needs further study, for lupulin and hops often contain hard resins which may be precipitated under the same conditions.

J. H. LANE.

Determination of the colour of malt. V. BERMANN (Woch. Brau., 1926, 43, 309—312).—Adler has constructed an apparatus for measuring the colour of malt extracts in accordance with the method previously described (B., 1925, 820; 1926, 170). In terms of Ostwald's theory of colour, the apparatus actually measures the "white-content" of the colour of the malt extract, and this by difference from unity gives the real colour-content ("Vollfarbe"), since for moderately clear liquids the "black-content" is negligibly small.

J. H. LANE.

Colorimeters based on Ostwald's theory. F. MESTAN (Woch. Brau., 1926, 43, 312—316).—From a comparison of Adler's apparatus (see preceding abstract) with Hahn's instrument and Ostwald's chrometer, the author concludes that the first is likely to prove most suitable for the examination of malt extracts, but it could be improved in several important respects.

J. H. LANE.

Development and nutrition of yeast. III. A. TAIT and L. FLETCHER (J. Inst. Brew., 1926, 32, 385—414; cf. B., 1922, 724 A; 1923, 792 A).—Yeast kept for a day or two in its own beer after complete fermentation and then seeded into fresh wort exhibits a lag-phase in growth corresponding to a building-up process during which there is an appreciable assimilation of nitrogen. The influence of temperature, during storage of the yeast, in increasing the lag-phase points to the endocellular changes being of an enzymic nature, the velocity of the changes being affected by the hydrogen-ion concentration of the storage liquid. Storage of yeast in a nitrogen-free carbohydrate solution lessens the lag period. There is a marked difference between unwashed and water-washed yeast during storage, the washed yeast giving a greatly lessened lag-phase and a smaller proportion of dead cells. The washed and unwashed yeasts differ in their behaviour when stored in air, carbon dioxide, and nitrogen. With one exception, *i.e.*, the acid content of yeast stored in carbon dioxide, the amounts of acid, "total" nitrogen, and "formol" nitrogen are greater in the unwashed yeast. The washing almost entirely removes the interstitial and internal alcohol, the presence *per se* of which, however, has no effect upon the changes taking place during storage. Substances adsorbed on the surface of the yeast, and having a clogging effect, may be partially removed by washing, while the internal hydrogen-ion concentration of the cells is hardly, if at all, affected. Washing brings about an alteration in the course of autofermentation. Pressed unwashed yeast cannot get rid of its poisonous by-products, suffers from toxic substances, and quickly dies, this being followed by rapid liquefactive autolysis. Placed in fresh wort the unwashed yeast cells have first to expel the accumulated poisonous by-products and then, or concurrently, to absorb the carbohydrate and nitrogenous matters necessary to rebuild the starved plasma; in the case of the washed variety, although by-products and

toxicity may be less in amount, still the yeast has also to rebuild its depleted plasma. Accordingly, the length of the lag-phase period must be some function of the extent of the cell disintegration.

C. RANKEN.

Colour changes of beer during the primary fermentation. W. WANDERSCHECK (Woch. Brau., 1926, 43, 391—395, 403—408).—As a rule brewery worts become appreciably lighter in colour during fermentation. In the production of very pale bottom-fermentation beers of the Pilsen type it was found that in some cases the fermented beers were slightly darker than the original worts. A study of these cases led to the following conclusions:—The colour of wort fermented by freshly propagated pure-culture yeast diminishes immediately after pitching and throughout the fermentation. The yeast takes from the wort certain colouring matters, possibly protein-tannin compounds, which accumulate on it during successive fermentations until they begin to redissolve. In the cases studied this occurred after 6—7 fermentations. The re-solution of the colouring matters is inhibited by air but favoured by carbon dioxide. Hence, yeast which has been repeatedly used may extract colour from wort at the commencement of fermentation and yield colouring matters to the wort during the later stages. This is much more likely to occur with bottom-fermentation than with top-fermentation, owing to the much greater duration of the former. Washing the yeast with water and sifting, between successive fermentations, only partially removes the colouring matters in question, but treatment with dilute sodium carbonate solutions is much more effective. Darkening of wort during fermentation is not caused by the use of discoloured hops, nor by flocculent matters precipitated during wort boiling; if the latter remain in the beer during fermentation they tend to decolorise rather than to colour the wort. The main factors which affect the phenomenon are the character of the yeast and particularly the method employed for cleansing it, and the character of the malt used. The changes in the colour of wort during fermentation do not correspond with the changes in the concentration of hydrogen ions.

