# BRITISH CHEMICAL ABSTRACTS

# B.—APPLIED CHEMISTRY

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JUNE 8, 1928.

# I.--GENERAL ; PLANT ; MACHINERY.

Centrifugal mixers. E. BUHTZ (Chem. Fabr., 1928, 211-213).-Several types of mixers are described, the essential feature being that the two liquids, or the liquid and solid, to be mixed are fed continuously on to a rapidly rotating dish, over the edge of which the finished mixture passes. Provision is made for heating the dish and inlet pipe, for removal of gases evolved, etc. as necessary. Their use is suggested for the acid treatment of crude oils, the salting out of soaps, and the refining of fats. In some cases spiral baffles are provided. Another type of apparatus has an upper rotating plate and a lower funnel-shaped dish with common axes revolving in opposite directions. This is intended for the mixing of colours, moistening of powders, etc. C. IRWIN.

Instrument to record the carbon dioxide content of a gaseous mixture. R. GORDON and J. F. LEHMANN (J. Sci. Instr., 1928, 5, 123-126).-An instrument is described the action of which depends on the variation in electrical conductivity of a solution of barium bicarbonate in equilibrium with a saturated solution of barium carbonate. The readings are independent of the other constituents of the gaseous mixture, which is passed through the solution. C. W. GIBBY.

Value of aluminium and its alloys in chemical and allied industries. H. BUSCHLINGER (Chem. Fabr., 1928, 209-211) .- The corrosion of aluminium, as of other metals, is affected by crystalline structure and surface quality, the absence of inclusions (e.g., Al<sub>3</sub>Fe), and particularly by the quality of the water used if aqueous solutions are being handled. The possibility of the application of aluminium to plant used in tar distillation is discussed. The penetration of the protective surface layer of oxide is often the decisive factor in starting corrosion. Large aluminium vessels should on structural grounds be cylindrical rather than hemispherical. The rapid diminution in strength with rise of temperature is pointed out. Whilst the walls of vessels to withstand pressure are constructed on a theoretical basis, the minor parts are usually designed according to empirical rules. In jacketed vessels the steam inlet must be designed to minimise mechanical wear. In all cases corners and angles should be avoided to facilitate cleaning. In the case of aluminium vessels which are to contain edible preparations, rollers etc. should be used exclusively for aluminium, and the sheets should be pressed so as to give a smooth surface capable of polishing both inside and out. Suitable packing material, quite free from acid, should be used.

C. IRWIN.

Strength of curved walls exposed to external pressure. C. A. ANDSTEN (Ind. Eng. Chem., 1928, 20, 364-366).-Equations derived from experimental work

by Bach on the collapsing pressures of spheres and cylinders under external pressure are plotted graphically. A separate curve is obtained for each metal which may be used, and by using Bach's constants the necessary data are obtained by which the ratio of thickness to diameter is correlated with the pressure to be used.

- BULL ; GAS ; TAR ; MINTRA

C. IRWIN.

Gas cleaners. HARBORD.-See X. Glass extractor. FREY and REED,-See XV. Flammability of refrigerants. JONES .- See XXII.

#### PATENTS.

Rotary furnaces. GEWERKSCHAFT SACHTLEBEN, and J. KÜPPERS (B.P. 286,590, 30.9.27. Ger., 5.3.27. Addn. to B.P. 268,308; B., 1927, 927) .- The inner lining of the muffle furnace consists of fireproof material of high thermal conductivity, whilst that of the outer muffle is of low conductivity. H. ROYAL-DAWSON.

Apparatus for carrying out exothermic catalytic gas reactions. H. HARTER (B.P. 275,983, 8.8.27. Ger., 10.8.26).-The catalytic tube is divided transversely into a number of separate chambers, through each of which a corresponding fraction of the gas current passes from a common conduit, and the reaction products and uncombined gases are withdrawn through a common collecting conduit. The reaction is thus distributed as uniformly as possible over the whole catalyst and local heating is avoided. The collecting conduit may surround the contact chambers in the form of a cylinder, tube, cooler, or heat exchanger. A. B. MANNING.

Temperature-regulating system. C. E. ANDREWS, Assr. to Selden Co. (U.S.P. 1,666,251, 17.4.28. Appl., 19.2.27).—The b.p. of a two-phase, liquid-vapour, temperature-regulating system which includes reflux condensation of the vapour, and in which the liquid contains a number of components of which at least one is normally solid, is regulated by varying the composition of the liquid condensed in the reflux apparatus before it is returned to the main body of the liquid.

L. A. Coles.

Production of emulsions. C. A. AGTHE (U.S.P. 1,668,136, 1.5.28. Appl., 12.3.23. Ger., 13.3.22).-See B.P. 210,307; B., 1924, 239.

Apparatus for comparing the colour of liquids by reflected and transmitted light. H. A. ELLIS, Assr. to BRIT. DRUG HOUSES, LTD. (U.S.P. 1,667,249, 24.4.28. Appl., 25.2.25. U.K., 12.11.24).-See B.P. 235,458; B., 1925, 616.

Measuring the density of gas. G. KÖNIG (U.S.P. 1,664,752, 3.4.28. Appl., 3.7.24. Ger., 6.3.20).-See B.P. 159,845; B., 1922, 692 A.

Friction surfaces (B.P. 264,471) .- See X. Gastesting apparatus (B.P. 288,908) .- See XI.

# II.—FUEL; GAS; TAR; MINERAL OILS.

Coke for crucible steel melting. R. V. WHEELER (Fuel, 1928, 7, 148-151).-The preference shown by crucible steel workers for beehive over by-product coke is due to the readiness with which the former can be broken into pieces of suitable size without undue formation of breeze, and the fact that it burns uniformly at an economical rate and that the clinker formed does not stick to the furnace lining. These advantages arise from the absence of cross-fractures and the uniform cell-structure in beehive coke, combined with possession of the requisite "combustibility value." A special coke produced in a by-product oven from a blend of coking slack and anthracite duff behaved satisfactorily in trial melts as regards manner of burning, but was difficult to break into suitable pieces and gave rather a high proportion of breeze. A. B. MANNING.

Reactivity of coke. J. H. JONES, J. G. KING, and F. S. SINNATT (Iron and Steel Inst., May, 1928. Advance copy. 20 pp.; cf. B., 1928, 146).-A comparison of the reactivities of a number of metallurgical cokes has shown that the cokes from Yorkshire coals are distinct from those of South Wales or Durham coals, and possess a higher reactivity. The reactivity curves for the South Wales and Durham cokes are similar, the latter, however, giving slightly higher average values than the former. The differences are probably due to differences in the character of the coals used, and there is some indication that the ash is an important factor. Attempts to correlate reactivity values of metallurgical cokes with the results of "shatter" tests have met with no A. B. MANNING. success.

Reactivity of coke and a new method of determining it. R. A. DENGG (Polytechn. Weekblad, 1926, 20, 246; Fuel, 1928, 7, 152-154).-Three cokes were prepared in the laboratory by carbonising an anthracite, a coking coal, and a gas coal, respectively, at 950°. Their relative "reactivities" were determined by passing air at a constant rate (5 litres/hr.) over 0.2 g. of the coke, of uniform size, placed in a boat in the tube of a furnace maintained at a constant temperature, and measuring the amount of carbon dioxide produced. This was done by observing the variation in electrical conductivity of a solution of barium hydroxide through which the issuing gas was passed. The coke from the anthracite had the least reactivity, and that from the gas coal the greatest. The distinction between "reactivity" and "combustibility" is emphasised. A. B. MANNING.

Specific heats of amorphous carbon and semicokes. E. TERRES and H. BIEDERBECK (Gas- u. Wasserfach, 1928, 71, 265—268, 297—303, 320—325, 338—345). —Three samples of amorphous carbon have been prepared, (a) by the action of sodium on barium carbonate at 550°, (b) by the action of chlorine on turpentine below 600°, and (c) by the catalytic decomposition of methane below 600°, and determinations made of the mean sp. heat of each sample, over temperature ranges from 20—300° to 20—1200°. The method used is described in detail (cf. Terres and Schaller, B., 1923, 214 A), all the precautions being taken necessary to ensure accuracy. The results for all three samples of

carbon were similar; the mean sp. heat rose rapidly with the temperature (i.e., the upper temperature of the range) up to about 600°, and thence only slightly to about 1150°, the value then agreeing with that of graphite. The values obtained at the lower temperatures were at first greater than the corresponding values for graphite, but after the carbon had been heated to 1200° the mean sp. heats re-determined at the lower temperatures approximated closely over the entire range to those of graphite. Semi-cokes were prepared from four coals at temperatures from 400° to 900°, and their sp. heats have been determined over ranges up to their respective temperatures of carbonisation. The sp. heat of each coke rises with the temperature, and for cokes from the same coal is higher the lower the temperature of carbonisation. The sp. heat of the coke-substance of all the semi-cokes investigated over temperature ranges from 550° to 900° was constant and equal to  $0.3825\pm$ 0.0025. Below 550° the sp. heat is higher for semi-cokes from gas coals than from other coals.

#### A. B. MANNING.

Correlation of Explosions in closed vessels. pressure development with flame movement. O. C. DE C. ELLIS and R. V. WHEELER (Fuel, 1928, 7, 169-178; cf. B., 1925, 388).-By recording on the same film the movement of the flame produced when mixtures of carbon monoxide and air are ignited centrally in a spherical vessel and the reading of the pressure gauge, it has been shown that, except for mixtures in which the flame front travels very slowly, the moment of maximum pressure coincides with the arrival of the flame front at the boundary of the vessel within less than half a millisecond. Similar records of explosions within a cubical vessel show that pressure continues to be developed slowly after the flame front has touched the walls of the cube and while it is squeezing itself into the corners. The moment of attainment of maximum pressure does not synchronise exactly with the moment of "complete inflammation" of the mixture, but is antecedent to it, probably owing to cooling of the gases at the faces of the cube before the flame front has arrived at the corners.

#### A. B. MANNING.

Limits of inflammability of gases and vapours. H. F. COWARD and G. W. JONES (U.S. Bur. Mines Bull. 279, 1928. 99 pp.).—All the available data in the literature relating to the limits of inflammability of mixtures of gases and vapours with air, oxygen, or other "atmospheres," and to the variation of the limits with the initial conditions of pressure, temperature, turbulence, humidity, etc., have been collected and co-ordinated.

A. B. MANNING.

Use of active charcoal in the gas industry. A. ENGELHARDT (Gas- u. Wasserfach, 1928, 71, 290—297; cf. B., 1922, 659 A).—The properties of active charcoal are discussed in relation to its use as an adsorbent or as a catalyst in the gas industry. Methods for the determination of benzol in coal gas, and details of the largescale plant for benzol recovery by absorption in active charcoal, are described. The benzol is now recovered from the charcoal and the latter regenerated by the use of wet instead of superheated steam, the charcoal being maintained at 100—120° by means of a heater within

the filter. Loss of material due to dust formation is small ; some loss of activity occurs due to the absorption of unsaturated hydrocarbons which polymerise, depositing resinous substances within the pores of the charcoal. Hydrogen sulphide is removed from coal-gas or water-gas by catalytic oxidation in the presence of active charcoal. Small quantities of air (3-4%) and ammonia  $(0.3 \text{ g./m.}^3)$  are added to the gas, which is then passed through a charcoal filter. The sulphur formed is adsorbed by the charcoal, which is regenerated by extraction with ammonium sulphide followed by steaming. The polysulphide solution obtained is decomposed by heat and the sulphur recovered in a pure state. Details of the necessary plant are briefly described. A. B. MANNING.

Coke-oven and gas-works gas: a comparative study of gas quality. R. NÜBLING and R. MEZGER (Gas- u. Wasserfach, 1928, 71, 304—310).—The importance of establishing suitable standards of gas quality is emphasised, particularly in view of the recent developments in long-distance transmission. The standards of calorific value, density, sulphur content, etc. of the gas supplied to which the gas and coke-oven industries respectively conform are compared in detail. Those of the coke-oven industry are not sufficiently rigid to satisfy general industrial demands. A. B. MANNING.

Application of low-temperature carbonisation to gas-producer practice. S. UCHIDA (Fuel, 1928, 7, 179—185).—The results obtained by the carbonisation of Japanese coals, lignite, slurry, and an oil shale in a specially designed producer are summarised; tar and gas yields and analyses etc. are tabulated, and carbon, hydrogen, and thermal balances calculated. The plant has a capacity of 3 tons/24 hrs., and consists of a producer proper surmounted by a retort, in which the coal undergoes a preliminary carbonisation at about 500°. The plant operates smoothly provided that the coal used is not too strongly swelling. The tar yield from Bibai coal (moisture 3.74%, volatile matter 42.74%, "fixed carbon" 45.07%, ash 8.45%) was 10.6%, corresponding with 81.6% of that produced in the Fischer assay. A. B. MANNING.

K.S.G. low-temperature process. J. N. HAZELDON (Fuel, 1928, 7, 155-160).-The low-temperature carbonisation plant in operation near Essen is described. The retort comprises two concentric mild steel drums, the outer 72 ft. long and 9 ft. 9 in. in diam., the inner 84 ft. long and 5 ft. 7 in. in diam., which are inclined at a slight angle to the horizontal and are supported on rollers; in operation they are rotated at  $\frac{3}{4}$  r.p.m. Fine coal is fed continuously into the lower end of the inner drum and is carried by helical flanges to the upper end; it passes then into the annular space between the drums and, under the combined effect of gravity and the rotation, returns to the lower end and is discharged as coke. Carbonisation is effected by a combination of external and internal heating. The retorts are enclosed in heating chambers through which circulate hot gases from a combustion chamber below, and superheated steam is admitted into the annular space between the drums in such a way that it passes through the mass of the coal. The gases and vapours evolved pass through a dust catcher to a condensing system. After separation of the fines, which may amount to 20% and are utilised for blending purposes, the lump coke forms a suitable domestic fuel. It contains 10— 12% of volatile matter. A Welsh coal (moisture 3%, volatile matter 32.5%, ash 5%) yielded 15 cwt. of coke, 16.1 gals. of tar, 3500 cub. ft. of gas (850 B.Th.U./ cub. ft.), and 2 gals. of light spirit. The heat required for carbonisation of 1 lb. of coal is about 700 B.Th.U. A. B. MANNING.

Composition of tar from low-temperature carbonisation of Utah coal. I. R. L. BROWN and B. F. BRANTING (Ind. Eng. Chem., 1928, 20, 392-396).-Utah coal distilled in a continuous vertical retort with superheated steam at a maximum temperature of 700° yielded 9.6% of solid tar with air condensation and a further 3.9% of fluid tar in a water condenser. The aqueous condensate was neutral to litmus, but contained small quantities of tar acids (including phenol and the cresols), ammonia, pyridine bases, and carboxylic acids. The fluid tar ( $d^{20}$  0.9337) contained 23.4% of phenols, 66% of neutral oils, 1.7% of crude bases, and 0.25% of carboxylic acids. The phenols distilled over the range 180-205° and contained phenol, all the cresols, xylenols, and pyrocatechol. The neutral oil gave liquid distillate fractions varying from  $d \ 0.79$  to  $d \ 0.90$ , and higherboiling fractions containing large quantities of paraffin wax and "resins." C. IRWIN.

Report of test by the Director of Fuel Research on the Crozier retort installed by Mineral Oils Extraction, Ltd., at Wembley. ANON. (Dept. Sci. Ind. Res., Fuel Res. Bd., 1927. 21 pp.).-The retort is of cast iron, and consists of two parallel walls 1 ft. 3 in. apart joined by semi-circular ends; the height is 15 ft. and the greatest width 5 ft. At each of five levels are three inclined cross-flues passing from one flat side of the retort to the other. Offtakes, which are carried to the centre of the retort and are hooded, are situated at four different levels. The material to be carbonised is fed into two hoppers at the top of the retort, and after passing through is extracted into a coke chamber by means of a rotating helix. The retort is enclosed in a brickwork chamber divided into compartments through which pass heating gases supplied from a furnace burning either solid or gaseous fuel. After condensation of the tar and liquor the gas is oil-scrubbed to recover the light spirit therein, and the greater part of the scrubbed gas is returned to the furnace for heating the retort. The coal used for the test was a Scottish splint coal (Tullygarth), and the throughput was about 4 tons per day. Some difficulty was encountered owing to holding up of the charge in the retort. The temperature of carbonisation varied from about 800° at the base of the retort to about 280° at the top. The yields of products per ton of dry coal were: coke, 15.4 cwt.; gas, 7390 cub. ft. of 323 B.Th.U./cub. ft.; tar, 16.4 gals.; spirit (crude), 0.8 gal.; ammonium sulphate, 9.1 lb. The coke was not completely carbonised, the volatile matter being 14.6%. It gave a satisfactory, but not quite smokeless, fire in a domestic grate.

A. B. MANNING.

Phenols in petroleum distillates. LER. G. STORY and R. D. SNOW (Ind. Eng. Chem., 1928, 20, 359-364).—The disposal of phenols which gradually accumulate in the alkaline solutions used for petroleum washing presents some difficulty, especially with cracked distillates, where the quantity is larger. A sample of such a caustic soda solution was acidified and distilled, and the distillate again extracted with caustic soda, acidified, and the phenols obtained were refractionated. The b.p. of the product ranged from 192° to 299°. It contained small proportions of cresols. Tests for other specific compounds gave negative results. A phenol mixture of lower b.p. range was extracted from lowboiling cracked distillate. With straight-run distillates the yields of phenols were very low, but the cresols were again identified. The general character of these phenols is similar to that of the phenols found in lowtemperature tar. The quantity even in cracked distillates is usually less than 0.01%. C. IRWIN.

Carbon dioxide recorder. GORDON and LEHMANN. Aluminium vessels. BUSCHLINGER.—See I. Blastfurnace gas. HARBORD.—See X. Graphite for electrotype. WINKLER and BLUM.—See XI.

#### PATENTS.

Coking retort oven. H. KOPPERS, ASST. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,664,632, 3.4.28. Appl., 31.8.21. Ger., 7.11.19).—An inclined retort is provided with vertical heating flues, of which those arranged alongside the lower part of the retort have their lower ends lying in a line parallel to the retort, whilst those alongside the upper part have their lower ends horizontal. Each set of flues communicates with a corresponding regenerator below, that for the lower flues having its upper part arranged stepwise, each step being directly connected with a vertical flue. Above the flues and connected with them runs a waste-gas collecting channel, over the horizontal portion of which a charging platform is constructed. A. B. MANNING.

Coking retort oven. [A] N. G. PARRY, [B] J. VAN ACKEREN, Assrs. to KOPPERS Co. (U.S.P. 1,664,633 and 1,664,649, 3.4.28. Appl., [A, B], 6.9.21. [B] Renewed 29.1.26).—(A) A coke oven consists of a series of coking chambers with heating walls constituted of vertical combustion flues disposed in pairs. Vertical outflow channels in each heating wall are connected with a pair of combustion flues, the individual outflow channels of one heating wall being connected, respectively, with those of an adjacent heating wall by ducts beneath the chambers. Crosswise regenerators parallel with the coking chambers are divided into inflow and outflow operating pairs, each regenerator of each pair being connected with the combustion flues of a pair of adjacent heating walls. (B) The coking chambers are heated by vertical combustion flues, with each pair of which vertical outflow channels are connected. The outflow channels of alternate heating walls are connected with the corresponding outflow channels of intermediate heating walls by ducts beneath the chambers. Crosswise regenerators are disposed as in (A), both regenerators of each pair corresponding to and being connected with combustion flues of only a single heating wall.

A. B. MANNING.

Manufacture of fuel briquettes. A. L. STILLMAN, Assr. to GEN. FUEL BRIQUETTE CORP. (U.S.P. 1,664,998, 3.4.28. Appl., 21.1.27).—A mixture of bituminous coal and a bituminous binder is subjected to an intense grinding operation at a temperature above the fusion point of the binder and in the presence of water vapour, until volatile constituents are liberated from the coal and dissolve in the molten binder. A. B. MANNING.

**Production of hard low-temperature coke.** K. JAHNKE (U.S.P. 1,665,665, 10.4.28. Appl., 19.10.26. Ger., 2.5,24).—A mixture of pulverised coal, spathic iron ore, and a bituminous material is subjected to a low-temperature carbonisation process. A. R. POWELL.

Manufacture of active carbon from soot. G. HUGHES. From T. FUJISAWA (B.P. 287,982, 13.1.27).— Soot obtained by the incomplete combustion of oils, fats, hydrocarbon gases, etc. is heated in a thin layer to a dull red heat and stirred in contact with air until all the volatile impurities have been removed. It is then quickly transferred to a closed vessel and cooled.

#### A. B. MANNING.

Cooling of coke or like material and utilising the sensible heat thereof. FRANKFURTER GAS-GES., F. P. TILLMETZ, and E. SCHUMACHER (B.P. 280,514, 14.10.27. Ger., 11.11.26).-The sensible heat of incandescent coke is utilised in stages by an arrangement in which the coke passes over successive grates through which inert gases are blown. E.g., in the higher stage, during which the coke cools from about 1000° to 700°, the hot gases may be conducted through a high-pressure boiler, and in the lower stage, from about 700° to 300°, the gases may traverse a low-pressure boiler or water preheater. The first grate may be a stepped grate and the second either a stepped or a travelling grate. The coke leaving the last grate passes into a bunker in which it is completely cooled by warm water or low-pressure steam. A. B. MANNING.

Preparation of charges for combustion in internalcombustion engines. G. B. ELLIS. From MOORE INVENTIONS CORP. (B.P. 287,996, 7.2.27).-In order to secure complete combustion of the fuel in an internalcombustion engine the charge is formed by producing a flow of mixed fuel and air and delivering into admixture therewith a flow of exhaust gas and air, the temperature of the charge being controlled by the temperature of the air mixed with the exhaust gas. Hot air may be mixed with exhaust gases at low engine compression pressures and relatively cold air at higher compression pressures. The charge is compressed and ignited in a cylinder having inclined vanes adjacent to its head, the gases as they pass these vanes being thereby given a turbulent motion. A. B. MANNING.

Balanced motor fuel. E. F. CHANDLER (U.S.P. 1,665,621, 10.4.28. Appl., 23.12.24).—A motor fuel comprises a liquid hydrocarbon, an oxide of nitrogen, and a solvent for the latter. A. R. POWELL.

Distillation of coal and similar carbonaceous substances. Low TEMPERATURE CARBONISATION, LTD., and C. H. PARKER (B.P.287,584-7 and 287,830, 25.9.26). -(A) A retort for the production of smokeless fuel by the distillation of coal at low temperatures consists of

a number of vertical chambers, substantially rectangular in cross-section and slightly tapered (e.g.,  $4\frac{1}{2} \times 11$  in. at the upper end,  $5\frac{1}{2} \times 12$  in. at the lower end, and 9 ft. high), formed in a single casting and arranged to present a large external heating surface. The upper and lower ends of the casting have flanges so that a hood may be fitted above the retorts and a hinged bottom plate below. (B) In a process for the distillation of coal in two stages, in the second of which the material is transferred to a retort of considerable volume, water is passed into this retort and vaporises therein, the material being thus cooled and quenched in an atmosphere of steam at a slight pressure. (c) Coal is carbonised at low temperatures by being filled into cylindrical containers and then feeding these into a retort through which they pass by rolling on inclined tracks from the charging to the discharging positions. The containers are open at both ends and are provided with perforated covers. The retorts are made up of a number of connected sections of opposite inclination one above the other, which the filled containers traverse in succession. Each section of retort is mounted in a corresponding retort chamber; heating gases circulate through the chambers, which communicate with one another. The volatile products of distillation are withdrawn from a point near the uppermost end of the retort, or from points near the junctions of the sections. Automatically operating means are provided for filling the containers, charging them into and discharging them from the retort, sealing the retort, and discharging the coke from the containers. (D) In the distillation of coal described under (c) the material is subjected to a preliminary drying by causing the filled containers to traverse a chamber through which hot waste gases pass. (E) In an apparatus for the distillation of coal in which the process is carried out in two stages, first in an externally heated upper retort and then in a separate lower retort without further application of heat, means are provided for the withdrawal of gas from the upper end of the lower retort and conducting it through an auxiliary ascension pipe up and into the main ascension pipe at a point behind A. B. MANNING. the anti-dip valve.

Distillation and gasification of solid carbonaceous material. A. K. CROAD. From ZIELEY PROCESSES CORP. (B.P. 287,999, 14.2.27).—Liquid hydrocarbons of low mol. wt., e.g., gasoline, naphtha, are produced from coal or other carbonaceous material by impregnating the latter with a chemical such as sodium chloride, and subjecting it to distillation and gasification in a current of air and steam at 1370°. The sodium chloride, which is resolved into its constituents at this temperature, acts only as a catalyst. A. B. MANNING.

Distillation of solid carbonaceous or bituminous substances. W. E. EVANS. From KOHLENVEREDLUNG A.-G. (B.P. 287,627, 22.12.26).—The carbonaceous material in the form of a fine powder is carried in a continuous stream with the heating gas, the initial temperature of which is above about 800°, and is maintained in contact therewith for only the short time necessary for the low-temperature distillation of the fuel. Although the initial temperature of the heating gas is above 800° the heat taken up by the preliminary drying of the fuel particles causes this to fall considerably before actual carbonisation commences, so that the latter process then occurs without overheating of the fuel or cracking of the tar vapours. A. B. MANNING.

Distillation of solid carbonaceous materials. H. NIELSEN and B. LAING (B.P. 287,381, 4.12.26, 21.2. and 18.5.27).-In the distillation of bituminous coal etc. cracking of the oil vapours evolved is prevented by so regulating the heating that the temperature difference between the periphery and the centre of any coal particle or any suitable thickness of fuel bed never exceeds 150°. The material may be crushed below 2-in. mesh and passed continuously, during about 2 hrs., through zones of progressively higher temperature produced by heating gases having a temperature gradient of approximately 650° down to 110°, the oil vapours being withdrawn from the apparatus at approximately the temperatures at which they are evolved. The process may be carried out by the use of a large volume of an inert gaseous heating medium as in B.P. 287,037 (B, 1928, 356) and yields products similar to those therein described. A. B. MANNING.

Apparatus for the low-temperature distillation or coking of fuels. J. PLASSMANN (B.P. 287,313, 9.3.27).—A retort comprises a number of superposed annular distillation chambers alternating with heating chambers. The former are closed but the latter are open and in communication with one another at the outer periphery. A centrally disposed shell, which can be rotated, supports the charging and discharging devices, and also the internal peripheral closing means for the distillation chambers. The material is charged into the distillation chambers from a central feeding shaft by means of radially disposed screw conveyors. The heating gases are circulated through the heating chambers by fans. The above system of distillation may be combined with that described in B.P. 240,800 (B., 1926, 228), to form a single unit. A. B. MANNING.

Gas producer. C. W. LUMMIS, ASST. to MORGAN CONSTRUCTION CO. (U.S.P. 1,666,604, 17.4.28. Appl., 19.12.21).—The shell of a gas producer and the waterseal pan are connected and arranged so that they can be rotated together. An ash-plough is supported on the water-seal pan, and, by means of a pivot through the centre of the seal, can be rotated at a different speed from the pan and shell. T. A. SMITH.

Apparatus for making carburetted water-gas. HUMPHREYS & GLASGOW, LTD., Assees. of C. S. CHRIS-MAN (B.P. 282,034, 21.11.27. U.S., 10.12.26).—In a carburetted water-gas set a regenerator is interposed between the superheater and the wash box in order to recover the sensible heat of the carburetted water-gas and to utilise it for preheating the air or superheating the steam supplied to the generator. The regenerator may be provided with a shaking grate carrying a bed of clinker, or may be filled with chequer brick.

A. B. MANNING.

Manufacture of mixed water-gas and distillation gas from fuels of volatile content. HUMPHREYS & GLASGOW, LTD., Assees. of C.S. CHRISMAN (B.P.279,060, 27.9.27. U.S., 13.10.26).—A plant for the complete gasification of fuels of high volatile content consists of

two generators, two stoves for heat storage, a tubular heater, and a wash box. The generators are divided into a lower gasification zone and an upper carbonisation zone. The first operation of the cycle consists of a simultaneous blasting in the gasification zones of both generators, the resulting blow gases passing through the stoves, in which most of their heat becomes stored. The second operation consists of up-steaming both zones in one generator and simultaneously down-steaming the lower zone in the other, and passing the water-gas produced in the latter through one of the heated stoves and then through the upper zone of the former generator together with the water-gas generated in the lower zone thereof. The resulting mixed water-gas and distillation gas is collected. The blow gases entering the stoves may be burnt therein with secondary air preheated in the tubular heater by waste gases from the stoves. Some operating cycles slightly modified from the above are described. A. B. MANNING.

