# BRITISH CHEMICAL ABSTRACTS

## **B.**—APPLIED CHEMISTRY

APRIL 2, 1926.

#### I.—GENERAL; PLANT; MACHINERY.

Distillation and rectification. L. GAY (Chim. et Ind., 1926, 15, 3-13, 173-183).-The study of the problem of the complete separation of a mixture of three constituents (ibid., 1923, 811, 1026) is extended to the case of four or more constituents. As in each step in rectification the pure constituents may be separated as either vapour or liquid, the number of ways of proceeding increases rapidly with the complexity of the mixture. A quaternary mixture may be represented in space by a regular tetrahedron on which the composition of the liquid or vapour at any point in the first rectification column may be represented. The calculation of the minimum heat required to operate a column rectifying a quaternary mixture is very complex but may be simplified in the case of a mixture of four successive members of a homologous series, for which the molecular latent heats of evaporation at a given temperature are approximately the same. For ternary mixtures of this type a graphical method is developed for calculating the relation between heat flow and the composition of either phase at any point in the two columns by means of the representation of composition as a triangle.

C. IRWIN.

Determination of the size and number of pores of porous filtering plates. H. Ruoss (Chem.-Ztg., 1926, 50, 83-84).-The size of the pores of a porous plate may be determined roughly by fixing the plate to a glass tube by means of a rubber band, saturating it with water, suspending it just below the surface of the water in a glass vessel, and observing the pressure required to be applied to the upper side of the plate to cause bubbles of air to form on the lower. Unless the pores are of approximately equal size and regular in cross-section this method gives simply the diameter, at the surface, of the largest pores. The average diameter of the pores is ascertained by measuring the rate of flow of water through the plate under constant pressure, the weight of water flowing through the plate being directly proportional to the pressure. A. R. POWELL.

#### PATENTS.

Separation of vaporised organic substances from gaseous mixtures. J. J. V. ARMSTRONG. From N. V. ALGEM. CHEM. PRODUCTENHANDEL (E.P. 245,856, 21.9.25).—An adsorbent such as carbon is contained in a gas-tight casing and is subdivided into thin layers by a number of pipe coils which occupy 8—12% of the internal space. A cooling medium is passed through the pipe coils during adsorption and a heating medium in the same coils is used in conjunction with a vacuum over the adsorbent for regeneration.

B. M. VENABLES.

Treatment of liquids, gases, or solutions with active carbon. H. E. Potts. From N. V. ALGEM. NORIT MAATSCHAPPIJ (E.P. 245,912, 22.2. and 9.5.24) .- Liquids or gases are treated with carbon which is much more active than Norit, and having a true sp. gr. (determined in ether) of not less than 2.0, a heat of adsorption of benzene of more than 40 cal. per g., and one or more of the following properties :- elementary carbon not less than 97% calculated free of ash and moisture, an organised structure derived from vegetable raw material, an apparent sp. gr. of less than 0.1, a moisture adsorption of not less than 20% from an atmosphere at ordinary temperature and relative humidity of 60%; 0.5 g. of the carbon adsorbs more than 80% of the iodine in 50 c.c. of 0.1N-solution. The active carbon is prepared from pine wood or other vegetable matter by known means (cf. E.P. 228,582, 228,812, 228,954, and 230,293; B., 1925, 275, 346) in a current of gases (e.g., superheated steam) which is adjusted to such a rate of flow as to draw off the activated carbon of very low apparent sp. gr., leaving the unactivated carbon behind in the retort. B. M. VENABLES.

Machines for emulsifying and disintegrating solids suspended in liquids. G. C. HURRELL (E.P. 245,929, 2.1.25).—In a high-speed emulsifying machine, to prevent the induction of air, the material is introduced into the shearing zone through a hollow shaft and radial passages in the rotor, and the rate of flow of material is regulated by restriction of the outer ends of the radial passages, one means of adjustment being grub screws which partially obstruct the passages. The machine may conveniently have a vertical shaft, the hollow end of which extends downwards into a supply reservoir. B. M. VENABLES.

Utilisation of products of combustion in steam generators. G. AND J. WEIR, LTD., and J. G. WEIR (E.P. 245,960, 25.2.25).—Products of combustion containing a large amount of water vapour are passed through an ordinary boiler and then through a low-pressure boiler, and the products of combustion under both boilers are maintained at considerable pressure (say 10 lb. per sq. in.) so that the water vapour in them will be condensed while under the low-pressure boiler and give up its latent 224 CL. II.-FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

heat. The pressure in the flues is maintained by a fan at the inlet and a turbine at the outlet, and the latter may drive the former. B. M. VENABLES.

[Electrostatically] separating suspended particles from flowing gases or vapours. Lodge-Cottrell, Ltd. From Lurgi Apparatebau-G.M.B.H. (E.P. 246,046, 5.8.25).—An electrostatic gas cleaner is arranged with horizontal electrodes, and the collecting electrodes are composed of a liquid, preferably flowing at about the same rate as the gases. B. M. VENABLES.

Process for making a reflocculated product. G. W. ACHESON (U.S.P. 1,563,713, 1.12.25. Appl., 22.11.24).—Fuller's earth, china clay, or the like is deflocculated by grinding with a deflocculating agent and a limited quantity of water, e.g., 28%, as described in U.S.P. 1,253,556 and 1,345,305-6; B., 1918, 174 A; 1920, 564 A), and then reflocculated by adding an electrolyte and more water, up to about 40%, while continuing the grinding. The resulting paste is filter-pressed, dried, and if necessary (e.g., for use as an adsorbent) calcined. Reflocculated materials prepared as described have very large surface per unit weight and are of special value for use as fillers and adsorbents. R. B. CLARKE.

Annealing furnace. G. H. COLE, Assr. to WESTINGHOUSE ELECTRIC AND MANUF. Co. (U.S.P. 1,569,356, 12.1.26. Appl., 8.8.22).—A number of refractory heat-insulating walls enclose an open-top furnace chamber. An inner cover of low thermal resistance and an outer cover of relatively high thermal resistance are sealed in members of channel section disposed at the top of the walls near the inner and outer edges respectively. M. COOK.

Refrigerant. R. R. STITT (U.S.P. 1,570,080, 19.1.26. Appl., 2.2.25).—A product for use as a refrigerant or for other similar purposes consists of methyl chloride and methyl alcohol. W. CLARK.

Filtering apparatus. P. H. CRAWFORD and J. F. ABBEMA (U.S.P. 1,570,374, 19.1.26. Appl., 31.3.21).—A number of independent filtering units are capable of rotation on a carrier and the carrier also rotates so as to dip them in succession into a trough containing the material to be filtered.

#### B. M. VENABLES.

Refrigerating apparatus. W. A. McNUTT, Assr. to KITSON ENGINEERING CO. (LONDON), LTD. (U.S.P. 1,571,633, 2.2.26. Appl., 12.12.23).—See E.P. 214,736; B., 1924, 543.

Preparation of colloid solutions. C. S. SMITH (E.P. 246,570, 28.10.24).—See U.S.P. 1,514,737; B., 1925, 991.

Closed furnace and method of operating it. C. STEENSTRUP, ASST. to GEN. ELECTRIC Co. (U.S.P. 1,571,742, 2.2.26. Appl., 20.12.23).—See E.P. 226,559; B., 1925, 853.

Rotary furnace. A. H. PEHRSON, Assr. to A. P. PEHRSON (U.S.P. 1,572,805, 9.2.26. Appl., 15.6.25).— See E.P. 238,928; B., 1925, 867. Centrifugal separator. F. W. McENTIRE (U.S.P. 1,572,299, 9.2.26. Appl., 18.6.23).—See E.P. 225,396; B., 1925, 418.

Apparatus for heat treatment by means of a bath of molten metal. A. McD. DUCKHAM, Assr. to THERMAL INDUSTRIAL AND CHEMICAL (T.I.C.) RESEARCH CO., LTD. (U.S.P. 1,572,779, 9.2.26. Appl., 20.6.23).—See E.P. 198,625; B., 1923, 753 A.

Gas-fired furnaces. Askania-WERKE A.-G. (E.P. 234,791, 24.4.25. Conv., 31.5.24).

Removal of residue from furnaces, retorts, or the like. M. SCHWABACH (E.P. 240,467, 23.9.25. Conv., 23.9.24).

Pulverulent fuel furnaces for water-tube boilers. KOHLENSCHEIDUNGS-GES. (E.P. 241,940, 23.10.25. Conv., 25.10.24).

Steam regenerative accumulators. W.Müller and P. M. HEINRICH (E.P. 246,678, 17.2.25).

[Means for jarring collecting electrodes in] electrical precipitation apparatus. Lodge-Cot-TRELL, LTD., and N. STALLARD. From METALLBANK U. METALLURGISCHE GES. (E.P. 246,757, 23.9.25. Addn. to 176,713).

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

Coal ash and clean coal. R. LESSING (J. Soc. Arts, 1926, 74, 182-197, 205-218, 224-241; Fuel, 1926, 5, 17-23, 69-76, 117-124; cf. B., 1925. 33; J.S.C.I., 1925, 277 T, 345 T).-Recent work on the origin of the ash in coal is summarised. A study of the alumina : silica ratio shows that durain ash, in spite of its high aluminium content, cannot be derived from lycopods, but is practically identical in composition with clay, whilst the ashes of clarain and vitrain closely resemble plant ashes. The four constituents of banded bituminous coal from the same seam show remarkable variations in ash content; e.g., the values for the Hamstead Thick Seam are :—fusain 15.59%, durain 6.26%, clarain 1.22%, vitrain 1.11%. Full analyses of the banded constituents from many coals prove that the ashes of clarain and vitrain represent the original plant ash, that the durain ash is derived mainly from clay, and that much of the ash in fusain comes from the carbonates or other salts of infiltered water. The bulk of the ash in commercial coal comes from the roof, floor, and dirt bands of the seam. The microstructure of the banded constituents throws some light on the relationship between them. It is suggested that the ash constituents will be found a useful index of the component parts of a coal seam. The chemical changes occurring during incineration are discussed in relation to ash analysis. Where a complete ash analysis is not made, a proximate analysis into water-soluble, acid-soluble, and acid-insoluble portions gives useful information. The methods of

determining the ash structure and distribution by examination of ash skeletons, and by X-ray photographs, are briefly described. Large blocks of massive coal can be disintegrated by the action of dilute acid on the cleat and partings. The proposed process for winning coal by the use of sulphur dioxide in this way is described. The attack on the cleat and partings by sulphuric acid produced by the oxidation of pyrites facilitates the disintegration of coals containing the latter, and thus has a bearing on the question of spontaneous combustion. Methods in use for the removal of ash from coal in preparing it for the market (coal washing and cleaning processes) are reviewed. In considering the influence of the mineral constituents on the utilisation of coal the following factors arise :---(1) the total amount of ash present; (2) catalytic activity of the ash in promoting the various chemical processes involved; (3) fusibility of the ash. From the point of view of amount only, the ash acts as a diluting impurity. The work of Fieldner and his collaborators on the fusibility of coal ash is discussed, and a distinction is drawn between the behaviour of the ash in the furnace and when finely ground and mixed for m.p. tests. The most important function of the mineral matter in the carbonisation and combustion of coal is its catalytic activity. The yield of coke from the carbonisation of sugar and collulose is greatly influenced by addition of small quantities of inorganic salts. The yield and composition of coke from bituminous coals is similarly influenced. Catalysts that increase the coke yield also tend to raise the carbon percentage in the coke. The catalytic influence of the inorganic matter has a bearing also on the gasification of coal and coke in producers, and on the hydrogenation of coal by the Bergius process. Future developments in the treatment of raw coal will probably tend to a more complete elimination of ash before putting the coal on the market. A. B. MANNING.