J. H. LANE.

See also A., Oct., 1061, **Action of oxygen on alcoholic fermentation of sugar** (GORR and PERLMANN); **Action of ammonium salts on yeast fermentation** (ZELLER); **Fermentation of sugars etc. by yeast juice and fresh yeast** (NEUBERG and KOBEL).

PATENTS.

Process for making fresh yeast lasting. L. J. J. LINDEMANN, AssT. to T. P. Hodge (U.S.P. 1,596,279, 17.8.26. Appl., 15.9.24).—Finely-divided yeast is suspended in lime or other alkaline water at 33—43° with or without the addition of a small quantity of sugar, and the whole gently agitated until tests show that the glycogen has been removed.

F. R. ENNOS.

Refining alcohol. E. A. BARBET (U.S.P. 1,598,548, 31.8.26. Appl., 14.11.22. Conv., 15.11.21).—See E.P. 189,136; B., 1923, 1193 A.

XIX.—FOODS.

The miscometer: an apparatus for obtaining composite samples [e.g., of milk]. J. HOUSTON (Analyst, 1926, 51, 453—455).—Miscible liquids, e.g., milk samples, may be quickly and accurately mixed, and a definite quantity delivered, without the use of pipettes, by using an apparatus consisting essentially of two cylinders, one graduated for measuring and the other for mixing, and both fixed to a stand. They are connected at their upper ends through a four-way stopcock (which in one position connects the measuring chamber and suction pump and at the same time allows air to enter the mixing cylinder, while when turned through 90° the connexions are reversed) to a device for creating partial vacuum, and at their lower ends by a glass tube. At the lower end of the mixing chamber is a stopcock so made that when closed a measured quantity (11 c.c.) flows into the narrower end, and when opened into a butyrometer, and at the same time the residue flows out through the side tube. By suitable manipulation the samples are successively drawn into the measuring chamber, the mixture is transferred to the mixing chamber, and air bubbled through. The 11 c.c. and residue of the composite sample leave the stopcock at the same time.

D. G. HEWER.

Nutritive value of synthetic fats containing fatty acids of an odd number of carbon atoms. J. OZAKI (Proc. Imp. Acad. Tokyo, 1926, 2, 12—14).—Feeding experiments with rats, in which the fat was supplied in the form of the pure glycerides of fatty acids with odd numbers of carbon atoms up to 17, prove that such fats have a nutritive value equal to or greater than that of natural fats and in general that this value tends to diminish as the acid involved increases in mol. wt. A definitely noxious effect was, however, observed with propionin (glyceryl tripropionate), undecylein, and isovalerin, the last being particularly marked in its effects. Saturation of undecylein to undecylin rendered it nutritious.

G. M. BENNETT.

Composition and maturity of maize. T. H. HOPPER (N. Dakota Agric. Exp. Sta. Bull., 192, 1925).—The percentages of the following constituents increased with maturity—ear: dry matter, ether extract, nitrogen-free extract, also ash, protein, and fibre in the dry matter; stover: dry matter, also ash, and fibre in the dry matter; fodder: dry matter, ether extract, nitrogen-free extract. In the fodder the ash, protein, and fibre decreased, whilst in the stover the ash, protein, and nitrogen-free extract in the dry matter remained unchanged. The chemical evidence indicates that the maize plant is mature at the glazed stage.

A. A. ELDRIDGE.

Determination of acid-hydrolysable carbohydrates in green plant tissue. V. H. MORRIS and F. A. WELTON (J. Agric. Res., 1926, 33, 195—199).—With samples of green plant tissue preserved in alcohol, it is unnecessary to clear, or even neutralise, the hydrolysed solution before determining the reducing power. The only disadvantage of omitting clearing is that the precipitate of cuprous oxide is liable to be very finely divided; the use of a centrifuge is recommended.

C. T. GIMINGHAM.

See also A., Oct., 1065, Preservation of vitamin-C in dried orange juice (HUMPHREY).

Pectin. NANJI and NORMAN.—See XX.