Production of gas. R. LEDERER (B.P. 270,318, 28.4.27. Austr., 28.4.26.).-A mixture of water-gas and coal gas is produced from a bituminous fuel, and coke obtained as a by-product, by the use of a generator and a separate coking chamber so arranged that the producer gas formed during the "blow" is led either directly, or after partial combustion, first through the lower zone of the coking chamber containing fuel which has been distilled, for the purpose of hardening the coke therein, and then through one or more heat accumulators. The mixture of water-gas and coal gas produced during the "run" is conducted through the coking chamber, or through a heat accumulator, in order to decompose the tar vapours in the coal gas and thereby increase the calorific value of the gas produced. A. B. MANNING.

Manufacture of gas. J. HANSFORD and J. S. HUGHES (B.P. 287,213, 13.12.26).—A mixture of liquid or powdered fuel is injected into a combustion chamber wherein the fuel undergoes partial combustion in the presence of iron or other metal shavings. The gas may be enriched by feeding oil into the combustion chamber, or its hydrogen content may be increased by blowing in steam. The burner comprises two semicircular nozzles arranged to produce a conical combustion zone, and may be placed in the lower or the upper end of the combustion chamber. A valve operated by the pressure in the combustion chamber controls the supply of fuel to the burner. A. B. MANNING.

Drying of fuel gases. D. M. HENSHAW, C. COOPER, and W. C. HOLMES & CO., LTD. (B.P. 287,678, 5.2.27).— The gases are partially dried by treatment with a hygroscopic liquid at the ordinary temperature, and then subjected to a similar drying process with the application of artificial cooling. The liquid is reconcentrated either periodically or continuously. Cooling may be effected by having two vessels in communication with one another, one containing a hygroscopic liquid and the other water, and maintaining a reduced pressure in the apparatus, evaporation of the water producing the required reduction of temperature. Means may be provided for agitating the water in the apparatus and so promoting evaporation. A. B. MANNING. Recovery of phenol from waste water containing ammonia. F. RASCHIG (B.P. 287,226, 15.12.26).— The removal of phenol from waste water by passage of the latter through a column counter-current to a suitable extracting liquid, *e.g.*, benzol, is made very efficient by the use of 15-mm. Raschig rings as filling material for the column. The waste water is preferably heated to  $50-60^{\circ}$  by using it for condensing the benzol vapour in the subsequent distillation of the benzolphenol mixture. A. B. MANNING.

Production from coal tar of neutral lubricating oil free from salts, stable in the air, and capable of admixture with mineral oils. "Rex" MINERALÖL-GES. STEPHAN, BOOK, & ZIEGLER (G.P. 445,679, 10.4.24. Addn. to G.P. 444,958; B., 1928, 116).—The vapours generated during the distillation of coal tar pass over solid alkali hydroxides, and, after the distillate has been cooled rapidly to a low temperature, preferably *in vacuo*, any constituents which settle out are removed. L. A. COLES.

Conversion of petroleum hydrocarbons. W. M. CROSS (U.S.P. 1,666,119, 17.4.28. Appl., 20.7.25).—Oil is heated in a tubular still, vaporised in a separate chamber, and the unvaporised residue is returned to the still for re-treatment; the re-treated oil is passed to a separate vaporising and condensing apparatus. The degree of heating to which the charging stock and re-cycled oil are subjected is controlled by adjusting their rates of circulation through the still.

#### C. O. HARVEY.

Separation of gasoline from crude [mineral] oil. Distillation of mineral oil. A. E. PEW, JUN., and H. THOMAS, Assrs. to SUN OIL CO. (U.S.P. 1,666,300-2, 17.4.28. Appl., 23.2.24).-(A) The heated oil is sprayed into a chamber at successively higher elevations and diminishing temperatures, and gives up its heat to a downflowing stream of oil with which it is not in actual contact. (B) The oil is heated to a temperature adequate for the degree of vaporisation required, and is sprayed in a number of streams into a vaporising chamber. The streams are recombined and the operation is repeated a number of times at progressively increasing heights in the chamber, and correspondingly decreasing temperatures. The unvaporised oil is finally allowed to flow down the column under conditions favouring heatinterchange, but out of direct contact with the hot oil streams. (c) The downflowing stream of oil is augmented by an independent stream of mineral oil which finally passes to the heating zone. C. O. HARVEY.

Distillation of oils. A. E. HARNSBERGER, ASST. to PURE OIL CO. (U.S.P. 1,666,597, 17.4.28. Appl., 12.11.26).—Mineral oil is submitted to vacuum distillation in a pipe still, which is built of progressively increasing cross-sectional pipe area from the inlet to the outlet ends, to allow for the increasing volume of the oil and vapours and to reduce back-pressure.

T. A. SMITH.

Distillation of [mineral] oils. C. B. BUERGER, Assr. to GULF REFINING Co. (U.S.P. 1,666,042, 10.4.28. Appl., 28.8.23).—The oil in a horizontal cylindrical still, fitted with a system of partitions and a steam jet, is maintained in a state of constant motion, deposition of carbon being thereby prevented. C. O. HARVEY.

Distillation of hydrocarbon oils. W. P. DEPPÉ and L. L. SUMMERS (U.S.P. 1,666,051, 10.4.28. Appl., 5.1.24).—Crude oils and mixtures of oils with other liquids are passed through tubular stills at progressively increasing temperatures, and the mixture of vapours and liquid is subjected to series of dephlegmating operations. The resulting vapours of predetermined composition are combined, and the residues pass back for re-treatment. C. O. HARVEY.

Refining of heavy mineral oils by means of sulphur dioxide in a continuous operation. L. EDELEANU, K. PFEIFFER, K. GRESS, and P. JODECK (U.S.P. 1,666,560, 17.4.28. Appl., 25.6.27. Ger., 12.6.24).—Mineral oil and liquid sulphur dioxide are continuously run into a mixing vessel fitted with stirring device. The mixture is passed to a larger vessel, being delivered about the middle of the latter vessel, which is full of liquid. Oil is continuously drawn from the top of the separating vessel and sulphur dioxide solution from the bottom. T. A. SMITH.

Manufacture of a gasoline substitute. J. B. NEUENDORFF (U.S.P. 1,666,976, 24.4.28. Appl., 6.8.26). —Petroleum distillates for use in internal-combustion engines are subjected, in a liquid state, to the action of clear lime water. F. G. CROSSE.

Recovery of aluminium chloride from sludge. P. DANCKWARDT (U.S.P. 1,665,406, 10.4.28. Appl., 23.4.27).—Aluminium chloride "sludge" and a gas are injected into a mass of molten metal, and the aluminium chloride is condensed. C. O. HARVEY.

Anti-sludging means for oil. D. C. Cox, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,665,845, 10.4.28. Appl., 28.9.26).—Mineral oil is heated in contact with fuller's earth in an electrically heated vessel. T. A. SMITH.

Manufacture of carbonised fuel. W. W. STRAF-FORD, Assr. to FUELITE Co., LTD. (U.S.P. 1,667,906, 1.5.28. Appl., 21.4.24. U.K., 15.5.23).—See B.P. 221,526; B., 1924, 932.

Manufacture of valuable products from combustible materials. A. H. PEHRSON, ASST. to DUN-FORD & ELLIOTT (SHEFFIELD), LTD. (U.S.P. 1,667,217, 24.4.28. Appl., 13.8.23. Swed., 19.8.22).—See B.P. 202,625; B., 1924, 859.

Gas-treating process. C. LOURENS, ASST. to GEN. NORIT CO., LTD. (U.S.P. 1,667,426, 24.4.28. Appl., 11.11.22. Holl., 14.11.21).—See B.P. 188,666; B., 1923, 914 A.

Exothermic gas reactions (B.P. 275,983).—See I. Rock asphalt (U.S.P. 1,664,846). Bituminous emulsion (Swiss P. 119,219). Bituminous product (U.S.P. 1,665,881). Pavements (B.P. 287,799).— See IX. Furnace for shales (U.S.P. 1,665,546). Recovery of reaction products (B.P. 263,859 and 288,056).—See XI. Lamp blacks etc. (B.P. 270,658).— See XIV.

#### **III.—ORGANIC INTERMEDIATES.**

Synthesis of alcohols higher than methyl alcohol from carbon monoxide and hydrogen. K. FROLICH and W. K. LEWIS (Ind. Eng. Chem., 1928, 20, 354-359).—The comparative efficiency of various catalysts in the formation of higher alcohols from carbon monoxide and hydrogen was investigated, the pressure used being 204 atm. Steel or chromium-steel turnings impregnated with potassium hydroxide gave unsatisfactory results, the loss as gaseous products in most cases varying from 30 to 70%. A low-chromium steel with a minimum of added alkali appeared the most effective. The highest oil yield obtained was  $13 \cdot 4\%$ , and increase of oil yield was associated with increase of gas losses. In combination with a methyl alcohol catalyst, gas losses were less, but only traces of oil were obtained, the product being a mixture of methyl alcohol and higher alcohols. Metallic oxide catalysts, such as a mixture of zinc oxide, chromic oxide, and barium hydroxide on copper, which yield methyl alcohol at 300-350°, produced higher alcohols at 450-500°. The best yield recorded, however, was 13.9%, chiefly propyl alcohol. The loss was rather less than with iron-alkali catalysts, and the product was largely alcoholic in place of the complex mixture of alcohols, aldehydes, ketones, etc. obtained in the other case. C. IRWIN.

Phenols in petroleum distillates. STORY and SNOW.—See II. Furfuraldehyde from wood. RITTER and FLECK.—See V. Tests for ether. MIDDLETON and HYMAS.—See XX. Carbon disulphide. EggERT.— See XXIII.

#### PATENTS.

Oxidation of alcohol. S. GOLDSCHMIDT (U.S.P. 1,666,447, 17.4.28. Appl., 16.12.27. Ger., 12.11.26).— Silver at 380—440° catalytically converts a mixture of ethyl alcohol and air into acetaldehyde and acetic acid. B. FULLMAN.

Manufacture of acetaldehyde from gaseous mixtures containing acetylene. I. G. FARBENIND. A.-G. (B.P. 278,324, 2.8.27. Ger., 28.9.26).—Acetylene, in gas mixtures containing it, is converted into acetaldehyde by treating the mixture, in a finely-divided state (e.g., by passage through microporous glass plates), under at least 2 atm. pressure, and (where the acetylene content is low) at 80° or above, with acid solutions containing mercury salts. The aldehyde is separated from the gas (e.g., by passage through water) prior to expansion of the latter. B. FULLMAN.

Manufacture of formic acid. G. H. BUCHANAN and J. L. OSBORNE, Assrs. to AMER. CYANAMID Co. (U.S.P. 1,666,437, 17.4.28. Appl., 10.12.23).—Formic acid is added to an alkaline-earth formate, and the mixture treated with a non-volatile acid capable of decomposing it; the formic acid formed is then boiled off. H. ROYAL-DAWSON.

Extraction of butyric acid. Soc. DES BREVETS ÉTRANGERS LEFRANC ET CIE. (B.P. 276,617, 11.2.27. Fr., 27.8.26).—Alkali or alkaline-earth butyrates are treated with a slight defect of hydrochloric acid. The liquid separates into two layers. The upper contains the greater part of the butyric acid in 85-90% concentration, which may be further concentrated as usual. The lower contains the metallic chloride and some butyric acid, which may be recovered, *e.g.*, by distillation with superheated steam. B. FULLMAN.

Coagulation or peptisation of products containing unsaturated carbon compounds. L. AUER (B.P. 287,943, 30.9.26).—Isocolloid substances containing unsaturated carbon compounds undergo a physical or physico-chemical change when heated with "electrolytes," under which term are included salts, metal derivatives of organic substances, and solid organic acids of the aromatic and lower aliphatic series; liquid inorganic or organic acids may be used in conjunction with these. The "electrolyte" is added dry to the isocolloid and is dissolved in it with the aid of heat and. if desired, by grinding. The treatment may be followed or accompanied by a treatment with a gas or gases at reduced, ordinary, or increased pressure. The result is to convert liquid starting materials (vegetable oils, liquid unsaturated hydrocarbons, etc.) into pasty or hard solids, and solid starting materials (resins etc.) into pasty or thin liquids; rubber, tars, asphalts, etc. may also be used. The properties of the final product may be modified by adding to the starting materials purely organic substances (phenols, naphthols, naphthalene, chloroform, acetone, alcohols); siccatives may also be added. The products may be vulcanised during or after the modifying process, e.g., by means of sulphur chloride, sulphur, or sulphur and an accelerator. The examples refer to linseed oil and rosin, the products being useful as rubber substitutes, linoleums, varnishes, etc. The process appears to be physical rather than chemical. and is reversed if by prolonged washing or grinding with a solvent the "electrolyte" is moved. C. HOLLINS.

Manufacture of alkyl  $\beta$ -halogenoethyl ketones. Schering-Kahlbaum A.-G. (B.P. 282,412, 14.12.27. Ger., 15.12.26).—Ethylene reacts with acyl halides in presence of aluminium chloride or bromide to give alkyl  $\beta$ -halogenoethyl ketones. Examples are methyl  $\beta$ chloroethyl ketone, b.p. 50—55°/16 mm.; methyl  $\beta$ bromoethyl ketone, b.p. 55—60°/15 mm. C. Hollins.

Manufacture of 1-amino-8-naphthoic acid. R. HERZ and F. SCHULTE, ASSTS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,668,148, 1.5.28. Appl., 9.5.27. Ger., 29.11.24).—See B.P. 276,126; B., 1927, 808.

Continuous dehydration of volatile fatty acids. E. RICARD, Assr. to Soc. ANON. DES DISTILLERIES DES DEUX-SÈVRES (U.S.P. 1,668,380, 1.5.28. Appl., 26.12.24. Belg., 28.12.23).—See B.P. 226,822; B., 1925, 737.

Manufacture of chlorinated amines from chlorinated hydroaromatic ketimino-compounds [chloroketimides]. T. VOLTZ, ASST. to DURAND & HUGUENIN Soc. ANON. (U.S.P. 1,667,336, 24.4.28. Appl., 21.5.23. Ger., 3.6.22).—See B.P. 217,753; B., 1924, 769.

Condensation products of anthraquinone (B.P. 263,178).—See IV.

# IV.—DYESTUFFS.

Some azo dyes soluble in non-aqueous solvents. C. E. MAY and H. HUNT (Ind. Eng. Chem., 1928, 20, 384—388).—A number of new *azo dyes*, soluble in hydro-

carbons, linseed oil, and carbon tetrachloride, but insoluble in water, have been prepared. In the following list the name of the diazotised intermediate is followed by that of the substance with which it was coupled, and the m.p. of the resulting dyestuff: 3-o-xylidine,  $\beta$ -naphthol, 125—130°; 3-o-xylidine, 1-amino- $\beta$ -naphthol, 140—145°; 4-m-xylidine,  $\beta$ -naphthol, 132—136°; 4-m-xylidine, 1-amino-β-naphthol, 140-144°; 4-mxylidine, phenol, 172-174°; 4-m-xylidine, resorcinol, 275-280°; 4-m-xylidine, guaiacol, 110-112°; pxylidine, β-naphthol, 115-120°; p-xylidine, 1-aminoβ-naphthol, 105-106°; aminoazo-4-m-xylene, β-naphthol,  $145-150^{\circ}$ ; aminoazo-4-*m*-xylene, 1-amino- $\beta$ -naphthol,  $140-150^{\circ}$ ; aniline,  $\alpha$ -naphthol,  $148-150^{\circ}$ ; aniline, 1-amino-\$\beta-naphthol, 110-115°; aniline, 1nitroso-B-naphthol, 106°; o-aminoazotoluene, salicylic acid, 125-130°; o-aminoazotoluene, resorcinol, above 360°; o-aminoazotoluene, a-naphthol, 153-156°; oaminoazotoluene, phenol, 290-295°; aminoazo-pxylene,  $\beta$ -naphthol, 180—185°; aminoazo-p-xylene, 1-amino- $\beta$ -naphthol, 110—115°;  $\beta$ -naphthylamine,  $\alpha$ naphthol, 146°; β-naphthylamine, dimethylaniline, 153-158°; benzidine, 2 mols. of α-naphthol, 255-260°; benzidine, 1 mol. of phenol and 1 mol. of  $\alpha$ -naphthol, above 360°; benzidine, 2 mols. of *a*-naphthylamine, above 360°; o-anisidine, 1-amino-B-naphthol, 135-140°; aminoazo-β-naphthylamine, β-naphthol, 107-109°; commercial xylidine and  $\alpha$ -naphthol, 113–118°; 6-nitro-as-m-xylidine, β-naphthol, 180-185°; 1-aminoβ-naphthol, dimethylaniline, 135-140°. The dyestuffs had the expected composition, except for the lastmentioned, which had apparently acquired a nitrosogroup. The relative solubilities of these and other similar known dyes in toluene and in carbon tetrachloride, expressed as the smallest amount of solvent which dissolved completely 0.5 g. of the sample, were determined. Reduction of these dyes was found to be impossible with stannous chloride and hydrochloric acid, but was readily accomplished by means of sodium amalgam and alcohol at 100°. On reduction of the dye from 4-mxylidine and 1-amino-\beta-naphthol by this method, the diaminonaphthol first produced was very readily oxidised and the substance isolated was a dark red powder, apparently 4-amino-1: 2-naphthaquinone (m.p. 136°). When this substance was diazotised and the diazonium salt coupled with  $\beta$ -naphthol a bright red dye resulted. W. J. POWELL.

# PATENTS.

Manufacture of new dyes of the anthanthrone series. L. CASSELLA & Co., G.M.B.H. (B.P. 287,020, 3.11.26. Addn. to B.P. 260,998; B., 1928, 225).—The bromination and/or chlorination of anthanthrone is performed in presence of excess of liquid bromine or sulphuryl chloride as diluent; alternatively, anthanthrone is treated with gaseous halogen, either reaction being assisted, if desired, by addition of a carrier (iodine, ferric chloride). Orange vat dyes similar to those of the prior patent are obtained. C. HOLLINS.

Manufacture of condensation products of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 263,178, 17.12.26. Ger., 17.12.25).—o-Aminothiolan-thraquinones are condensed with halogenohydrins (ethylene chlorohydrin, propylene chlorohydrin,  $\alpha$ -chloro-

hydrin) to give thioethers (not thiomorpholines), which are acetate silk dyes and also intermediates. Examples are the  $\beta$ -hydroxyethyl thioethers of 1-amino-2-thiolanthraquinone (m.p. 171—173°), 2-chloro-1: 4diamino-3-thiolanthraquinone, 1-amino-4-p-toluidino-2thiolanthraquinone, etc. C. HOLLINS.

Manufacture of condensation products and vat dyes of the benzanthrone series. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 261,757, 17.11.26. Ger., 17.11.25. Addn. to B.P. 249,891; B., 1927, 647).—Halogenated benzanthronyl mercaptans, sulphides, or disulphides are condensed with phenols or mercaptans, and the products give by alkaline fusion reddish-blue to violet vat dyes. Examples are dibromo-1: 1'-dibenzanthronyl sulphide with phenol,  $\beta$ -naphthol, or p-thiocresol. C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 273,656, 7.3.27. Ger., 1.7.26).—Vat dyes are obtained by treating with acid condensing agents (boric and sulphuric acids, aluminium chloride) the benzanthronylaminoanthraquinones obtained from  $\alpha$ -aminoanthraquinones and 4-chloro- or 4 : 8-dichloro-benzanthrone. 4'-Benzanthronyl- $\alpha$ -aminoanthraquinone gives a bright orange-red vat dye, 4 : 8-di- $\alpha$ -anthraquinonylaminobenzanthrone a brown, 6-chloro-4'-benzanthronyl-1-aminoanthraquinone a red-brown, 6-amino-4'-benzanthronyl-1aminoanthraquinone a violet-brown, 8'-chloro-4'-benzanthronyl-1-amino-4-methoxyanthraquinone a brown. C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 278,651, 17.3.27. Ger., 8.10.26).—Aminobenzanthrone is diazotised in concentrated sulphuric acid, and the mixture diluted and boiled; the hydroxybenzanthrone, m.p. 287—288°, so produced is alkylated (e.g., with methyl toluenesulphonate to form methoxybenzanthrone, m.p. 167—168°), and finally fused with alkali. In the case of methoxybenzanthrone the green vat dye obtained is identical with that of B.P. 181,304 and 218,255. C. HOLLINS.

Manufacture of vat dyes of the dibenzanthrone series. I. G. FARBENIND, A.-G. (B.P. 262,819, 13.12.26. Ger., 14.12.25).—3:3'-Dibenzanthronyls, unsubstituted in the 2:2'-positions, are reduced in alkaline media to give leuco-vat dyes. For example, 3:3'-dibenzanthronyl is boiled with dilute sodium hydroxide, and sodium formaldehydesulphoxylate is gradually added; the resulting vat is oxidised with hypochlorite to precipitate the new vat dye. C. HOLLINS.

Manufacture of vat dyes of the dibenzanthrone series. I. G. FARBENIND. A.-G. (B.P. 263,861, 30.12.26. Ger., 30.12.25. Addn. to B.P. 218,255; B., 1925, 538).— The dyes of the prior patent are obtained in better yield and purer condition by alkaline fusion of suitable dialkoxy-3:3'-dibenzanthronyls, e.g., 2:2'-dimethoxy-3:3'-dibenzanthronyl, m.p. 387—390°, obtained by heating with copper the 3-iodo-2-methoxybenzanthrone, m.p. 248°, prepared from 2-methoxybenzanthrone by nitration, reduction, and the Sandmeyer reaction.

C. HOLLINS.

Manufacture of grey to black vat dyes. I. G. FARBENIND. A.-G. (B.P. 264,502, 12.1.27. Ger., 12.1.26).—Aminodibenzanthrone is treated with methyl

sulphate or methyl alcohol and sulphuric acid at 170° in the absence of acid-binding agents to give a complex condensation product which dyes cotton grey to black shades from a blue vat. C. HOLLINS.

Manufacture of azo dyes. J. R. GEIGY A.-G. (B.P. 275,223, 22.7.27. Ger., 29.7.26).—An aminosulphobenzoic acid in which the sulphonic group is ortho to the amino-group is diazotised and coupled with the usual components (excluding H-acid and cyanuric derivatives). The dyes, especially those from 2-chloro-5-amino-4-sulphobenzoic acid, show superior fastness to light compared with the non-carboxylated dyes. The  $\beta$ -naphthol coupling gives a barium or calcium lake superior to Lake Red C; the  $\gamma$ -acid coupling is a bluish-red wool dye, little changed in shade by after-chroming. C. HOLLINS.

Manufacture of trisazo dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 287,232, 16.12.26. Addn. to B.P. 248,230. Cf. F.P. 598,894; B., 1926, 311).-1:2-Aminonaphthyl ethyl ether is used as third component in place of the sulphonic acid of the prior patent, and/or other J-acid derivatives in place of J-acid and aryl-J-acids, the coupling of the end-component being effected in presence of pyridine. Examples are : aniline-2:5-disulphonic acid  $\rightarrow \alpha$ -naphthylamine or its 7-sulphonic acid  $\rightarrow$  1-amino-2-naphthyl ethyl ether > phenyl-J-acid or ethyl-J-acid (greenish-blue); o-toluidine-3: 5-disulphonic acid  $\rightarrow \alpha$ -naphthylamine-7-sulphonic acid  $\rightarrow$  1-amino-2-naphthyl ethyl ether  $\rightarrow$  6-hydroxy-2-m-aminophenyl- $\alpha$ -naphthiminazole-8sulphonic acid (from 1: 2-diamino-5-naphthol-7-sulphonic acid ; bluish-green) or 1-phenyl-3-methyl-5-pyrazolone (green); aniline-o-sulphonic acid  $\rightarrow \alpha$ -naphthylamine-7sulphonic acid  $\rightarrow$  2-ethoxy- $\alpha$ -naphthylamine-7-sulphonic acid  $\rightarrow$  *m*-aminobenzoyl-J-acid (greenish-blue).

C. HOLLINS.

Manufacture of dyes containing chromium. I. G. FARBENIND. A.-G. (B.P. 262,418, 25.11.26. Ger., 1.12.25).—Chromable dyes are heated in open or closed vessels with chromic salts in presence of a solution of a mineral acid salt of a metal which does not combine with the dye (*i.e.*, a non-mordanting metal). The shades obtained are different from those obtained in the absence of the non-mordant salt. *E.g.*, the dye, 4-nitro-2-aminophenol-6-sulphonic acid  $\rightarrow \beta$ -naphthol, heated with chromium formate solution and sodium chloride, gives a black wool dye, whereas in the absence of sodium chloride the shade is violet brown. The amount of nonmordant salt must be at least equal to the weight of dye. C. HOLLINS.

Manufacture of dyes [of the perylene series]. F. BENSA (B.P. 278,325, 9.8.27. Austr., 2.10.26).— When 3:9-dichloro-4:10'-diacetyl- (or dipropionyl-) perylene is heated with copper cyanide in boiling quinoline (or pyridine under pressure at 190—200°), the chlorine atoms are replaced by nitrile groups, and further condensation occurs with production of reddish-violet vat dyes. The starting materials are obtained from 3:9-dichloroperylene and acetyl and propionyl chlorides in presence of aluminium chloride at 15°.

C. HOLLINS.

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Manufacture of triarylmethane dyes. BRIT. DYESTUFFS CORP., LTD., and T. A. SIMMONS (B.P. 287,995, 5.2.27).—Tetraethyldiaminobenzhydrol is isolated (from the oxidation product of the methane with lead peroxide) in the form of its 2:7-naphthalenedisulphonate, and this salt is condensed in sulphuric acid to the leuco-compound of Lissamine Green V, from which the dye is obtained in the usual manner.

C. HOLLINS.

Manufacture of colour lakes. I. G. FARBENIND. A.-G. (B.P. 270,750, 7.5.27. Ger., 10.5.26).—Colour lakes made from complex phosphorus, tungsten, molybdenum, and silicon acids and basic dyes are improved by heating in paste form under 2—4 atm. pressure either during or after the manufacture. The treatment may be carried out in presence of the usual substrates, dispersing agents, and, if desired, a further quantity of complex acid. C. HOLLINS.

Manufacture of black copying colours. I. G. FARBENIND. A.-G. (B.P. 282,804, 28.12.27. Ger., 27.12.26).—A dialkylphenosafranine is diazotised and coupled with an alkylated or aralkylated 2-amino-*p*cresol (cf. B.P. 283,777; B., 1928, 226), *e.g.*, 2-ethylamino- or 2-benzylamino-*p*-cresol. C. HOLLINS.

Manufacture of vat dyes. J. G. DINWIDDIE, Assr. to E. I. DU PONT DE NEMOURS & Co. (Re-issue 16,939, 24.4.28, of U.S.P. 1,558,252, 20.10.25).—See B., 1925, 983.

Manufacture of azo dyes. H. FRITZSCHE, E. KRUMMENACHER, H. GUBLER, and O. KAISER, ASSIS. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,667,312, 24.4.28. Appl., 21.12.23. Fr., 9.1.23).—See B.P. 209,723; B., 1925, 437.

Manufacture of azo dyes. A. L. LASKA and A. ZITSCHER, ASSTS. to I. G. FARBENIND. A.-G. (U.S.P. 1,667,667, 24.4.28. Appl., 10.9.25. Ger., 3.10.24).— See G.P. 430,580; B., 1927, 275.