Initial decomposition of coal by heat. M. J. BURGESS and R. V. WHEELER (Fuel, 1926, 5, 65-68). -A Lancashire caking coal was heated in vacuo and the gaseous products evolved over different temperature ranges were examined separately. Up to 200° the gases were mostly paraffin hydrocarbons which had been occluded in the coal. Between 270° and 300° the nature of these gases underwent a change, oxides of carbon appearing in quantity. Traces of a reddish-brown oil appeared also. These observations are considered to point to a decomposition of the coal substance initiated at  $270^{\circ}$ —280°. The coal was further heated from  $300^{\circ}$  to  $400^{\circ}$  and the gaseous distillation products were fractionated by liquid air to separate the paraffin hydrocarbons, the diversity of which could not have been disclosed by the ordinary methods of gas analysis. The presence of members of the paraffin series up to pentane was demonstrated. R. A. A. TAYLOR.

Conditions underlying the caking of coal. E. AUDIBERT (Compt. rend., 1926, 182, 316-318).— The temperature of incipient distillation of bituminous coal lies somewhat below that of incipient fusion, which latter sets in at temperatures between  $325^{\circ}$  and  $450^{\circ}$ , whatever the method of heating. On subjecting samples of French coals containing 21-42% of volatile matter to sudden heating at various temperatures, definite fusion-temperatures lying between the above limits have been found. If, however, the heating is gradual, caking sets in at temperatures  $25-35^{\circ}$  higher, or else not at all; this is ascribed to loss of volatile matter. Coals such as are used for metallurgical coke require a rate of heating of at least 1° per min. to give caked masses. W. A. CASPARI.

Influence of temperature on the coke yield in crucible tests. H. STRACHE and C. MIKA (Gas-u. Wasserf., 1926, 69, 105-107).-The specification of the height of flame and the position of the crucible in the volatile matter determination for coal is shown to be an insufficient control of temperature, and the actual temperature inside the crucible should be determined. The nature of the crucible (platinum, nickel, V2A steel) only influences the rapidity of coking and not to any appreciable extent the final coke yield (cf. J.S.C.I., 1922, 372 T). The method recommended is to heat 1 g. of coal in a metal crucible of 20-35 mm. diam. with a wellfitting lid for 7 min. so that the interior of the crucible rapidly attains a temperature of 900° and is kept at that temperature during the test.

#### A. C. MONKHOUSE.

Importance of combustibility of the coke in the combustion of solid fuels on the fire-grate. A new heat theory. D. J. W. KREULEN (Brennstoff-Chem., 1926, 7, 54-57).--Korevaar's theory of "heat compression" ("Combustion in the gas producer and the blast furnace," 1925) is applied to the combustion of fuels on the fire-grate, and the following relation deduced between the height (h) of the combustion zone, and the average temperature (T):

 $h = [x(667 - 0.24t - 0.228T) - (T - T_k)K_2]/(T - T_0)K_2$ where x is the weight of air in kg. at temperature t, entering the layer of coke per minute,  $T_k$  is the temperature of the boiler plate, and  $T_0$  is the average temperature of the place where the heat loss occurs. Thus, other conditions being constant, T rises when h decreases. Korevaar has shown that the height of the oxidation zone in the gas producer is dependent on the combustibility of the coke in such a way that an increase of combustibility decreases h. This concept of combustibility involves the four factors, particle size, porosity, activity, and ash content. From the point of view of its behaviour on the fire-grate, a fuel is fully characterised by its moisture content, ash content, and the yield and combustibility of its coke. The theory is used to explain the apparently abnormal formation of A. B. MANNING. clinker with certain coals.

Detection of carbon monoxide in fire-damp, combustion gases, and after-damp. L. WEIN (Glückauf, 1925, 61, 1623—1625; Chem. Zentr., 1926, I., 1456).—0.02—0.03% of carbon monoxide in fire-damp can be detected with certainty by 5% palladous chloride solution. The test fails for combustion gases containing more than 1% of hydrogen. Unsaturated hydrocarbons must first be removed by absorption in bromine. In testing the after-damp from explosives any hydrogen sulphide formed must also first be removed. A. B. MANNING.

Condensation and wet [gas] purification. T. V. BLAKE (Gas World, 1926, 84, 102-105).-The efficiency of the condensing plant of a Glover-West continuous vertical retort installation carbonising a mixture of English and Welsh coals was examined. 3.7% of the ammonia was removed in the cyclone tar extractor, 74.1% in the airand water-cooled condensers, 8.1% in the Livesey washers, and 13.9% in the rotary brush washers. 9.3% of the tar fog was removed in the extractor, 18.3% in the condensers, 52.4% in the Livesey washer, and 16.9% in the rotary washers. The naphthalene, hydrogen sulphide, carbon dioxide, and cyanogen contents of the gas, together with the temperatures, and composition of the liquor and tar were also determined at the various points of the system.

A. C. MONKHOUSE.

Artificial petroleum from Balkash sapropelite. N. D. ZELINSKY (Brennstoff-Chem., 1925, 6, 365-369; 1926, 7, 35-37).-The oils obtained by the dry distillation of a sapropelite from Lake Balkash have been examined. The sapropelite consists of a dry, elastic mass of characteristic waxor tallow-like odour, and is remarkable for its high content of organic matter. Analysis gave moisture 1.8%, ash 4%; for the organic matter: C 73.76%, H 10.91%, S 1.03%, N 0.56%, P trace, O 13.74%. Organic solvents extracted from 27% (ether) to 39% (carbon tetrachloride) of the organic matter, the extract consisting principally of free fatty acids and their esters (waxes). 4930 g. of the material were distilled in two stages, giving (a) 2248 g. of tar and 562 g. of liquor; (b) 307 g. of tar and 795 g. of coke. The first tar fraction was mobile, of an intense garnet-red colour, and had d 0.82. It was washed with dilute sulphuric acid, distilled with superheated steam, and the distillate washed with potassium hydroxide solution and fractionated. giving 383 g. of light oil boiling to 150°, 1228 g. b.p.  $150^{\circ}$  (760 mm.) to  $200^{\circ}$  (20 mm.), and 353 g. boiling above  $200^{\circ}$  (20 mm.). These products, which contained from 10 to 40% of unsaturated compounds, and the second tar fraction were refined by treatment with sulphuric acid, and again fractionated. The crude light oil fractions were stable, those boiling below 120° showing no change after being kept under ordinary conditions for several years. The refined fractions resembled closely in physical properties (odour, density, and refractive index) and chemical composition the corresponding fractions from natural petroleum. The refined light oil had a boiling range of  $58^{\circ}$  to  $137^{\circ}$ ,  $d^{11}$  0.717, and n 1.4071. It consisted principally of paraffin hydrocarbons from hexane to nonane, and contained also some aromatic hydrocarbons and naphthenes. In particular the presence of methylcyclohexane was

proved, and that of derivatives of cyclopentane shown to be probable. Crystalline solid paraffins separated from the higher fractions. These fractions exhibited weak optical activity. On treatment with anhydrous aluminium chloride they gave products of the same chemical character as those obtained from Russian petroleum by the action of the same reagent. The close similarity between the oils from the Balkash sapropelite and the natural petroleum fractions gives further experimental support to the hypothesis of the organic origin of petroleum. A. B. MANNING.

Hydrogenation and desulphurisation of Norfolk shale oil. H. G. SHATWELL (J. Inst. Petrol. Tech., 1925, 11, 548-555).-Crude shale oil having d 0.9850, sulphur content 6.81%, and iodine value (Wijs) 882, was submitted to Bergius treatment with hydrogen in a gas-heated cylindrical autoclave. The pressure rose to about 100 atm., and the temperature was maintained at about 400° for 3 hrs. A similar experiment was made in the presence of nitrogen, the pressure rising to about 150 atm. The loss in treatment with hydrogen was 10% and with nitrogen 14%. The final pressures in the two experiments were 4 atm. below and 3 atm. above the respective initial pressures. The products had d 0.9463 and 0.9712 respectively. They were refined with 10% of 10% caustic soda and 2% of 98% sulphuric acid, again treated with alkali, and washed with water. The refining losses for the hydrogenated product were 35.4% for the petrol fraction and 24.7% for the kerosene. The refined products darkened slowly on keeping. The refined petrol had a sulphur content of 2.78%, only reduced to 2.31% by a second refining with 6% loss. The refined kerosene had a sulphur content of 5.39%. The hydrogenation greatly increased the yield of lighter fractions (from 6% to 22.3% at 175°).

H. MOORE.

Action of sulphuric acid on cracked spirit. C. M. HOUGHTON and S. BOWMAN (J. Inst. Petrol. Tech., 1925, 11, 583-586).-The behaviour of motor spirit in an engine depends on the proportion of paraffin, aromatic, naphthenic, and unsaturated hydrocarbons present. To ascertain the strength of sulphuric acid most effective in polymerising olefines, 100 c.c. of spirit were distilled, the final b.p. and the amount of reflux into the flask on removing the flame being noted. Another sample was distilled up to the final b.p. of the original spirit after treat-ment with acid. The difference between the volume remaining and the reflux in the first case was taken as a measure of the polymerisation. The cracked spirit used was entirely soluble in 95% acid. A portion was diluted with an entirely saturated spirit, and the mixture had a final b.p. of 108°, whilst 2% of reflux collected. Its fraction up to 100° was shaken for 5 min. with 50 c.c. of acid of 95, 92.5, 90, 87.5, and 85% strength, respectively. A drop in the reduction in volume between 95 and 92.5% acid probably indicated the minimum strength for solution of aromatic hydrocarbons. A rise in

high-boiling fractions occurred at about 91%. Experiments using the entire original spirit mixed with saturated spirit and treated twice with acid gave similar results, and 91% seems to be the optimum strength of acid for producing polymerisation in spirit of this class. H. MOORE.

Determination of unsaturated constituents in petroleum spirit. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petrol. Tech., 1925, 11, 533-536).-The formation of gummy substances is probably due to substances of the diene class and not to monoolefines. Unsaturated constituents may be determined by subtracting the quantity of aromatic constituents present from the total loss on treatment with strong sulphuric acid (98%); by addition of the gum value to the loss with 80% acid; or by calculation from the bromine value and the molecular weight. The Hanus test for bromine absorption was used. Molecular weights were determined by the Victor Meyer method, allowance being made for the resinous residue left in the tube. Aromatic constituents were determined by the Tizard-Marshall method (J.S.C.I., 1921, 20 T), and acid loss by shaking 100 c.c. of spirit with 300 c.c. of acid and keeping overnight. In (i.) a mixture of No. 3 spirits, (ii.) a shale petrol, and (iii. and iv.) typical cracked spirits, the percentages of unsaturated constituents found were, by the first method,  $3\cdot5$ ,  $10\cdot0$ ,  $5\cdot4$ , and  $9\cdot6$ ; by the second method,  $2\cdot1$ ,  $12\cdot5$ ,  $5\cdot1$ , and  $8\cdot1$ , and by the third method,  $3\cdot8$ ,  $37\cdot8$ ,  $13\cdot6$ , and  $9\cdot4$ , respectively. The results given by bromine absorption appear to be too high. H. MOORE.