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

Pectin: Micro-method for the determination of methyl alcohol, and its application to the study of the conditions governing the de-esterification of pectinogen. D. R. NANJI and A. G. NORMAN (J.C.S.I., 1926, 45, 337—340 T).—Methyl alcohol in dilute solution is oxidised completely to carbon dioxide and water by boiling with alkaline permanganate solution for 3 min. under reflux. Oxalic acid is added, followed by sulphuric acid while hot, and the mixture titrated back with *N*/20-permanganate solution. The alcohol can be removed quantitatively from dilute solution by a single distillation, with certain precautions. As other volatile substances are absent from pectinogen and de-esterified pectinogen solutions, the alcohol can be determined directly in the distillates. The total methoxyl content of pectinogen is determined by treating it overnight in a closed vessel with *N*/20-sodium hydroxide solution, adding sulphuric acid to give p_H 4.5, and determining the alcohol directly in the distillate. In considering the results, it is essential to determine the yield of calcium pectate from pectinogen, as unesterified uronic acid complexes are often present to the extent of as much as 30%. The methoxyl contents hitherto recorded have been uniformly too low, as the presence of such impurities has been overlooked. The methoxyl content of a pure pectinogen preparation (11.76%) is found to agree closely with that required for a tetramethyl ester of pectic acid. Tri-, di-, and mono-methyl esters exist having methoxyl contents of 8.94, 6.04, and 3.06% respectively. The degree of de-esterification of the pectinogen molecule is shown to be the most important factor in jellying phenomena, satisfactory jellies being only given by the tetra- and tri-methyl esters. All those conditions likely to produce de-esterification above a certain degree must therefore be avoided during the manufacture of pectin substances, jams, etc. Results are given showing the degree of de-esterification produced under various conditions.

Treatment of sheep-rot virus with aldehydes. E. DUCLOUX and G. CORDIER (Compt. rend., 1926, 183, 486—488).—Vaccines prepared by attenuation of the virus of sheep-rot by addition of fatty aldehydes confer immunity on treated sheep and are safe in use. Preparations from rot pustules, to which formaldehyde was added at the rate of 2.5 pts. per 1000 or less, when injected subcutaneously (1 c.c. per animal) caused little or no reaction and protected against subsequent injection, 15 or 16 days later, of virulent lymph. Similar results were obtained with acetaldehyde and butaldehyde. The vaccines so prepared kept well.

C. T. GIMINGHAM.

Essential oils of Sardinian aromatic plants.
III. Essence of *Thymus herba barona* and of *Thymus capitatus*. E. PUXEDDU (Annali Chim. Appl.,

1926, 16, 323—332; cf. B., 1925, 690).—The following data have been determined, the first values being those of the original oil, the values in brackets those of the oil re-distilled in steam. *Thymus herba barona* gives 1—1.36% of oil, red (yellow), d_{20}^{18} 0.9304 (0.9232), $[\alpha]_D^{18}$ (—4.83°), n_D^{20} 1.4925 (1.4970), solubility in 80% alcohol 1 : 1.5 (1 : 1.4), acid value 7.35 (9.90), ester value 3.80 (6.00), saponification value 11.22 (15.90), after acetylation 183.46 (187.26); phenol content 60% (mainly carvacrol). *Thymus capitatus* gives 0.3—0.6 of oil, d_{20}^{13} 0.9385, $[\alpha]_D^{13}$ —1.6°, n_D^{13} 1.5030, solubility in 80% alcohol 1 : 2, acid value 10.57, ester value 1.28, phenols 66%, these values referring to the oil after distillation in steam.
E. W. WIGNALL.

See also A., Oct., 1012, Catalytic oxidation of methane (MEDVEEV). 1042, Identity of uncineol with eudesmol (PENFOLD). 1049, Determination of methoxyl (NIERENSTEIN). 1062, Isolation of insulin (FUNK). 1063, Crystalline insulin (ABEL). 1064, Ovarian hormone (RALLS and others, also HARTMANN and ISLER.).

Aktivin in analysis. JANGMICHL and HACKL.—See VI.

PATENTS.

Process of combining ethylene with sulphuric acid. J. N. COMPTON, Assr. to CARBIDE AND CARBON CHEMICALS CORP. (U.S.P. 1,598,560, 31.8.26. Appl., 29.5.23).—Ethylene is absorbed in a bath containing 20—90 mols. of ethylene per 100 mols. of sulphur trioxide. The composition of the bath is maintained by regulated additions of sulphuric acid. B. FULLMAN.

Dehydrating formic acid. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 250,199, 12.3.26. Conv., 6.4.25).—The acid is treated with an anhydrous compound capable of retaining water of crystallisation with great tenacity, in sufficient quantity to form a stable hydrate with the water present in the acid, e.g., with sufficient anhydrous copper sulphate or magnesium sulphate to yield a salt containing 1 mol. of water of crystallisation, after which the acid is recovered by distillation at the ordinary pressure or *in vacuo*, after filtering off the hydrated compound, if necessary.
L. A. COLES.