Manufacture of [azo] dyes [containing metal]. F. STRAUB and H. SCHNEIDER, ASSTS. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,667,333, 24.4.28. Appl., 12.3.26. Switz., 28.3.25).—See B.P. 249,884; B., 1927, 674.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Wetting of pine wood. C. G. SCHWALBE and W. LANGE (Papier-Fabr., 1928, 26, 238-241).-Pine heartwood absorbs a much larger quantity of magnesium bisulphite solution than sapwood in the same time, whereas with sodium bisulphite the amounts taken up are not markedly different. Sapwood in cylindrical form (3 cm. long, 3 cm. diam.) was shaken for 8 hrs. at 35-70°, and allowed to remain for 24 hrs. with solutions of sodium sulphite, sodium bisulphite, and sodium bisulphite containing free sulphurous acid; the quantities of solution absorbed per 100 g. of air-dry wood were  $104 \cdot 2, 85 \cdot 0, 120 \cdot 2$  g., indicating that the presence of free sulphurous acid is necessary to ensure rapid wetting of the chips. The amount of water taken up under the same conditions was only  $89 \cdot 0$  g., that of sulphurous acid solution (4.5% SO.) 128.7 g., and sodium hydroxide solution 134.0 g. On cutting up the chips which had

been immersed in sodium sulphite solution, exterior portions were found to contain a solution of the same composition as the bulk, whilst inner portions were acid to litmus and contained a solution with a considerable sodium bisulphite content. Inner portions of chips after immersion in sodium bisulphite solution with or without excess of sulphurous acid, contained sulphurous acid solution only. This selective absorption did not take place when corresponding calcium and magnesium salts were used in place of sodium salts. W. J. POWELL.

Furfuraldehyde and carbon dioxide from wood before and after chlorination. G. J. RITTER and L. C. FLECK (Ind. Eng. Chem., 1928, 20, 371-373).-A study of the Cross and Bevan method of cellulose isolation (alternate treatment of wood with chlorine and sodium sulphite solution). No substances capable of producing furfuraldehyde on boiling with dilute hydrochloric acid were formed during the chlorination of a sample of wood, but a considerable amount of a substance which yielded carbon dioxide with boiling dilute hydrochloric acid was obtained, chiefly during the first chlorination, this material differing from oxycellulose in that it was soluble in sulphite solution and yielded no furfuraldehyde. These results indicate that there is no danger of forming oxycellulose in the determination of cellulose by the Cross and Bevan method. The original wood yielded 0.86% of carbon dioxide with boiling dilute hydrochloric acid, and  $56 \cdot 6\%$  of the unstable pentosans present were rendered soluble in sulphite solution during the first chlorination. W. J. POWELL.

Micro-method for the determination of the nitrogen content of nitrocellulose. Fractionation of nitrocellulose by diffusion. D. KRÜGER (Z. angew. Chem., 1928, 41, 407-408).-A sample of nitrocellulose (5-10 mg.) is moistened with a small quantity of alcohol and hydrolysed by heating at 50-60° with 5 c.c. of 30% sodium hydroxide solution and 0.5 c.c. of 30% hydrogen peroxide solution until a clear solution is obtained. The nitrate is then reduced to ammonia by addition of 10 c.c. of 30% sodium hydroxide solution and 0.25 g. of Devarda's alloy, and the ammonia is steamdistilled into N/70 hydrochloric acid, the excess of acid being titrated with standard sodium hydroxide solution. If a solution of nitrocellulose in an organic solvent is allowed partially to diffuse into the pure solvent, it is found that a separation into two fractions of different physical behaviour and of different nitrogen content has taken place. H. F. GILLBE.

Deflocculation and detergency. CHAPIN.—See XII. Carbon disulphide in viscose manufacture. Eggert.—See XXIII.

# PATENTS.

Degreasing of raw wool. R. BRAUCKMEYER (G.P. 445,792, 28.5.25. Denm., 18.7.24. Addn. to G.P. 411,334; B., 1925, 842).—Wool dried until it contains only 2—3% of moisture is degreased with acetone and subsequently treated with water. L. A. COLES.

Lowering the viscosity of nitrocellulose in solutions by mechanical action. G. H. TOZIER, ASST. to EASTMAN KODAK CO. (U.S.P. 1,666,049, 10.4.28. Appl., 5.4.26).—The viscosities of solutions of nitrocellulose CL. VI.-BLEACHING; DYEING; PRINTING; FINISHING.

are reduced to 80% of their original value by intensive mechanical agitation. C. O. HARVEY.

Recovery of camphor [from celluloid]. A. FRIEDEN, Assr. to R. F. BACON and A. HIRSCH (U.S.P. 1,666,645, 17.4.28. Appl., 7.3.27).—Celluloid etc. is treated with an alkaline solution of such strength that the greater part of the nitrocellulose is not decomposed. On steam-distillation the camphor is removed.

B. FULLMAN.

Preparation of artificial silk of improved physical structure. T. IWASAKI and K. HAGIWARA (B.P. 288,655, 8.10.26).—See F.P. 621,543; B., 1928, 187.

# VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Primary action of chromic acid on animal fibre. M. A. ILJINSKI and D. J. KODNER (J. Russ. Phys. Chem. Soc., 1928, 60, 193-207).—The action of chromic acid on wool fibre under various conditions was investigated. The fibre readily adsorbs the chromic acid in the cold, forming a bright yellow, stable complex. The amount adsorbed is proportional to the time of reaction and concentration of solution, and reaches a maximum at about 85% adsorption. The complex is, in every case, one of the basic fibre with dichromic acid, irrespective of the nature of the original chromium salt. The maximum amount taken up by the fibre is 0.09-0.1 g.-mol./100 g. of wool. Addition of sulphuric acid above a certain limit, as also rise of temperature, have no effect on the final adsorption. The dichromate complexes are not hydrolysed by water, like the sulphuric and hydrochloric acid complexes-both the latter are quantitatively displaced by addition of chromic acid. Silk fibre, likewise, forms dichromic acid complexes in the cold, and this property affords a means of dyeing woollen and silk fibre with lake colours and effecting an economy of more than 90% in the use of steam in mordanting. For colours which are easily oxidised, the dichromic acid complex can be reduced on the fibre by means of bisulphite.

M. ZVEGINTZOV.

Mordanting of basic dyes. M. MICHELS (Giorn. Chim. Ind. Appl., 1927, 9, 580-588).—The various theories as to the nature of the dyeing process are discussed, especially as regards basic dyes. The author's results confirm those of Caille (B., 1926, 355), and indicate that absorption of basic dyes by Celanese is due, not to the cellulose acetate, but to the impurities present. Basic dyes are fixed more or less well by all silicates, provided that these are sufficiently finely divided, the capacity of the semi-colloidal clay, fuller's earth, etc. in this direction being more marked than that of any other natural silicate; the fastness of the dye towards light and washing increases with the micellar dispersion of the silicate. Quartz, even when very finely powdered, exhibits no tinctorial affinity, and silicic acid fixes basic dyes only when amorphous, the age and mode of precipitation of the acid exerting a pronounced influence on the composition of the lake precipitated. The fact that colloidal silicic acid precipitates only those basic dyes which contain two benzene nuclei is regarded as due to the fact that the freshly precipitated acid is imperfectly colloidal and thus partly ionised,



carrying a negative electric charge. Maturation of the silicic acid is accompanied by saturation of the secondary valencies producing the positive electric charge, with formation of non-dissociable and noncrystallisable complexes. The latter are able to exert tertiary affinities, by means of two of which they combine with the dye. Silica precipitated in pulverulent forms absorbs basic dyes to a slight extent, this being an adsorption or surface phenomenon. Such dyes may, indeed, be purified by absorbing them from their impure solutions by means of silica powder and subsequently liberating them by treatment with pyridine. The trioxides of chromium, molybdenum, tungsten, and uranium combine with basic dyes, including those derived from triphenylmethane, the solubility and stability of the precipitates varying progressively throughout the homologous series with the degree of complexity of the oxide. The fixation of basic dyes by zinc sulphite is brought about by the formation with water of compounds such as 5ZnSO<sub>2</sub>,8Zn(OH)<sub>2</sub>, these fixing the dyes by means of tertiary valencies. Zinc ferrocyanide and zinc thiocyanate also form basic dyes containing zinc salts. Cuprous thiocyanate, used in photography for the production of coloured films and diapositives, acts well only when it contains a certain proportion of alkali thiocyanate-that is, when complex salt is formed. Better results are obtained when the silver image is transformed into one of cupric ferrocyanide; dyeings on this salt exhibit moderate fastness against light, but less against washing. Cuprous ferrocyanide forms an excellent mordant for basic dyes. The four conditions for the stable fixation of basic dyes are that: (1) the mordant contains active groups, not in the state of ions, but as radicals forming part of a complex molecule; (2) the active groups are distributed in heterogeneous molecules-that is, those containing also radicals inert towards basic dyes; (3) the compounds containing active groups are not crystalline, so that the molecules do not assume reciprocally symmetrical positions; and (4) these compounds occur in as highly disperse a state of aggregation as possible. T. H. POPE.

#### PATENTS.

Dyeing of artificial [viscose] silk. BRIT. DVE-STUFFS CORP., LTD., J. BADDILEY, P. CHORLEY, and R. BRIGHTMAN (B.P. 287,010, 8.9.26. Addn. to B.P. 281,410; B., 1928, 121).—Level dyeings on regenerated cellulose materials are obtained by the application of secondary disazo dyes made by coupling a diazotised aminoazo compound with N-substituted derivatives of 2-amino-8-naphtholsulphonic acids, such as phenyl- $\gamma$ -acid,  $\beta$ -naphthyl-H-acid,  $\beta$ -naphthyl- $\gamma$ -acid, o-anisyl- $\gamma$ -acid, 2:4-dinitrophenyl- $\gamma$ -acid, ethyl- $\gamma$ -acid, benzoyl- $\gamma$ acid, acetyl- $\gamma$ -acid, and 8-hydroxy-6-sulpho- $\beta$ -naphthylglycine. The shades obtained are browns, navy-blue, violet to black. C. HOLLINS.

Production of fast dyeings [on acetate silk, pelts, hairs, feathers, etc.]. A. G. BLOXAM. From I. G. FARBENIND. A.-G. (B.P. 287,651, 5.1.27. Addn. to B.P. 262,537; B., 1927, 104).—The goods are padded with a mixture of a coupling component, an arylamine, and a nitrite, and the bath is acidified

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to develop the colour; the nitrite assists the absorption of the azo components. The two-bath process of the prior patent for dyeing acetate silk is extended to the dyeing of pelts, hairs, feathers, etc. Degreasing ("killing") and protective materials may be added to the dye-bath. In the printing process of the prior patent the arylamine may be omitted from the printing paste and applied afterwards, preferably with addition of an organic acid. In the case of *m*-diamines, the goods are padded with the diamine and printed with acid and nitrite. Twenty-one examples are given.

C. HOLLINS.

Dyeing of regenerated cellulose materials. BRIT. DVESTUFFS CORP., LTD., and J. BADDILEY (B.P. 287,609, 10.12.26. Addn. to B.P. 283,319; B., 1928, 189).— Viscose silk is dyed in level blue shades by means of secondary disazo dyes (other than those of the prior patent) of the type: sulphonated or carboxylated *p*-nitroarylamine  $\rightarrow$  middle component  $\rightarrow$  an aminonaphtholsulphonic acid (except 2:8-aminonaphtholsulphonic acid) or *N*-derivatives thereof. The nitrogroup may be reduced to give developing dyes. The dye *p*-nitroaniline-*o*-sulphonic acid  $\rightarrow$  cresidine  $\rightarrow$ 1:8-aminonaphthol-4-sulphonic acid gives bright blue shades. C. HOLLINS.

Dyeing of regenerated cellulose materials. BRIT. DYESTUFFS CORP., LTD., and J. BADDILEY (B.P. 287,214, 13.12.26).-Viscose silk is dyed in level brown-black to black shades with secondary disazo dyes made by coupling a diazotised nitroarylamine containing no sulphonic or carboxylic groups with a middle component, rediazotising, and coupling in alkaline medium with a sulphonated 1:8-aminonaphthol or an Nsubstituted derivative thereof. The nitro-group may be reduced. Examples are: p-nitroaniline  $\rightarrow \alpha$ -naphthylamine-6(7)-sulphonic acid  $\rightarrow 1$ : 8-aminonaphthol-4sulphonic acid (dull blue); p-nitroaniline  $\rightarrow$  cresidine  $\rightarrow$ H-acid, reduced (blue-black). The reduced dyes may be diazotised on the fibre and developed, e.g., with *m*-tolylenediamine. C. HOLLINS.

Mordanting and dyeing of materials made with or containing cellulose derivatives. H. DREYFUS (B.P. 286,761 and 287,204, 3.12.26).-(A) Cellulose esters or ethers are mordanted with solutions of mordant metal salts (other than ferric salts) at concentrations of 15% or higher and are then dyed with mordant dyes. If temperatures above 75-80° are used, a protective agent is added to prevent delustring. Examples are alizarin on an aluminium mordant, Modern Heliotrope DH on a chrome mordant. Pattern effects are obtained in the usual manner by printing or stencilling, and by local application of a resist. (B) The mordant metals are best applied in the form of their aliphatic salts, especially salts of hydroxylated acids, e.g., lactates, glycollates, citrates, acetates, in concentrations of 5% or higher. Mixed salts, such as aluminium sulphate-acetate, nitrate-acetate, etc., and basic salts may be used.

C. HOLLINS. Dyeing of artificial silk. J. BADDILEY, P. CHORLEY, and C. BUTLER, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,667,524, 24.4.28. Appl., 2.4.27. U.K., 4.6.26). —See B.P. 276,450; B., 1927, 812. Production of figured fabrics. G. HEBERLEIN, Assr. to HEBERLEIN PATENT CORP. (U.S.P. 1,667,892, 1.5.28. Appl., 27.7.25. Ger., 29.7.24).—See B.P. 237,909; B., 1926, 485.

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

Titrometric determination of calcium and magnesium carbonates in limestone. J. S. PIERCE, W. C. SETZER, and A. M. PETER (Ind. Eng. Chem., 1928, 20, 436-437).-The carbonate is dissolved in a known excess of standard acid, the solution boiled, and titrated back with standard alkali until neutral to bromothymolblue. There is then added 1 c.c. of saturated alcoholic trinitrobenzene for each 10 c.c. of solution, and alkali is run in until the indicator shows a dark brick-red colour. A blank test correction to the same colour is run immediately, as the colour is not permanent. The result indicates completion of magnesium precipitation. Results of analyses are in good agreement with those obtained gravimetrically. Aluminium, if present, must be filtered off at neutrality, but iron does not interfere unless present in such quantity as to mask the colour.

C. IRWIN.

Determination of very small quantities of iodides. J. T. DUNN (Analyst, 1928, 53, 211—212).—The following method, which is a modification of Hunter's method for iodine in thyroid glands (B., 1910, 784) and Brubaker's for natural waters (B., 1926, 630), is satisfactory. About 50 g. of the sample are dissolved in 250 c.c. of water, and a few drops of sodium hypochlorite solution are added, followed by 1—5 c.c. of 40% phosphoric acid. The chlorine is driven off by boiling, 1 or 2 c.c. of 1% potassium iodide are added to the cooled solution, and the liberated iodine is titrated with about 0.005N-thiosulphate. D. G. HEWER.

Properties of high-calcium lime. RAY and MATHERS.—See IX.

PATENTS.

**Production of high-percentage nitric acid.** N. CARO and A. R. FRANK (B.P. 273,718, 28.6.27. Ger., 29.6.26).—Increased concentrations of ammonia and oxygen are made possible in the catalytic formation of nitric acid by a layer of cooling liquid circulating in front of or under the contact layer and extending over its whole width so closely that no travelling explosion wave can be formed. The gaseous mixture bubbles under pressure through this liquid (water, ammoniacal liquor, etc.) in such a way that heat radiated from the contact material is absorbed and liquid is evaporated into the mixture. Devices for cooling the walls and for absorbing radiation heat on the nitric oxide side of the contact layer are indicated. W. G. CAREY.

**Combustion of ammonia.** H. PAULING (B.P. 279,511, 24.10.27. Ger., 22.10.26).—Uniform distribution of the gas current over a large cross-section in the catalytic combustion of ammonia with air is obtained by a series of diffuser chambers in the form of orifices in a thick plate of refractory material, narrow ribs being left between the orifices upon which the contact mass is supported. The diffuser element is set in operation

by heating with combustible gas and air which flows axially into each diffusion chamber; the combustible gas is then replaced by ammonia. W. G. CAREY.

Manufacture of hydrofluoric acid. W. J. HAR-SHAW and C. S. PARKE, ASSTS. to HARSHAW, FULLER, & GOODWIN CO. (U.S.P. 1,665,588, 10.4.28. Appl., 19.2.25). —A fluoride in broken, fragmentary form (not ground) is heated with a suitable acid to liberate hydrofluoric acid, any sulphate coating on the fluoride fragments being removed to allow further action. W. G. CAREY.

Manufacture of silica gels. H. N. HOLMES and J. A. ANDERSON (U.S.P. 1,665,264, 10.4.28. Appl., 10.8.23).—A soluble silicate and a metallic salt are mixed in solution, and the resulting mixture of hydrated silica, soluble salt, and insoluble metallic oxide is dried in a manner to produce a rigid structure without material shrinkage; the oxide and salts are subsequently dissolved out. W. G. CAREY.

Wide-porous active silica. Active silica gels with fine capillary pores. I. G. FARBENIND. A.-G. (B.P. 263,198-9, 20.12.26. Ger., 19.12.25).-(A) Active silica gels with wide pores, particularly suitable for the absorption of gases and vapours at high partial pressures and saturated or almost saturated vapours, are prepared by washing a silica jelly, before or during drying, with such a solution as to impart to it  $p_{\rm H}$  7—10. E.g., a jelly prepared by stirring 550 litres of sodium silicate solution, d 1.164, with 51 kg. of sulphuric acid, 46 kg. of ice, and 60 litres of water, and subsequently heating the mixture, is washed until it gives a red-violet coloration with cresol-red, preferably with water treated with alkalis to give the same coloration, after which it is dried in a current of air at 150-200°. (B) Gels with fine capillary pores, having a high absorptive capacity for gases and vapours at low partial pressures, are prepared by a process similar to that described in (A), except that the jelly is washed until it has  $p_{\rm H} 2 - 5 \cdot 5$ , *i.e.*, until it gives a yellow to blue-green coloration with bromocresolgreen, water acidified with hydrochloric to give this coloration being used for the washing. L. A. COLES.

Manufacture of prepared calcium chloride. A. K. SMITH, Assr. to Dow CHEM. Co. (U.S.P. 1,660,053, 21.2.28. Appl., 12.10.25).—A saturated solution of calcium chloride is prepared at 190°, cooled below 175° under pressure, and allowed to solidify before it is powdered. H. ROYAL-DAWSON.

Continuous transformation of oxides into chlorides by exothermic reaction. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 274,048, 25.5.27).— Chlorine is introduced into a shaft furnace containing a mixture of the oxide with a suitable amount of an inert solid substance, *e.g.*, retort carbon, to absorb the excess heat of reaction. The inert material is of such granular size that the separation of the chloride can be brought about by screening. W. G. CAREY.

**Production of complex fluorides.** A. F. MEYER-HOFER, Assee. of M. BUCHNER (B.P. 269,491, 2.3.27. Ger., 13.4.27).—A suitable gaseous fluoride, with admixture of neutral gas if necessary, is injected into a tall column of liquid containing a metal fluoride, a salt of the base the fluoride of which is required, an acid, and a solvent. The influx of gas causes the liquid to overflow, and it is returned to the lower part of the column while the solid reaction products are conducted downwards into a conically widened chamber. The process may be conducted under pressure or *in vacuo* and assisted by heating or stirring or by baffles in the column.

W. G. CAREY.

Preparatory treatment of crude heavy spar. K. EBERS (B.P. 288,498, 25.10.27).—Heavy spar containing little or no lime and in which silica and compounds of iron and manganese are present yields a pure white barium sulphate if it is roasted, quenched in water without cooling, and treated with hydrochloric acid. W. G. CAREY.

Production of hydrated iron oxide from iron metal. J. WAGNER (B.P. 287,702, 8.3.27).—Scrap iron or steel free from rust is treated in an open tank with a continuous current of an aqueous solution containing up to 3% of sodium chloride and up to 1.5%of chlorides having catalytic properties, *e.g.*, ferric chloride, manganese chloride, etc., the liquid being continuously aerated with air to which about 10% of dust-free carbon dioxide has been added. The overflow from the top of the tank passes into a filter to remove the suspended hydrated iron oxide before the liquor is pumped back through an inlet at the lower end.

L. A. Coles.

Production of artificial fogs [from phosphorus]. A. V. JERNBERG (U.S.P. 1,665,267, 10.4.28. Appl., 14.7.26. Swed., 22.7.24).—A continuous supply of melted phosphorus subjected to fluid pressure is passed through a perforated device, and the finely-divided material so produced is delivered to the open air.

F. G. CLARKE.

Production of helium from natural gas. P. E. HAYNES, ASST. to LINDE AIR PRODUCTS CO. (U.S.P. 1,664,412, 3.4.28. Appl., 7.8.19).—Natural gas is compressed, cooled, and expanded to liquefy the greater portion of the hydrocarbons, the liquid is rectified to recover dissolved helium, and the mixed gases are further cooled without compression to liquefy progressively the other constituents, which are collected separately and utilised separately in progressively cooling the residual gas until it consists of practically pure helium. A. R. POWELL.

Concentration or purification of caustic soda or other suitable material. C. F. HAMMOND, Assr. to W. SHACKLETON (U.S.P. 1,668,504, 1.5.28. Appl., 4.9.26. U.K., 5.9.25).—See B.P. 265,252; B., 1927, 251.

Separation of compounds [nitrates] of potassium from compounds [nitrates] of aluminium. H. J. FALCK, ASST. to NORSK HYDRO-ELEKTRISK KVAEL-STOFAKT. (U.S.P. 1,667,968, 1.5.28. Appl., 5.1.25. Norw., 27.2.24).—See B.P. 230,045; B., 1925, 758.

Production of alkaline-earth sulphides from alkaline-earth sulphates. T. LICHTENBERGER and K. FLOR (U.S.P. 1,667,423, 24.4.28. Appl., 4.3.26. Ger., 8.5.25).—See B.P. 251,942; B., 1927, 75.

Manufacture of arsenates of manganese. W. L. TANNER, Assr. to GRASSELLI CHEM. Co. (Re-issue 16,933, 10.4.28, of U.S.P. 1,591,795, 6.7.26).—See B., 1926, 743. Catalytic combustion of ammonia-oxygen mixtures. I. W. CEDERBERG (U.S.P. 1,666,958, 24.4.28. Appl., 8.12.24. Ger., 4.12.23).—See B.P. 225,821; B., 1925, 589.

Aluminium chloride from sludge (U.S.P. 1,665,406). —See II. Formic acid (U.S.P. 1,666,437).—See III. Gases treated in electric arcs (B.P. 263,859 and 288,056).—See XI. Fumigating mixtures (B.P. 271,514).—See XXIII.

#### VIII.—GLASS; CERAMICS.

Influence of iron oxide on the properties of glass. S. English, H. W. Howes, W. E. S. TURNER, and F. WINKS (J. Soc. Glass Tech., 1928, 12, 31-44).-In a series of glasses of the general molecular formula 6SiO<sub>2</sub>,(2-x)Na<sub>2</sub>O,xFe<sub>2</sub>O<sub>3</sub>(FeO), although the iron had been added to the batch as ferric oxide, there was a ratio of ferrous oxide to total iron, as ferric oxide, varying between 9.4% and 21.6%. Melting of the batches presented no difficulty over the range prepared  $(2 \cdot 91 - 26 \cdot 35\%)$  of iron oxides as  $Fe_2O_3$ , but the higher members gave a heavy boil in the later melting stages. Little attack by the glass was observed on the sillimanite pot used for melting. The density of the glasses increased continuously with increase of iron oxide content, and from the data the specific volume constants for ferrous and ferric oxides were deduced as 3.0 and 4.8, respectively. Annealing temperatures varied somewhat irregularly, but indicated that replacement of sodium oxide by ferric oxide increased, replacement by ferrous oxide decreased, the annealing point. There was, in general, a diminution of linear thermal expansion when soda was replaced by iron oxide, but the values were affected by the relative proportions of ferrous and ferric oxides present. In the thermal expansion curves, in addition to the upper and lower critical points, two change points occurred in the normal region, the first at 130-140°, the second at 240-250°, and at each there was an increase of the linear coefficient of 8-15%. A. COUSEN.

Relationship between chemical composition and the resistance of glasses to the action of chemical reagents. II. Glasses containing iron oxides. V. DIMBLEBY and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 52-57).—The chemical durability of the series of soda-iron oxide-silica glasses previously described (cf. preceding abstract) was determined and compared with that of other glasses previously tested (B., 1927, 12). Resistance to attack by water, hydrochloric acid, and sodium carbonate progressively increased as iron oxide replaced soda, but the resistance continuously decreased in the same circumstances when caustic soda was the solution employed.

#### A. COUSEN.

Casing of colourless by cobalt blue glass. I. Thermal expansions. W. E. S. TURNER and F. WINKS. II. Setting rate. S. ENGLISH and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 57—74, 75—82).— I. Increasing quantities of cobalt oxide were incorporated in a series of soda-lime glasses up to 0.49%, and in a potash-lead oxide series up to 0.9%. Thermal expansions were determined in a specially designed apparatus, details of which are given. In the soda-lime series the expansion was but little altered by the introduction of the colouring agent, whilst upper and lower annealing temperatures suffered a maximum variation of only about 5°. The total variation of expansion of the lead series was little more than 3%, but the cobalt affected the upper and lower annealing temperatures to a greater degree than with the lime glasses. Thermal expansion measurements of ruby, green, and blue commercial casing glasses, as compared with the parent glass, indicated that ruby deviated most in expansion rate in the normal region, whilst the green glass differed most in its upper and lower annealing temperatures. In practice, the ruby glass was most difficult to flash, proving the decisive factor to be coefficient of expansion.

II. Increasing amounts of cobalt caused the glasses to set more quickly in working. Viscosity measurements over the range 1400° to 700° in the case of the lime glasses gave lower values for the coloured members than for the parent glass throughout the range, whilst, below 750°, members with higher cobalt content set with greater rapidity than the colourless glass. Temperature measurements of the surface and interior, during cooling, indicated that the important factor inducing quicker setting in practice in the case of the blue glasses was the greater radiating power which they possessed. A. COUSEN.

Calculation of glass constants on the basis of recent investigations. E. ZSCHIMMER (J. Soc. Glass Tech., 1928, 12, 82-118).-The best method of expressing the composition of glass, in order to relate this to its physical properties, for commercial purposes, is that of oxide percentage, and for systematic comparison it is most convenient to set out the continuous succession of all possible percentage combinations in terms of the substituents (or permutants) used. Most recent work has followed the permutant method, which is described and explained. From this point of view, an examination of modern research on the physical properties of glass was commenced, properties first treated being the devitrification constants of sodalime-silica glasses, the working viscosity of these glasses at high temperature, the effect of magnesia and alumina on the working properties of soda-lime-silica and of boric oxide on soda-silica glasses. A. COUSEN.

Ultra-violet light transmission of some colourless bottle glasses. D. STARKIE and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 27–29).—Light transmission in the region 6500 to 2500 Å. was measured in the case of glasses from samples used as food containers. There was a very definite increase of transmission in the ultra-violet with decrease of iron oxide content of the glass. A. COUSEN.

Analysis of opal and alabaster glasses. J. D. CAUWOOD, J. H. DAVIDSON, and V. DIMBLEBY (J. Soc. Glass Tech., 1928, 12, 7—16).—An account of the methods of analysis adopted for the determination of constituents, particularly the opacifying agents, in opal and alabaster glasses. The agents considered were fluorine compounds, phosphates, tin oxide, and alkali sulphates and chlorides. A. COUSEN.

Analysis of opal glasses. W. SINGLETON and R. C. CHIRNSIDE (J. Soc. Glass Tech., 1928, 12, 18-24). —A description of methods considered best, from the authors' experience, for the analysis of opal glasses. A. COUSEN.

# PATENTS.

Manufacture of glass. E. E. FISHER, Assr. to G. W. BATCHELL (U.S.P. 1,665,693-4, 10.4.28. Appl., [A] 5.4.27, [B] 6.4.27).—(A) A transparent glass, capable of decolorisation by selenium, is prepared from a batch containing a substantial proportion of barium sulphate. (B) A batch for glass-making contains, as a primary component, barium sulphide. A. COUSEN.

Oven or kiln for firing pottery and other ware. F. BENNION, H. J. PLANT, and J. B. CLARKE (B.P. 287,693, 22.11.27).—A circular kiln with a number of fire-mouths has a perforated tube or conduit in communication with a central hole in the oven floor leading to the chimney, the upper or inlet end of the tube extending nearly to the crown or is joined to it.