Composition of cracked spirit. H. MOORE and R. B. HOBSON (J. Inst. Petrol. Tech., 1925, 11, 587-592).-Investigations were made of the composition of a cracked spirit, made from Russian kerosene in a Cross cracking unit, and slightly refined with acid and soda. It had d 0.748, initial b.p.  $34^{\circ}$ , and final b.p. 167°. It was divided into four fractions : up to  $95^{\circ}$ ; from  $95^{\circ}$  to  $122^{\circ}$ ; from  $122^{\circ}$  to  $150^{\circ}$ , and residue at 150°. The mean mol. wts. of the fractions were calculated to be 91, 105, 119, and 140. The iodine value of the spirit was 56.3, and the figure for the unsaturated content was 24.9%. Naphthenes were determined by the aniline cloud test method after treatment with 98% sulphuric acid, and found to be 47.7%. An attempt was made to remove olefines by washing with 80% sulphuric acid. The product after washing gave an iodine value of 25.3. It was found that equilibrium was reached when 55% of the olefines had been removed. Further experiments showed that 90% acid was necessary to remove the olefines effectively. Assuming that no aromatic hydrocarbons had been removed, the aromatic hydrocarbons were responsible for lowering the aniline point  $3.9^{\circ}$ , corresponding to an aromatic content of 4.7%. Further experiments indicated that 90% acid removed the unsaturated hydrocarbons almost completely, and 1.1% of the aromatics. By separate analysis of the fractions there were found 22.5% of unsaturated hydrocarbons, 50% of naphthenes, 6.8% of aromatics, and 19.6% of paraffins, a result not greatly differing from that obtained by

the other methods. The results obtained are very different from those of Ormandy and Craven (cf. preceding abstract). H. MOORE.

Determination of the molecular weight of petrol. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petrol. Tech., 1925, 11, 539-542).-In determinations by the Victor Meyer method erratic results were obtained at temperatures above 220°. This was particularly the case with heptane, but better results were obtained when fragments of porous earthenware were introduced into the tube. With benzene no irregularity was observed. This effect seems to be due in part to cracking, but also to actual combustion of the hydrocarbon. Cracking action does not seem to increase with rise of temperature above the critical, and mostly takes place in the liquid state. Better results are obtained by replacing the air in the tube by coal gas or carbon dioxide. The molecular weight of a petrol was found by the Victor Meyer method to be 100-101, and by the freezing-point method to be 118. The formation of carbon dioxide in mixed heptane vapour and oxygen passed over heated iron begins at 241°, and with toluene and oxygen at 210°. When petrol was heated in an atmosphere of its own vapour no cracking was observed up to 500°. H. MOORE.

Valuation of motor spirit and lubricating oils. H. MOORE (J. Inst. Petrol. Tech., 1925, 11, 571-575). -The tests essential for the valuation of lubricants are specific gravity, viscosity, cold test, flash point, coke test, and appearance. The gravity, in connexion with other values, may indicate the origin of the oil; high flash point indicates close fractionation in refining. The readings of viscosimeters in general use are not direct functions of absolute viscosity. Load factor and age of engine may decide the viscosity of oil to be used, but the size of the engine alone is not determinative. Pennsylvanian oils fall off less in viscosity with rise in temperature than do Texas and Californian oils. The heavier blends of cylinder oils contain at least two different bases. The presence of paraffin wax leads to high cold-test results. Wax-free oils may be obtained with cold tests below -7°, The coke test indicates liability to carbon deposit in cylinder, engine conditions being the same. In the same class, the lighter coloured oil is generally the better. Compounded oils have the faculty of maintaining a film under extreme pressure. The tests for motor spirit are specific gravity, Engler distillation, corrosion test, and aniline cloud test. The higher the specific gravity the greater the power obtainable per unit volume. Ease of starting is determined by the amount of spirit distilling over from initial b.p. to 100°. The aniline cloud test is a measure of the aromatic hydrocarbons present, and of the tendency to detonate. Lead tetraethyl as an anti-detonator is objectionable because of its poisonous qualities. Ethyl alcohol may come to be used for this purpose. H. MOORE.

Adsorption and electrical properties of various charcoals. Ogawa.—See A., Mar., 239.

#### PATENTS.

Peat drying machine. C. A. WILLMARTH (U.S.P. 1,569,345, 12.1.26. Appl., 16.11.23).— Horizontal drums open at the ends and provided with screw conveyors are arranged in two rows, an upper and a lower, within a heating chamber, and project through the walls of the chamber into compartment heads common to all the drums. One of the heads has an inlet opening, the other a discharge opening. R. A. A. TAYLOR.

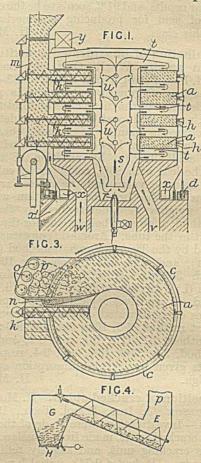
Burning fuel. FULLER FUEL Co., and G. H. KAEMMERLING (E.P. 224,511, 4.10.24. Conv., 6.11.23).—The system is designed essentially for effecting in small space the complete combustion of gaseous, liquid, and, in particular, pulverised fuels and consists of a combustion chamber with a number of burners pointing through holes in the wall tangentially directed to an imaginary circle in the middle, the jet from each burner impinging on that from the burner before it. The result is a rotating zone of combustion in the chamber, which is wellshaped with respect to a circulating chamber. Various modifications are described and provision is made for cooling the combustion chamber walls by air or water. R. A. A. TAYLOR.

Preparation of combustible and heating materials from peat and the like. CHEM. FABR. HEPPES U. Co., and J. B. CARPZOW (G.P. 421,734, 24.3.23).—In preparing fuels from humic substances by converting them into coal-like substances by biological processes under the action of bacteria and fungi, fresh sea or river-slime (sapropelic mud) may be mixed with the material. The mixture is left in pits to mature, then dried, and pressed.

#### R. A. A. TAYLOR.

Vertical retorts for the distillation of shale and like materials. R. H. CROZIER (E.P. 245,499, 7.10.24).—A retort is built up of flat standard sections, formed with flanges so that they may be bolted together, and semi-cylindrical end sections, the opposite flat sections being connected by bracing members in the form of oval internal flues. The retort may be extended and its capacity increased by detaching the sections at one end, attaching additional flat sections, and re-attaching the end sections. The internal bracing flues may be provided with means for withdrawing the vapours from the retort, and for supplying steam thereto. A. B. MANNING.

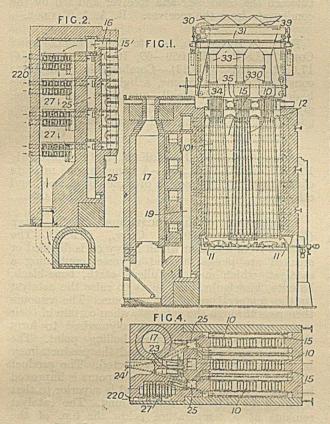
[Retort for] carbonising fuels. J. PLASSMANN (E.P. 240,800, 17.8.25. Conv., 30.9.24).\*—A carbonising retort consists of superposed flat annular chambers, *a*, heated both above and below from the interior. The stationary superposed chambers are enclosed by a rotary jacket, *c*, to which are attached the supplying and discharging means. Bands, *h*, also attached to the outer jacket, extend into the carbonising chambers, and prevent the fuel from falling cut, while allowing the passage of gases and vapours. The fuel is supplied to the chambers from a central bunker above the retort under the pressure of the screw feeds, *k*, whereby a particularly dense semi-coke is obtained. The coke is removed by a rotating scraper, n, and is quenched in such a way as to retain sufficient heat to evaporate the



water adhering to the coke. For example, it may be discharged into an inclined conduit, e (Fig. 4), containing water, and conveyed thence into a collecting chamber, g. The chambers are heated by a central heating cell, provided with a burner, r, the heating gases being led around the chambers by the annular guide discs, t. The gases and vapours formed are removed by suction through the channel, w. A. B. MANNING.

Manufacture of active charcoal. P. M. A. LEBEAU (F.P. 595,400, 18.6.24).—The substance to be carbonised, e.g. wood, is cut into the form of cubes, prisms, cylinders, etc. These are impregnated by prolonged immersion in a solution containing dilute sulphuric acid and a metal sulphate, e.g. aluminium sulphate, chromium sulphate, etc. Carbonisation is effected in two stages. The temperature is slowly raised to 500-600° to avoid sudden loss of volatile matter and shattering of the blocks. It is then raised to 600-900°; during this stage the metal sulphate is reduced and R. B. CLARKE. carbonisation completed

Carbonisation of fuel. S. R. Illingworth, Illingworth Carbonization Co., Ltd., R. DempsTER AND SONS, LTD., and H. J. TOOGOOD (E.P. 245,190, 25.9.24).\*—The setting consists of a producer, 17, and cast iron vertical retorts, 10, made



of tapering tubes, 2 in.—7 in. diam., or of plates of H-section, each tube being a separate unit and easily removable. The retorts are enclosed by horizontal fireclay flues, 15, which are so arranged that the heat can be varied at different levels, and a chamber for cooling the fuel or for further carbonisation is provided below the retorts. The setting is particularly suitable for the manufacture of shaped fuel from blended or non-swelling coals. A. C. MONKHOUSE.

Conversion of poorly coking coals into useful metallurgical coke. H. KOPPERS (G.P. 415,846, 31.8.22).—A finely ground mixture of a poorly coking coal and a more gassy coal is briquetted and from the briquettes a product similar to the coal cakes obtained by the usual method of tamping, but higher and narrower, is prepared. This is charged into horizontal ovens with narrow high chambers. R. A. A. TAYLOR.

Operation of carbonising and gasifying plant. P. ILLIG (G.P. 416,540, 21.4.23).—In plant where the gasifying portion is separated from the carbonising retort by an intermediate chamber provided with a conveyor, the passage of the charge from the retort is so regulated that only carbonised material in a loose (divided) condition enters the gasifying chamber, and sufficient room is left in the intermediate chamber to admit of cooling the gas passing from the gasifying chamber to the retort by any desired means such as added gas, steam, or a built-in heat exchanger.

#### R. A. A. TAYLOR.

Carbonisation of bituminous substances. F. LEITNER (G.P. 417,211, 24.10.23).—The charge is carbonised in an externally heated continuous inclined or vertical retort, and gas or a mixture of steam and gas heated by sensible heat derived from the spent charge is introduced into the retort. By suitable means the charge in the retort is subdivided into numerous small portions, so that the gas or gas-steam mixture is deflected alternately from the retort wall to the charge and vice versa. R. A. A. TAYLOR.

Vertical retort for low-temperature carbonisation. W. PFEIFFER (G.P. 420,810, 31.12.22). —The fuel passes through a container consisting of co-axial vertical cylinders connected by screwblades; the fuel does not fill the space at its disposal. The retort moves to and fro about its vertical axis, and the heating gases circulate in the annular space between the two cylinders. The screw-blades are provided with steps or weirs to ensure mixing and turning over of the fuel. Inside the inner cylinder is a collector with dustseparator for the distillation gases.

#### R. A. A. TAYLOR.

Carbonisation process for wood, peat, shale, and the like. NORDDEUTSCHE TORFKOKEREI A.-G. (G.P. 421,119, 21.10.24).—In intermittent vertical retorts where the charge is admitted a layer at a time, hot gases are admitted above and pass downwards long enough to bring each fresh layer to about 200°, when they are cut off until a fresh layer has been introduced. R. A. A. TAYLOR.