Making halohydrins. H. ESSEX and A. L. WARD, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,594,879, 3.8.26. Appl., 22.11.19; cf. U.S.P. 1,477,113, B., 1924, 200).—By leading chlorine gas into an agitated mixture of allyl chloride and water below 20°, two isomeric glycerol dichlorohydrins are formed in satisfactory yield. It is of advantage to neutralise from time to time the hydrochloric acid formed. When the water present has become saturated with the dichlorohydrins these separate and may be recovered directly. On heating with sodium carbonate solution they afford glycerol in over 90% yield.
T. S. WHEELER.

2 : 4-Dihydroxydiphenylethane. E. KLARMANN, Assr. to LEHN & FINK, INC. (U.S.P. 1,596,613, 17.8.26. Appl., 15.12.25).—Resorcinol and phenylacetonitrile are condensed in dry ether by the action of hydrogen

chloride in presence of zinc chloride to 2 : 4-dihydroxyphenyl benzyl ketimine hydrochloride,



which on boiling with water gives 2 : 4-dihydroxyphenyl benzyl ketone, m.p. 121°, and this on reduction with zinc and hydrochloric acid yields 2 : 4-dihydroxydiphenylethane, m.p. 131°. It is a valuable non-toxic internal antiseptic especially as regards *B. typhosus*.

T. S. WHEELER.

Manufacture of compounds from 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone and halogenated alcohols or their esters with carbamic acid. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 257,816, 1.2.26).—Halogenated aliphatic monohydric alcohols and their carbamates react with equimolar proportions of 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone, on fusion or on mixing solutions of the components, yielding compounds which are analgesics. Trichlorobutyl alcohol yields a compound of m.p. 61—63°, and trichloroethyl carbamate a compound of m.p. 75—76°.
B. FULLMAN.

Manufacture of colourless products of [the reaction of] dialkylbarbituric acids with 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 255,434, 26.6.26. Conv., 16.7.25).—The yellow colour of the products obtained on melting together at 110—115° 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone with *CC*-dialkylbarbituric acids (such as diethyl- or isopropylallyl-barbituric acid) is due to atmospheric oxidation of the pyrazolone. White products are obtained by carrying out the melting and subsequent cooling in presence of an inert gas, such as carbon dioxide, hydrogen, nitrogen, etc.
B. FULLMAN.

Pharmaceutical product [analgesic]. W. SCHULEMANN and K. MEISENBURG, Assrs. to WINTHROP CHEM. Co. (U.S.P. 1,596,769, 17.8.26. Appl., 15.5.25).— Δ^1 -cycloHexenylethylbarbituric acid and 4-dimethylamino-1-phenyl-2 : 3-dimethyl-5-pyrazolone when mixed in ether or melted together, form a compound, m.p. 134—135°, which has strong analgesic and soporific properties.
T. S. WHEELER.

Preparation of substituted allophanic alkyl esters. E. MERCK, Asses. of C. DIEHL (G.P. 427,417, 14.6.24).—Esters of the type : $NHR_1 \cdot C(X) : N(R) \cdot CO_2 \cdot \text{alkyl}$ (R = H or alkyl; R_1 = alkyl or aryl; X = O or S), are prepared by the action of the corresponding carbamides or thiocarbamides on neutral alkyl carbonates and sodium alkoxide, under slight pressure if necessary. For example, monomethylcarbamide is heated with diethyl carbonate and sodium ethoxide for 2 hrs. under pressure at 80°, the alcohol distilled off, the residue dissolved in water and acidified faintly with mineral acid, when ethyl N : ω -methylallophanate, m.p. 136—137°, separates in a pure state. The following esters are prepared similarly from the appropriate components: methyl N : ω -methylallophanate, m.p. 163°; ethyl N : ω -dimethylallophanate, m.p. 77—80°; ethyl N : ω -allylallophanate, m.p. 69—70°; ethyl N : ω -phenylallophanate, m.p. 105°; ethyl N : ω -allylthioallophanate, m.p. 47—49°. A. DAVIDSON.