W. G. CAREY.

Ovens or kilns for firing tiles, bricks, etc. J. and J. W. Moss (B.P. 288,383, 15.1.27).—Alternate firemouths in a circular oven communicate with up-flues between the wall of the kiln and a lining, and the flame, after striking the crown, is drawn downwards through a conduit in the centre of the floor and extending to about half the height of the oven. The other firemouths communicate with down-flues leading to openings in the bottom of the oven lining.

W. G. CAREY.

Manufacture of porous refractory bricks etc. I. SETTERBERG (B.P. 281,254, 8.11.27. Swed., 26.11.26).— Finely-powdered refractory clay, chamotte, kaolin, quartz, etc. is mixed with water and a substance which reacts with water to produce gas, *e.g.*, aluminium powder, calcium, zinc, calcium carbide, etc., and when evolution of gas has ceased the plastic dough is formed into blocks and burnt. W. G. CAREY.

Tank furnaces for the manufacture of glass. GEN. ELECTRIC CO., LTD., W. W. WARREN, and J. F. HVSLOP (B.P. 288,668, 13.12.26).

#### IX.—BUILDING MATERIALS.

Effect of temperature and time of burning on the properties of high-calcium lime. K. W. RAY and F. C. MATHERS (Ind. Eng. Chem., 1928, 20, 415-419).-Samples of limestone were burned at temperatures varying from 900° to 1300° and for periods from 2 to 10 hrs. The resultant limes were tested for rates of slaking (time required to attain maximum temperature), rate of settling of the hydrates, plasticity, and time required for thickening of the slurry. The effect of increasing time of burning was small, but in the same direction as that of increase of temperature of burning. Increase of temperature reduced the rate of slaking and decreased the plasticity of the putty. A medium temperature of burning gave the slowest settling and greatest volume of settled suspension, also the slowest thickening of the slurry. The plasticity of the putty made by adding water to the dry hydrate is usually less than of that made direct from quicklime. In this case the most plastic putty is given by quicklimes made at 1100-1250°. In

general, a temperature of about 1100° seems most desirable considering all factors. C. IRWIN.

#### PATENTS.

Rotary tubular kilns for treating cement etc. O. BOUZIN (B.P. 288,114, 8.12.27. Addn. to B.P. 266,939; B., 1927, 333).—The refractory lining of the kiln immediately behind the high-temperature zone is also provided with a ring-shaped projection, and the tubular clinker cooler is fitted internally with similar projections of refractory material in the upper portion of the cooler and of metal in the lower portion.

W. G. CAREY.

Manufacture of pavements. R. HADDAN. From WEST PROCESS PAVEMENT Co. (B.P. 287,799, 5.10.27).— A soft asphalt or flux is heated with pulverised hard asphalt and sand, with or without a filler.

L. A. Coles.

Treatment of rock asphalt. W. P. BENTLEY (U.S.P. 1,664,846, 3.4.28. Appl., 17.2.28).—A bituminous flux is added to the rock asphalt, previously reduced to a usable size, in amount insufficient in itself to fill the voids between the particles, but just sufficient to do so when added to the naturally contained bitumen in the rock. A. B. MANNING.

Production of aqueous emulsions of bituminous material. H. MATTI (Swiss P. 119,219, 27.5.25).— The emulsions are prepared by adding hot water to mixtures containing bituminous material, a fatty oil which has been treated with concentrated sulphuric acid, and a sufficient quantity of an alkaline reagent to neutralise the sulphonic acids and free fatty acids.

L. A. COLES.

Manufacture of a bituminous product. E. HUTZENLAUB, Assr. to P. LECHLER (U.S.P. 1,665,881, 10.4.28. Appl., 26.6.26. Ger., 8.7.25).—A stable paste of a creamy consistency and non-miscible with water at ordinary temperatures consists of a tarry substance, water, and an emulsifying agent, the latter being insufficient in quantity to form an aqueous emulsion. F. G. CLARKE.

**Preservation of wood.** J. HIMMELSBACH (F.P. 604,897, 20.10.25. Ger., 20.10.24).—Fresh wood, before impregnation with, *e.g.*, mercuric chloride solution, is thoroughly wetted with water and subjected for about 7 hrs. under reduced pressure to the action of the moist vapour of neutral solvents, *e.g.*, methyl alcohol or carbon tetrachloride, and formaldehyde, at a maximum temperature of 80—85°. L. A. COLES.

Production on wood of a protective coating impervious to water. GEBR. HIMMELSBACH A.-G. (F.P. 600,290, 2.7.25. Ger., 15.1.25).—Wood is painted with a homogeneous mixture of bitumen or pitch with a solvent, *e.g.*, carbon tetrachloride. L. A. COLES.

Mixing machines [for concrete]. J. M. VER MEHR (B.P. 288,365, 5.1.27).

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

Blast-furnace data and their correlation. E. C. Evans and F. J. BALLEY (Iron and Steel Inst., May, 1928. Advance copy. 64 pp.).—From the operating

results of more than 120 blast furnaces, data in relation to output and fuel consumption have been correlated on a common basis. Other factors being equal, the greater the effective time of contact between the reducing gases and the ore before the hearth is reached the lower is the fuel consumption. Among the factors influencing the effective time of contact are (a) slow driving of the furnace, (b) preparation of the burden before charging, (c) methods of charging, (d) furnace design, and (e) quality of coke. Over a range of operation including a wide variation in furnace design, composition of ores, metals, and slags, the mean operating hearth temperatures are approximately constant, *i.e.*, metal  $1450^{\circ}\pm50^{\circ}$ , slag  $1500^{\circ}\pm100^{\circ}$ . The basic heat loss from the hearth zone is represented as necessitating the combustion of K/D lb. of carbon per sq. ft. of hearth area, where K is a constant and D the diameter of the hearth. By development an average equation for all the furnaces examined became  $C_1 + C_2 = 500/D +$ 0.56I + 0.28S, in which  $C_1$  is the carbon gasified at tuyères,  $C_2$  the sensible heat in blast in terms of carbon, I amount of iron (lb.) made, S the amount (lb.) of slag made, all figures being per sq. ft. of hearth area per hr.; in 80% of the cases examined coke consumptions calculated from these factors came within 10% of the actual amount. The amount of carbon required for external heat losses from the hearth is much greater than was generally thought, consequently slow driving is an uneconomical method of obtaining increased time of contact. The hearth losses are inversely proportional to the diameter of the hearth, and the heat consumption in the hearth is directly affected by the quality of the iron as represented by the content of silicon. Improved thermal efficiency, together with a possible increase in the effective rate of reaction between gases and ore, results from the use of dry blast. Constructional and operating details are given for 123 blast furnaces.

C. A. KING.

Comparison of the most important methods employed in the cleaning of blast-furnace gas. V. HARBORD (Iron and Steel Inst., May, 1928. Advance copy. 28 pp.).-Results obtained from comparative tests on dry- and wet-cleaning plants showed that the dust content of blast-furnace gas could be reduced to 0.4 - 0.5 g./m.<sup>3</sup> by wet cleaners with spray towers only and by electrostatic cleaners. Spray towers followed by Theisen disintegrators and Halberg-Beth filtration plants gave the best results, dust being reduced to 0.02 or even 0.004 g./m.<sup>3</sup> after a final spray for engine purposes. General considerations taking into account the maximum heat development and minimum erosion of refractories in relation to the cost of cleaning seem to indicate that a gas containing not more than 0.2 g./m.<sup>3</sup> gives sufficiently good results. The electrostatic plants showed most favourable results on power and working costs, though the capital cost is high; the dust content of the cleaned gas was greater than with disintegrator washers, and the handling of dry dust presented some disadvantages. If due attention is given to the renewal of bags the Halberg-Beth cleaner gives a lower dust content, and its cost of operation is no more than that of the more efficient wet cleaners. Again high capital cost and dry dust are objections. The Kling-Weidlein

plant which depends on gas being passed through wirewool mattresses is regarded only as a primary cleaner in conjunction with some other form, and its cost is too high for a first-stage cleaner. C. A. KING.

Theory of blast-furnace smelting [of iron ores]. F. Wüst (Stahl u. Eisen, 1928, 48, 505-506).-To account for the considerable economy in fuel that follows from the use of a hot blast, the author outlines a new theory of the mechanism of the processes taking place during the blast-smelting of iron ores. For efficient working the depth of the oxidising zone just above the tuyères must be as small as possible; this may be effected by widening the furnace at this part, by the use of an oil spray, by means of a hot blast, or by substituting wood charcoal for coke. The thermal decomposition of carbon monoxide favours the reduction process and a hot blast increases this decomposition. The carbon liberated from this reaction effects the socalled direct reduction of iron, phosphorus, and silicon in the zone just above the tuyères, the indirect reduction higher up the furnace being brought about by hydrogen reduced by the carbon monoxide from the water vapour present. The water formed in the reduction is then again reduced to hydrogen by further quantities of carbon monoxide. The iron formed in the upper zones of the shaft takes up the greater part of the impurities found in the pig iron by cementation and not by direct reduction from the slag. The analysis of the flue gases or of gases taken from any part of the shaft is not sufficient to permit of any inferences being drawn as to the working of the blast furnace. Experimental details on which the theory is based are withheld for future publication. A. R. POWELL.

New plant of the Appleby Iron Co., Ltd. -A. CROOKE and I. THOMSON (Iron and Steel Inst., May, 1928. Advance copy. 35 pp.).—A general survey of the plant, including blast furnaces, open-hearth furnaces, power and blowing plant, rolling, slabbing, and plate mills, basic slag plant, etc. C. A. KING.

Blast-furnace practice in Natal. J. E. HOLGATE and R. R. F. WALTON (Iron and Steel Inst., May, 1928. Advance copy. 26 pp.).—A description of the materials and operation of the only blast furnace in S. Africa. One of the principal ores is a carbonate ore from Prestwick containing 5% of free carbon which renders the ore self-calcining. Northern Natal possesses the only extensive areas of coking coal in S. Africa, and a dolomite has been used from near Johannesburg (180 miles), replacing limestone previously obtained from Bechuanaland (400 miles). Costs of production are controlled largely by the high rates of carriage. C. A. KING.

Influence of varying proportions of scrap and pig iron on the economical working of the openhearth process [for the manufacture of steel]. G. BULLE (Stahl u. Eisen, 1928, 48, 329—338, 368—370). —Comparative data of the costs and working results of six steelworks using various ratios of pig iron to scrap in the Siemens-Martin furnace are tabulated and briefly discussed. A. R. POWELL.

Peculiar graphite crystallisation in pig iron and cast iron. H. PINSL (Stahl u. Eisen, 1928, 48, 473-477).—Fifteen photomicrographs of peculiar forms in which graphite has crystallised in pig iron and cast iron are reproduced, and the factors which have probably influenced their formation are briefly discussed. The graphite-ferrite eutectic may frequently be found along veins of graphite segregations on the outer layers of very large castings. In pig iron with a high phosphorus content, the graphite veins often consist of numerous thin lamellæ of graphite separated by thin layers of iron phosphide, and sometimes a gradual transition from a vein of steadite to one of graphite may be observed. The mechanism of the formation of graphite nuclei and the structure of temper carbon are briefly discussed. A. R. POWELL.

Twin-like crystals in annealed a-iron. H. O'NEILL (Iron and Steel Inst., May, 1928. Advance copy. 9 pp.).-A metallographic examination has been made of certain small ferrite grains embedded in a large single crystal of iron prepared by the method of straining and annealing. Pressure figures, etch pits, and the form of the grain boundaries indicated that the grains were twins (relative to the large crystal), the twins being bounded by (112) planes, the twinning being of the fluorite type. They are regarded as true annealing twins produced by cold-work and recrystallisation.

W. HUME-ROTHERY.

Rate of dissolution of graphite in molten ironcarbon alloys. F. SAUERWALD and A. KORENY (Stahl u. Eisen, 1928, 48, 537-540).-The rate of dissolution of graphite in cast iron has been determined at 1255° and at 1350°. In both cases the rate approached closely to that calculated from the formula of Noyes, Withney, and Nernst ("Theoretical Chemistry," Stuttgart, 1926). It is proportional to the degree of saturation of the molten metal, the contour of the graphite particles, the temperature, and the time of contact. The amount of graphite dissolved in a given time increases rapidly with the temperature, and that dissolved at a given temperature increases at first rapidly with the time, then more slowly, the curve being hyperbolic in form.

#### A. R. POWELL.

Graphite in cast iron and its influence on the tensile strength. P BARDENHEUER and K. L. ZEYEN (Mitt. K.-W.-Inst. Eisenforsch., 1928, 10, 23-53; Stahl u. Eisen, 1928, 48, 515-519) .- The higher the temperature at which cast iron is heated the smaller is the amount of graphite which separates on cooling; cast iron even with a high silicon content has a white fracture after heating above 1500°. At very high temperatures (e.g., 1700-1800°) air or occluded gases and sometimes the walls of the crucible cause the separation of graphite nuclei, and the metal solidifies wholly or partly as grey cast iron (cf. Pivovarsky, B., 1925, 805; Hanemann, B., 1927, 678). The tensile strength of cast iron is very appreciably increased by casting in chill moulds and annealing at 850-900° for 6 hrs. owing to the finer state of subdivision of the graphite compared with that in sand castings. The ground mass in sand castings is chiefly pearlite, and that in annealed chill castings chiefly ferrite; hence it appears that the state of subdivision of the graphite is the factor which determines the mechanical properties of cast iron, and that the nature

of the ground mass has little influence. Numerous photomicrographs of the structure of cast iron heated at temperatures between 1200° and 1800° and cooled at varying rates are included. A. R. POWELL.

Influence of nickel on iron-carbon-silicon alloys containing phosphorus. A. B. EVEREST and D. HANson (Iron and Steel Inst., May, 1928. Advance copy. 23 pp.; cf. B., 1927, 782) .- The influence of up to 3% of nickel on synthetic iron alloys containing 3.5% C, 1.2 and 2.5% Si, and over a range 0.2 to 1.2% P has been investigated ; the experiments were afterwards repeated with a commercial refined iron containing 3.05% C and 1.87% Si. In all cases it was found that phosphorus increases the tendency of the iron to chill; this chilling effect may be counteracted by the addition of small quantities of nickel, and is less pronounced in the high-silicon irons. Phosphorus increases the hardness of the iron; in the absence of chill, nickel produces its normal hardening action. Phosphorus impairs the machinability of the iron. Both nickel and phosphorus have a refining effect on the graphite in the iron, but with a high phosphorus content the effect of nickel is small and inappreciable when 1.2% P is present.

M. E. NOTTAGE.

Annealing transformer sheet [iron]. M. VON Moos, W. OERTEL, and R. SCHERER (Stahl u. Eisen, 1928, 48, 477-485).-Recrystallisation tests on 4% silicon-iron for transformer sheets showed that the presence of oxygen has almost as great an effect on the wattage losses as the presence of carbon; it prevents the formation of an even, regularly oriented crystal structure during annealing and restrains the grain growth. The harmful action of carbon and oxygen in transformer sheets may be overcome by annealing in hydrogen, which reduces the oxides and produces a sheet which, in the absence of other harmful constituents, has a very low wattage loss. A period of 1 hr. at 900-1000° suffices for most sheets; longer annealing at 1000° produces slightly higher losses. A. R. POWELL.

Resistance of over-stressed wrought irons and carbon steels to salt-water corrosion. J. N. FRIEND (Iron and Steel Inst., May, 1928. Advance copy. 12 pp.).-Bars of wrought iron and carbon steels were deformed by stretching, twisting, or compression, and were then immersed for one or two years in salt water, made by dissolving rock salt in water to give a solution resembling sea water. To examine the effect of surface conditions the bars were immersed both in the natural state after deformation, and also after machining to shape. Under these conditions the corrodibility of wrought iron is not increased by stretching even to rupture, by twisting, or by compression up to 30%. The corrodibility of carbon steels (0.025-1.35% C)is not enhanced by stretching even to rupture. Torsion and compression do not affect the corrodibility of 0.11%carbon steel, but with 0.20% carbon steel the corrodibility is slightly increased. Wrought irons and carbon steels (0.025-0.32% C) deformed by stretching, and then tested in the natural condition without machining the surface, were in nearly every case less corroded than the same material in the unstressed state; in the case of the 0.11% carbon steel the loss on weight of the

stretched bar was  $20 \cdot 4\%$  less than that of the unstressed bar. W. HUME-ROTHERY.

**Properties of materials at high temperatures. III. "Creep " of Armco iron.** H. J. TAPSELL (Dept. Sci. Ind. Res., Eng. Res., Spec. Rep. No. 6, 1928. 11 pp.; cf. Tapsell and Clenshaw, B., 1927, 525).— "Creep" tests made on Armco iron at 136° under a load of 25.7 tons/in.<sup>2</sup> showed that creep had practically ceased in 22 days. Stress-strain relations of specimens which had been creep-tested at 150°, 237°, 325°, and 390° indicated that plasticity progressively decreased and the new limit of proportionality acquired by the material was at all temperatures much greater than the value of the original material. It is considered that a hardening process occurs during creep similar to that taking place in the neighbourhood of the slip planes during a fatigue test. C. A. KING.

Fatigue-resisting properties of 0.17% carbon steel at different temperatures and at different mean tensile stresses. H. J. TAPSELL (Iron and Steel Inst., May, 1928. Advance copy. 13 pp.; cf. B., 1927, 525).-Fatigue limits at different mean tensile stresses and at temperatures up to 500° were determined in a Haigh machine under direct pulsating stresses applied at the rate of 2400 cycles/min. for 107 cycles. For practical use, the results obtained must be correlated with a knowledge of the yield-points and limiting creep stresses at different temperatures. It is shown that the endurance of the steel above 300° is wholly dependent on the rate of alternations applied ; at air temperature the rate has little, if any, effect. Large ranges of stresses can be applied at all temperatures. The superior limit of ranges of stress applied at 2400 cycles/ min. for 10<sup>7</sup> cycles can exceed the ordinary ultimate strength values. The "practical" fatigue limits under different mean tensile stresses do not appreciably alter for temperatures up to 200° or 250°, and they are dependent on the yield-point of the material at the specified temperature. Above about 200° the practical fatigue limits are dependent on a suitable proof M. E. NOTTAGE. stress.

Rapid normalising of overstrained steel. W. E. WOODWARD (Iron and Steel Inst., May, 1928. Advance copy. 10 pp.).—The elastic properties and resistance to fatigue of specimens of mild steel which have been seriously overstrained can be rapidly and completely restored by the passage of a direct current of about 600 amp. through the material so as to heat it above the upper critical point. Normalisation by this method produces remarkably uniform results. An alternating current of about 2000 amp. at 6 volts can also be used, but the results are less uniform. The results obtained by both methods compare very favourably with those obtained by the ordinary process of normalising.

#### M. E. NOTTAGE.

Heat-resisting steels. II. Mechanical properties. W. H. HATFIELD (Iron and Steel Inst., May, 1928. Advance copy. 22 pp.; cf. B., 1927, 445).— To compare the effects of the introduction of special elements into steel on its mechanical strength at high temperatures, tensile tests were made at 800°. For ordinary steels, 7 and 8 tons/in.<sup>2</sup> indicates the limiting maximum stress value at 800°; high-speed tool steels give slightly higher values. Silicon-chromium heatresisting steels are already softened at 800°. With steels containing 0.07 and 0.09% C, the presence of 14 and 18% of chromium, respectively, has no influence on the resistance to softening at high temperatures; increasing the carbon raises the strength. The addition of nickel has no influence even with a high carbon content, but nickel and chromium together increases the strength to a moderate extent. The introduction of tungsten results in a marked increase in strength when added together with nickel and chromium; 14% of chromium is about the requisite amount, but the amount of nickel may vary within wide limits. The addition of molybdenum, and to a lesser degree silicon, strengthens the iron-nickel-chromium steels at high temperatures. Carbon plays an important part, and there is an optimum percentage for the maximum strength in each combination in the alloys. It is probable that these results would be modified by varying the heat-treatment accorded to the material. The author discusses the phenomena accompanying deformation and rupture at high temperatures, and the effect of added elements on the steel from the point of view of the space lattice.

M. E. NOTTAGE.

Structure of the iron-chromium-carbon system. A. WESTGREN, G. PHRAGMÉN, and T. NEGRESCO (Iron and Steel Inst., May, 1928. Advance copy. 18 pp.).-X-Ray analysis shows that the iron-chromium alloys at ordinary temperatures consist of an unbroken series of solid solutions, the size of the unit cube increasing regularly from a = 2.861 Å. for pure iron to 2.878 Å. for chromium. In the ternary iron-chromium-carbon system the following phases are met with : (1)  $\alpha$ -Solid solution (body-centred cubic structure). (2) y-Solid solution (face-centred cubic structure). (3) Cementite (Fe,Cr)<sub>3</sub>C derived from ordinary cementite Fe<sub>3</sub>C by the substitution of chromium for iron; the chromium content may rise to rather more than 15%. The lattice dimensions of cementite decrease slightly as the iron is replaced by chromium. (4) Cubic chromium carbide, probably (Cr,Fe)<sub>4</sub>C derived from Cr<sub>4</sub>C (cf. Westgren and Phragmén, B., 1922, 418; 1924, 518) by the substitution of iron for chromium up to a maximum of about 25%, the lattice parameter decreasing with rising percentage of iron. (5) Trigonal chromium carbide (Cr,Fe)<sub>7</sub>C<sub>3</sub>, derived similarly from Cr<sub>7</sub>C<sub>4</sub> (cf. Westgren and Phragmén, loc. cit.), in which the iron content may rise to 55%, the lattice parameter decreasing with rising percentage of iron. (6) Orthorhombic chromium carbide, (Cr,Fe)<sub>3</sub>C<sub>2</sub>, in which only a few per cent. of chromium may be substituted by iron. None of the above phases is a true double carbide, *i.e.*, a carbide requiring both chromium and iron atoms for its formation. In an annealed chromium steel such as is used for ball bearings almost all the chromium is in the cementite, and the constituent commonly called " double carbide " is simply ternary cementite in large crystals. In stainless steel the carbide is cubic chromium carbide saturated with iron. The equilibrium diagram of the ternary system is discussed in the light of these results.

W. HUME-ROTHERY.

Heterogeneity of steel ingots. Report II (Iron and Steel Inst., May, 1928. Advance copy. 147 pp.; cf. B., 1926, 490, 883).—The design of moulds and the effect of inclusions in steel on the heterogeneity of ingots are discussed, and macrographic and analytical representations of segregation in ingots of nickel, nickelchromium, and nickel-chromium-molybdenum steels are given. Interim reports on the density and viscosity of molten steel, and on the freezing and melting ranges of various types of steel are included. C. A. KING.

Chromium steel rails. T. SWINDEN and P. H. JOHNSON (Iron and Steel Inst., May, 1928. Advance copy. 14 pp.).—The advisability of obtaining increased resistance of rails to wear by abrasion by increasing the carbon range and reducing the maximum manganese permitted is considered not beyond doubt, and from results of trials of steels containing up to 2% Cr, a chromium content of approximately 1% was adopted as being most satisfactory, other constituents being about 0.5% C, 0.8% Mn, 0.22% Si, 0.03 S, and 0.04% P. The effect of increasing the carbon content from 0.51%to 0.61% was to increase the maximum stress from  $59 \cdot 2$  to  $65 \cdot 12$  tons/in.<sup>2</sup>, and the Brinell hardness value from 269 to 293. As rolled, the chromium steel shows a dense sorbitic pearlite matrix with a fine broken network of ferrite, the effect of the chromium addition being to reduce the proportion of the soft ferrite constituent and to emulsify the carbide, both conditions tending to contribute to the greater hardness and toughness of the chromium steel. C. A. KING.

Application of metallography in improving the quality [of iron and steel]. H. MEYER (Stahl u. Eisen, 1928, 48, 506—515).—Many examples are given with photomicrographs of various faults which can be found in iron and steel castings and forgings by the use of the microscope and by mechanical testing. The effect of transverse crystallisation, of segregation, and of blowholes on the properties of the metal is discussed briefly with reference to certain specific examples.

#### A. R. POWELL.

Properties of nickel steels, with special reference to the influence of manganese. J. A. JONES (Iron and Steel Inst., May, 1928. Advance copy. 36 pp.).-The transformation points, mechanical properties, and microstructures of nickel steels containing 0.2-0.55% C and 3-12% Ni have been examined with special reference to the effects of manganese and chromium. In nickel steels the Ac1 point is lowered by about 10° for each 1% of nickel, whilst with from 3 to 5% of nickel the Arl point is lowered by from  $25^{\circ}$  to  $30^{\circ}$  for each 1%of nickel, the exact value depending on the manganese and carbon contents; the rate of cooling used was 4°/min. Consequently with low manganese content there is no advantage in increasing the nickel beyond 6% on account of the limitation on the tempering temperature imposed by the lowering of Ac1. Little effect on the critical ranges is produced by less than 0.8% of manganese; an increase in manganese content from 0.4 to 0.8% in steels with 4% Ni lowers the Ac1 and Ar1 by 3° and 4°, respectively, the rate of cooling being 4°/min. Chromium produces a decided rise in Ac1, but has little effect on Ac3, Ar3, or Ar1. On increasing the

manganese content of steels containing 3-4% Ni, a sudden improvement in mechanical properties takes place at a definite manganese content which increases with the size of the section treated; in no case should less than 0.5% of manganese be present. The mass effect met with in quenching these alloys has been studied in detail. W. HUME-ROTHERY.

Effect of silicon on tungsten magnet steel. J. SWAN (Iron and Steel Inst., May, 1928. Advance copy. 9 pp.) .- The "spoiling" or loss of coercive force on annealing above the Ac point of a tungsten magnet steel of normal composition is due to the gradual formation and segregation of free tungsten carbide, which is only slightly soluble in austenite at the ordinary hardening temperatures, so that the steel loses in available carbon and tungsten content. By increasing the silicon content to 0.25% or above this segregation can be retarded, but not prevented, as is shown by the fact that loss of coercive force still occurs on annealing. Silicon, up to 0.5%, has, if anything, a slightly beneficial effect on the magnetic properties of the steel; 1.0% of silicon reduces the loss of coercive force on annealing, but has a harmful effect on the general magnetic properties. It is probable that less loss due to ageing is shown by steels with a high silicon content.

M. E. NOTTAGE.

Iodometric determination of vanadium in special steels and in ferrovanadium. K. ROESCH and W. WERZ (Z. anal. Chem., 1928, 73, 352-355).-For the determination of vanadium in a special steel, 3-5 g. are dissolved in 30-50 c.c. of hydrochloric acid and sufficient nitric acid to effect complete oxidation, the solution is evaporated to 25 c.c., the tungstic acid and silica are filtered off, and the iron is removed by the etherextraction method. The aqueous layer is evaporated to dryness and the residue dissolved in 20 c.c. of 1:1 nitric acid. Manganese sulphate is added, followed by ammonia to precipitate manganese vanadate and chrom-The precipitate is collected, washed, dried, ate. ignited in an iron crucible, and fused for 10 min. with 3-4 g. of sodium hydroxide in an atmosphere of hydrogen. After cooling, the sodium vanadate is extracted with hot water, the insoluble chromic oxide filtered off, and the filtrate acidified with 75 c.c. of phosphoric acid  $(d \ 1 \cdot 7)$ . To ensure that all the vanadium is present as vanadate the solution is boiled for 15 min. with 0.5 g. of ammonium persulphate. The cold solution is treated with potassium iodide and the liberated iodine titrated with thiosulphate (cf. Heczko, A., 1926, 1020). For the analysis of ferrovanadium, 1 g. is fused in a nickel crucible with 10 g. of sodium hydroxide. the product leached with water, and an aliquot part of the filtrate treated with phosphoric acid and potassium iodide as above. A. R. POWELL.