Cooling coke. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 245,702, 15.10.25).— Coke is cooled, and at the same time a useful fuel gas produced, by passing carbon dioxide, or a gas rich in carbon dioxide, through it. The sensible heat of the resulting gas may be utilised prior to its combustion, *e.g.*, by passing it through the tubes of a steam boiler. A. B. MANNING.

Manufacture of water-gas. HUMPHREYS and GLASGOW, LTD., Assees. of O. B. EVANS (E.P. 231,866, 31.3.25. Conv., 3.4.24).—The plant consists of one or two generators situated between two sets of carburettor and superheater. The air or steam, superheated in one set, enters the first generator at the middle of the fuel bed and the gases leave at the top and bottom; they then enter the second generator at the top and bottom and leave at the middle to heat up the second carburettor and superheater. In the succeeding "blow" and "run" the direction of flow is reversed. The hot gases do not pass through the grates, and if coal be used provision is made for the introduction of secondary air at the top of the fuel bed. A. C. MONKHOUSE.

Gas producer. S. A. Moss, Assr. to GEN. ELECTRIC Co. (U.S.P., 1,570,314, 19.1.26. Appl., 27.2.23).—The air-steam blast for the producer is supplied by means of a steam jet, the area of the throat of the discharge tube being not more than 100 times greater than the steam nozzle throat area. A. C. MONKHOUSE.

Removal of sulphuretted hydrogen from gas. WOODALL-DUCKHAM, LTD., E. W. SMITH, and T. C. FINLAYSON (E.P. 245,575, 10.12.24).-Gas to be purified is washed in a tower scrubber with a solution of alkali containing a suspension of iron compounds. The spent liquor withdrawn from the bottom of the tower is regenerated by repeated emulsification with air, supplied under such a pressure as to lift the liquor during this process into one compartment of the tank at the top of the tower. Sulphur which is formed during regeneration rises as a froth to the surface of the regenerated liquor and overflows into a centrifuge, where it is separated. If necessary the liquor may be treated for regeneration more than once and then utilised for the purification of more foul gas. S. PEXTON.

Gas washer and scrubber. J. F. BLAKELEY (E.P. 245,814, 12.9.24).—The scrubber is of the horizontal type and consists of chambers in which rotate a number of circular brushes on separate shafts, the direction of rotation of which can be automatically reversed. The gas enters a preliminary chamber through a strainer and comb dipping into the liquid in order to remove the heavier constituents of the gas before passing to the brushing chambers. A. C. MONKHOUSE.

Purification of acetylene. A. WACKEE, GES. FÜR ELEKTROCHEM. IND., Assees. of W. GRUBER (G.P. 419,729, 20.9.24).—The gas, with small quantities of oxygen, or a gas containing oxygen, is passed over silica gel at elevated temperatures. The phosphorus and sulphur compounds present as impurities are oxidised, and the oxidation products can easily be removed from the gel by water or steam. R. A. TAYLOR.

Increasing the safety and activity of porous masses for the storage of acetylene. Autogen GASACCUMULATOR A.-G., Assees. of E. STELL (G.P. 420,150, 2.11.24).—Organic substances used as porous masses for storing explosive gases such as acetylene may be treated, before or after insertion into the container, with calcium chloride and water-glass or with solutions of zinc or copper salts and resin soap, whereby finely-divided inorganic matter is deposited on the porous material, rendering the organic substance non-inflammable and decreasing the size of its pores, thus enhancing capillary action. R. A. A. TAYLOR.

Distillation of [benzol] wash-oil. ZECHE M. STINNES, Assees. of A. WEINDEL and H. KIEMSTEDT (G.P. 419,570, 7.2.24).—To increase light oil yields, the first runnings and some of the benzol are collected as a first fraction and are passed to a washer situated between the condenser and receiver and fed by the condensate from the receiver. R. A. A. TAYLOR.

Fuel. G. HAMMOND, Assr. to FUEL DEVELOPMENT CORP. (U.S.P. 1,570,059-60, 19.1.26. Appl., [A] 17.6.22, [B] 3.5.23).—Combustible mixtures consisting of (A) kerosene, a light readily ignitible hydrocarbon, and small quantities of acetone, glycerin, and butyl alcohol; or (B) kerosene, gasoline, butyl alcohol, and benzol, are claimed. W. CLARK.

Motor fuel. R. H. McKEE (U.S.P. 1,570,161, 19.1.26. Appl., 19.11.23).—A homogeneous mixture of the gasoline-like fraction of shale-oil, alcohol, and ethylene. L. M. CLARK.

Purification of hydrocarbons. T. T. GRAY (E.P. 222,481, 23.9.24. Conv., 26.9.23).-The vapours from a still in which heavy oil is distilled or cracked are passed around a vessel containing a catalyst, which may be fuller's earth, bone-black, bauxite, kieselguhr, etc., so as to raise the catalyst to the temperature of the vapours. The vapours then pass through the catalyst, when certain of their unsaturated constituents are polymerised to form liquid compounds of higher boiling point. The reaction may be exothermic, and the products may be returned to the still or collected. The product obtained by condensation of the vapours is improved in quality, odour, and colour, and is superior to that obtained by sulphuric acid refining. The catalyst remains at a temperature below the b.p. of the polymerides. H. MOORE.

Vapour separation and condensation particularly for use with [petroleum] oils. POWER SPECIALTY Co. (E.P. 239,208, 25.8.25. Conv., 27.8.24).—The vapours are treated in a number of rectifying columns at successively lower temperatures and are finally exhausted to a condenser, part of the liquid from the condenser being used as reflux liquor in the final column. The condensate formed at any stage is passed through pipe coils within the lower parts or condensate sumps of all later stages and serves to re-distil the later condensates, and each condensate after passing through a final cooler is partly returned as reflux liquor to the next earlier stage. B. M. VENABLES.

Cracking hydrocarbon oils. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,568,400, 5.1.26. Appl., 4.2.25).—The oil is passed from an elongated heating coil in a furnace to an enlarged reaction zone, and thence the vapours pass to a dephlegmator. Unvaporised oil may be withdrawn from the reaction zone without re-entering the heating coil. Reflux oil from the dephlegmator may be pumped into the heating coil under pressure, or may be admitted continuously to the coil without pumping. H. MOORE. Treatment of low-boiling hydrocarbons. W. F. DOWNS (U.S.P. 1,568,812, 5.1.26. Appl., 6.5.22).— The yield of gasoline is increased and its quality improved by condensing the vapours of hydrocarbon oils which boil below 260° in a porous mass containing anhydrous metal chlorides. The liquid condensate is continuously withdrawn from contact with the chloride. H. MOORE.

Purifying hydrocarbon liquids. W. F. Downs (U.S.P. 1,568,813, 5.1.26. Appl., 6.5.22).—Hydrocarbons are passed in liquid form through a filter bed containing a metal chloride heated to a temperature below the b.p. of the hydrocarbon and below the vaporising point of the chloride. H. MOORE.

Refining hydrocarbon oils. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,568,904, 5.1.26. Appl., 27.4.23).—The oil is subjected separately to the action of sulphuric acid and of caustic soda containing litharge, sulphur dioxide being afterwards added. H. MOORE.

Treating [hydrocarbon] oil. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,569,855, 19.1.26. Appl., 20.8.20).— Vapours generated in a still for treating hydrocarbon oils, pass through a filter bed situated in the vapour space above the oil level, and above the oil inlet into the still, and then into a dephlegmator. Vapour leaving the dephlegmator passes to a condenser, whilst condensed liquid is returned to the still through an opening situated below the filter bed.

#### L. A. Coles.

Refining [mineral] oils. Refining hydrocar-Refining cracked hydrocarbon oil. bon oil. J. C. MORRELL, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,569,870-2, 19.1.26. Appl., [A] 28.8, [B, c] 31.8.22).—Oil is treated with alkali plumbite solution, and after removing the solution as completely as possible, is treated with (A) a metal sulphide capable of hydrolysing in water to complete the reaction of the plumbite solution, or (c) an aqueous sulphide solution, precipitated material and sulphides being subsequently removed. (B) Oil is treated successively with sodium plumbite solution, with an aqueous sulphide solution, and with a flocculating agent to precipitate the other products of reaction, the aqueous solutions and precipitated material being removed after each stage. L. A. COLES.

Medium for bleaching, cracking, and desulphurising petroleum and other hydrocarbon compounds. H. and H. REINBOLD (U.S.P. 1,570,005, 19.1.26. Appl., 21.1.24).—Oil is treated with hydrated silicic acid in combination with a metal chloride. L. A. COLES.

Treating [cracking] petroleum oils. R. B. DAY (U.S.P. 1,570,131, 19.1.26. Appl., 5.8.21).— High-boiling hydrocarbon oil is partially cracked by heating in a chamber connected with an upper chamber by means of a pipe extending from the top of the lower to the bottom of the upper chamber. The lower chamber and the pipe are filled, and the upper chamber is partly filled, with the oil under treatment. Vapours generated during the treatment are withdrawn from the upper chamber. Deposition of carbon on the walls of the heating chamber is prevented by continuously agitating the oil therein. L. A. COLES.

Desulphurisation of oils, hydrocarbons, and tars. A. A. M. MASSENET (G.P. 421,263, 28.6.22).— The liquid is intimately mixed with and distilled over a small quantity of desulphurising material, such as a metal, metallic compound, alloy, or amalgam, e.g., a 50% emulsion of a metal, such as sodium, with oil, tar, or petroleum. The emulsion may be prepared from finely-powdered or molten metal by shaking at elevated temperature with a hydrocarbon, preferably one which is solid at ordinary temperatures, such as naphthalene or transformer oil or a fatty oil. R. A. A. TAYLOR.

Improvement of cracked distillates. OBER-SCHLESISCHE KOKSWERKE U. CHEM. FABR. A.-G., F. RUSSIG and A. SUPAN (G.P. 421,558, 1.1.22).— The crude products are treated with a moderate quantity of sulphuric acid (d 1.53), whereby the evilsmelling constituents are converted into substances with an aromatic odour. The products are separated by distillation into light products similar to benzene and a residual oil, the latter being returned to the cracking process. R. A. A. TAYLOR.

Preparation of a catalyst for recovering light hydrocarbons from heavy mineral oils and shale oils. G. GRISARD (Swiss P. 111,354, 30.7.24). —Aluminium chloride is mixed with an alkali or alkaline-earth, a silicate, and a mineral oil having a high content of sulphur and unsaturated hydrocarbons, to give a pitchy mass, which is but slightly hygroscopic, and can be transported and stored. The product is used as a catalyst for cracking oils, being mixed with the latter by heating and stirring. R. A. A. TAYLOR.

Emulsion. J. C. MORRELL (U.S.P. 1,556,005, 6.10.25. Appl., 18.5.23).—By mixing together emulsions having as internal phase liquids of different viscosities, partially or completely miscible with one another, an emulsion having an internal phase of intermediate viscosity is obtained. For example an emulsion of a very viscous asphalt can be made more fluid by adding an emulsion of a creosote oil. T. S. WHEELER.

Product derived from acid sludge. H. T. BENNETT, G. B. MURPHY, and LE R. G. STORY (U.S.P. 1,568,261, 5.1.26. Appl., 20.12.24).—Acid sludge containing asphaltic substances is converted into useful products by mixing it with a solvent containing alcohol and a coal-tar product, *e.g.*, benzol, and distilling off the excess solvent.

H. MOORE.

Recovery of aluminium chloride [from oil residues]. A. McD. McAFEE, Assr. to GULF REFIN-ING Co. (U.S.P. 1,568,171, 5.1.26. Appl., 25.1.22).— To recover aluminium chloride and oil, oily residues are heated to a temperature sufficient to volatilise the chloride, under a minimal partial pressure of aluminium chloride vapour. H. MOORE.