Production of tetrazoles. K. F. SCHMIDT (E.P. 257,418, 21.9.25. Addn. to 252,460; cf. U.S.P. 1,564,631, B., 1926, 216).—Carbonyl compounds, when treated with 2 mols. of hydrazoic acid in the presence of concentrated mineral acid, yield tetrazoles; if 3 mols. of hydrazoic acid are used, C-aminotetrazoles are formed. For example, cyclohexanone with 2.5 mols. of hydrazoic acid yields 70% of tetrazoles, including (?) 1:5-pentamethylene-1:2:3:4-tetrazole, acetone with 2 mols. of hydrazoic acid yields 1:5-dimethyl-1:2:3:4-tetrazole, m.p. 71°; and benzophenone with 3 mols. of hydrazoic acid yields 5-phenylamino-1-phenyl-1:2:3:4-tetrazole, identical with phenyliminophenyltetrazolone (of A., 1900, i., 415).

B. FULLMAN.

Arsenic compounds [arsenoxides] of the aromatic series. A. J. RANSFORD. From L. CASSELLA & Co., G.M.B.H. (E.P. 257,361, 9.6.25).—1:2-Dihydrobenzoxazolone arsenoxides, having therapeutic value in spirochaetal infections, are obtained by reducing the corresponding arsenic acids with hydriodic acid and sulphur dioxide. The following are described: 1:2-dihydrobenzoxazolone-5-arsenoxide; 4-methyl-1:2-dihydrobenzoxazolone-5-arsenoxide; 1:2-dihydrobenzoxazolone-4-arsenoxide; and 6-chloro-1:2-dihydrobenzoxazolone-4-arsenoxide.

B. FULLMAN.

Production of 3-chloro-5-amino-4-hydroxyphenyl-1-arsinic acid. I. G. FARBENIND. A.-G., Asses. of L. BENDA and W. SCHMIDT (U.S.P. 1,595,498, 10.8.26. Appl., 23.12.25. Conv., 2.3.25).—3-Chloro-4-hydroxyphenylarsinic acid is nitrated to the 5-nitro-compound, which is reduced and treated with acetic anhydride to yield the acetamido-derivative, and this, on hydrolysis with 16% aqueous caustic soda at 100°, gives 3-chloro-5-amino-4-hydroxyphenylarsinic acid, which is of therapeutic value and is unaffected by light.

T. S. WHEELER.

Process of making camphor. H. D. GIBBS and A. W. FRANCIS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,597,877, 31.8.26. Appl., 23.11.20).—A mixture containing 1 g. of isoborneol (as vapour) per 0.5–10 litres of air (measured at 20° and 760 mm.) is subjected to the action of a catalyst kept at 200–600°

B. FULLMAN.

Manufacture of esters of borneol and isoborneol. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (E.P. 250,551 and 257,275, 12.3.26. Conv., 11.4.25).—(A) Camphene and a fatty acid are heated together at atmospheric pressure, first at about 100°, rising to about 150°, the formation of esters of borneol and isoborneol occurring quickly and completely. Especially when the acid is anhydrous esters of high m.p. are obtained in improved yield. For example, when 136 pts. of camphene are heated with 58 pts. of anhydrous formic acid, the reaction product contains 98% of ester. (B) If a considerable excess of fatty acid is used the reaction takes place at the ordinary temperatures.

B. FULLMAN.

Manufacture of synthetic camphor [conversion

of bornyl chloride into camphene]. G. H. DUPONT and G. BRUS (E.P. 248,404, 2.3.26. Conv., 2.3.25).—Crude or pure bornyl chloride (pinene hydrochloride) is converted into camphene by treatment with metal resinates dissolved in excess of resin, at about 180°, a slightly volatile solvent, e.g., resin oil, paraffin, etc., being added if necessary. The camphene is removed by steam-distillation, and separated from bornyl chloride by vacuum distillation. The residual resinic acid, if necessary after washing out the metal chloride formed, may be used again, or in the preparation of oil of resin, resin soap, etc. All the operations may be effected in the same vat provided with an agitator and reflux condenser. For example, 43 g. of dry sodium carbonate are added to 604 g. of colophony at 180–200°, 65 g. of bornyl chloride are added, and the mixture is heated for 2–3 hrs. at 180° under reflux, yielding finally 80% of camphene. Instead of pure bornyl chloride, the crude reaction product of pinene and hydrogen chloride may be used.

B. FULLMAN.

Extraction of the active biological principles from the ether extract of male fern. F. FUMAROLA (E.P. 256,768, 17.7.25).—A mixture of 1 pt. by weight of the ether extract with, e.g., 4 pts. of magnesium oxide is digested with about 6 times its weight of distilled water at 40°, and filtered after about 5 hrs. The solution is acidified with dilute hydrochloric acid, and the precipitate is removed, dried below 40°, and treated for a few days with a mixture of, e.g., 60 pts. of carbon tetrachloride and 40 pts. of ether, after which the residual silicic acid is filtered off, washed with a similar mixture until the wash liquor is colourless, dried at 40–80°, and desiccated on glass plates.