Rapid determination of gases in metals, especially oxygen in steel. W. HESSENBRUCH and P. OBER-HOFFER (Arch. Eisenhüttenw., 1927-8, 1, 583-603; Stahl u. Eisen, 1928, 48, 486-487).—The efficient removal of occluded and combined gases from steel requires a temperature of 1500-1600° and a high vacuum, and the only suitable container for the specimen during the test is a graphite crucible which has previously

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been degassed at  $1500-1600^{\circ}$  in vacuo. The apparatus used comprises a high-frequency induction furnace, a pump for producing a high vacuum, and the usual gasanalysis apparatus for the determination of carbon monoxide, carbon dioxide, hydrogen, and nitrogen. The graphite crucible is placed inside a magnesia crucible which is inside a closed cylindrical, silica container connected to the vacuum pump and placed inside the coil of the furnace. The specimen for analysis should consist of a solid block of the metal and not of turnings. Usually 45-60 min. at  $1600^{\circ}$  suffices to reduce all oxides and expel occluded gases. When more than 0.05% S is present in the metal errors may be caused by the formation of sulphur dioxide or hydrogen sulphide in the test. A. R. POWELL.

Relative corrodibilities of ferrous and non-ferrous metals and alloys. I. Results of four years' exposure in the Bristol Channel. J. N. FRIEND (Inst. Metals, March, 1928. Advance copy. 23 pp.).-Bars of ferrous and non-ferrous metals were exposed to sea action in the Bristol Channel for four years, under conditions in which they were totally immersed for slightly more than 93.5% of the total time. Results are given for 14 non-ferrous, and 4 ferrous bars, losses in weight, depths of pitting, reduction in diameter, and fall in tensile strength having been measured. The detailed behaviour of each bar is described. Of brasses, both Muntz metal and Naval brass underwent dezincification, but not Nergandin brass or screw metal; these last two brasses corroded very uniformly with no local pitting. Aluminium was badly pitted, some holes penetrating to a depth of  $5 \cdot 8$  mm, in a bar of  $2 \cdot 87$  cm, diam. Taking pitting, loss in tensile strength, and total loss in weight into account, the non-ferrous metals are arranged in the following order of merit : English common ingot tin, high-grade pure tin, commercial nickel, antimonial lead, commercial soft lead, screw metal (Cu 60.02%, Zn 38.61%, Pb 1.37%, As < 0.05%), nickel-copper (1.75 Ni), arsenical copper (0.45% As), ordinary copper, Nergandin brass (Cu 70.05%, Zn 27.93%, Pb 2.02%), Naval brass, Muntz metal, commercial zinc, and commercial aluminium. W. HUME-ROTHERY.

Annealing of non-ferrous metals in the electric furnace. R. M. KEENEY (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 10 pp.).-In a discussion of the relative merits of oil, gas, and electric furnaces for the annealing of non-ferrous metals, it is emphasised that the over-all cost of the finished product should be the deciding factor. The cost of thermal energy alone does not give a true basis of comparison, since items such as cost of labour, maintenance, rejects, pickling, etc. may vary greatly according to the source of heat adopted. Data are given for the electric furnace annealing of brass and copper tubing and nickel-silver stampings at Connecticut, and the saving in over-all costs of these processes as compared with oil-fired furnaces is estimated. It is concluded that the continued use of oil-fired furnaces in rolling mills is largely due to failure to consider economies in over-all cost which the introduction of electric furnaces might produce in this field. H. J. T. ELLINGHAM.

Changes of a nickel-copper alloy in the vapour of superheated water at about 350–400°. J. F. SAFFY

(Compt. rend., 1928, **186**, 1116—1118).—A plate 3 mm. thick of an alloy of nickel  $(68 \cdot 6\%)$ , copper  $(28 \cdot 9\%)$ , manganese  $(1 \cdot 6\%)$ , and carbon  $(0 \cdot 2\%)$ , which was originally pliable, broke with a deep crack after 2 weeks in steam at 500° when bent at an angle of 180°. After 7 weeks at 400°, it broke when folded to 90—120°, and after 8 weeks at 350°, cracks of varying depths appeared when it was bent to 180°. In some cases traces of a white oxide were noted, but there was little or no gain in weight. J. GRANT.

Behaviour of metals and alloys during hotforging. W. L. KENT (Inst. Metals, March, 1928. Advance copy. 18 pp.) .- Small cylindrical specimens of tin, lead, zinc, aluminium, copper, and brass (both 70:30 and 60:40, with and without small amounts of lead) were forged with a standard blow of 50 ft.-lb. at temperatures up to the m.p. The deformation produced, and the Brinell hardness of the samples before and after deformation, were measured. When a material is worked at high temperature it strain-hardens in much the same way as, but to a slightly less extent than, at normal temperatures; the decrease is, however, small. With copper at 600°, recrystallisation after deformation is practically instantaneous and continuous forging is possible, but with aluminium residual hardness remains even after forging at 650°, although here the actual softness of the metal aids hot-forging. The forgeability of 70:30 brass increased but little up to 750°, but that of 60:40 brass increased steadily from 400° to 650°, and then rapidly to the m.p. The presence of lead caused cracking in brasses forged from 350° upwards, but in 60:40 brass the cracks disappeared at 700° and the lead was beneficial. In general, both forging and Izod notched-bar impact tests are necessary to give a true measurement of malleability, neither test alone being satisfactory. W. HUME-ROTHERY.

Deterioration of lead cable sheathing by cracking, and its prevention. S. BECKINSALE and H. WATERHOUSE (Inst. Metals, March, 1928. Advance copy. 25 pp.).-The intercrystalline failure sometimes found in lead cable sheathing has been investigated by laboratory experiments and by the examination of samples which have failed in practice. The failure is a fatigue phenomenon, and test pieces submitted to alternating stresses fail by the development of intercrystalline cracks identical with those occurring in cable sheathing, but similar cracks could not be obtained by any other methods. In agreement with this, failures in practice usually occur only in places where the lead cable is subjected to vibration. The probability of failure can be greatly reduced by using binary or ternary alloys of lead with small quantities of tin, antimony, or cadmium in which the fatigue limit is much higher than in pure lead; alloys containing 0.25% Cd and 1.5% Sn, or 0.25% Cd and 0.5% Sb, are particularly suitable. No evidence could be found for any allotropic transformation of lead between  $-75^{\circ}$  and  $+350^{\circ}$ . The effects of simultaneous corrosion and tensile stress were also investigated, whilst other possible causes of intercrystalline fracture in lead are shown to be untenable.

W. HUME-ROTHERY. Alloys of zirconium. I. T. E. ALLIBONE and C. SYKES (Inst. Metals, March, 1928. Advance copy. 14 pp.).—The microstructures of the alloy systems copper-zirconium to 35% Zr, nickel-zirconium to 55% Zr, and iron-zirconium to 30% Zr have been investigated, the alloys being prepared in a vacuum highfrequency induction furnace which is described. Copper dissolves only about 0.2% of zirconium in solid solution ; the alloys contain a compound Cu<sub>3</sub>Zr (m.p. above 1000°), which forms a eutectic with the copper solid solution at 964° and 12.5% Zr. The solid-solubility of zirconium in nickel is less than 0.5% at room temperature; the alloys contain two compounds, Ni4Zr and Ni<sub>3</sub>Zr, the former forming a eutectic with the nickel solid solution at 16% Zr. The addition of zirconium to iron lowers the Ar3 change point, but does not affect the Ar2 point. Iron dissolves about 0.3% of zirconium in the solid state at room temperatures; a eutectic alloy is formed at 15% Zr, but the second constituent of this eutectic has not been isolated. The hardness, tensile strength, and ductility of some of the alloys were investigated. W. HUME-ROTHERY.

Speiss and the metals of the platinum group. H. RUSDEN and J. HENDERSON (J. Chem. Met. Soc. S. Africa, 1928, 28, 181-196).-In recovering the platinum metals from certain concentrates on the Rand, the material is smelted to a speiss in which all the platinum metals are collected. A typical speiss contains 19.5% As, 56% Fe, 8% Cu, 8% Ni, 3.3% Pb, and 4% S, with about 100 oz./ton of platinum metals. By smelting this speiss in a cupel bed with 8 pts. of litharge, 1.36 pts. of sodium carbonate, and 0.7 pt. of sand, the platinum metals were obtained in a lead bottom weighing 4 times as much as the original speiss. This could be cupelled to about one third the weight of the speiss treated without serious loss of the precious metals, and by dissolution of the remaining lead in nitric acid a residue containing 13% of platinum metals was obtained. The amount of lead required in the above treatment was considerably reduced by roasting the speiss previous to smelting. Direct electrolysis of the speiss in 5% sulphuric acid yielded an anode slime weighing 26% of the original speiss ; this slime lost 26% of its weight on roasting and, after fusion with iron and a borosilicate slag, gave a new speiss weighing 25% of the slime, together with a matte and slag practically free from the platinum metals. Electrolysis of the new speiss yielded 90% of the platinum metals in the anode slime, which contained 3.37% of these metals. Instead of smelting the first slimes to obtain a new speiss, the platinum metals could also be separated by roasting the slimes and smelting with litharge to obtain a lead bottom for acid treatment. All the above methods gave extractions well below 100%. A. R. POWELL.

Ball hardness and cold-working of soft metals and eutectics. F. HARGREAVES (Inst. Metals, March, 1928. Advance copy. 27 pp.).—The relation between the diameter of the impression in the Brinell hardness test and the time of application of the load has been investigated for pitch, vulcanite, and a number of soft metals and eutectics in cast, annealed, or cold-worked states. The relation may be expressed by the general equation  $d = ct^s$ , where d is the diameter of the impression, t the time of application of the load, and c and s are constants depending on the material, the diameter of the ball, and the applied load. Generally speaking the harder the metal the smaller are both c and s. The factor s is probably a measure of the rate of spontaneous annealing, and for a given metal it increases when the material is tested in the cold-worked state. The factor c for a given metal is almost the same for cast, worked, or annealed specimens. For soft metals the exact temperature at which the test is carried out is of great importance, the diameter of the impression being affected by as much as 0.005 mm. per 1° F. The significance of the ordinary Brinell numbers is discussed. W. HUME-ROTHERY.

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Recovery and sinking-in or piling-up of material in the Brinell test, and effects of these factors on correlation of the Brinell with other hardness tests. A. L. NORBURY and T. SAMUEL (Iron and Steel Inst., May, 1928. Advance copy. 15 pp.).-Measurements have been made of the "sinking-in" or "pilingup," and "flattening" and elastic recovery of 10 mm.ball Brinell impressions on vulcanite and a number of metals in different conditions. If expressed as a percentage of the measured depth of impression, the sinkingin or piling-up of the material round Brinell impressions has a constant value for a given material, irrespective of the size of the impression. In cast materials the sinking-in, if expressed as above, may be as much as 30%, whilst in cold-worked materials the piling-up may reach this value. Hence depth indicators measuring the depth from the original surface may give differences of  $\pm$  30% if the hardness be calculated from the indicated depths instead of from the diameter. The effects of these phenomena on the correlation of Brinell tests with scleroscope, pendulum, and cone indentation tests are discussed. W. HUME-ROTHERY.

Adsorption processes in flotation. K. KELLER-MANN and E. PEETZ (Kolloid-Z., 1928, 44, 296-308).-The adsorption of caprylic (octoic) acid by quartz and by galena was studied by measurements of the refractive index, electrical potential, electrical conductivity, and surface tension. Examination of the system quartz-octoic acid showed that, although quartz does not act as an adsorbent, a portion of it goes into colloidal solution under the peptising influence of octoic acid, and in this state it acts as an adsorbent. Galena is attacked by octoic acid, and the measurements were carried out in presence of a solution saturated with respect to lead octoate. Galena adsorbs octoic acid well, although the dried material does so poorly. The constants of the adsorption isotherms have been calculated from the experimental data, and also the molecular thickness of the adsorption layers. Under the experimental conditions, galena is readily floated, whilst quartz is not; the parallel between flotation and adsorption is discussed in regard to flotation practice. E. S. HEDGES.

Aluminium and its alloys. BUSCHLINGER.—See I. Coke for steel making. WHEELER.—See II. Magnet steels. SANFORD. Chromium. LUKENS. Nickel electrotyping. WINKLER and BLUM.—See XI.

#### PATENTS.

Blast furnace. W. P. Power and E. Lewis, Assrs. to BROKEN HILL PROPRIETARY CO., LTD. (U.S.P. 1,664,832, 3.4.28. Appl., 20.1.27. Austral., 9.12.26).—The upper part of a blast furnace is lined with refractory material which is supported against the shell independently of the lining in the lower part of the furnace. A. R. POWELL.

**Cupola.** A. H. COPLAN (B.P. 288,072, 18.7.27).— The refractory lining of a cupola is replaced by an inner casing of heat-resisting steel having ribs in helical formation on its outer surface in order to impart a cyclonic motion to a current of air passing between the inner and outer casings. The heated air is then conveyed to the air box and tapering tuyères of the cupola.

C. A. KING.

**Open-hearth furnace.** G. L. DANFORTH, JUN., Assr. to OPEN HEARTH COMBUSTION Co. (U.S.P. 1,665,086, 3.4.28. Appl., 13.7.22).—The furnace is provided with slag pockets which are connected by means of passages to the regenerative chambers and to the furnace ports. Vertical dampers are provided to close certain of the passages between the regenerative chambers and the slag pockets and to place them in direct communication with the furnace ports. A. R. POWELL.

Tilting-hearth tray furnace. A. D. KEANE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,664,749, 3.4.28. Appl., 28.3.27).—The furnace comprises an annular chamber, means for moving a number of trays through the furnace, and means for tilting the trays in the furnace and for returning them to their normal position. A. R. POWELL.

Working-up ores and metallurgical products of various kinds containing volatilisable metals. F. KRUPP GRUSONWERK A.-G. (B.P. 287,745, 24.5.27. Addn. to B.P. 252,679; B., 1927, 583).—Additions of materials, e.g., limestone, sand, intended for stiffening metallurgical substances in a refining furnace are introduced wholly or in part into the reaction zone and there mixed with the charge, enabling a higher temperature to be used. Other materials, e.g., potassium nitrate or fine-grained ore, may be blown in together with additional fuel if desired. C. A. KING.

Metallurgical briquette and process of using it. W. B. RUNYAN (U.S.P. 1,666,312, 17.4.28. Appl., 31.3.21).—The briquette, free from admixed ore, consists of ground coke and equal parts of a silicon sand and lime in sufficient quantities to serve as a binder.

H. ROYAL-DAWSON.

Production of beryllium. Production of metals by electrolysis. E. C. R. MARKS. From KEMET LABORATORIES Co., INC. (B.P. 287,734 and 287,762, 20.4.27).-(A) Anhydrous beryllium chloride, obtained by sublimation from a mixture of the oxide and carbon heated in chlorine, carbonyl chloride, or carbon tetrachloride, is added to a bath of molten sodium chloride and the mixture is electrolysed at 5-8 volts in an inert atmosphere, the temperature being raised slowly from 730° to 820° during electrolysis. The beryllium deposits in flakes on the anode. When electrolysis is finished the molten sodium chloride is poured out and the beryllium collected, washed free of soluble salts, dried, pressed into rods or discs, and melted under a layer of 10% of barium fluoride and 90% of barium chloride. (B) The vessel in which the electrolysis is carried out is made of a nickel-chromium or iron-chromium alloy containing more than 5% Cr, preferably 20% Cr. A. R. POWELL.

Manufacture [refining] of aluminium. ALUMIN-IUM-IND. A.-G. (B.P. 265,170, 7.1.27. Ger., 27.1.26).— Current is passed between an aluminium anode to be refined and a cathode through an electrolyte, *e.g.*, aluminium sodium chloride, of lower m.p. than the anode and cathode, whereby fusion of these electrodes is avoided. J. S. G. THOMAS.

Production of nickel or ferro-nickel. M. STERN (B.P. 269,133, 22.11.26. Ger., 7.4.26).—Materials containing nickel or nickel oxide are melted together with iron, and the content of nickel is increased either by preferential oxidation of the iron or by the addition of further quantities of nickel. C. A. KING.

Recovery of nickel from compound sheets of copper and nickel. E. BREUNING (B.P. 274,064, 27.6.27. Ger., 10.7.26).—Copper may be dissolved from the surface of a nickel sheet by treatment at 80° with neutral ferric sulphate solution in the presence of an oxidising agent such as air, oxygen, ozone, or a persulphate, and an oxygen carrier such as ceric sulphate. The nickel becomes passive and remains unattacked.

A. R. POWELL.

Separation of tin from alloys and mechanical mixtures. F. BISCHITZKY (B.P. 288,049, 23.5.27).— White metal alloy scrap with a high content of tin is treated in counter-current with a boiling solution of hydrochloric acid and calcium chloride until the liquid ceases to dissolve more metal. The solution deposits lead chloride on diluting and cooling, and the tin may be recovered by precipitation with lime or calcium carbonate. The residues from the acid treatment may still contain tin; they are washed free from acid, allowed to oxidise by exposure to the air, and returned to the treatment vats. A. R. POWELL.

Treatment of impure [silver, lead, or tin] metals or alloys [for the removal of copper]. H. HARRIS (B.P. 288,004, 21.2.27).—The molten metal or alloy is treated with sulphur or a sulphur-bearing material such as hydrogen sulphide in the absence of air or in a reducing atmosphere. To cause complete separation of the copper sulphide from the purified metal, resin or other similar carbonaceous material is stirred in after the sulphur treatment. A. R. POWELL.

Purification of metals. J. J. CONLIN, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,665,844, 10.4.28. Appl., 6.4.27).—A readily oxidisable metal may be freed from non-metallic impurities by inclosing it in a container with an upper valve, and submerging the container below the surface of a molten salt bath which does not react with the metal, but causes the impurities to separate from the metal. A. R. POWELL.

Bearing [metal] alloy. I. R. VALENTINE, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,667,641, 24.4.28. Appl., 25.9.25).—The alloy contains 70 pts. of copper, a hardening metal, about 25 pts. of lead, and a trace of arsenic. F. G. CROSSE.

Alloy. M. G. CORSON, Assr. to ELECTRO METALLUR-GICAL CO. (U.S.P. 1,667,966, 1.5.28. Appl., 21.6.26).— A bronze contains up to 9% Al, above 0.5% Cr in solid solution, and sufficient nickel to hold the chromium in solution. F. G. CROSSE.

Production of a refractory material [alloy]. Alloy and material employing the same. J. G. DONALDSON and H. L. COLES, ASSTS. to GUARDIAN METALS CO. (U.S.P. 1,668,306—7, 1.5.28. Appl., [A] 16.1.25, [B] 19.5.23).—(A) A copper alloy contains 0.60-0.75% Si and less than 1% C. (B) A compound plate consists of a core (an alloy containing 99.385— 99.230% Cu, 0.015-0.020% C, and 0.60-0.75% Si) surrounded by other metal to which it is united, *e.g.*, by alloying therewith. F. G. CROSSE.

Apparatus for cleansing metals from oil and other impurities. Dr. A. WACKER GES. F. ELEKTRO-CHEM. IND. G.M.B.H. (B.P. 273,307, 22.6.27. Ger., 25.6.26).—An apparatus for cleaning articles with organic liquids, *e.g.*, trichloroethylene, benzine, carbon tetrachloride, consists of two closable vessels arranged one above the other. The upper vessel contains the articles in baskets which may be rocked, and a pump is used to force the cleaning liquid from the bottom chamber to the one above or in the reverse direction. Heat may be applied to the lower chamber, or a current of hot air for drying the cleaned articles, a condenser being mounted on top of the chambers. C. A. KING.

Increasing the elasticity of metal articles. G. L. KELLEY, Assr. to BUDD WHEEL Co. (U.S.P. 1,667,476, 24.4.28. Appl., 14.9.26).—Rings produced by coldworking blank stock are heated at  $93-427^{\circ}$  for  $\frac{1}{2}$ —30 min. J. S. G. THOMAS.

Friction materials for surfacing the friction elements of brakes, clutches, etc. Soc. Anon. FRANÇ. DU FERODO (B.P. 264,471, 20.12.26. Fr., 13.1.26). —The friction surfaces are impregnated with lead or an alloy of lead by either mechanical or chemical means.

C. A. KING.

Solder bars. H. A. MEINHARDT (B.P. 282,007, 20.9.27. Ger., 10.12.26).—In a composite soft-solder bar the tubular sheath of solder has a lower m.p. than the core. The core may also contain fluxes or deoxidising substances. C. A. KING.

Flotation processes. D., M., S. R., and S. GUGGEN-HEIM, J. K. MACGOWAN, and E. A. C. SMITH [GUGGEN-HEIM BROS.], Assees. of A. H. FISCHER (B.P. 275,561, 29.4.27. U.S., 3.8.26).—Flotation agents comprising derivatives of thiocarbonic acid, such as dixanthogen or xanthic anhydride, or their alkyl, aryl, or aralkyl homologues, are claimed. A. R. POWELL.

Process of welding. R. APPLEGATE, Assr. to Ohio Brass Co. (U.S.P. 1,666,026, 10.4.28. Appl., 10.10.21). —In welding copper bands to steel rails by the arc process, calcium silicide is used as a deoxidiser.

A. R. POWELL.

Manufacture of plated goods. E. G. BEK (B.P. 287,641, 24.12.26. Addn. to B.P. 244,487; B., 1927, 633).—In the process of plating base-metal articles with a precious metal by electrodeposition followed by an annealing operation, the colour of the deposit may be modified by depositing a second layer of the same or another metal and again subjecting the article to an

annealing operation to cause diffusion between the layers to take place. To coat a gold-plated article with platinum, several repetitions of the process using an annealing temperature of 700—800° are necessary. To convert a coating of red gold into one of white gold the gold-plated article is further plated with nickel and heated for 2 min. at 700°; or into green gold by plating with a thin layer of cadmium and heating at 300—600°. A yellow gold coating is converted into one of white gold by plating with chromium and heating at 600—800°. A platinum coating may be applied to a base metal or gold article by plating first with cadmium, zinc, or silver and heating at 400°, then plating with platinum and heating at 600—700°. A. R. POWELL.

Magnetic separation of minerals. R. RADE-MACHER and E. GOEBEL (B.P. 274,889, 21.7.27. Ger., 23.7.26).—Ferrous and non-ferrous constituents of a mineral are separated by magnetic treatment of the mixture at a temperature either between 300° and 600° or above 1100°. The residue, after separation of the ferrous constituent, may be re-treated.

J. S. G. THOMAS.

Cupola furnace. O. WEICHEL and W. HOLLINDER-BÄUMER (U.S.P. 1,668,133, 1.5.28. Appl., 7.7.27. Ger., 2.6.26).—See B.P. 272,216; B., 1928, 160.

Magnetic alloy. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (U.S.P. 1,667,746, 1.5.28. Appl., 21.1.27. U.K., 8.9.26).—See B.P. 281,763; B., 1928, 128.

Magnetisable material [alloy]. E. GUMLICH (U.S.P. 1,668,115, 1.5.28. Appl., 30.11.26. Ger., 30.11.25).—See B.P. 262,153; B., 1927, 560.

[Wire-]enamelling ovens. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of C. L. HEISLER (B.P. 282,740, 12.12.27. U.S., 29.12.26).

Application of protective coatings to metal pipes etc. W. R. HUME, and HUME STEEL, LTD. (B.P. 287,656, 10.1.27).

Manufacture of electrodes, welding rods, etc. for use in arc welding and depositing metal. B. TURNER and FERRO-ARC WELDING CO., LTD. (B.P. 285,128-9, 9.11.26).

Furnace for ores (U.S.P. 1,665,546).—See XI.

# XI.—ELECTROTECHNICS.

Principles governing the choice and utilisation of permanent-magnet steels. R. L. SANFORD (U.S. Bur. Stand., Sci. Paper 567, 1927, 22, 557-567).-Magnet steels with a high cobalt content have coercive force values 2-5 times as great as those of the chromium or tungsten steels generally used in the manufacture of permanent magnets, and the residual induction is only very slightly lower, but cobalt steels cannot be satisfactorily substituted for chromium or tungsten steels without changing the design of the magnet. From tests of numerous types of magnet steels it is shown that the product of the coercive force and the residual induction is the best criterion of the value of a steel for this purpose. Magnet steels can be stabilised against permanent deterioration from transient demagnetising fields by subjecting them to partial demagnetisation; this method is particularly applicable

to the treatment of cobalt steels owing to their high coercive force. A. R. POWELL.

Influence of the cathode on the electrodeposition of chromium. H. S. LUKENS (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 7 pp.) .- A solution containing 250 g. of commercial chromic acid and 3.3 g. of chromic sulphate per litre was electrolysed at 45° in a diaphragm cell for 1 hr. and the composition of the catholyte after the electrolysis was determined. Experiments were made with cathodes of electrolytic nickel, electrolytic copper, and sheet lead at various current densities. The greatest amount of tervalent chromium is produced in the catholyte at the lowest current density used (5 amp./dm.2), and at given current density the amount is least with a nickel cathode and greatest with a lead cathode, i.e., it increases with increasing hydrogen overvoltage of the cathode metal. Of the three cathode metals, copper yields practical deposits of chromium over the widest range of current densities, nickel over the narrowest range. When the concentration of chromic salt in a chromium bath exceeds a low limiting value, the conductivity of the bath becomes impracticably low: such a solution can be rejuvenated by electrolysis using a very large lead anode and a small cathode surrounded by a porous diaphragm. In attempting to deposit chromium on the surface of nickel which had been electrodeposited on graphitised wax, the surface of the nickel which had been in contact with the wax was treated with an abrasive after removing the wax by application of hot water. The very narrow limits of current density required for the deposition of chromium on this surface and the behaviour of the surface towards chemical reagents indicate that it is in a passive state.

# H. J. T. ELLINGHAM.

Properties of graphite used in electrotyping. J. H. WINKLER and W. BLUM (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 19 pp.).-The fineness, electrical conductivity, and carbon content of five samples of graphite were determined, and wax moulds were coated with films of graphite by application of aqueous suspensions of the various samples. The rate at which nickel deposits from a specified electrotyping solution under specified conditions on the various graphite films was determined (cf. Blum and Winkler, following). From such tests carried out both in the laboratory and in commercial electrotyping plants, tentative specifications for graphite for use in electrotyping are suggested. For graphite applied from aqueous suspension ("wet leads"), the material should contain at least 92% C; at least 99% should pass through 100-mesh, 95% through 200-mesh, and 50% through 325-mesh; it must readily form a suspension with water, and the film should be covered with nickel in 10 min. under the specified conditions. Slightly different specifications are given for "polishing leads." H. J. T. ELLINGHAM.

Nickel electrotyping solutions. W. BLUM and J. H. WINKLER (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 15 pp.).-Experiments have been made on the rate of "covering" of a graphitised wax slab by nickel electrodeposited from solutions of various compositions under various conditions. Electrical

connexion to the graphitised surface was made by means of a brass strip. The physical character of the nickel deposits is recorded. The presence of an ammonium salt in the bath is found to be essential, but addition of boric acid is generally unsatisfactory in that the nickel deposit tends to crack and curl. The best nickel concentration is found to be about 0.5N; higher concentrations increase the tendency to crack and curl. The rate of covering increases somewhat with increasing conductivity of a bath of given nickel content; it also increases with rise of temperature, but a duller and softer deposit is then obtained. If the current density is too high, "burnt" or cracked deposits are produced, but, since the current density is changing continuously during deposition, it is usual to operate at a constant specified voltage. It is recommended that a solution which is N to nickel sulphate and 0.1N to ammonium chloride should be used, the  $p_{\rm H}$  value being maintained at  $6 \cdot 0 - 6 \cdot 4$  at 25-35° in a well agitated bath. With anodes and cathodes 7.5-10 cm. apart, the voltage applied may be about 4. The use of this bath, which is more concentrated in nickel than is usual for electrotyping baths, has proved generally satisfactory in commercial operation over an extended period. Recommendations are also given for nickel deposition on lead moulds. In this case the solution may be still more concentrated and of higher  $p_{\rm H}$  value, and can be operated at 4-5 volts at 30-45°. H. J. T. ELLINGHAM.