Reclaiming and converting asphaltum and other materials from waste material [with production of lampblack]. R. D. DIVINE (U.S.P. 1,569,462, 12.1.26. Appl., 18.1.24).—The material is heated in an inclined retort, and melted material (asphalt) drops from the lower end of the retort on to a hot plate contained in a chamber connected with a lampblack collection chamber, the latter also being connected with the upper gasdischarge end of the retort. J. S. G. THOMAS.

Dust-like fuel. F. W. BRANDES (U.S.P. 1,571,621, 2.2.26. Appl., 3.7.24).—See E.P. 215,771; B., 1925, 793.

Method of making fuel briquettes. P. E. WELTON (U.S.P. 1,572,629, 9.2.26. Appl., 19.5.24). —See E.P. 244,971; B., 1926, 182.

Container for testing coal and method of testing. W. J. KLAIBER, Assr. to KOPPERS Co. (U.S.P. 1,572,391, 9.2.26. Appl., 12.9.23).—See E.P. 221,778; B., 1925, 390.

Apparatus for carbonising bituminous fuels. A. BESTA (U.S.P. 1,571,927, 9.2.26. Appl., 27.12.24). —See E.P. 226,809; B., 1925, 700.

Gas producer. J. F. ROGERS, ASST. to WELLMAN-SEAVER-MORGAN CO. (U.S.P. 1,572,041, 9.2.26. Appl., 12.11.21. Renewed 31.3.24).—See E.P. 188,669; B., 1923, 1213 A.

Purification of gas. J. W. COBB and H. J. HODSMAN (U.S.P. 1,571,624, 2.2.26. Appl., 5.1.25). —See E.P. 233,011; B., 1925, 533.

Fuel for internal-combustion engines. B. H. MORGAN (U.S.P. 1,572,750, 9.2.26. Appl., 4.4.25).— See E.P. 225,685; B., 1925, 91.

Heat-exchange device [for fractionating hydrocarbons]. H. L. ALLAN (U.S.P. 1,572,374, 9.2.26. Appl., 15.12.19).—See E.P. 152,791; B., 1920, 813 A.

Apparatus for production of gasoline. F. TINKER (U.S.P. 1,572,626, 9.2.26. Appl., 7.6.23).— See E.P. 205,289; B., 1923, 1213 A.

Apparatus for removing water from coal after washing. BUTTERLEY Co., LTD., and G. F. WRIGHT (E.P. 247,012, 10.2.25).

See also pages 236, Ammonia (F.P. 594,712); Hydrogen-nitrogen mixture (G.P. 419,860); Ammoniacal liquor (G.P. 420,498). 237, Vanadium compounds from petroleum (U.S.P. 1,570,170); Carbides (G.P. 421,268).

#### IIL—TAR AND TAR PRODUCTS.

Manufacture of prepared tar for road-surfacing. C. AB-DER-HALDEN (Chim. et Ind., 1926, 15, 14—17).—The binding qualities of road tar are injured by the presence of water-soluble constituents (phenols) and of volatile constituents (naphthalene). A continuous tar-dehydration plant is described in which distillation is effected in steam at  $180^{\circ}$  under reduced pressure. The tar produced contains 0.05%of water, 1.5% of naphthalene, and 0.03% of phenols, the distillate being separated into two fractions in a rectifying column. C. IRWIN.

Normal composition of the light oil from the low-temperature tar from bituminous coals. H. BROCHE (Brennstoff-Chem., 1926, 7, 37-39).-The benzol and phenol contents of the low-temperature tar produced from Hardenberg coal have been determined (0.5% and 0.16% respectively), and shown to conform with the values generally accepted as characteristic of a low-temperature tar. The tar from the same coal, investigated by Schütz (B., 1923, 175 A, 488 A, 595 A, 1119 A; 1924, 367, 460), who found considerable quantities of aromatic hydrocarbons in the neutral oil boiling below 200°, and a phenol content of 1.35%, cannot have been a true low-temperature tar, but must have undergone some cracking. This conclusion is supported by other recent work on low-temperature tars (Klein, B., 1926, 148; Brittain, Rowe, and Sinnatt, B., 1925, A. B. MANNING. 795).

Free sulphur in benzol. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petrol. Tech., 1925, 11, 556-559).-To determine free sulphur 100 c.c. of benzol are shaken with 1.2 c.c. of mercury in a tap funnel, and a small quantity of dilute hydrochloric acid is added. The mixture is filtered under vacuum through a Gooch crucible with double filter paper at the bottom. The crucible with the precipitate is placed in a beaker and covered with aqua regia, and boiled until no mercury globules are left. It is then washed with water, the solution is neutralised with ammonia, 1 c.c. of hydrochloric acid added, and the liquid made up to 350 c.c. After filtering, barium nitrate is added to the filtrate, which is boiled and filtered, sulphur being determined as barium sulphate. The results are reasonably accurate. H. MOORE.

#### PATENTS.

Recovery of phenols and pyridine bases from tar distillation products or other mixtures. P. PAPILLON (F.P. 594,687, 30.5.24).—Before the usual fractional distillation, tar oils are washed at a little above 30° with alkali, the alkali extract then being run off and treated to recover the phenols. The residue is fractionally distilled, and the 90—160° fraction, which contains the greater part of the bases, is extracted with dilute sulphuric acid and the extract worked up in the usual way.

R. A. A. TAYLOR.

Distillation of benzol wash-oil (G.P. 419,570). --See II.

Desulphurising tars (G.P. 421,263).-See II.

#### **IV.**—DYESTUFFS AND INTERMEDIATES.

Solubility of the nitroanilines in various liquids. Collett and Johnston.—See A., Mar., 237.

Passivity of zinc dust in alkaline reduction of nitro-compounds. IZMAILSKI and KOLPENSKI. —See A., Mar., 248.

#### PATENTS.

Manufacture of quinizarin. H. DODD, W. C. SPRENT, and UNITED ALKALI CO., LTD. (E.P. 245,584, 29.12.24).—A product, which is substantially quinizarin, is obtained by heating o-chlorophenol or a mixture of o- and p-chlorophenols (crude monochlorophenol) with phthalic anhydride in presence of sulphuric and boric acids to temperatures above 200°. For example, 80 g. of o-chlorophenol in 150 g. of sulphuric acid (preferably monohydrate) are heated at 90-100° for 1 hr. A mixture of 120 g. of phthalic anhydride and 50 g. of boric acid in 750 g. of sulphuric acid is run into the first mixture with slow agitation. The mixture is slowly raised to about 250° during 16 hrs., cooled to 40-50°, and then run into 1500 c.c. of cold water. The quinizarin (about 88% yield) separates as a reddish-brown powder identical with that from pure p-chlorophenol. A. COULTHARD.

Sulphonated [hydr]oxydianthraquinonylamine dyestuffs. W. MIEG and H. RAEDER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,564,091, 1.12.25. Appl., 17.8.25).-The products obtained by sulphonating dianthraquinonylamines as described in U.S.P. 814,137 (cf. F.P. 357,138, B., 1906, 66) are mixtures of sulphonic acids and hydroxysul-phonic acids of the amines. According to the present patent by controlling the conditions of sulphonation products consisting entirely of hydroxysulphonic acids are obtained which are superior as dyestuffs to those obtained previously. For example, 1:1'or 1:2'-dianthraquinonylamine is treated with oleum containing 15-30% SO<sub>3</sub> at 120-150° in presence or absence of boric oxide or mercuric oxide or other oxygen carriers. The resulting hydroxydianthra-quinonylaminesulphonic acids dye wool from an acid bath brownish-violet to blue shades. With after-chroming, or if chrome-mordanted wool is used, brownish-violet to bluish-black shades are produced. The dyestuff from 1:1'-dianthraguinonylamine is converted into 1:1'-dianthraquin-onyloxazine when heated to 140° with 95% sulphuric acid. T. S. WHEELER.

Manufacture of azo-dyestuffs. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 245,674, 13.7.25).—Azo dyestuffs of good solubility in water, which give yellow shades on both animal and vegetable fibres, can be obtained by the introduction of one or more sulphonic acid groups into disazo dyestuffs derived from diaminodiarylcarbamides and 2 mols. of salicylic acid or one of its derivatives. In order to introduce the sulphonic groups the disazo dyestuffs may be directly sulphonated, or the tetrazotised sulphonic acids of diaminodiarylcarbamides may be combined with salicylic acid derivatives, or the aminoazo dyestuff, aminoarylenesulphonic acid-azosalicylic acid compound, may be treated with phosgene. For example, 10 pts. by weight of the disazo dyestuff from pp'diaminodiphenylcarbamide and two mols. of salicylic acid, are added at room temperature to 100 pts. of oleum (23%) while stirring. After 2 hrs. at about 75° the mass is poured on ice and the dyestuff filtered off and purified. A. COULTHARD.

Manufacture of intermediate compounds and of azo dyestuffs. BRITISH DYESTUFFS CORP., LTD., and K. H. SAUNDERS (E.P. 245,865, 21.10.24). -Aromatic o-hydroxycarboxysulphochlorides, such as salicylic acid sulphochloride, o-cresotic acid sulphochloride, and 3-chlorosalicylic acid sulphochloride, are converted by treatment with reducing agents other than alkaline sodium sulphite into the corresponding sulphinic acids, and these are condensed with aromatic nitro-compounds having a labile halogen atom, such as 4-chloro-3-nitrobenzenesulphonic acid, 4-chloro-3-nitrobenzenecarboxylic acid, 4-chloro-3-nitrobenzaldehyde, 2:4-dinitrochlorobenzene, 3:5-dinitro-4-chlorobenzenesulphonic acid, and 1-chloro-2:4-dinitronaphthalene, yielding nitrosulphones. The aminosulphones obtained by reducing the nitrosulphones, on diazotisation and coupling with azo components, yield dyestuffs having the chrome-fixing group screened from the chromophore group, so that very little change in shade takes place on mordanting. Disazo or polyazo dyestuffs may be produced, e.g., by coupling with azo components containing aminogroups, and subsequently diazotising these and coupling with further azo components. For example, 2.8-3 pts. of zinc dust are added in small quantities, to prevent too great a rise in temperature, to a solution in 20 pts. of alcohol of 10 pts. of salicylic acid sulphochloride, which is obtained as described in E.P. 18,430 of 1913 (cf. G.P. 264,786; B. 1913, 1003). After removing the solvent by filtration, the residue is dissolved in strong sodium hydroxide and the sulphinic acid is precipitated by slowly adding acid until the solution is acid to Congo Red. A mixture of 10 pts. of the sulphinic acid, 13 pts. of sodium 4-chloro-3-nitrobenzenesulphonate, 5.3 pts. of sodium carbonate, and 25 pts. of water, is boiled for several hours, and the *nitrosulphone*,  $(1) OH \cdot (2) COOH \cdot C_6 H_3 \cdot (4) SO_2 \cdot C_6 H_3 \cdot (2) NO_2 \cdot (4) SO_3 H,$ is precipitated by acidifying the hot solution, or is converted into its barium salt, or is reduced direct to the corresponding aminosulphone, e.g., by the addition of iron filings or zinc dust and an acid, or stannous chloride or sodium sulphide, or it may be chlorinated by treatment with an alkali hypochlorite before reduction. The amine on diazotisation and coupling with  $\beta$ -hydroxynaphthoic anilide,  $\beta$ -naphthol,  $\beta$ -naphthylamine, ethyl- $\beta$ -naphthyl-amine, phenylmethylpyrazolone, and ethylbenzylaniline, yields dyestuffs giving salmon-pink, orange, scarlet, bluish-red, yellow, and orange shades, respectively, on printing with a chrome mordant L. A. COLES. on cotton.