L. A. COLES.

Preparation of colloidal water-soluble bismuth for injection purposes. E. MERCK (G.P. 427,669, 27.10.22).—An alkaline bismuth solution is reduced, in presence of protective colloids, with sodium hyposulphite, air being excluded and a high temperature avoided. The colloid is precipitated by alcohol or a similar precipitant, and is carefully dried. For example, potassium sodium tartrate is dissolved in water and sodium hydroxide, and, while warming, basic bismuth nitrate is added gradually. To the solution, diluted with water, is added a 9% gum solution, sodium hydroxide, and an aqueous solution of sodium hyposulphite. After shaking for 1 hr. in a closed vessel a sol is obtained which is stable for a day in air. To obtain the gel, 87% alcohol is added to the solution, the precipitate washed four times with 58% alcohol, and dried by spreading on glass plates. The water-soluble gel contains about 40% of bismuth and 44% of organic matter. On injection, the product has no corrosive action on living tissue.

A. DAVIDSON.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Relation between intermittent and non-intermittent sector-wheel photographic exposures. R. DAVIS (U.S. Bur. of Standards, Sci. Papers No. 528,

1926, 21, 95—139).—The difference between intermittent and equal non-intermittent exposures varies with the emulsion used, the illumination, the number of interruptions, and the rest periods between. When the illumination intensity is above a certain level, a greater effect results from the intermittent exposure. For lower intensities a loss is obtained. The gain or loss is accentuated by increased intermittence. The results were confirmed for six different types of emulsion. In explanation it is suggested that after extinguishing the illumination the latent image is subject to modification by two opposing influences, one tending to intensify it, the other to fade it; both decrease rapidly with time. At higher illumination values the resultant effect is a growth in density; at lower values, fading results. It is suggested that the reaction between light and the silver halide is not a single step, but that an intermediate condition exists between the first effects of light and the latent image proper. The first product of the reaction is in a nascent form, which, under the continued action of light, completes the reaction and forms the latent image. If the light is extinguished during this process, the nascent product, in the dark, partly returns to its former state, and partly completes the reaction, the degree of each depending on the illumination used.

W. CLARK.

Mordanting of silver images by cupric thiocyanate. A. and L. LUMIÈRE and A. SEYEWETZ (Bull. Soc. chim., 1926, [iv], 39, 1246—1249).—In the Christensen mordanting bath containing copper sulphate, alkali thiocyanate, potassium citrate, and acetic acid, the active agent is cupric thiocyanate, which acts as well in simple aqueous solution as in presence of potassium citrate and acetic acid. The addition of these two substances increases the solubility of the cupric salt and the stability of the solutions. The white precipitate which slowly forms in the mordanting solution is cuprous thiocyanate. The mordanted image appears to consist of a complex of cuprous and silver² thiocyanates. The composition is variable, but in general the substance contains 1 mol. of copper salt to 2 mols. of silver salt. Finely divided free silver, in the absence of gelatin, does not fix copper when shaken in the mordant bath.

W. CLARK.

Recovery of silver from exhausted fixing baths. J. I. CRABTREE and J. F. ROSS (Brit. J. Phot., 1926, 73, 522—526).—The recovery of silver on a large scale from exhausted fixing baths by means of sulphide, zinc, sodium hyposulphite, and electrolytic methods is discussed. For large-scale use, precipitation with sodium sulphide is the most economical method. Precipitation with zinc dust is less rapid, but it is efficient and has the advantage that no hydrogen sulphide is formed. Although restoration of a fixing bath by de-silvering is possible, it is just as economical, and far preferable, to use a fresh bath. To revive a bath after de-silvering, it is necessary to add a further amount of hardener equal to that originally present, and also a quantity of thio-sulphate equal to 50% of the original amount used. The resulting bath has approximately three-quarters the life of a fresh bath.