Effect of temperature and other factors on the performance of storage batteries. G. W. VINAL and C. L. SNYDER (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 12 pp.) .--- Using small experimental batteries having either one positive plate and two negatives or one negative and two positives set up in acid of various concentrations, which, however, remained practically constant during operation, measurements of capacity were made at various rates of discharge and at various temperatures ( $-17^{\circ}$  to  $25^{\circ}$ ), the capacities of the positive and negative plates being separately determined. At given temperature and discharge rate, the capacity of the positive plates increases with the concentration of the acid used, but that of the negative plates decreases. Hence the best concentration of electrolyte is a compromise between the requirements of the positive and negative plates. Since under extreme conditions, as in aeroplane service, the negative plates are likely to become the limiting factor in determining the capacity, more dilute acid will then be desirable. With given acid concentration and discharge rate, a fall of temperature reduces the capacity of both plates, but the effect is greater with the negative than the positive plates. Also the temperature effect is least at higher electrolyte concentrations in the case of the positive plates, whereas the reverse is true for the negatives. In a battery where the capacity is limited by the positives, the change in capacity with temperature is inversely proportioned to the change in the viscosity of the solution. The conditions under which localised freezing may occur at lower temperatures are considered. The possible effects of these various factors on the operation of aeroplane batteries are discussed. H. J. T. ELLINGHAM.

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Dry cell service and tests. C. A. GILLINGHAM (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 6 pp.).—The processes occurring in a dry cell may be classified as (a) chemical depolarisation, which occurs only when the cell is under load, (b) physical depolarisation, including escape of hydrogen gas and diffusion processes, which occurs during and after the delivery of current by the cell, and (c) shelf reactions, including the chemical dissolution of zinc, which occur at all times. The relative importance of these three groups in determining the efficiency of the cell depends on the nature of the service in which it is employed. Tests intended to give a true indication of the suitability of a cell for various types of service are suggested.

#### H. J. T. ELLINGHAM.

Gelatinisation of corn starch in dry cell electrolytes. W. D. STALEY and A. J. HELFRECHT (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 7 pp.).—A mixture of 2 pts. of corn starch and 1 pt. of corn meal was added to solutions of zinc chloride or of zinc and ammonium chlorides at 10°, and after thorough mixing the time taken for the solutions to gelatinise on being kept in surroundings at 18° was determined. With 35 g. of the cereal in 80 c.c. of zinc chloride solution, the solution does not gelatinise unless the zinc chloride concentration is as high as about 35%; with increasing concentration of zinc chloride the setting time decreases to a minimum at about 42% and then increases rapidly Similar results are obtained with solutions again. containing ammonium chloride and zinc chloride, but the former salt is only about half as effective in promoting setting as compared with the weight of the latter which it replaces. By the use of two solutions, one of which contains ammonium chloride and most of the zinc chloride and the other contains ammonium chloride, a little zinc chloride, and all the cereal, mixing in suitable proportions gives a solution which will set in a convenient time for the preparation of dry cells.

# H. J. T. ELLINGHAM.

Carbon dioxide recorder. GORDON and LEHMANN. —See I. Transformer sheet iron. von Moos and others. Normalising of steel. Woodward. Tungsten steel. Swan. Annealing non-ferrous metals. KEENEY. Cable sheathing. BECKINSALE and WATERHOUSE. Speiss and metals of platinum group. RUSDEN and HENDERSON.—See X.

#### PATENTS.

Electric furnace. H. WIGGIN & Co., LTD., and A. G. LOBLEY (B.P. 285,147, 11.11.26).—Portions of the shanks of resistance-suspenders or connectors are, after insertion through the furnace bricks, bent over to form keys which are engaged with hook-locating grooves or seatings formed either in the brick or in bonded or nonrotatable sleeves or insulators applied to the brick. J. S. G. THOMAS.

Electric furnace. E. C. SASNETT, ASST. to C. B. FOLEY, INC. (U.S.P. 1,660,209, 21.2.28. Appl., 24.9.19. Renewed 7.7.27).—A structure lined with refractory material is adapted to hold molten metal in the shape of a superposed pool with a subjoined loop, the crosssectional area of which is a minimum at one point at which it joins the pool and increases gradually and continuously to a maximum at the other. Electrical induction means are threaded through a transverse opening within the loop. J. S. G. THOMAS.

Electric furnace. A. J. ASCH (U.S.P. 1,661,026, 28.2.28. Appl., 4.2.27).—A heating element composed of platinum foil is supported within the furnace, and a source of current is connected to portions thereof extending outside the furnace wall. J. S. G. THOMAS.

Electric furnace for treating ores, shales, etc. F. E. HATCH (U.S.P. 1,665,546, 10.4.28. Appl., 4.9.26). —An electrically heated, horizontal, rotatable drum is lined with refractory material. The heads of the drum are closed and have inlet and exit for material. Pipes pass through the drum to a collector at one end, and means are provided for causing a current of air to pass through the pipes and collector. T. A. SMITH.

Ironless induction furnaces or heating apparatus. SIEMENS & HALSKE A.-G. (B.P. 274,888, 21.7.27, and Addn. B.P. 275,249, 28.7.27. Ger., [A] 24.7.26, [B] 29.7.26).—(A) Heat produced by induction in the charge is generated by a heating coil dipped or inserted into the charge in the furnace interior so that a large part of the external electromagnetic lines of force produced by the heating coil pass through the charge. (B) In addition to the heating coil dipping into the charge, another coil is arranged round the outside wall of the furnace.

J. S. G. THOMAS.

Recovery of reaction products from gases treated with electric arcs. I. G. FARBENIND. A.-G. (B.P. 263,859 and Addn. B.P. 288,056, 30.12.26. Ger., 30.12.25).—(A) Small outlet tubes or pipes are arranged in combination with gas conduits in order to separate gas rich in reaction products from gas weak in reaction products. (B) The process is applied for producing pure hydrogen from mixtures containing hydrogen and methane which are treated in electric arc furnaces whereby the methane is largely converted into a cetylene. The reaction products are separated into a portion containing practically no unconverted methane and a portion containing very little acetylene. The process is also applied to the separation of oxides of nitrogen produced by the fixation of atmospheric nitrogen.

#### J. S. G. THOMAS.

Net-cathode for electrolytic cells. A. ERGANG (B.P. 287,760, 11.7.27).—The wire gauze net-cathodes used, e.g., in electrolysing alkali chlorides, are replaced by cathodes formed from sheet metal by perforating with rectangular holes by means of a punch so constructed that the upper surfaces of the bars between the holes are rounded.

[Electrolyte for] voltaic cells. FRIGAMIN G.M.B.H. (B.P. 283,559, 5.7.27. Ger., 14.1.27).—Hydrochlorides of the organic amines, more especially methylamine hydrochloride, to which may be added one or more weak organic acids, *e.g.*, cinnamic acid, sulphonic acids, are employed as electrolytes in cells of the Leclanché type. J. S. G. THOMAS.

**Production of a storage-battery plate.** C. W. JENNER (U.S.P. 1,665,962, 10.4.28. Appl., 2.6.27).---Oxidised, finely-ground electrolytic lead is mixed with dilute sulphuric acid and moulded into a plate.

J. S. G. THOMAS.

Glow cathode and electron tube containing same. E. FRIEDERICH, ASST. to RADIO CORP. OF AMERICA (U.S.P. 1,667,471, 24.4.28. Appl., 28.11.25. Ger., 1.12.24).—A highly refractory filamentary core coated with a mixture of carbon and a metal of high electron-emitting capacity is heated so that a carbide of the metal is formed. J. S. G. THOMAS.

Magnetic separation. F. M. SIMONDS, Assr. to A. F. HYDE (U.S.P. 1,660,362, 28.2.28. Appl., 3.10.25).— A mixture comprising magnetic and non-magnetic particles moves across a magnetic field, and the aggregates formed are subjected to disintegrating blows as they move towards the source of magnetic attraction. J. S. G. THOMAS.

**Treatment of magnetisable materials.** A. F. BANDUR, ASST. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,666,191, 17.4.28. Appl., 12.12.24).—Magnetisable material is locally flash-heated to a high temperature and cooled, the rate of cooling being controlled so that there is a rapid loss of heat from a temperature not below 500°. J. S. G. THOMAS.

Treatment of wax surfaces [for electrotype] with graphite. W. McLAUGHLIN and H. KNOWLES (B.P. 288,709, 11.1.27).—Wax surfaces are treated with a colloidal suspension of graphite, preferably a suspension in water containing a protective colloid, *e.g.*, tannic acid, and a deflocculating agent, *e.g.*, sodium silicate, glycerin, or eucalyptus oil. Thus, "Aquadag" may be employed. J. S. G. THOMAS.

Gas-testing apparatus. C. ENGELHARD, INC., Assees. of R. H. KRUEGER (B.P. 288,908, 17.10.27. U.S., 27.8.27).—In gas-analysis apparatus of the catharometer type, controllable means, *e.g.*, tubular insulating thimbles, are provided in order to regulate the heat dissipated in the chambers containing gases and the resistances forming arms of the Wheatstone bridge. J. S. G. THOMAS.

Lead accumulator. R. OPPENHEIM, Assr. to Soc. ANON. LE CARBONE (U.S.P. 1,667,435, 24.4.28. Appl., 18.4.25. Fr., 14.6.24).—See B.P. 235,530; B., 1926, 591.

Use of gas-filled photo-electric cells. GEN. ELECTRIC Co., and N. R. CAMPBELL (B.P. 288,747, 24.1.27, and 288,783, 25.2.27).

Electric mercury-vapour apparatus [rectifier] etc. GEN. ELECTRIC CO., LTD., and E. WEINTRAUB, Assees. of Soc. Alsacienne de Constructions Mécaniques (B.P. 272,958, 20.6.27. Fr., 19.6.26).

Beryllium (B.P. 287,734 and 287,762). Refining of aluminium (B.P. 265,170). Plated goods (B.P. 287,641).—See X.

# XII.—FATS; OILS; WAXES.

Bleaching of [linseed] oil. K. WÜRTH (Farben-Ztg., 1928, 33, 1852—1853).—Linseed oil was treated with ozonised air, a large, moving surface of oil being exposed during the process. The oil was rapidly bleached and its consistency unchanged, but, on gentle heating, the oil assumed a brownish colour and a strongly smelling gas was evolved. Similar results were obtained by treating the oil with air while exposed to ultra-

violet light from a mercury lamp. The presence of moisture in the air or in the oil may influence the processes involved. S. Coffey.

Standardisation of cod-liver oil. W. H. DICKHART (Oil and Fat Ind., 1928, 5, 82-84).-A rapid sorting test, to distinguish good oils from those contaminated with oils other than those from the livers of fish related to the cod, consists in adding 10 mg. of uranium nitrate to 3 c.c. of oil placed in a 6-in. glass tube of inside diam. 20 mm. The tube is placed upright in a 400 c.c. beaker containing 80 c.c. of water which is heated just at the b.p. for 20 min.; afterwards the oil is allowed to cool and the colour measured in a Wesson tintometer using standard Lovibond glasses. Experiments and analyses proved that fish oil changed to give a definite colour reading with uranium nitrate. Comparison of data on colour readings for a number of commercial cod-liver oils showed but one passing the U.S.P. test for unsaponifiable matter, and that oil gave characteristics similar to whale oil, and yet all the oils were cod-liver oils. It is suggested that for medicinal cod-liver oil a maximum of 1 yellow, 0.1 red before heating with uranium nitrate and 35 yellow, 3.8 red after heating be allowed on a basis of a 10 mm.  $\times$  20 mm. column of oil; that for poultry oil 1.5 vellow, 0.2 red in the cold and 35 vellow, 5.0 red after heating be the established standard in the uranium nitrate test conducted as described.

H. M. LANGTON.

Analysis and technology of evening primrose oil. A. EIBNER and E. SCHILD (Chem. Umschau, 1927, 34, 339-342; cf. Heiduschka and Lüft, B., 1919, 426 A).—The oil has  $d^{15}$  0.9312; viscosity (Engler) at 24° 8.14,  $n_{\rm D}^{_{40}}$  1.47154, acid value 4.51, saponif. value 187.39, iodine value (Hanus) 147.89, glycerin content 10.01%, unsaponif. matter 1.60%, total fatty acids 95.52% (iodine value 156.65). Separation of the fatty acids (Twitchell) gave 5.37% of saturated acids and 94.63% of unsaturated. Analysis of that part of the unsaturated brominated acids soluble in light petroleum shows the existence of an isomeric y-linolenic aicd. The results obtained are (%):  $\gamma$ -linolenic acid 2.69, isomeric  $\gamma$ -linolenic acid 6.00,  $\alpha$ -linoleic acid 29.55, 3-linoleic acid 22.23, oleic acid 23.56, saturated acids 5.13, unsaponifiable matter 1.60, and glyceryl radical 4.11. This oil does not attain the drying properties of the pine seed oils, which, according to Reitter (B., 1926, 593), contain 6.6% of  $\alpha$ -linolenic acid and  $17 \cdot 2\%$  of  $\beta$ -linolenic acid. Since  $\gamma$ -linolenic acid as a symmetrical molecule has not such strong drying qualities as the  $\alpha$ -linolenic acid of perilla and linseed oil, quantitative analysis of an oil throws light on its drying qualities, in which respect it is shown to be midway between linseed and poppyseed oil.

H. M. LANGTON.

Chemical composition of ergot oil. W. F. BAUGHMAN and G. S. JAMIESON (Oil and Fat Ind., 1928, 5, 85-89; cf. Rosenheim and Webster, A., 1927, 1224; Dieterle, Diester, and Thimann, B., 1927, 799; Matthes and Schütz, B., 1927, 891).—The ergot oil examined was extracted by light petroleum from a composite sample of Russian, Spanish, and Austrian ergot containing  $5 \cdot 50\%$  of moisture and  $32 \cdot 75\%$  of oil. The oil, dark red by transmitted light with a dark brownish yellow fluorescence by reflected light, had  $d_{25}^{25}$  0.9222,  $n_{\rm D}^{23}$  1.4691, acid value 3.02, iodine value (Hanus) 73.8, saponif. value 196.9, unsaponifiable matter 1.18%, acetyl value 7.3, R.-M. value 0.3, Polenske value 0.4, saturated acids 27.2% (iodine value  $2 \cdot 6$ ), unsaturated acids + unsaponifiable matter  $68 \cdot 6\%$ , iodine value of unsaturated acids  $101 \cdot 2$ , saturated acids (corr.) 26.5%, unsaturated acids (corr.) 68.1%. The composition of the unsaturated acids was determined from their saponif. and iodine values, that of the saturated acids being determined by fractional distillation of their methyl esters. The composition of ergot oil is summarised thus : glycerides of oleic acid 62.5%, of linoleic acid 8.7%, of myristic acid 0.3%, of palmitic acid 21.5%, of stearic acid 5.3%, of arachidic acid 0.7%, unsaponifiable matter 1.2%. No confirmation was obtained of the presence of daturic acid or of hydroxylated acids previously reported in the oil. H. M. LANGTON.

Determin tion of sodium sulphate in sulphonated oils. W. HERBIG (Chem. Umschau, 1927, 34, 330—331). —The oil (10 g.) is shaken with 200 c.c. of absolute alcohol in an Erlenmeyer flask which is then lightly closed. On keeping, the sodium sulphate separates very quickly as a yellowish-brown precipitate which, after decanting the alcoholic solution, is washed on to a filter with absolute alcohol. The precipitate is then dissolved in hot water and converted into barium sulphate in the usual way. Two different qualities of oil gave sodium sulphate (as SO<sub>3</sub>) (a) 1.97 and 2.05%, (b) 0.68 and 0.75%. H. M. LANGTON.

Deflocculation and detergency. R. M. CHAPIN (Oil and Fat Ind., 1928, 5, 95-106) .- Following on his earlier work (cf. B., 1927, 117; 1928, 4) on the deflocculation of carbon black, the author has experimented on fabrics soiled by the same substance with the object of determining whether deflocculation and detergency are correlative phenomena. A laboratory washing machine is described, also a Kober-Klett colorimeter which had been converted into a reflectometer for the evaluation, by numerical expression, of soil remaining on a fabric. Detergent experiments were made on new cotton fabric (previously cleansed by boiling with soap) soiled in simple aqueous suspensions of non-oily carbon black. The most powerful detergent appeared to be an alkaline solution of sodium stearate or palmitate at 70° or above. In this effect alkali deflocculation and detergency are parallel, but other possible correlative factors were only vaguely defined. The method is not sufficiently selective of the superior amongst competing commercial soaps. Comparative tests were made on the power of soap solutions to prevent adsorption of suspended carbon black by clean fabric. Acid soaps were the most effective, probably owing to masking of adsorptive affinities by an oily film of fatty acid. Power to prevent adsorption appears not to be correlative with detergent power in the special case of the prevention of adsorption of suspended carbon by fabric. This result is based on tests using soap solu-H. M. LANGTON. tions.

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Rancidity determinations and a possible source of error in the Kreis test. W. C. POWICK (Oil and Fat Ind., 1928, 5, 107—108).—In the examination of fats and oils for rancidity by the Kreis test with hydrochloric acid and phloroglucinol, misleading results may be obtained through the presence of nitrosyl chloride in the acid. The acid so contaminated gives a "blank" test with phloroglucinol, and under such conditions rancid fats will generally appear to be sweet, whilst sweet fats may occasionally appear to be rancid unless the positive reaction be controlled spectroscopically. In the absence of spectroscopic control, positive tests obtained from cottonseed oils are not sufficient evidence of rancidity (cf. B., 1923, 376 A). H. M. LANGTON.

**Precision of iodine-bromine value determinations.** L. W. WINKLER (Arch. Pharm., 1928, **266**, 189— 193).—The latest official (German) method of determining iodine values, in which iodine is used only as an indicator, gives very trustworthy results. The error in repeated determinations is less than 0.55%. The determination is rapidly carried out, but the time allowed for interaction should be adjusted according as the iodine value of the oil under examination is low or high, *i.e.*, for iodine values below 40, 10 min. suffices; for those between 40 and 140,  $\frac{1}{2}$ —1 hr.; and so on (tables are given). Cod-liver and linseed oils are exceptional, and special tables are given for these. The method has the further advantage of using cheap reagents.

W. A. SILVESTER.

E. HOLMES.

Behaviour of beeswax towards trichloroethylene at ordinary temperatures. G. BUCHNER (Chem.-Ztg., 1928, 52, 319).—Treatment in the cold causes the removal of a large portion of the hydrocarbons present in the beeswax. The soluble portion has m.p.  $54 \cdot 5^{\circ}$ , acid value  $3 \cdot 45$ , ester value  $24 \cdot 36$ , and saponif. value  $27 \cdot 8$ , whilst the corresponding values for the insoluble fraction are  $67 \cdot 5^{\circ}$ ,  $15 \cdot 6$ ,  $54 \cdot 0$ , and  $69 \cdot 6$ . The ratio of cerotic acid to ester in the original wax was  $1: 6 \cdot 5$ , that in the soluble portion 1: 12, and in the insoluble portion  $1: 6 \cdot 3$ . H. INGLESON.

Examination and evaluation of beeswax. E. ELSER (Schweiz, Chem.-Ztg., 1928, 27-32, 40-45).-A precise method of determining m.p. of waxes, using a 1.5 mm. capillary tube in a bath, the temperature of which is raised 4° per min. from an initial temperature not above 10-15°, is described. Tables are given showing the variation of m.p. of three fractions of beeswax when mixed with varying amounts of carnauba wax, ceresin, Japan wax, spermaceti, and paraffin waxes (m.p. 58° and 48°). Each wax is considered in some detail and its characteristic effect on the m.p. of beeswax demonstrated in a series of curves. The author concludes that modern chemical and physico-chemical research has shown that the thermal method of analysis of beeswax is simple and exact, and permits the detection not only of the nature but the percentage of adulteration.

#### PATENTS.

Purification of olive oil and other edible oils. E. FORAY (Addn. No. 31,181, 17.11.25, to F.P. 605,389; B., 1926, 795).—The oil, after neutralisation and addition of salt, is washed three times with half its weight of boiling water to remove traces of dissolved soaps, treated at 60° with 2% of regenerated fuller's earth (or with 1.5-3.0% of fresh earth for dark-coloured oil), passed hot through a filter press, and deodorised with 0.05% of manganese dioxide. L. A. COLES.

Bleaching of fatty substances of vegetable and animal origin. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 286,794, 11.12.26).-A combination of the oxidising and reducing processes for bleaching is claimed to give improvements in the colour and odour of fatty materials. The substances may be subjected to a reducing bleach immediately preceded or followed by treatment with one or more inorganic, non-gaseous, oxidising agents, or it may be treated with an absorptive agent simultaneously with the oxidising agent. Fatty substances so treated may be saponified and then bleached with hypochlorite to give light-coloured soaps. Where there is a tendency to form emulsions, from which the manganese dioxide or chromic hydroxide can be removed only with difficulty, it is an advantage to add an equivalent of mineral acid at the outset. E. HOLMES.

Sulphonation of fatty acids and their esters. H. T. BÖHME A.-G. (B.P. 261,385, 10.11.26. Ger., 11.11.25).—Difficulties arising from the formation of water in the sulphonation of fatty acids and their esters (e.g., castor oil) by sulphuric acid are obviated by carrying out the reaction in the presence of anhydrous organic acids or their anhydrides or chlorides, especially acetic acid. B. FULLMAN.

**Production of strongly frothing soaps.** CHEM. FABR. STOCKHAUSEN & Co. (G.P. 445,848, 9.6.25).— A mixture of about equal parts of turpentine with perchloroethylene or other chlorinated hydrocarbons is added to the usual ingredients of soap. L. A. COLES.

Production of stable moistening and degreasing agents. G. ZIMMERLI CHEM.-TECHN. FABR. (Swiss P. 119,114, 23.3.26. Addn. to Swiss P. 111,767).—A mixture of hydrogenated naphthalene with sulphonated fat having a sulphonic acid content of over 40% of the total fats is neutralised and emulsified with water, yielding a product not affected by hard water and possessing good emulsifying, degreasing, and purifying properties. L. A. COLES.

Purification of fat solvents after use. R. A. A. JEANNIN (F.P. 622,238, 28.9.26).—Fat solvents, e.g., benzol, benzine, and trichloroethylene, are treated successively with sand acidified with sulphuric acid and sand made alkaline with sodium carbonate solution, the solvent being decanted off from the residue after each treatment. L. A. COLES.

Alkali fusion [saponification of glycerides]. V. R. KOKATNUR (U.S.P. 1,667,480, 24.4.28. Appl., 22.1.26).—See B.P. 251,290; B., 1927, 755.

Coagulation or peptisation (B.P. 287,943).— See III. Products from cashew nut-shell oil (B.P. 262,134).—See XIV.

# XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Analyses of paints with plumbago base. R. JACQUES (Ann. Chim. analyt., 1928, [ii], 10, 98-100).—A 25 g. sample was repeatedly extracted with ether and

filtered through a tared Gooch crucible. The residue was then washed with 95° alcohol to extract resins, the precipitate was dried at 105°, and the filtrate evaporated at 130—140°. If the solid colouring matter and the oil so determined show a deficiency from 100%, the difference is probably light spirit. This can be determined by distilling a sample with four times the weight of water. The solid base was tested for loss on ignition and another portion extracted with hydrochloric acid and the residue weighed in a Gooch crucible. If carbonates are present they must be separately determined ; otherwise the loss on ignition gives the proportion of graphite present. The analysis of the insoluble residue for silica and metallic bases was completed in the usual way. C. IRWIN.

Mechanism of the drying of red lead and white lead pigments. W. VAUBEL (Z. angew. Chem., 1928, 41, 181-183).-In the drying of pigments prepared from red lead and white lead and linseed oil, although the oxidation of the linseed oil to linoxyn is the main process, subsidiary reactions occur as follows :--(1) neutralisation of the free acids present in the oil by the lead pigment, (2) hydrolysis of the linseed oil by the lead compound, affording glycerol and lead linoleate, (3) reaction between the lead compound and glycerol, (4) neutralisation by the lead compound of the mono- and diglycerides present or formed in the drying process, (5) neutralisation of the acetic acid or formic acid formed by oxidation in the drying process. Such hydrolysis of the linseed oil may be a source of streakiness in the pigment, owing to the hygroscopic character of the glycerol liberated, and the presence of glycerol so formed constitutes a danger to the effectiveness of the "anti-rust" pigments containing lead, white lead and red lead reacting only very slowly with glycerol. Litharge reacts rapidly with glycerol, affording a solid compound,  $C_3H_5(0 \cdot PbOH)_3$ , which on heating at 120–130° to constant weight is converted into Pb[O·C<sub>3</sub>H<sub>5</sub>(OPb)<sub>2</sub>O]<sub>2</sub>, which can be used in the determination of glycerol. Lead hydroxide reacts very slowly, the reaction being accelerated by heat. Lead peroxide does not react cold, but reacts rapidly when heated; lead sulphate, barium sulphate, and lithopone are without action on glycerol. Zinc oxide reacts more slowly than red lead, but faster than white lead. In addition to their advantages in covering power, lead pigments thus possess advantages in their reactivity towards decomposition products formed in the drying process.

R. BRIGHTMAN.

Detection of sulphur and sulphurous acid in mineral colours. A. NOLL (Farben-Ztg., 1928, 33, 1849—1850).—The colour is ignited with sulphur-free iron powder and the melt dropped into dilute hydrochloric acid, when the production of hydrogen sulphide indicates sulphur. The test gives positive results, no matter how the sulphur is combined. The presence of sulphite is ascertained by digesting the colour with hot distilled water and filtering. Sulphurous acid is detected in the filtrate either by reduction with zinc and dilute acid to hydrogen sulphide or by reduction with zinc dust alone to hyposulphite. The latter is detected by its action on indanthrene-yellow G, methylene-blue, or sodium anthraquinone- $\beta$ -sulphonate. S. COFFEY.

#### PATENTS.

Manufacture of a dispersing powder. E. R. ALLEN and W. E. KAUFMANN, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,665,945, 10.4.28. Appl., 3.7.25).—An inorganic pigment not readily miscible with water is agitated with water and a foam-producing dispersing agent, and the resulting foam is dried and ground. The product readily disperses in water to a smooth pulp. F. G. CLARKE.

Manufacture of condensation products of urea and formaldehyde. I. G. FARBENIND. A.-G. (B.P. 288,346, 1.10.26. Addn. to B.P. 258,289; B., 1928, 376).—Carbamide and formaldehyde are condensed in a solution maintained by buffer compounds at  $p_{\rm H}$  4—6, and the product is evaporated or dehydrated at  $p_{\rm H}$  6—7. Clear products are thus obtained with comparatively small quantities of formaldehyde, and when the molar ratio of formaldehyde : carbamide is less than 2:1 (1.5—1.7:1) the evaporation or dehydration may be dispensed with partly or entirely, the condensation product separating on cooling (preferably while adding salts) as a liquid colloid. B. FULLMAN.

Condensation products. "SILUR" TECHN. U. CHEM. PROD. GES.M.B.H. and I. THORN (B.P. 287,727, 9.4.27).— Plastic condensation products are obtained by mixing carbamide or thiocarbamide with formaldehyde or compounds yielding it, and then adding starch (or substances containing it, especially flour) in a quantity which is a multiple of the weight of carbamide. Resins, fillers, etc. may be added. B. FULLMAN.

Furfuraldehyde resins. Furfuryl alcohol resins. Furan derivatives. Resinous substance. J. P. TRICKEY and C. S. MINER, Assrs. to QUAKER OATS CO. (U.S.P. 1,665,233-7, 10.4.28. Appl., [A, B] 26.5.24, [C] 31.5.24, [D] 5.7.24, [E] 23.8.24. Renewed [A, B, D, E] 27.2.28).-Furfuraldehyde is catalytically treated with (A) a metallic salt, (B) a metal, to yield a resin. (c) Treatment of furfuryl alcohol alone yields an infusible and insoluble resin. (D) Furfuraldehyde, on treatment with alkali, yields furfuryl alcohol and a salt of pyromucic acid. On acidification and heating, the pyromucic acid is set free and the alcohol resinified. (E) Mixtures of furan derivatives individually capable of resinification are converted into compound resins (e.g., an alcohol and an aldehyde). B. FULLMAN.

Water-resistant polyhydric alcohol-carboxylic acid resins. C. R. Downs and L. WEISBERG, Assrs. to BARRETT Co. (U.S.P. 1,667,197—1,667,200, 24.4.28. Appl., [A] 22.12.20, [B-D] 7.9.21).—See B.P. 173,225; B., 1923, 667 A.

Accelerator for hardening phenol-urea products. H. V. POTTER and J. W. CRUMP, ASSTS. to DAMARD LACQUER CO., LTD. (U.S.P. 1,667,675, 24.4.28. Appl., 11.9.26. U.K., 23.10.25).—See B.P. 264,601; B., 1927, 228.