Azo dyes containing chromium. BADISCHE ANILIN- U. SODA-FABR., Assees. of H. KÄMMERER (G.P. 419,825, 31.7.23).—Azo dyes capable of chroming are treated first with chromium salts and then with alkaline substances. The chromium compounds obtained in this way can be separated from unused chromium salts by salting out. They dye wool and silk in fast shades from an acid bath. A. COULTHARD.

o-Hydroxyazo dyes. Ges. für Chem. Ind. IN BASEL (Swiss P. 107,204 and 107,520, 8.11.22).-Double compounds of an azo dye and  $\beta$ -naphthylamine are obtained by the action of 1 mol. of the diazo compound from 2-aminophenol-4-sulphonic acid or from 4-chloro-2-aminophenol-5-sulphonic acid, on 2 mols. of  $\beta$ -naphthylamine. By the action of sodium other hydrolysing hydroxide. or substance,  $\beta$ -naphthylamine is split off, leaving solutions from which, after filtration, the dyes may be salted out. They give orange shades on wool from an acid bath and can be used for the preparation of other dyes. A. COULTHARD.

Preparation of azo dyes. GES. FUR CHEM. IND. IN BASEL (Swiss P. 111,123 and 111,497—9, 12.4.24).—Fast bordeaux and black dyes are obtained by combining 2:4:6-tri-*p*-hydroxynaphthyl-1:3:5-triazine with the diazo compounds from 4-chloro-2-aminodiphenyl ether, aminoazobenzene, 4-amino-2'-ethoxyphenylazonaphthalene, or the tetrazo compound from 4:4'-diamino-5-methoxy-2-methylazobenzene, either in solution or on the fibre.

A. COULTHARD.

Azo dyes. CHEM. Fabr. ROHNER A.-G. PRATTELN (Swiss P. 111,495—6, 22.5.24. Addns. to 109,706; B., 1926, 149).—(A) A dye which gives a lake fast to light, and is yellowish-red when produced on the fibre, is obtained by coupling the diazo compound from monobenzoyl-*m*-phenylenediamine with 2:3-hydroxynaphthoic acid anilide. (B) Coupling the diazo compound from *p*-benzamido-*o*-toluidine with 2:3-hydroxynaphthoic acid anilide gives a fast, red lake (bluish-red when produced on the fibre).

A. COULTHARD.

Preparation of hydroxy-compounds [dyes] of the dibenzanthrone series. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING, Assees. of K. SCHIRMACHER and K. ZAHN (G.P. 414,924, 6.2.23. Addn. to 414,203; B., 1925, 799).—Benzanthrones having hydroxy-groups in the 3-, 6-, or 7-position to the CO-group of the naphthalene nucleus, are fused with alkali. The hydroxybenzanthrone obtained by heating 3-methoxy-1-benzoylnaphthalene with aluminium chloride gives a blue vat cotton dye. The products can also be used as intermediates.

A. COULTHARD.

Production of dyestuff emulsions. C. E. J. GOEDECKE, and COLLOISIL COLOUR CO., LTD. (E.P. 245,678, 9.4.25; cf. E.P. 241,331, B., 1925, 983).— Dyes such as Brilliant Green or Malachite Green, in solution, are converted into colloidal emulsions or suspensions by working up with non-lake-forming materials, such as oil, fat, water-glass, soap, dextrin, starch, or glue in a colloid mill. In this way the dyestuff acquires an increased staining power beyond what it possesses when thickened in the usual way. For example, 1 lb. of Brilliant Green base is converted into chloride with the exact amount of hydrochloric acid required, and the solution made up to 2 lb. with water, 2 lb. of mineral oil are added, and the mixture is emulsified in a colloid mill. The products are used in calico printing and in the preparation of pigments. A. COULTHARD.

Sulphonating  $\beta$ -naphthol. M. L. CROSSLEY and G. S. SIMPSON (U.S.P. 1,570,046, 19.1.26. Appl., 14.10.22).— $\beta$ -Naphthol is sulphonated with sulphuric acid in the presence of an oxy-compound of boron. H. ROYAL-DAWSON.

New dyestuffs of the pyrone series. W. CARP-MAEL. From FARBENFABR. VORM. F. BAYER U. CO. (E.P. 247,003, 31.1.25).—See U.S.P. 1,532,790; B., 1925, 494.

Sulphurising organic compounds [for production of sulphur dyestuffs]. F. W. ATACK (U.S.P. 1,571,181, 2.2.26. Appl., 22.1.23).—See E.P. 196,993; B., 1923, 647 A.

Solid stable diazo compounds. BADISCHE ANILIN- U. SODA-FABR., Assees. of F. GÜNTHER and F. LANGE (U.S.P. 1,572,715, 9.2.26. Appl., 22.7.24). --See E.P. 238,676; B., 1925, 840.

Dyes and dyeing (E.P. 245,587).-See VI.

Solutions of dyes (G.P. 419,223).-See XIII.

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

Bacterial decay of textile fibres. II. Deterioration of artificial silk through the action of micro-organisms. A. C. THAYSEN and H. J. BUNKER (Biochem. J., 1925, 19, 1088-1094).-Four samples of artificial silk were tested for their resistance to the action of cellulose-decomposing micro-organisms. They were submitted to three tests: (1) the samples were placed anaerobically in a nutrient solution favouring the development of cellulose-decomposing bacteria; (2) the samples were buried on frames vertically in a light garden soil reaching to a depth of 30 cm., and (3) the samples were submerged on fixed frames in sea-water. Cellulose acetate silk was found to be the most resistant of the four samples tested. The varying degree of resistance among the other three types were in the following order : nitro-silk, viscose silk, and cuprammonium silk. The reasons for the different rates of destruction are discussed. S. S. ZILVA.

See also A., Mar., 241, Nature of solutions of cellulose in cuprammonium hydroxide (NEALE). 243, Swelling and dispersion of colloidal substances in ether-alcohol mixtures (MARDLES).

Aeroplane dopes. Deschiens .- See XIII.

#### PATENTS.

Apparatus for the manufacture of artificial threads. N. B. GRILLET, Assr. to Soc. POUR LA

FABR. DE LA SOIE "RHODIASETA" (U.S.P. 1,571,474, 2.2.26. Appl., 5.3.24).—See E.P. 233,384; B., 1925, 587.

Apparatus for drying textile or fibrous material. H. HAAS (E.P. 246,655, 17.1.25).

Emulsifying or mixing apparatus [for preparing size for paper]. R. B. BEST (E.P. 246,608, 17.11.24).

Coating welding electrodes (E.P. 230,831).----See X.

Solutions of cellulose esters (G.P. 419,223).— See XIII.

Tans from sulphite-cellulose (U.S.P. 1,563,010). —See XV.

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

#### PATENTS.

Dyes and dyeing. [Preparation of derivatives of leuco-compounds of vat dyestuffs.] J. I. M. JONES, B. WYLAM, J. MORTON, and MORTON SUNDOUR FABRICS, LTD. (E.P. 245,587, 12.7 and 30.7.24).—Derivatives of leuco-compounds of vat dyestuffs, particularly those of the anthraquinone series, e.g., leucoflavanthrone, leucoindanthrone, and leucoanthraquinone-2:1-naphthacridone (Caledon Red BN), for use in dyeing and printing, are prepared by treating the leuco-compounds with alkyl chlorosulphonates, e.g., methyl or ethyl chlorosulphonate. For example, 13 pts. of methyl chlorosulphonate are added during <sup>3</sup>/<sub>4</sub> hr. to a well stirred suspension of 5 pts. of dry leucoflavanthrone in a mixture of 25 pts. of carbon disulphide and 8 pts. of pyridine at  $20^{\circ}$ . When addition is complete, the temperature is raised to 80° for 10 min., and the product is precipitated by shaking with water, and removed. The product dissolves in dilute alkalis, yielding a blue-violet solution which gives bright blue shades on cotton without the use of a vat. The dry leuco-compounds are prepared by precipitation with acids from alkaline vats, the precipitated material being washed with air-free water and alcohol, and dried in a steam oven. or over sulphuric acid in vacuo, or in a rarefied atmosphere of a non-reacting gas, preferably below 50°.

L. A. Coles.

Stable vat dye printing pastes. FARBENFABR. VORM. F. BAYER U. CO., Assees. of R. FISCHER (G.P. 418,990, 7.5.24).—The pastes contain zinc hyposulphite-aldehyde compounds and alkali. Prints obtained with these pastes are stable in the air and can be kept for weeks before steaming.

#### A. COULTHARD.

Production of fast shades on wool with indigo or indigo derivatives. DURAND U. HUGUENIN A.-G. (G.P. 419,061, 25.12.23).—Wool is allowed to take up dehydroindigo-bisulphite compounds from an acid bath, after which the compounds are converted into the corresponding indigoid parent substances by known methods. For the development of the colour, the wool, dyed with the bisulphite compounds, is treated with dilute sulphuric acid, or with ammonia or alkali carbonates at ordinary or at higher temperatures. A. COULTHARD.

Dyeing cellulose acetates. R. CLAVEL (U.S.P. 1,571,320, 2.2.26. Appl., 5.10.22).—See E.P. 199,754; B., 1923, 826 A.

Azo dyes (Swiss P. 111,495-6).-See IV.

Dyestuff emulsions (E.P. 245,678).—See IV.

Dyeing wood (U.S.P. 1,570,575).—See IX.

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

Unsoundness of a large chlorine tank at the Wilhelmsburg tin works. F. BÖRNER (Chem.-Ztg., 1926, 59, 85-86).-A large tank containing 10-12 tons of liquid chlorine was allowed to stand idle in the Wilhelmsburg tin works for several years. As a result serious corrosion of the valve on the manhole cover took place and it became necessary to empty the tank without delay. In order to do this a drill working in a gas-tight tube connected to a horizontal valve was made and fixed on the top of the tank, using lead washers to make the joint gastight. By operating a lever the tank was drilled and the chlorine delivered through the valve into milk of lime. The time consumed in this operation was so great that the valve corroded and a serious leakage took place. This was finally stopped after much trouble by reducing the pressure in the valve by pushing the drill further into the tank and affixing new washers. This hole was then abandoned and a second valve inserted in the same way, except that the drill fitted into a conical chamber so that in case of further accidental leakage the hole could be stopped efficiently by pushing in the drill. A. R. POWELL.

See also A., Mar., 217, Critical potentials of hydrogen in presence of catalytic nickel and copper (WOLFENDEN). 232, Heat capacities of metal oxides (PARKS and KELLEY); Melting point of graphite (RYSCHKEWITSCH and MERCK). 235, Partial pressures of water vapour and sulphuric acid vapour over concentrated sulphuric acid at high temperatures (THOMAS and BARKER). 236, Solubilities of sodium, potassium, and calcium ferrocyanides (FARROW). 237, Barium dithionate, and solubility of calcium dithionate (ISHIK-AWA and KIMURA). 240, Stability of carbon dioxide solutions (KLING and LASSIEUR). 244, Mass action equation for condensed gases, with application to Haber equilibrium data (GILLESPIE). 246, Basic salts of copper (BRITTON); Dissociation and specific heats of chlorine and hydrogen chloride at high temperatures (WOHL and KADOW); Thermal dissociation of sodium carbide (GUERNSEY and SHERMAN). 249, Action of boric acid on alkali carbonates in solution

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(VANZETTI). 250, Union of carbon monoxide and oxygen in contact with nickel, copper, and their oxides (BONE and ANDREW). 256, Double salt of ammonium nitrate and sulphate (WöHLER and SCHÄFFER). 257, Thiosulphuric acid (RIESEN-FELD and GRÜNTHAL). 258, Hypochlorous acid and alkali hypochlorites (MÜLLER). 260, Purification of phosphoric oxide (FINCH and FRASER); Determination of small amounts of bromine (HIEBARD).