W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Metalamines and hydrazinates of the bivalent heavy metal chlorates and perchlorates as primary explosives.—W. F. FRIEDERICH and P. VERVOORST (Z. ges. Schiess- u. Sprengstoffw., 1926, 21, 49—52, 65—69, 84—97, 103—105, 123—125, 143—146).—The metalamines of the chlorates and perchlorates of copper, cadmium, cobalt, zinc, and nickel are sensitive explosives, but only tetramminocupric chloride is suitable for use as a primary explosive. Their energy contents and detonation velocities are intermediate between those of mercury fulminate or lead azide and those of tetranitroaniline, T.N.T., or tetryl. The ammine chlorate compounds are considerably more sensitive than the corresponding perchlorate compounds, but cannot as a rule be used in technology as they lose ammonia rapidly and hydrolyse in damp air. The metalamines were prepared by saturating the cooled alcoholic solution of the metal chlorate or perchlorate with gaseous ammonia. The hydrazinates were obtained by cooling the aqueous or alcoholic solutions of the chlorate or perchlorate of the metal, and adding an aqueous or alcoholic solution of hydrazine hydrate with stirring, when the hydrazinate was precipitated. The *hydrazinates* of nickel chlorate and perchlorate and of cadmium perchlorate have not hitherto been prepared. The formulæ assigned to them are $\text{Ni}(\text{ClO}_3)_2 \cdot 3\text{N}_2\text{H}_4$, $\text{Ni}(\text{ClO}_4)_2 \cdot \text{NiClO}_4 \cdot \text{OH} \cdot 5\text{N}_2\text{H}_4 \cdot 3\text{H}_2\text{O}$, and $\text{Cd}(\text{ClO}_4)_2 \cdot \text{Cd}(\text{OH})_2 \cdot 3\text{N}_2\text{H}_4 \cdot 2\text{H}_2\text{O}$ respectively. The hydrazinates of the chlorates are very sensitive primary explosives, and surpass mercury fulminate and lead azide in brisance. The explosion points of some are below 100°. The perchlorate hydrazinates are less sensitive. The explosive properties of all metalamines and hydrazinates examined are summarised in a table which gives their formulæ, explosion points, sensitiveness to shock in the fall-hammer test, Trauzl lead block values, and ability to initiate explosion in tetryl, T.N.T., and trinitroanisole.

S. BINNING.

Apparatus for routine testing of the stability of a smokeless powder by determining the loss in weight [at 115°]. J. VON MEERSCHERDT-HÜLLESSEM (Z. ges. Schiess- u. Sprengstoffw., 1926, 21, 137—141).—In testing the stability of a smokeless powder the hot-storage test and the method in which the loss of weight of the powder when heated at a definite temperature is determined give more reliable results than any other. The loss-of-weight method has the disadvantage that it requires considerable time, and is therefore impracticable when many samples require to be tested. An apparatus, drawings of which are given, has therefore been designed that enables 37 samples to be tested in 8 days of 12 hrs., or if a duplicate apparatus is available 74 samples in 16 days of 6 hrs. It consists of a bath which supports a frame capable of taking 38 test-tubes, which are immersed in the heating medium consisting of an aqueous glycerin solution boiling at 115°, the temperature chosen for the test. The apparatus also includes means for raising the frame with its test-tubes out of the bath, and lowering it into a box where the samples cool to room temperature in an atmosphere free from moisture, after which the loss of weight is determined.

S. BINNING.

PATENT.

Stabiliser for nitrated organic compounds [explosives]. W. A. PATRICK, Assr. to SILICA GEL CORP. (U.S.P. 1,596,622, 17.8.26. Appl., 18.2.21).—Silica gel of the type described in U.S.P. 1,297,724 (B., 1919, 363 A), when mixed with organic nitro-compounds which are used as explosives, *e.g.*, nitroglycerin or nitro-cellulose, rapidly absorbs any oxides of nitrogen formed and stabilises the explosive more efficiently than the chemical substances hitherto employed. Its use in dynamite renders unnecessary the inclusion of anhydrous copper sulphate hitherto employed as an absorbent for moisture.

T. S. WHEELER.

XXIII.—SANITATION; WATER PURIFICATION.