[Lacquer-]enamelling furnaces. INTERNAT. GEN. ELECTRIC Co., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 283,591, 14.1.28. Ger., 15.1.27).

Coagulation and peptisation (B.P. 287,943).— See III. Colour lakes (B.P. 270,750).—See IV. Pigments (B.P. 270,658).—See XIV.

# XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rotatory powers and melting points of the resinous constituents of gutta-percha, balata, and allied gums. S. MINATOYA and H. KANEKO (Res. Electrotech. Lab., Japan, 1928, No. 223, 18 pp.).—The resinous constituents were separated into three divisions, fluavile, spherical albane, and needle albane. The m.p. of the fluaviles of gutta-percha lie between 37° and 90°, whilst those from balata are liquid. As a rule the specific rotatory powers of the fluaviles of gutta-percha are higher than those of the corresponding constituents from balata. The albanes are present only in small quantities. S. COFFEY.

Elastic behaviour of india-rubber. G. B. DEODHAR and D. S. KOTHARI (Indian J. Physics, 1928, 2, 305-318; cf. Phil. Mag., 1923, [vi], 45, 471).-A dynamical method is described for determining the variation of the modulus of rigidity of rubber with stress. Tests on three samples showed that the rigidity increases linearly with stress up to breaking-point. A qualitative study of the effects of heat shows that a stretched band, when heated to about 100°, contracts, but if the heating is stopped the band extends until its length exceeds that before heating, and this extension is permanent. It is possible that, just after heating, the rigidity falls considerably, but after some time it regains almost its initial value at that load. The logarithmic decrement, unlike that of a metal, decreases with the load. The behaviour of metals is similar, the difference being one of magnitude only. In variation of "torsional stiffness" with load, the behaviour of rubber seems to resemble that of annealed wire. M. S. BURR.

#### PATENTS.

**Porous soft and hard vulcanised rubber goods.** H. ZIEGNER (B.P. 283,566, 21.11.27. Ger., 14.1.27).— An absorbent filler such as sawdust saturated with a swelling agent, *e.g.*, benzine, is introduced rapidly into a mixture of rubber with the usual compounding ingredients. The absorbed solvent causes the rubber on the surface of the particles to swell, and during subsequent drying the swollen rubber shrinks, producing capillaries. The porosity of the vulcanised product may be increased by filling the capillaries before or during vulcanisation with a non-swelling substance such as water.

D. F. Twiss.

**Production of rubber goods directly from latex.** DUNLOP RUBBER CO., LTD., G. W. TROBRIDGE, and E. A. MURPHY (B.P. 287,946, 20.10.26).—Articles are manufactured by immersing in an aqueous dispersion of organic material, such as rubber, a hollow porous mould which has previously been dipped in a liquid, *e.g.*, a solution of calcium chloride or other coagulant. Suction may be applied to the interior of the mould during the immersion in the dispersion to expedite deposition of the material and/or after subsequent removal to hasten drying. Completion of drying and vulcanisation are then effected by known processes. D. F. TWISS.

Production of articles from rubber and other thermoplastic substances. A. T. GUSTAFSON (U.S.P. 1,665,355, 10.4.28. Appl., 4.1.26. Swed., 12.2.25).— In the production of moulded articles from rubber and other materials which become plastic on heating, a thin layer of cellulose derivatives is placed between the mould and the material. L. A. COLES.

Vulcanisation of rubber substances. GRASSELLI CHEM. Co., Assees. of W. B. BURNETT and I. WILLIAMS (B.P. 265,930, 29.12.26. U.S., 12.2.26).—Vulcanisation is accelerated by a condensation product of a primary amine, e.g., n-butylamine, aniline, or o-tolyldiguanide, with more than a bimolecular proportion of an  $\alpha\beta$ saturated aldehyde, e.g., propaldehyde, n-butaldehyde, or heptaldehyde, prepared in the presence of a weak organic acid, e.g., acetic, stearic, or salicylic acid, as condensing agent. D. F. Twiss.

Treatment of pulverulent [rubber] fillers and pigments having a carbon base, particularly lamp blacks, carbon blacks, etc. COMP. LORRAINE DE CHARBONS, LAMPES, & APPAREILLAGES ELECTRIQUES (B.P. 270,658, 28.3.27. Fr., 10.5.26).—The particles of lamp black, carbon black, and similar carbonaceous powders are coated with a very thin covering of rubber, *e.g.*, by mixing with 1% of dissolved rubber and subsequently removing the solvent. Black so treated shows no objectionable dustiness, is compact for storage and carriage, and can be incorporated in rubber without difficulty. D. F. Twiss.

Manufacture of products [artificial rubber] from cashew nut-shell oil. M. T. HARVEY (B.P. 262,134, 27.11.26. U.S., 28.11.25).—Artificial rubber compounds may be prepared by heating cashew nutshell oil and glycerin in varying proportions and for different periods to a minimum initial temperature of 240°. The thickened material, having the general appearance of rubber, may be vulcanised to give an elastic product which is soluble in the usual commercial rubber solvents. E. Holmes.

Coagulation and peptisation (B.P. 287,943).— See III.

# XV.-LEATHER; GLUE.

Practical glass extractor [for tanning materials]. R. W. FREY and H. C. REED (J. Amer. Leather Chem. Assoc., 1928, 23, 124-128).-The extractor consists of a glass tube  $2\frac{1}{8}$  in. diam. and  $8\frac{3}{4}$  in. long, closed at its lower end. A central vapour tube  $10\frac{1}{4}$  in. long projects through the bottom, so that only  $3\frac{1}{2}$  in. remains outside it. This tube is closed at the top, and has two vapour outlets near the top and two ears near the bottom of the large tube. A further side tube is fitted into the large tube as near the bottom as possible. In use, a pad of cotton wool covers the open ends of the ears, upon which the tanning material rests. A filterpaper thimble placed over the vapour tube acts as a spreader for the condensed vapours. For extraction without refluxing, the extract is led off through the side tube. By sealing the side tube the extract collects in the large tube and flows through the ears into the boiling flask to which it is attached. A condenser on the top of the large tube completes the apparatus. D. WoodRoffe.

#### PATENTS.

Preparation of mineral acid-free synthetic tanning agents. J. R. GEIGY A.-G. (B.P. 276,014, 12.8.27. Ger., 16.8.26).—Synthetic tanning agents obtained from aromatic hydroxysulphonic acids and formaldehyde are

neutralised with sodium hydroxide, dried, and 100 pts. of the dry product are mixed with a fluosilicate, thereby liberating the tanning acid. D. WOODROFFE.

Settish Chemical Adultation 3

Production of aromatic sulphonic acids for use as tanning agents. I. G. FAREENIND. A.-G., Assees. of A. STEINDORFF, K. DAIMLER, and G. BALLE (G.P. 445,569, 6.5.23).—The products are obtained by the sulphonation of diarylmethylene ethers prepared from mononuclear phenols. Suitable material for sulphonation includes methylene diphenyl ether and the product obtained by the condensation of methylene chloride with low-boiling low-temperature tar phenols. L. A. COLES.

Manufacture of leather dressings. BRIT. DVE-STUFFS CORP., LTD., T. H. FAIRBROTHER, and A. REN-SHAW (B.P. 287,222, 14.12.26).—A small amount (0.05%) of the alkali salt of a polyhalogenated phenol, particularly 2:4:6-trichlorophenol, is added to leather dressings to prevent mildew or other fungoid growths. D. WOODROFFE.

Tanning of hides and skins. E. G. STIASNY and B. JALOWZER (B.P. 287,221, 14.12.26).—Complex ferrisulphite compounds or substances forming the same, with or without the joint use of other substances used in the tanning industry, are utilised. These complex compounds may contain an organic acid radical, and/or organic compounds, especially carbohydrates. An example is given of a product prepared by mixing 1 mol. of ferric chloride with 1—5 mols. of sodium sulphite. D. WOODROFFE.

Production of a waterproofing bottom-filling composition for boots, shoes, etc. H. BROOMFIELD, and SHOE INVENTIONS, LTD. (B.P. 287,206, 7.12.26).

Coloured leather. A. C. LAWRENCE LEATHER Co., Assees. of C. P. KELLEY and E. W. WHITE (B.P. 262,780, 7.12.26. U.S., 8.12.25).

Fast dyeings (B.P. 287,651).—See VI. Diffusion process (B.P. 274,131).—See XVII.

# XVI.—AGRICULTURE.

Determination of the need of soils for phosphoric acid relative to the soluble silica content. A. NEMEC (Compt. rend., 1928, 186, 1060-1062).-The percentage increase in crops of various roots, grain, tubers, and grass has been correlated with the mean content in soluble silica [determined colorimetrically either by the method of Bell and Doisy (A., 1920, ii, 769) or of Atkins (B., 1924, 483)] of the cultivated and sub-soil. On the basis of 200 results it is found that when the mean silica content is greater than a certain minimum value, the addition of phosphorus as superphosphate has scarcely any effect on the crop yield. The following minimum values are found : sugar beet 12, wheat 11, barley and potato 10, rye 9, oats 7, and grass 6 mg./kg. of soil. J. W. BAKER.

Rapid determination of the phosphoric ion in soils and manures by coeruleomolybdimetry. G. DENIGÈS (Compt. rend., 1928, 186, 1052—1054).—If the phosphoric ion is water-soluble the determination is made on the diluted filtered extract by the author's method (A., 1928, 263), any colour being balanced by means of a similar boiling, acidified extract placed in front of the standard at the moment of matching. Otherwise 50 c.c. of a 2% solution of nitric acid may be used to extract 5 g. of soil, and the extract diluted 50 times. If more concentrated acid is required 1 c.c. of extract must be evaporated, and the residue calcined and extracted with boiling acidulated water. For soils containing 1-2% Fe 12 drops of reagent are used, but greater amounts (up to 50%) must be reduced by the preparation of the reagent *in situ* in the presence of 0.1 g. of copper turnings which serve to re-reduce the reagent after it has itself reduced the iron. Iron should be added to the standard. J. GRANT.

Sulphur cycle in soil. A. RIPPEL (J. Landw., 1928, 76, 1-10).—The sulphur cycle in soil resembles the nitrogen cycle. Mineralisation of humus sulphur takes place slowly. The addition of soluble carbohydrate to a compost soil causes the water-soluble sulphate to disappear. This change is occasioned by the growth of fungus mycelia, and can be demonstrated in pure culture. It is claimed that *Aspergillus* is capable of oxidising elementary sulphur, and that the sulphuric acid produced by micro-organisms substantially influences soil acidity and rock decomposition. H. J. G. HINES.

Measurement of suction power in the seedling stage. K. MEYER (J. Landw., 1928, 76, 11-24).— A preliminary communication describing modifications of Buchinger's apparatus and procedure. Seedlings are grown on glass lattices above sugar solutions in a shallow zinc tank. The changes in sugar concentration are determined polarimetrically. H. J. G. HINES.

Relation between mechanical composition and hygroscopic coefficient of a soil. F. GIESECKE (J. Landw., 1928, 76, 33-40).—No relation could be found between the hygroscopic coefficient and the clay content or the ratio of clay to fine silt.

H. J. G. HINES.

Extraction of soils by the hydrochloric acid method. E. BLANCK and A. RIESER (J. Landw., 1928, 76, 25—31).—Three modifications of the method proposed by the International Commission were compared with the Commission's method and substantial differences observed in the subsequent analyses. The method of preparation of the soil for analysis also affected the proportion dissolved by hydrochloric acid. H. J. G. HINES.

Determination of chlorophyll by Willstätter's method. K. MAIWALD (J. Landw., 1928, 76, 63-69). T. STECHE (*ibid.*, 71-73; cf. B., 1928, 136).—(A) An adverse criticism of Steche's modification of Willstätter's method and a description of the procedure recommended by the author. (B) A reply. H. J. G. HINES.

Influence of stimulants on the sprouting of potatoes. W. von VELSEN (J. Landw., 1928, 76, 41-62). —Chemical and physical methods of stimulating the sprouting of three varieties of potatoes were tested. The physical methods used were ineffective, but a number of chemical agents hastened the sprouting. Treatment with hydrocyanic acid, thiourea, ethylene dichloride, and potassium thiocyanate, or injection of diastase gave marked effects. The number and total weight of sprouts were increased also by these reagents. On following the development in spring and summer, however, no differences could be observed between treated and untreated tubers. H. J. G. HINES.

Citric acid-soluble phosphate in basic slag. A. SÜLLWALD (Arch. Eisenhüttenw., 1927-8, 1, 565-570; Stahl u. Eisen, 1928, 48, 547-548).-Basic slags containing sufficient calcium silicate to form the double compound with calcium tetraphosphate are unaffected by the rate of cooling under ordinary conditions, but rapid quenching or granulation in water causes a decrease in the citric acid-soluble phosphate content. When the slag is cooled in large blocks the total phosphate and the acid-soluble phosphate contents increase regularly from the outside towards the middle. Reheating a basic slag with a low silica content decreases the proportion of acid-soluble phosphate owing to the decomposition of the free calcium tetraphosphate into lime and the less soluble compound, oxyapatite. A. R. POWELL.

Fumigation of stored-product insects with certain alkyl and alkylene formates. R. T. COTTON and R. C. ROARK (Ind. Eng. Chem., 1928, 20, 380—382; cf. B., 1927, 862).—Quantitative laboratory experiments showed that the vapours of methyl, ethyl, propyl, *iso*propyl, *iso*butyl, *iso*amyl, and allyl formates are all toxic to the rice weevil, and have little or no effect on the germination of wheat. Addition of 60—75% by vol. of carbon tetrachloride renders these formates (with the exception of the methyl and ethyl compounds) "free from fire hazard," *i.e.*, the vapour of the mixture heated to 50° with air will not propagate a flame when sparked. Large-scale tests indicated that some of the mixtures appeared promising as fumigants against various stored-product insects. C. T. GIMINGHAM.

# XVII.—SUGARS; STARCHES; GUMS.

Natural alkalinity [of beet juices]. O. SPENGLER and C. BRENDEL (Z. Ver. deut. Zucker-Ind., 1927, 801-816).-By the natural alkalinity of beet juice is understood the residual alkalinity after treatment with lime and precipitation of this by carbon dioxide, and it represents free potash and soda originally present as salts. The greater part of the alkali metals in fresh juice from sound beets is combined with acids which are precipitable by lime. In carbonatation, therefore, it is not necessary to leave any free lime to supply the requisite final alkalinity, the liberated potash and soda sufficing for this. These are present as hydroxides at the end of the first carbonatation, and should be converted into carbonates, but not into bicarbonates, by the second. In old or damaged beets the alkali metals are combined with acids which are not precipitable by lime, and they remain as neutral salts after carbonatation, so that there is a deficiency of natural alkalinity, and it becomes necessary to leave some free lime after carbonatation to obtain the necessary final alkalinity. To avoid this, since lime alkalinity is particularly undesirable during evaporation, a suitable amount of sodium carbonate may be added to the juice, e.g., before the final carbonatation. As a guide in ascertaining how much to add, the authors describe methods for

determining the "theoretical" and the "practical" residual alkalinity, on filtered juice from the first The "theoretical" residual affinity carbonatation. is the excess of the total alkalinity (to phenolphthalein) over the lime content (determined by soap solution), both being expressed as CaO. Probably, however, the "practical" residual alkalinity will afford a closer estimate of the condition of the juice after the final carbonatation. It is found by neutralising to phenolphthalein with 0.2N-hydrochloric acid, then adding an equal volume of 0.2N-sodium carbonate, heating in boiling water, filtering from calcium carbonate, cooling, and determining the residual alkalinity with 0.0357Nhydrochloric acid. Full working details and precautions are given. Factory experience alone can decide which of these methods is the more useful. J. H. LANE.

Copper-reducing substances contained in beetroots and diffusion juice. L. BRANCOURT (Bull. Assoc. Chim. Sucr., 1928, 45, 251-254).-In diffusion juice from beets which have been stored or slightly injured by frost or other influences, the copper-reducing power is not a sure measure of the invert-sugar content. Decomposition of cellulosic and protein constituents of the beets is liable to occur, especially in the last units of the diffusion battery, and give rise to reducing substances without a corresponding disappearance of sucrose. These reducing substances differ from invert sugar in that, with proper treatment of the juice, they are not so liable as the latter to give rise to coloured substances on liming and evaporation. In badly frozen beets the decompositions which set in after thawing result in the formation of viscous products. Reducing substances are then apt to occur in widely varying amounts, and on liming they yield soluble calcium compounds. The precipitate formed on carbonatation is thereby reduced in amount and of a character difficult to filter even when considerable amounts of sodium carbonate are used. In such cases the author recommends heating the juice to actual boiling, at least after the first carbonatation, and adding a granular form of calcium carbonate to assist filtration. J. H. LANE.

Drying of sugar products and determination of moisture. D. SIDERSKY (Bull. Assoc. Chim. Sucr., 1928, 45, 247-249).-Heating of samples in a capsule inserted in a tube through which dry air was passed and to which a calcium chloride tube was attached, showed that at 108° constant weight both of sample and of calcium chloride tube could be attained in 2 hrs. with first-product beet sugars. With second and lower beet products the calcium chloride tube reached constant weight in 21-3 hrs., but the samples underwent a slight further loss (about 0.1%) during the next hour. With raw cane sugars, constancy of weight could not be reached even in 6 hrs., probably owing to slow decomposition of lævulose. For exact determinations of moisture heating in vacuo below 95° is recommended, although the ordinary method serves well enough for commercial analyses. J. H. LANE.

Electrometric determination of the ash of sugar factory products. O. SPENGLER and F. TÖDT (Z. Ver. deut. Zucker-Ind., 1928, 1-12).—Electrometric ash

determinations on raw sugars are best made on solutions of 5° Brix, for although errors due to small variations in sugar concentration are least in solutions of about 30° Brix, the results obtained at this concentration are liable to deviate much more widely from the results by incineration than those obtained with 5% solutions, the conductivities of the individual salts present being affected to different extents by high concentrations of sugar. In general, the influence of the conductivity of the water used is depressed in presence of the sugar and its salts. Zerban and Sattler (Facts about Sugar, 1926, 1158) found that water of very low conductivity,  $3 \times 10^{-6}$ , exerted its full influence, but water of conductivity  $40 \times 10^{-6}$  contributed only  $27 \cdot 4 \times 10^{-6}$  to the conductivity of 5% raw cane sugar solutions. The factor Crepresenting the ratio between conductivity and ash content (by incineration) was found by Zerban and Sattler to range from 1476 to 2022 for cane sugars of various origin in 5% solutions. For sugars from the same district it was fairly constant, being 1786 on the average for Cuban, and 1560 for British West Indian sugars. For 50 raw beet sugars tested by Kayser (B., 1926, 685) it ranged from 1680 to 1880. According to Lundén (B., 1926, 927) it is lower for beet factory products than for refinery (i.e., affined) beet products; and, in general, as the authors have confirmed, there is an inverse relation between C and the quality of beet products. A high value of C is associated with a high ratio of organic to inorganic salts, which indicates inferior quality. For raw cane sugars Zerban and Sattler found that if k is the specific conductivity of a filtered solution containing 10 g. of sample in 200 c.c., and  $k_1$  that for a similar solution containing also 5 c.c. of 0.25N-hydrochloric acid, the ash content is given by the formula:  $0.0001757(9.13k + 1935 - k_1)$ . The results show much more uniform agreement with incineration results than when a single electrometric factor is used. By this formula variations in the amount of organic salts present are largely compensated. since they correspondingly lessen the value of  $k_1$  by the replacement of part of the added mineral acid by feebly dissociated organic acids. J. H. LANE.

Applicability of the methods of beet sugar factories to the production of lactose. O. UNGNADE (Chem.-Ztg., 1928, 52, 69-71).-The whey which forms the usual raw material of lactose manufacture contains about 4.83% of lactose, 1.17% of organic non-sugars, and 0.64% of ash. It contains much more acid than raw beet juice, and is not adapted to liming and carbonatation, as calcium salts not precipitable by carbon dioxide would be formed. The protein is best coagulated by heat, the neutralised whey being brought to 90° in steam-heated vessels and kept for  $\frac{1}{2}$  hr. Heating by injection of steam is often practised, but causes unnecessary dilution. The clear upper liquid is drawn off for evaporation, and the sludge is passed to filter presses which, under a liquid head of 2-3 m., work very satisfactorily and are much preferable to the bag filters still used in some factories. In evaporation great economy would result from the replacement of the single-unit evaporators now generally employed, by small-scale multiple-effect plant, in which the whey could be safely

brought to a dry substance content of 50%. Crystallisation is usually carried out in water-cooled copper tanks, but a modified form of "malaxeur," as used in sugar factories for crystallisation in motion, gives better results. The purging of the crystals in the centrifuge is best effected by a fine water spray delivered under 3-4 atm. pressure. The raw sugar contains lactose  $92 \cdot 7\%$ , water  $1 \cdot 85\%$ , organic non-sugar  $3 \cdot 34\%$ , and ash  $2 \cdot 14\%$ . The refining of the product could be rendered more efficient by improved conditions of crystallisation and the use of decolorising carbons, sodium hyposulphite, or sulphur dioxide. J. H. LANE.

Clerget factor and the Deerr method of double polarisation. C. E. COATES and C. SHEN (Ind. Eng. Chem., 1928, 20, 70-74).-With Deerr's method for determining sucrose (B., 1915, 503) the authors found inversion constants consistently about 1.5 units lower than those given by Deerr. To avoid possible sources of error they propose certain modifications. The direct polarisation is made on a solution treated only with barium hydroxide and aluminium sulphate solutions in equivalent amounts. The inversion is carried out on a separate portion of the original solution with 0.4N-sulphuric acid by heating to  $68^{\circ}$  in 3 min. and maintaining this temperature for 7 min.; after cooling, the same volume of aluminium sulphate solution is added as was added to the other, and the liquid is exactly neutralised (to rosolic acid) with the barium hydroxide solution, filtered, and read. Corrections are made for the volumes of the precipitates. The inversion constant found for half sugar normal solutions is 141.7, and that for quarter sugar normal solutions 140.89. J. H. LANE.

Decomposition of sucrose by adsorbent carbons. E. LANDT (Z. Ver. deut. Zucker-Ind., 1927, 834—840).— A mathematical exposition of Vašátko's results (B., 1928, 29.) J. H. LANE.

Corn starch. STALEY and HELFRECHT.-See XI.

# PATENTS.

Diffusion process and apparatus. K. KOMERS and K. CUKER (B.P. 274,131, 11.7.27. Czechoslov., 10.7.26).-Materials with undamaged cell membranes (e.g., beet slices, sugar cane wood, bark, etc.) are freed mechanically from superficially adhering juices, and then treated with a current of preheated air, flue gases, or other gas, which, without injury to the cell membranes, removes part of the internal moisture of the cells and oxidises the cell conglomerates (the oxidation, which may be done separately, converts leuco-bases and coagulates colloids). In practice, the material, freed from superficial juices before entering the diffuser or between individual elements of a battery of diffusers, is passed through a vessel for treatment with gases. By this process beets may be completely leached out in 25 min. B. FULLMAN.

Production of starch soluble in cold water. PFEIFFER, and Dr. SCHWANDNER G.M.B.H. (G.P. 445,557, 1.11.24).—Hot water in a finely-divided form is added to dry starch, in quantity sufficient to work it up into a thick paste which only needs drying in the air.

L. A. Coles.

Preparation of [pure] tricalcium saccharate. C. STEFFEN, JUN. (U.S.P. 1,667,446, 24.4.28. Appl., 27.4.26. Austr., 17.11.25).—See B.P. 261,693; B., 1927, 454.

Salts from fermented molasses (B.P. 288,390).— See XVIII.

# XVIII.—FERMENTATION INDUSTRIES.

Sarcinæ. K. SILBERNAGEL (Woch. Brau., 1928, 45. 143-148, 155-160, 165-168).-Sarcinæ were isolated on nutrient agar from beer and from air and classified into two groups. The yellow varieties in both the packet and non-packet forms exhibited a uniform and inherently related behaviour with regard to growth and other properties, whilst the colourless species, differing both from the yellow variety and also among themselves, showed marked individuality. Two of the most suitable sources of carbon for the reproduction of the micro-organism were lactic acid and pyrotartaric acid, whilst, of the carbohydrates, achroodextrin, inulin, and lævulose were assimilated most easily. Lactic acid had a specially stimulating action, and a few drops of its solution, when added to the nutrient, produced a rapid development of the organism. The nitrogen requirements of the sarcinæ were best satisfied by ammonium salts, and their presence produced rapid reproduction. Urea was assimilated very easily, but peptone induced a lag period prior to later development. The ease with which amide-nitrogen was assimilated depended on the complexity of the compound. Rapid growth accompanied the use of alanine, whereas the nitrogen from asparagine and leucine was assimilated with difficulty. Glycine was not so effective a source of nitrogen as alanine, whilst methylamine, nitrates, and nitrites yielded no nitrogen. Among the enzymes present in the sarcinæ were amylase, lipase, and the proteolytic enzymes in moderate amounts. Catalase and peroxidase were found in greater quantity, whilst invertase and amidase occurred as traces. Of the other enzymes tested, maltase, inulase, and lactase were absent. C. RANKEN.

Taka-invertase. R. WEIDENAGEN (Z. Ver. deut. Zucker-Ind., 1928, 125—134).—Taka-diastase was found to be without action on melibiose, but to hydrolyse raffinose and sucrose at velocities in the ratio of 1:1.96. It is concluded that taka-invertase is a fructo-invertase and is not associated with a melibiase as supposed by Leibowitz and Mechlinski (A., 1926, 865). The decomposition of raffinose was complete within. 24 hrs., and even after 96 hrs. no carbohydrates other than melibiose and lævulose could be detected. The optimum  $p_{\rm H}$  value for taka-invertase is about 5.0. Evidence for and against the existence of gluco- and fructo-invertases is considered. F. E. DAY.

Evaluation of a dried yeast in relation to its biological action. W. WEICHARDT and H. UNGER (Pharm. Ztg., 1928, 73, 526-527).—A quantitative examination of the biological value of "Levurinose," a commercial dried yeast. E. H. SHARPLES.

English barleys of 1927. Н. М. Снивв (J. Inst. Brew., 1928, 34, 214—217).—Owing to the very wet season about 30% of the barley crop was unfit for malting. The general average of the available crop was poor, but the best barleys malted well. Local conditions and environment were greater factors than usual, and good barleys were found in small quantities all over the country, the best being on the edge of the chalk in West Norfolk, Suffolk, and East Cambridge.

# C. RANKEN.

Critical analysis of malt. A. T. HENLEY (J. Inst. Brew., 1928, 34, 223-225).—Samples of malt are separated into fractions of differing sp. gr. by flotation liquids which are prepared by mixing benzol and carbon tetrachloride. Both of these liquids are substantially without effect on the malt, and can be removed easily by a current of dry warm air. Examination of the quantity of extract obtained from each of the several fractions allows information to be drawn as to the reasons for any want of modification of the malt, whilst any trace of overheating in the kiln will be reflected almost entirely in the analytical values obtained from only one of the fractions. C. RANKEN.

Finings. H. W. HARMAN, J. H. OLIVER, and P. WOODHOUSE (J. Inst. Brew., 1928, 34, 203-213).-In the absence of other colloids, maximum precipitation of the finings occurs in the neighbourhood of an isoelectric point of  $p_{\rm H}$  5.0, approximately, whilst with low rates of addition no precipitation takes place at  $p_{\rm H}$  3.7 and  $p_{\rm H}$  4.0. From a comparison of the result of fining beer and buffered solutions it is concluded that fining takes place chiefly owing to the mutual precipitation effect produced when two colloidal solutions of electrically opposite charges are mixed. A certain amount of finings are left in solution, which stabilises the other colloids in solution. This protective power varies according as the method of cutting the isinglass, and is entirely absent in finings which have been overcut. The action of finings may be retarded by the presence of wild yeast, excessive hop rates, and by variation of the temperature during fining, whilst the presence of carbon dioxide accelerates the action. Irish moss acts as a precipitant to isinglass fining. C. RANKEN.