#### PATENTS.

Concentration of nitric acid. M. KALTENBACH (F.P. 594,865, 7.3.25).—Concentrated sulphuric acid is introduced at the top of a column and meets halfway down a dilute solution of nitric acid. The heat evolved volatilises most of the nitric acid, which is carried upwards and condensed. The mixture passes to a heated cascade where the remaining nitric acid is volatilised, and the sulphuric acid is concentrated ready for re-introduction at the top of the column.

#### R. B. CLARKE.

Preparation of sulphuric acid of high concentration. METALLBANK U. METALLURGISCHE GES. (G.P. 419,559, 27.2.24).—Sulphuric acid of 66° B. (d 1.84) and oleum are prepared by combining the contact process with the processes described in G.P. 370,369, 370,853, and 378,610 (cf. E.P. 149,648 and 184,966; B., 1921, 693 A; 1922, 858 A).

#### S. S. WOOLF.

Purifying phosphoric acid and preparing pure phosphates. A.-G. FÜR CHEM. PROD. VORM. H. SCHEIDEMANDEL, Assees. of H. J. BRAUN (G.P. 420,173, 21.5.22).—Impurities are precipitated from crude phosphoric acid by addition of sufficient ammonia and then, according to whether phosphoric acid or ammonium phosphate is desired, the ammonia is wholly or partly removed by heating under normal or reduced pressure. The metaphosphoric acid or metaphosphate formed is converted into the orthocompound by the usual method. A. COUSEN.

Treatment of alkali liquors [obtained in purification of lead]. H. HARRIS (E.P. 245,479, 8.8. and 20.9.24) .- Sodium stannate is almost insoluble in cold sodium hydroxide solution (508 g. per litre) or in sodium chloride solution (400 g. per litre). Sodium arsenate is soluble in these solutions but becomes insoluble in a solution containing 280 g. of sodium hydroxide and 124 g. of sodium chloride per litre whereas sodium stannate is soluble. Sodium antimonate is insoluble in hot sodium hydroxide solution of 508 g. per litre con-Methods based on these facts, and centration. apparatus, for separating the impurities sodium stannate, sodium arsenate and, when present, sodium antimonate, from spent caustic soda used for the purification of lead (cf. E.P. 189,013; B, 1923, 60 A) are outlined. L. M. CLARK.

Carrying out circuit reactions [ammonia synthesis] under very high pressures. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 245,553, 12.11.24).—The difficulty of providing effective packing for circulating pumps moving gases at pressures of 100 atm. and above is avoided by placing the pump, of multistage centrifugal type, and motor, within the high-pressure circuit. In the case of ammonia synthesis a partition, through which the driving shaft passes, separates the pump and motor. This allows only the fresh gas mixture, containing no ammonia, to come in contact with the copper parts of the latter. C. IBWIN.

Production of catalysts for the synthesis of ammonia. I. W. CEDERBERG, ASST. to NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB (U.S.P. 1,570,333, 19.1.26. Appl., 14.7.24).—Compounds of metals of the iron group are mixed with cyanides in liquid, anhydrous ammonia and heated in a nonoxidising atmosphere, after removing excess of ammonia. H. ROYAL-DAWSON.

Hydrogen-nitrogen mixture for ammonia synthesis. CHEM. FABR. KALK G.m.b.H. (G.P. 419,860, 12.11.22).—The gaseous mixture is prepared from a mixture of water-gas and a gas containing nitrogen by burning the carbon monoxide contained therein by steam. Gas obtained by gasifying peat, lignite or similar fuels, is used as the nitrogen-containing gas, without previous condensation of steam. A. COUSEN.

**Production of ammonia.** E. DUGOUJON (F.P. 594,712, 3.6.24).—A mixture of 1 pt. of air and 2.5 pts. of steam is passed over glowing coal, which is impregnated with a mixture of salts, *e.g.*, chloride, sulphate, carbonate of sodium, magnesium, calcium, etc., obtained by calcining vegetable substances, such as seaweed. The water-gas produced contains 5% of ammonia, which is absorbed in sulphuric acid. The salts present in the coal act as catalysts.

R. B. CLARKE.

Working-up of crude ammoniacal liquor. F. RASCHIG (G.P. 420,498, 11.6.24).—In a continuous process for obtaining pure ammonia solution, the crude ammoniacal liqour is preheated by the spent ammonia-free liquor and then passed down a column which is packed so as to present a large surface area, and is heated to 96°. Volatile acids are thus removed. The liquor is then passed down another column containing similar packing. Steam is injected from the bottom and the ammonia is carried upwards and condensed in a cooling apparatus. The small quantity of fixed ammonia is lost but complications due to addition of lime are avoided and ammonia solution of about 20% concentration is obtained directly. R. B. CLARKE.

Recovery of thiocyanates and thiosulphates. M. DARRIN, ASST. to KOPPERS Co. (U.S.P. 1,570,047, 19.1.26. Appl., 16.1.23. Renewed 11.6.25).—A solution containing sodium thiocyanate, sodium thiosulphate and finely divided impurities, is concentrated till its boiling temperature is 130—135°/ 760 mm. and is then cooled to a temperature between the transition points of sodium thiosulphate pentahydrate and sodium thiocyanate hydrate. On seeding with crystals of hydrated sodium thiosulphate a gelatinous precipitate of sodium thiosulphate is formed which entraps the impurities; the solution is filtered and the filtrate concentrated till its boiling temperature is near that of a saturated solution of sodium thiocyanate, and cooled to cause crystallisation of anhydrous sodium thiocyanate, which is then separated from the mother liquor. H. ROYAL-DAWSON.

Manufacture of sodium thiosulphate. H. HOWARD, ASST. to GRASSELLI CHEMICAL CO. (U.S.P. 1,570,253, 19.1.26. Appl., 13.1.25).—In the production of sodium thiosulphate by the interaction of sodium sulphite with sulphur, the sulphur is used in gaseous form. H. ROYAL-DAWSON.

Recovering vanadium from petroleum hydrocarbons. A. OBERLE (U.S.P. 1,570,170, 19.1.26. Appl., 25.6.24).—The volatile constituents of the hydrocarbons are driven off and the residue is leached with a solvent to recover vanadium compounds. L. M. CLARK.

Ammonia-soda process. H. A. GALT, Assr. to PITTSBURGH PLATE GLASS Co. (U.S.P. 1,570,299, 19.1.26. Appl., 1.5.22).—Crude ammonia, concentrated brine, and carbon dioxide gas of low concentration are brought into contact with each other to form ammonium carbonate without material precipitation of sodium bicarbonate; carbon dioxide gas of high concentration is then passed into the mixture to precipitate sodium bicarbonate, which is subsequently calcined to yield soda ash and carbon dioxide gas of high concentration.

H. ROYAL-DAWSON.

Sodium carbonate and ammonium chloride. E. A. E. WACHÉ (F.P. 594,695, 31.5.24).—The reaction between ammonium carbonate and sodium chloride is carried out in a cold saturated solution of  $(NH_4)_2SO_4,Na_2SO_4,4H_2O$ , with a solution containing a fixed concentration of ammonium carbonate.

A. COUSEN.

Ammonium chloride and sodium sulphate. E. A. E. WACHÉ (F.P. 594,696, 31.5.24).—Calcium sulphate and ammonium carbonate are added at  $65-70^{\circ}$  to a solution of  $(NH_4)_2SO_4,Na_2SO_4,4H_2O$ , saturated in the cold. The precipitated calcium carbonate is removed, sodium sulphate is added, and by sudden cooling the double sulphate is crystallised out, this being converted into ammonium chloride and sodium sulphate by means of a solution of sodium chloride, saturated at  $30-35^{\circ}$ .

A. COUSEN.

Separation of didymium from cerium. DEUTSCHE GASGLÜHLICHT-AUER-GES. (F.P. 594,783, 15.1.25. Conv., 18.1.24).—Cerium peroxide is produced electrolytically in solutions of cerium salts. A. COUSEN.

Potassium salts from sea-water. E. NICCOLI (F.P. 594,904, 10.3.25).—Sea water which has been caused to deposit most of its sodium chloride by evaporation in the sun to  $29^{\circ}$  B. (d 1.25) is further concentrated to 34° B. (d 1.31) in open pans by solar heat, when a deposition of unimportant salts is effected. The decanted solution is transferred to another pan and evaporated to 37-38° B. (d 1.345-1.36). A mixture of potassium, sodium, and magnesium salts is deposited which is removed and dried. This is dissolved in cold water and mixed with a saturated solution of magnesium sulphate of  $33^{\circ}$  B. (d 1.3). On exposing to solar heat K<sub>2</sub>SO<sub>4</sub>,MgSO<sub>4</sub>,6H<sub>2</sub>O containing 1.0-1.5% of sodium chloride is deposited. The addition of magnesium sulphate may be avoided by suitable evaporation. A solution of the double salt is treated with the calculated quantity of milk of lime and filtered. Magnesium hydroxide and calcium sulphate are removed, and the potassium sulphate in the filtrate is crystallised out by solar heat. It has a purity of 97-98%. R. B. CLARKE.

Stable alkali polysulphide solutions. E. GLÜCKSMANN (G.P. 419,910, 24.9.24).—To the solution are added as stabilisers substances or mixtures of substances containing one or more OH-groups, substances capable of increasing the viscosity of the solution, protective colloids, or substances capable of forming complex compounds with certain gases, such as carbon dioxide or hydrogen sulphide.

A. COUSEN.

Stable solution of unstable halogen compounds of heavy metals. H. TRUTTWIN (G.P. 420,391, 31.5.24. Conv., 26.5.24).—Stable solutions of unstable halogen compounds of heavy metals, particularly of uranium and vanadium, or of corresponding complex compounds, are obtained by double decomposition in aqueous or alcoholic solution. The compounds,  $UO_2I_2$  and  $UO_2BII_5$ , are mentioned as examples. A. COUSEN,

Lead carbonate. BADISCHE ANILIN U. SODA-FABR., Assees. of L. BUB (G.P. 420,638, 30.12.23. Addn. to 336,767; cf. B., 1921, 583 A).—Lead carbonate is prepared by treating lead sulphate with ammonia and carbon dioxide, using an excess of the former over the latter gas. A. COUSEN.

Preparation of hydrogen sulphide. C. EHREN-BERG, H. WIEDERHOLD, C. KRUG, M. G. HOLSBOER, K. FISCHER, and STUDIENGES. FÜR AUSBAU DER IND. (G.P. 421,267, 11.11.23).—Sulphides or hydrosulphides are decomposed by humic acid or substances containing humic acid, *e.g.*, peat, with or without application of heat and pressure. Sodium chloride etc. may be added to the reaction mixture.

S. S. WOOLF.

Preparation of carbides. C. EHRENBERG, H. WIEDERHOLD, C. KRUG, M. G. HOLSBOER, K. FISCHER, and STUDIENGES. FÜR AUSBAU DER INDUSTRIE (G.P. 421,268, 30.11.23).—Humic coal, or humus-containing substances, such as peat, are mixed with lime and a suitable amount of water. The mixture, which quickly sets, is coked, yielding gas, tar, and ammonia as by-products, and the residue, which contains carbon and lime intimately mixed, is utilised as raw material for production of calcium carbide or calcium cyanamide. R. B. CLARKE.