Chemical and bacteriological examination of the London waters for the twelve months ended March 31, 1926. A. C. HOUSTON (Metropolitan Water Board, 29th Ann. Rept., 1926, 118 pp.; cf. B., 1925, 145, 939).—Chlorination of Thames river water has been continued throughout the year with marked success, some 24,540 million gallons of raw water being treated with 56.21 and 23.26 tons of bleaching powder and liquid chlorine respectively; treatment of the raw water in this manner, and allowing it to flow by gravity to the filters instead of first pumping it into the Staines reservoirs, has resulted in a net saving of £10,056. The average dose of chlorine applied was 0.4 p.p.m., and the average cost works out at 9.8d. per million gals. Of the samples taken of the chlorinated water 27.4% contained *B. coli* in 10 c.c., as compared with 41.2% of the samples taken of the Staines stored water during the period 1908—16. Chlorine was applied to New River water on 137 days during the winter months, 4490.7 million gals. of water being treated with an average dose of 0.283 p.p.m. of chlorine at an average cost of 6.5d. per million gals. treated. Permanganate, average dose 0.205 p.p.m., was added on 103 days at a cost of 11.8d. per million gals., and on 34 days ammonium chloride, instead of permanganate, was used as taste preventer. In December taste troubles, lasting for one or two days only, were encountered for the first time since chlorination was commenced in 1919. When the taste was noticed the dose of chlorine was reduced and, as an extra precaution, for a period of several days the dose of permanganate was doubled and ammonia added as well. For a period of 4 months, with intermissions, permanganate and ammonia treatments were alternated, with the object of comparing the two as regards sterilisation effect. The dose of permanganate applied was 0.2 p.p.m. and that of ammonia 0.2 p.p.m. in terms of nitrogen. The results were slightly in favour of chlorine plus ammonia. Other experiments, in which ammonia was added to water prior to chlorination, showed that the addition of ammonia increases the bactericidal effect of the chlorine, but more time is required to obtain the full sterilising effect. "Dichloramine" methods of treatment, consisting of the addition of a "mixture" of chlorine and ammonia to water in bulk, gave encouraging results. *Leptospiras* were found to be present in all raw sources of supply, and also in many filtered waters and pure deep-well waters. All the cultures made were

proved to be non-pathogenic, except in one instance (Deptford [Garden] Well), and this supply has since been chlorinated as a precautionary measure. The doses of chlorine used for the destruction of *B. coli* more than suffice for the death of *Leptospiras*. The experiments at Barn Elms, in which water is treated by rapid filtration methods prior to treatment on slow sand filters, have demonstrated that water so pretreated can be satisfactorily dealt with by slow sand filters at from 3 to 4 times the normal rate.

W. T. LOCKETT.

Determination of organic matter in water by means of potassium dichromate and sulphuric acid. W. E. ADENEY and (Miss) B. B. DAWSON (Sci. Proc. Roy. Dublin Soc., 1926, 18, 199—202).—The water (50 c.c.) is concentrated to 5 c.c. with a little sulphuric acid to remove chlorides, mixed with sulphuric acid (50 c.c., 50% by vol.) and 0.25*N*-potassium dichromate (50 c.c.), heated at 100—110° for 2 hrs., and the excess of oxidant then titrated with standard ferrous sulphate. The organic matter is oxidised almost completely to carbon dioxide, water, and ammonia.

G. M. BENNETT.

Rate and mechanism of aeration of water under open-air conditions. W. E. ADENEY (Sci. Proc. Roy. Dublin Soc., 1926, 18, 211—217; cf. A., 1920, ii, 686; B., 1923, 781 A).—Further experiments confirm the views already published.

G. M. BENNETT.

Determination of carbon monoxide in the air of workshops. F. SCHOofs (J. State Med., 1926, 34, 575—577).—During the removal of oxygen by pyrogallol, a considerable excess of alkali must be present to prevent the formation of carbon monoxide. (Cf. Drakeley and Nicol, B., 1925, 944.)

F. R. ENNOS.

Bactericidal action of cadmium compounds. E. A. COOPER and (Miss) L. I. ROBINSON (J.S.C.I., 1926, 45, 321—323 T).—Cadmium compounds are active germicides, being more efficacious than the salts of many metals, with the exception of silver and mercury. Cadmium salts are much more powerful disinfectants than the corresponding zinc compounds, and approach mercuric salts in germicidal power, although chemically cadmium is more closely allied to zinc. Organic derivatives of cadmium tend to be less effective as germicides than the inorganic salts. Solutions of cadmium hydroxide in ethylenediamine, however, exert a strong bactericidal action.

Insecticidal value of certain chemicals. BRINLEY.—See XVI.

PATENTS.

Preparation of chlorine water [for sterilising sewage]. I. G. FARBENIND. A.-G., Asses. of P. SIEDLER and W. MOSCHEL (G.P. 429,037, 13.7.22).—The chlorine water is made in an apparatus arranged to permit the flow of water to be kept continuously in contact with pure chlorine under slight pressure.

W. G. CAREY.

Insecticides. E. C. R. MARKS. From RUBBER SERVICE LABORATORIES Co. (E.P. 257,644, 29.4.25).—See U.S.P. 1,573,490; B., 1926, 379.

Insecticidal and fungicidal media (E.P. 257,171).—See XVI.