Samsu from rice. R. O. BISHOP and G. L. TEIK (Malay. Agric. J., 1928, 16, 14-19).-The process used by the Chinese in Malaya depends on the utilisation of a mould containing a ferment (Amylomyces Rouxii) which converts moist starch into alcohol. The mould is added in the form of "rice-cake" consisting of rice starch, soya beans, clay, vegetable tissue, and variable amounts of mucors and yeasts. The rice is boiled, allowed to cool, and the rice cake roughly mixed into it in the proportion of 3 pts. of rice to 1 pt. of rice cake. The mixture is placed in earthenware jars with water and left for 27 days. Distillation is carried out in a primitive apparatus which is described. A yield approximately 54% of the theoretical is obtained. This could be considerably increased if suggested improvements in the cooking, fermentation, and distillation were introduced; such changes would, however, probably alter the aroma and flavour of the beverage.

W. J. BOYD.

Uniform behaviour of bottom-fermentation beer

yeast in respect of fermentation, reproduction, and acid-formation, on storage under water at various temperatures. F. STOCKHAUSEN and F. WINDISCH (Woch. Brau., 1927, 44, 557—564, 573—579).—Similar experiments to the earlier ones (cf. B., 1928, 31) were made with three other typical bottom yeasts, and with the same results. J. H. LANE.

Sugar : alcohol ratio and the stability of sweet wines. P. MALVEZIN (Bull. Assoc. Chim. Sucr., 1928, 45, 396-399).-In connexion with his work on the preparation of vaccines for the prevention of secondary fermentation, the author noted that he was always successful when the sugar/alcohol ratio was less than 3, but never when the ratio exceeded 4. On reference to the data for over 100 samples of sweet wines received in his laboratory on account of secondary fermentation, all were found to give a ratio of over 4 and generally nearer 6. Samples which had been recorded as keeping well had mean sugar/alcohol ratios of: Gironde 1.3, Sauterne 3.65, Anjou 3.81, Gaillac 7.31. The author concludes that for sweet wines generally, the ratio should not exceed 3.5, but may reach 3.8 in Anjou wines, and 7 in Gaillac and similar wines in which secondary F. E. DAY. fermentation is usual.

#### PATENTS.

Clarification of beer, vinegar, and other like liquids. A. J. MURPHY, and MURPHY & SON, LTD. (B.P. 286,861, 18.2.27).—Finings for the clarification of beer, vinegar, and other liquids are produced by covering a suitable quantity of isinglass with water to which is added lactic acid in suitable proportion. As the isinglass progressively swells under treatment, more water is added and the mass stirred, churned, or sieved. Phosphoric acid or other suitable mineral or organic acid may be used in addition to the lactic acid.

C. RANKEN.

Collection of inorganic potassium salt, betaine salt, and glutamic acid from the waste liquor produced in distilling alcohol from fermented beet molasses. Y. TAKAYAMA (B.P. 288,390, 22.1.27. Cf. B.P. 233,196; B., 1925, 568).-The waste liquor is electrically or otherwise dialysed at 40-70°, yeast, proteins, etc. being thus removed. The dialysed liquor is concentrated, treated with hydrochloric acid, kept at 100° for 1 hr., and cooled, when potassium chloride separates. Alternatively, the concentrated liquor is treated with chamber sulphuric acid, heated, and, after removal of potassium sulphate, slaked lime is added to remove excess acid. On concentrating the motherliquor, betaine hydrochloride (containing potassium chloride) crystallises. The residual liquor is treated with hydrochloric acid and heated under pressure at 110-120° for 1-2 hrs. to convert glutimic into glutamic acid. On concentration, crude glutamic acid hydrochloride crystallises, and is purified by recrystallisation or conversion into calcium glutamate. B. FULLMAN.

Extraction of glycerin from distillery vinasses. Soc. DES ÉTABL. BARBET (B.P. 274,519, 19.7.27. Fr., 19.7.26).—Petroleum, toluol, benzol, or other like product is added to the concentrated distillation vinasses and the mixture superheated under a pressure of 6—8 kg. The pressure is suddenly lowered and self-evaporation produced, by means of which part of the glycerin is carried over with all the petroleum and part of the water. The first portion of the distillate is later added to the main bulk of the vinasses for re-treatment, whilst the second portion, after rectification, is purified by charcoal, filtered, and concentrated in a vacuum. C. RANKEN.

Recovery of nitrogen and acetone from vinasses and/or molasses. NOUVELLES INDUSTRIES CHIMIQUES Soc. ANON. (B.P. 287,829, 13.6.27. Addn. to B.P. 277,932; B., 1928, 345).—An alkaline-earth carbonate, or residue from a previous distillation, or a hygroscopic substance, *e.g.*, sawdust, is added to the mixture of vinasses or molasses and lime and subjected to destructive distillation up to 350°. H. ROYAL-DAWSON.

**Production of citric acid.** DISTILLERS Co., LTD., and A. M. PEAKE (B.P. 287,604, 27.11.26).—Fruit pulp, wood dextrins, gums, and similar pectinous substances are treated with citric acid enzymes present in the pith of fresh and preferably unripe citrus fruit from which the essential oils have been removed. A small amount of manganese or similar oxygen-carrying catalyst may be added. The reaction should be carried out at 25—35°, and moderate aeration is advantageous.

C. RANKEN.

Filters [for wines and liqueurs]. J. CUÉNOUD (B.P. 288,920, 8.11.27).

# XIX.—FOODS.

Use of Janus-green in the reductase test for milk. A. R. TANKARD (Analyst, 1928, 53, 213).-A comparison of results given in the reductase test by 35 samples of commercial milk using both Janus-green and methyleneblue gave similar results. The rising of the cream in the tubes appears to inhibit oxidation. Janus-green acts more slowly than methylene-blue, and the red colour produced in the first stage of the reduction shows early in the cream when the dye is about to be reduced throughout the milk, and in some instances may show when the full reaction does not occur within the time limit. A bacterial count shows the number of organisms to be generally lower than that indicated by either form of the test. D. G. HEWER.

Determination of salt in margarine. D. W. STEUART (Analyst, 1928, 53, 212—213; cf. B., 1928, 241). —If to 3 g. of melted margarine 10 c.c. of acetone and a few drops of chromate indicator are added, and the mixture is titrated with 0.1N-silver nitrate solution, the same result is obtained as if the aqueous extract of the solids-not-fat is used. D. G. HEWER.

By-products of the pineapple canning industry. V. R. GREENSTREET and G. L. TEIK (Malay. Agric. J., 1928, 16, 8—13).—Malayan methods of pineapple canning are inefficient compared with those employed in Hawaii, owing to the uneconomic disposal of waste matter. This material, consisting of cores, skins, and rotten and unripe fruit, is in the proportion of 2 pts. to each part of canned fruit. It contains about 90% of moisture, together with fibre, pentoses, sugars, organic acids and protein matter. Possible methods of disposal are: (a) incineration to produce a valuable manure (the ash contains about 6%  $P_2O_5$  and 24%  $K_2O$ ); (b) conversion into cattle food; (c) spreading the crushed waste in shallow layers on the ground so that it decomposes. All such methods involve a preliminary grinding and pressing whereby a liquid containing sugars is produced, disposal of which must also be considered. In Hawaii the waste is converted into "pineapple bran" —a valuable cattle food—containing water 10.63%, protein 3.62%, ether extract 1.01%, invert-sugar 11.96%, starch 42.15%, fibre 18.23%, and ash 3.70%. Trials showed that the fermentation and distillation of the juice, for the production of a potable alcoholic liquor similar to brandy or samsu, can be carried out without difficulty. W. J. Boyp.

Aluminium vessels. BUSCHLINGER.—See I. Ultraviolet light through glass. STARKIE and TURNER.— See VIII.

#### PATENTS.

Treatment of cereals. TREUHAND-GES.M.B.H. BARTMANN & Co. (B.P. 270,706, 2.5.27. Ger., 6.5.26).— The grains are steeped in water containing formaldehyde or lime to cause softening and swelling of the endosperm, during which intramolecular respiration is prevented by supplying oxygen to the grains by introduction of air or oxygen into the steeping liquor, by temporarily lifting the grains out of the liquor, or by transferring the grains from one steeping bath to another.

F. R. Ennos.

Preparation of vegetable food. E. H. MILES and G. REILLY (B.P. 274,051, 17.6.27. U.S., 8.7.26. Addn. to B.P. 256,765).—The juices extracted from vegetable material having essentially dietetic properties (e.g., lettuce, cabbage, etc.), and from other vegetable material having also preservative properties (e.g., carrots), are heated separately to 80°, cooled, and strained, then mixed together, again heated to 80° until hydrolysis of the sugars is well advanced, cooled to about 30°, and concentrated *in vacuo*, the total time of heating not exceeding 1 hr. L. A. COLES.

Manufacture of live-stock feeds. U.S. FARM FEED CORP., Assees. of C. R. MABEE (B.P. 272,447, 7.3.27. U.S., 11.6.26).—Farm roughage is loosely packed in a wire-mesh container and moistened either with water or with a solution containing a fermentative agent and other material, *e.g.*, malt with calcium and magnesium hydroxides, sulphur, and sodium chloride. It is then allowed to ferment for one or two days in a slowly moving current of air, which is made to circulate through the material by the application of heat or by utilisation of the heat of fermentation. F. R. ENNOS.

# XX.-MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Tests for impurities in ether. I. Test for peroxides. G. MIDDLETON and F. C. HYMAS (Analyst, 1928, 53, 201-209).—The relative sensitiveness of a number of different tests both for hydrogen peroxide and ether peroxide in ether was determined, the most

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useful being potassium iodide and starch, reduced phenolphthalein, leuco-methyl-green and peroxidase, and ferrous thiocyanate. If the maximum attainable purity is to be enforced the ferrous thiocyanate test should be used. It gives uniformly consistent results, and blank tests are readily obtained with pure ether. The amount of peroxide permissible might be defined by comparing the colour of the ether layer, after remaining for 5 min., either with a standard solution of cobalt sulphate or with tintometer glasses. In the former case the tint should not be greater than that of an equal depth of an aqueous solution containing 0.15 g. of crystalline cobalt sulphate and 2 c.c. of dilute sulphuric acid in 100 c.c. (equal to 0.3 red unit on the Lovibond scale for a depth of 30 mm. of liquid). The ferrous thiocyanate solution must be colourless, and may be freshly reduced by a dilute solution of titanous chloride (avoiding any excess of this reagent), or prepared from metallic iron, sulphuric acid, and potassium thiocyanate in an atmosphere of carbon dioxide, in which case it may be kept for several weeks if air is completely excluded. The usual pharmacopœia tests are regarded as unsatisfactory. D. G. HEWER.

Standardisation of ephedrine and its salts. J. B. PETERSON (Ind. Eng. Chem., 1928, 20, 388-391). -Standards are proposed for the control of the purity of ephedrine and its hydrochloride and sulphate. Ephedrine hydrochloride is soluble in chloroform, has m.p. 216-220°, [a]<sup>20</sup><sub>D</sub> - 33.0° to - 35.5°, whilst ephedrine sulphate has m.p. 240-243° (corr.; the rate of heating being strictly according to the method of U.S.P. X.),  $[\alpha]_{p}^{20} - 29^{\circ}$  to  $-30^{\circ}$ . On treating an aqueous solution of the sulphate with ammonia water, extracting with chloroform, and evaporating the chloroform extract, crystals of ephedrine hydrochloride are obtained, but the reaction is not quantitative as benzaldehyde is formed as a by-product; the hydrochloride may also be produced when a chloroform solution of ephedrine is evaporated. Ephedrine has m.p. 34-40°, b.p. above 200°, and  $[\alpha]_{0}^{20} - 6 \cdot 0^{\circ}$  to  $-7 \cdot 5^{\circ}$ . With copper sulphate and sodium hydroxide solution ephedrine and its salts yield a reddish-purple coloration which is partially removed on addition of ether. Crystallographic data are given for the hydrochloride and sulphate. W. J. POWELL.

Determination of yield of "digitaline cristalisée" [obtainable from *Digitalis* leaves]. E. PERROT and P. BOURCET (Compt. rend., 1928, 186, 1021—1024). —A method is described by which the yield of commercial "digitaline cristalisée" (Kiliani's digitoxin) from *Digitalis* leaves may be determined. The alcoholic extract of the powdered leaves is treated with basic lead acetate, evaporated, and the residue extracted with cold chloroform. After evaporation of the chloroform and extraction of the residue with pinene and ether, the solid is again extracted with chloroform and the "digitaline" precipitated by addition of ether and light petroleum. R. K. CALLOW.

Detection of colocynthin in colocynth extract. L. DAVID (Pharm. Ztg., 1928, 73, 525-526).—The isolation of colocynthin, colocynthidin resin, and colouring matter from colocynth extract is described. In the Keller colour reaction only colocynthin gives the crim-

son-coloured ring, and with the exception of the colouring matter, which forms a brown ring, the other constituents give no colour. The test has been modified as follows : 0.2 g. of the extract containing dextrin is mixed with lime paste to the size of a small bean and the mixture is dried on the water-bath. The pulverised residue is extracted twice with 1 c.c. of methyl alcohol each time, collected on a parchment filter moistened with alcohol, and the filtrate evaporated. The residue is dissolved in 2 c.c. of glacial acetic acid, one drop of ferric chloride solution is added, and 2 c.c. of concentrated sulphuric acid are run carefully down the side of the tube. A vivid, carmine-red ring is formed at the junction of the two liquids. The test is reliable and an extract containing only 2% of colocynthin gives a pale but quite perceptible colour. E. H. SHARPLES.

West Australian sandalwood oil. P. MAY (Pharm. J., 1928, 120, 368-369; cf. Perrot, B., 1928, 210).--The physical and chemical constants of 12 West Australian and Mysore sandalwood oils are compared and the alcoholic constituents of a "Plaimar" oil have been examined. West Australian sandalwood oil is now obtainable of good standard quality closely resembling the East Indian oil, to which it is equal in therapeutic value. The properties of the alcohols ( $d^{15}$  0.972,  $\alpha - 9.44^{\circ}$ ,  $n^{25}$  1.506) are not inconsistent with those of a mixture of  $\alpha$ - and  $\beta$ -santalols, but are quite incompatible with the fusanols of Rao and Sudborough (B., 1923, 575 A). E. H. SHARPLES.

Essential oil from Agathis australis. J. R. HOSKING (Rec. trav. chim., 1928, 47, 578—584).—The oil obtained in 0.16% yield by steam distillation of the leaves and shoots of Agathis australis (kauri pine) during December-February has  $d_{25}^{25}$  0.9340,  $n_{D}^{25}$  1.4952,  $[\alpha]_{5561}^{25} + 24.41^{\circ}$ , acid value 0.39. The chief constituents of the oil are citronellal (0.2%), d- $\alpha$ -pinene (64%), d-camphene (0.7%), d-limonene and dipentene (1%), cineole (0.6%), d-borneol (3.2%), bornyl acetate (1.4%), l-cadinene (7%), and a diterpene (13%) to which the name kaurene is given. Kaurene, probably C<sub>20</sub>H<sub>322</sub>, contains one double linking and has m.p. 57—58°,  $d_{29}^{20}$  0.9631,  $n_{20}^{20}$  1.5132,  $[\alpha]_{3461}^{25} \pm 0^{\circ}$ , when regenerated from its hydrochloride, m.p. 110—111°. The oil obtained during June from the leaves of A. australis has  $d_{25}^{24}$  0.9210,  $n_{25}^{25}$  1.4783,  $[\alpha]_{5461}^{26} \pm 33.5^{\circ}$ , and contains d- $\alpha$ -pinene (75%), kaurene (6%), and l-cadinene (3.5%). H. BURTON.

Determination of carvone in dill oil. J. REILLY and P. J. DRUMM (Analyst, 1928, 53, 209-211).-10 g. of dill oil in 120 c.c. of alcohol are added to a cooled solution of 6 g. of semicarbazide hydrochloride in 15 c.c. of water, followed by a solution of fused sodium acetate in 10 c.c. of hot water ; after 24 hrs. the semicarbazone crystallises. Water (840 c.c.) is now added and, after keeping, the precipitated carbazone is collected and dried. Values obtained by this method agree closely with those by the neutral sulphite method, and are approximately 4% less than by the oxime titration method. D. G. HEWER.

Determination of total geraniol content of citronella oil. A. RECLAIRE and D. B. SPOELSTRA (Perf. Ess. Oil Rec., 1928, 19, 143).—In the determination of citronellal by the Kjeldahl analysis of the oxime the factor previously given (B., 1927, 427) is not accurate for preparations containing a high percentage of citronellal. An exact formula is given and also a table for use with oils containing 28—55% of citronellal.

E. H. SHARPLES.

Cod-liver oil. DICKHART. Ergot oil. BAUGHMAN and JAMIESON.—See XII.

#### PATENTS.

Manufacture of thymol from umbellulone. E. K. NELSON, ASST. to U.S.A. (U.S.P., 1,666,342, 17.4.28. Appl., 27.9.27).—Thymol is obtained by passing the vapour of California laurel oil or umbellulone through a tube at 280°, being separated from the crude product in the former case by fractional distillation and in the latter by treatment with caustic alkali. B. FULLMAN.

Manufacture of ketones. (Sir) W. J. POPE (B.P. 287,967, 31.12.26).—The monoacyl derivatives of quinol and resorcinol are isomerised by heating with chloride of magnesium, zinc, aluminium, iron, or tin, or sulphuric or phosphoric acid, giving dihydroxyphenyl alkyl ketones, which may be reduced by the usual method to alkylresorcinols. The following compounds are described: p-hydroxyphenyl propionate; 2 : 5-dihydroxyphenyl ethyl ketone; m-hydroxyphenyl isobutyrate, n-hexoate, b.p. 200—210°/15 mm., laurate, and stearate; 2 : 4-dihydroxyphenyl isopropyl ketone; 4-isobutylresorcinol, m.p. 62—63.5°; 2 : 4-dihydroxyphenyl amyl ketone, m.p. 75—76°; 2 : 4-dihydroxyphenyl undecyl ketone, m.p. 90—91°. The ketones and the alkylresorcinols are bactericides. C. HOLLINS.

Manufacture of new pharmaceutical compounds [alkoxymethyltheobromines]. I. G. FARBENIND. A.-G., K. SCHRANZ, and C. LUTTER (B.P. 288,366, 6.1.27. Addn. to B.P. 242,296; B., 1926, 216).-Theobromine (or a metal salt) is treated with a chloromethyl alkyl ether, prepared from formaldehyde, hydrogen chloride, and a homologue of methyl alcohol, to give 3:7-dimethyl-1-alkoxymethylxanthines. These, unlike the 1-methoxymethyl compound, have no action on the heart, but are diuretics and give neutral solutions in aqueous sodium salicylate suitable for injection. 1-Ethoxymethyl- (m.p. 152-153°), 1-isopropoxymethyl- (m.p. 107-108°), 1-n-propoxymethyl- (m.p. 130-132°), 1-nbutoxymethyl- (m.p. 102-103°), 1-isobutoxymethyl- (m.p. 128-129°), 1-benzyloxymethyl- (m.p. 95-97°), 1-allyloxymethoxy- (m.p. 108-110°) derivatives are described; also chloromethyl n-butyl ether, b.p. 129-136°/15 mm., benzyl chloromethyl ether, b.p. 105-117°/15 mm., and allyl chloromethyl ether, b.p. 100-110°/15 mm.

# C. HOLLINS.

Preparation of addition compounds of deoxycholic acid possessing bactericidal properties. F. W. R. OSTEN (B.P. 287,965, 30.12.26).—Deoxycholic acid and its alkali salts form with safranine and acridine dyes non-toxic, bactericidal, and trypanocidal additive products. These dissolve insoluble therapeutic substances, *e.g.*, quinine. The deoxycholic acidtolusafranine additive product is described.

B. FULLMAN.

Manufacture of disodium salts of sulphomethylaminometalmercaptosulphonic acids. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 285,001, 9.8.27. Ger., 8.2.27. Addn. to B.P. 270,729; B., 1927, 860).— The formaldehyde-bisulphite compounds of 3-amino-4-aurothiolbenzenesulphonic acid, 4-(3'-amino-2'-pyridyl)amino-2-aurothiolbenzenesulphonic acid (from 3-aminothiophenol-6-sulphonic acid and 2-chloro-3-aminopyridine and subsequent reduction and introduction of gold), and 3-amino-4-argentothiolbenzenesulphonic acid, are described. C. HOLLINS.

Manufacture of a new syphilis remedy from diaminodihydroxyarsenobenzenemonomethylenesulphonic acid. T. Iwadare (U.S.P. 1,665,787, 10.4.28. Appl., 8.10.26. Japan, 14.12.25).—Diaminodihydroxyarsenobenzenemonomethylenesulphonic acid is treated with formaldehyde and sodium hydrogen sulphite. B. FULLMAN.

[Organic] arsenical compound. C. S. HAMILTON, Assr. to PARKE, DAVIS & Co. (U.S.P. 1,665,781, 10.4.28. Appl., 17.3.24).—A group containing hydroxyl is introduced into the amino-group of arsanilic acid derivatives by treating them with halogenohydrins.

B. FULLMAN.

Manufacture of new complex antimony compounds. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 288,370, 7.1.27. Addn. to B.P. 271,940; B., 1927, 573).—Antimonyl compounds of polyhydric phenols containing two ortho-hydroxyl groups are combined with neutral salts of carboxylated or sulphonated derivatives of the same or similar phenols, e.g., salts of gallic acid, carboxygallic acid, pyrocatecholdisulphonic acid, and 2:3-dihydroxynaphthalenedisulphonic acid. Examples are antimonylpyrocatechol with sodium gallate and with sodium pyrocatecholdisulphonate. The products are non-irritant trypanocides. C. HOLLINS.

Manufacture of farnesol. L. RUZICKA, ASSR. to M. NAEF & CO. (U.S.P. 1,663,817-8, 27.3.28. Appl., [A] 19.3.24, [B] 2.10.25. Switz., 22.3.23).—See B.P. 213,251; B., 1925, 691.

Production of diacylisothiocarbamide ether. H. SCHOTTE, ASST. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,667,053, 24.4.28. Appl., 12.7.26. Ger., 16.7.25).—See B.P. 255,466; B., 1927, 172.

Preparation of organic arsenic compounds. A. BINZ and C. RÄTH (U.S.P. 1,667,237, 24.4.28. Appl., 13.7.26. Ger., 22.7.25).—See B.P. 255,839; B., 1927, 829.

Reduction of arsinic acids. A. BINZ and C. RÄTH (U.S.P. 1,667,238, 24.4.28. Appl., 13.7.26. Ger., 22.7.25).—See B.P. 255,892; B., 1928, 36.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Mordanting of basic dyes. MICHELS.-See VI.

#### PATENTS.

Production of gelatin relief pictures. S. PROCOU-DINE GORSKY & CIE. [Soc. DE PHOTOCHIMIE, "ELKA"] (G.P. 445,513, 19.12.24).—Films comprising layers of gelatin and silver bromide, prepared separately in the usual manner from pure gelatin and silver bromide practically free from colloids, are exposed to light, developed with pyrogallol or pyrocatechol, and washed. L. A. COLES.

#### XXII.—EXPLOSIVES; MATCHES.

Flammability of refrigerants. Mixtures of methyl and ethyl chlorides and bromides. G. W. JONES (Ind. Eng. Chem., 1928, 20, 367-370).-With increasing depth of mines the use of refrigerants may be necessary, and such should preferably be non-flammable. The author's tests were carried out in a bell-jar resting in another bell inverted and containing mercury as a seal. A gas connexion, mixing fan, and electrodes were fitted. A mixture was considered to have propagated if the flame travelled to the top of the bell-jar and spread. The flammable limits for mixtures with air were found to be as follows: methyl chloride 8.25-18.70%, ethyl chloride 4.00-14.80%, methyl bromide 13.50-14.50%, and ethyl bromide 6.75-11.25%. Mixtures were found to follow Le Chatelier's law approximately (cf. Coward and others, B., 1919, 126 A), and no mixture was non-flammable. These compounds are more easily ignited by an intense electric spark than by a flame, with which the bromides are in fact non-flammable. Analysis of the mixtures was performed by diluting with a mixture of equal volumes of oxygen and air, adding gases from electrolysis of water to render the mixture thoroughly inflammable, and measuring the contraction on explosion and on absorption of carbon dioxide.

#### PATENT.

C. IRWIN.

Gelatin-dynamite explosive. R. B. SMITH and E. M. SYMMES, ASST. to HERCULES POWDER CO. (U.S.P. 1,667,083, 24.4.28. Appl., 27.11.26).—A low-strength dynamite explosive is composed of 17-18% of nitroglycerin, 2-4% of dinitrotoluol,  $0\cdot1-0\cdot2\%$  of nitrocotton, 60-65% of sodium nitrate, 9-12% of sulphur, 2-5% of starch, and a little chalk. F. G. CROSSE.

# XXIII.—SANITATION; WATER PURIFICATION.

Disinfectant action of sodium toluene-p-sulphochloroamide, particularly of "Chloramine-Heyden." R. DIETZEL and F. SCHLEMMER (Arch. Pharm., 1928, 266, 173—188).—"Chloramine-Heyden," together with several other brands of "chloramine-T" has been examined for bactericidal action on staphylococcic organisms by Paul and Krönig's method (Z. Hyg. Infekt., 1897, 25, 1). It is superior to phenol, but much inferior to mercuric chloride. It is also not so active as inorganic hypochlorites, but here the action is dependent to some extent on the free alkali present. The results obtained by Paul and Krönig's method are not directly comparable with those obtained in a different way.

# W. A. SILVESTER.

Carbon disulphide in viscose manufacture. J. EGGERT (Chem.-Ztg., 1928, 52, 289).—The importance of the protection of workers in factories where carbon disulphide is used is pointed out. The liquid, besides being very inflammable, acts as a poison when breathed and when allowed to come into contact with the skin. The action on the skin appears to be connected with the dissolving of surface grease and subsequent attack of the layer of fat just beneath the skin. Various symptoms of the poisoning are given, and suggestions are made for reducing the contact of the workers with the liquid or vapour to a minimum. H. INGLESON.

Iron and manganese in water. H. THIELE (Gasu. Wasserfach, 1928, 71, 289-290).-Processes for the removal of iron and manganese from water depend usually on the negligible solubility of ferric and manganic hydroxides as compared with the appreciable solubility of ferrous and manganous hydroxides. Oxidation of the latter by the air in a suitable filter is catalysed by the layer of ferric or manganic hydroxide first deposited on the contact material used, and also by the presence of certain micro-organisms. Manganese is often removed by the use of the dioxide as oxidising agent, in the form of pyrolusite, precipitated on coke, brick, etc., or in the form of manganese-permutit. Active charcoal will remove both iron and manganese, but its activity in that respect rapidly diminishes. A. B. MANNING.

Determination of plankton [in water]. W. F. LANGELIER (J. Amer. Water Works' Assoc., 1928, 19, 408-415).-For laboratory work the Sedgwick-Rafter method, described in the A.P.H.A. Standard Methods of Water Analysis, has many advantages, but the method of counting is tedious. In routine examination of water supplies, with the object of controlling the quantity of plankton present, Henson's net method is preferable. The nets used are conical with a copper or glass container. The volume of plankton collected is measured after settling or centrifuging and the results are quoted in c.c./m.<sup>3</sup> The main errors of this method depend on the velocity of haul and the ratio of net surface to area of circle. Certain small plankton pass through the net, but these have seldom been shown to have a deleterious effect on taste or odour. Their presence is often indicated by a loss of transparency by the water, and this is used to indicate the need for copper sulphate treatment. C. JEPSON.

#### Fumigants. COTTON AND ROARK .- See XVI.

#### PATENTS.

Production of fumigating mixtures containing hydrocyanic acid. DEUTS. GOLD- U. SILBER-SCHEIDEAN-STALT, VORM. ROESSLER (B.P. 271,514, 23.5.27. Ger., 22.5.26).—Mixtures of hydrocyanic acid and cyanogen chloride are prepared by the fairly rapid passage of chlorine into cooled liquid or concentrated (stronger than 92%) hydrocyanic acid, in the presence of non-alkaline substances (e.g., oxides, hydroxides, carbonates, etc.), which may also be added subsequently, to remove the hydrochloric acid formed. B. FULLMAN.

Plant for dealing with sewage. PULSOMETER ENGINEERING Co., LTD., and J. BJÖRNSTAD (B.P. 288,814, 11.4.27).

Apparatus for treating liquids [water] with zeolites. H. BARON. From W. NEUMANN (B.P. 288,660, 5.11.26, 25.4 and 1.7.27).