Preparation of table salt from powdered rock salt. GEWERKSCHAFT EINIGKEIT I. (G.P. 421,788, 13.9.24).—The powdered rock salt is suspended in a saturated salt solution, or is washed with water and then freed from the greater part of the washing liquid, a film of solution being thus formed around each of the grains. The resulting mass is quickly dried and a compact conglomerate is obtained, which is again disintegrated by a milling process.

R. B. CLARKE.

Extraction of helium from gases. R. R. Воттомя (U.S.P. 1,569,943, 19.1.26. Appl., 27.12.24). -The compressed gaseous mixture containing helium is led through one series of passages in a countercurrent heat exchanger, and then into a condenser where the constituents, including the greater part of the helium, are liquefied at high pressure by cooling to the lowest temperature reached in the process. On reducing the pressure by passing the resulting liquid through a throttle valve into a collector, the dissolved helium is released and passing into the gaseous phase is withdrawn. The liquids then pass into a condenser where they are again evaporated to the gaseous state, the vapour passing through a second series of passages in the heat interchanger at low pressure. H. ROYAL-DAWSON.

Decolorising diamonds. L. WOLFRUM (G.P. 419,476, 27.7.21).—Coloured diamonds are embedded in powdered carbon (preferably wood charcoal) and heated, first with exclusion of air, and then to redness in a stream of hydrogen. The smallest adhering carbon particles are then removed by treating with nitric acid and potassium chlorate, warming, and washing with water. A. COUSEN.

Obtaining pure sulphur. BADISCHE ANILIN-U. SODA-FABR., Assees. of F. WINKLER (G.P. 420,238, 2.10.24; Addn. to 358,700).—Pure sulphur is prepared as described in the chief patent (cf. E.P. 177,103; B., 1922, 373 A) from impure ammonium polysulphide solution. By varying the conditions a coarser form of sulphur may be obtained. In place of, or together with, the protective colloid, a surfaceactive material is used. A. COUSEN.

Purification of sulphur. BADISCHE ANILIN- U. SODA-FABR., Assees. of L. FRIEDERICI (G.P. 421,785, 22.7.24).—Sulphur is melted with water under pressure, in presence of dilute solutions of alkalis or alkaline compounds. S. S. WOOLF.

Obtaining hydrochloric acid free from arsenic. E. SCHMIDT, Assr. to GES. FÜR CHEM. PRODUKTION (U.S.P. 1,571,648, 2.2.26. Appl., 2.5.24).—See E.P. 216,129; B., 1925, 70.

Purification of crude ammoniacal liquors. A. WEINDEL, ASST. to ZECHE M. STINNES (U.S.P. 1,571,986, 9.2.26. Appl., 23.7.24).—See E.P. 230,705; B., 1925, 356. Removing hydrogen sulphide from gas (E.P. 245,575).—See II.

Recovering aluminium chloride from oil residues. (U.S.P. 1,568,171).—See II.

Lead oxide from storage-battery plates. (U.S.P. 1,570,438).—See XI.

#### VIII.—GLASS; CERAMICS.

Influence of boric oxide on the properties of chemical and heat-resisting glasses. I. W. E. S. TURNER and F. WINKS (J. Soc. Glass Tech., 1925, 9, 389-405).-Boric oxide was introduced into a typical Kavalier's glass (soda-potash-lime silicate) to replace the silica weight for weight and in successively increasing amounts up to 40% B<sub>2</sub>O<sub>3</sub>. The general formula of the glasses obtained was  $SiO_2$  (75.82—x)%,  $B_2O_3$  x%, CaO 8.56%,  $Na_2O$  6.86%,  $K_2O$  7.9%, with small amounts of  $Al_2O_3$ ,  $Fe_2O_3$ , and The average loss of B<sub>2</sub>O<sub>3</sub> in the melting MnO. operation was 15.8%. Substitution of B2O3 for SiO<sub>2</sub> caused an increase of the rate of melting at 1400°, but at temperatures below 1350° segregation occurred, giving, for a time, an upper layer of  $B_2O_3$ . With substitution of  $B_2O_3$  for  $SiO_2$  there was increase of pot attack, and the fluidity of the glass and the rate of setting also increased. Continued replacement of the SiO<sub>2</sub> by B<sub>2</sub>O<sub>3</sub> caused a rise in annealing temperature until a maximum was attained at 14.5% B<sub>2</sub>O<sub>3</sub>, followed by a rapid decrease. Similarly, the density gave a maximum at 13.8% B<sub>2</sub>O<sub>3</sub>, and the refractive index at 26% B<sub>2</sub>O<sub>3</sub>. The dispersion  $n_{\rm F}-n_{\rm C}$  remained practically constant over the whole range, and the  $\nu$  value (61-65) slowly increased with rise of  $B_2O_3$  content. A. COUSEN.

Optical glass. H. HEINRICHS and W. TEPOHL (Glastech. Ber., 1925, 3, 213-222; Chem. Zentr., 1926, I., 761-762).-Barium flint, flint, barium crown, and a series of other glasses are liable to attack by acids with formation of white iridescent spots. A measurement of durability to acids is obtained by heating 1 c.c. of the glass powdered to 0.15-0.3 mm. mesh with 20 c.c. of 0.5% acetic acid. The dissolved barium or lead is precipitated by boiling with 10 c.c. of 0.5N-potassium dichromate and after 1-2 hrs. is collected on a tared paper and weighed. The chromic acid in the filtrate is determined iodometrically. Tendency towards spotting increases rapidly with increase of lead oxide and barium oxide. Glasses containing boric anhydride with little alkali are liable to attack. In baryta crowns which contain lead oxide, barium oxide, and sometimes small amounts of boric anhydride the tendency to spotting is small, and from non-spotting crown glass no salts can be dissolved. Glasses containing antimony give an acid-soluble portion which is analytically determined. A classification of optical glasses into five groups based on the results of the test is given. The test gives no indication of susceptibility towards attack by salt solutions, hydrogen sulphide, etc. A. COUSEN.

Factors involved in the preheating of glass pots, with special reference to moisture control. W. W. OAKLEY (J. Amer. Ceram. Soc., 1926, 9, 23-28).—Mechanical strains are induced in the material of glass pots owing to varying conditions of humidity—and therefore rate of drying—outside and inside the pots. To minimise the effect of this, the atmosphere in the pot arch during the preheating process must be kept as humid as possible by the injection of steam etc. Natural gas is a very suitable fuel, since it gives the wettest flue gas. F. SALT.

Sillimanite in glass furnace practice. F. G. CLARK and W. J. REES (J. Soc. Glass Tech., 1925, 9, 383-388).-The linear shrinkage after burning at 1500°, the refractoriness, and the refractoriness under a load of 50 lb. per sq. in., of mixtures of sillimanite with dextrin, shellac, colloidal aluminium silicate, and ball clay were determined. Examination of the workability of the mixtures indicates that it would be best to use not less than 20% of ball clay as a bond for pot-making purposes, and 25% was finally adopted. The mixture was best when soaked for not less than four weeks before making up. From such a mixture a covered pot,  $54 \times 39 \times 43$ in., was successfully made in 16 days, and it was quite dry in 6 weeks. The total drying contraction was 1 in 100. Normal methods of arching and setting of the pot were used, but it only survived one week of melting operations, that is, two founds. Failure was due to a crack in the bottom, probably due to shock received in transferring from pot room to arch. Glass corrosion on both the pot and a ring of the same material was negligible. The cost of the pot was approximately 3 times that of a similar clay pot. Bricks made from sillimanite-bauxite-ball clay mixtures after firing at 1500° had a shrinkage of 2.8% and had a refractoriness equal to that of sillimanite itself. The high resistance of sillimaniteball clay bricks to corrosion by glass was proved by immersing these for a period of 6 weeks in a glass pot. A. COUSEN.

Sillimanite bricks and kaolin-sillimanite mixtures. H. S. HOULDSWORTH (J. Soc. Glass Tech., 1925, 9, 316-321).-Addition of sillimanite, of a grading as supplied commercially, to kaolin and to fireclay reduces the shrinkages on drying and on firing (to cone 14), whilst it increases the porosity. The refractoriness of the fireclay mixtures is also increased. Kaolin-sillimanite mixtures containing less than 60% of sillimanite are not so resistant to the solvent action of soda-lime glass as kaolin itself, whilst these mixtures are apparently more resistant than corresponding fireclay-sillimanite mixtures, the temperature at which vigorous interaction takes place being appreciably higher with the first-named mixtures. Kaolin-sillimanite mixtures give a regular reversible thermal expansion not affected by variations of the amount of sillimanite present. A commercial sillimanite brick was found to possess a porosity of 31.2% and a fusion point higher than that of cone 37. Ground samples of the brick heated in a gas-fired furnace for 2 hrs. at 1410° and then for 2 hrs. at 1500° gave a mean

Commercial sillimanite as a refractory material. I. Choice of bonding material and of grain size. A. COUSEN and W. E. S. TURNER (J. Soc. Glass Tech., 1925, 9, 334-346).-Microscopical examination of a sample of commercial sillimanite indicated much change of structure from the raw unfired material, suggesting the formation of a considerable proportion of mullite. Chemical analysis showed the presence of more than 6% of corundum, and the alumina content of the rest lay between that required for sillimanite and that for mullite. Comparative tests of plasticity, mechanical strength, and friability and also of porosity of mixtures of three different grades of the commercial sillimanite with ball clay, Stourbridge clay, and bentonite as bonding agents were carried out. Ball clay was the best bonding agent, the bentonite mixtures, although more plastic, giving a much greater drying shrinkage and a greater porosity. Comparison of ball clay mixtures amongst themselves showed that the one with 30 pts. of clay to 100 pts. of fine sillimanite (79%) through 120-mesh) gave the least porous material of the mixtures tried. A mixture with 4 pts. of fine and 2 pts. of the coarser fractions gave a porosity only 2% greater than that of the fine material, was little less plastic, and gave equally good results in melting operations. A. COUSEN.

Commercial sillimanite as a refractory material. II. Porosity, density, and mechanical strength of sillimanite-ball clay mixtures. A. COUSEN and W. E. S. TURNER (J. Soc. Glass Tech., 1925, 9, 347-356).-Two series of sillimanite mixtures, the one with the bulk of particles lying between 50- and 70-mesh, the other with all particles below 120-mesh, and both with ball clay as a bond, were compared as to porosity, density, and mechanical strength. The proportion of bond used was varied in each case to give 15, 30, and 40 pts. per 100 pts. of sillimanite. The tests served to show the advantage of the finer grade material, since this gave a lower porosity on firing and an increased mechanical strength. In all cases shrinkage and mechanical strength increased continuously with rise of firing temperature from  $100^{\circ}$  to  $1400^{\circ}$ , whilst the porosity decreased. In the fine sillimanite mixtures two zones of change were noted, namely  $800-1000^{\circ}$  and above  $1300^{\circ}$ , the former coinciding with that of the rapid shrinkage of ball clay, the latter probably indicating conversion of the mass into mullite. A mixture containing at least 30 and probably 40 pts. of ball clay per 100 pts. of sillimanite, and having much of the latter in a fine-grained form, proved most satisfactory, from the point of view of the properties examined, for the preparation of refractory A. COUSEN. articles.

CL. VIII .--- GLASS : CERAMICS.

Commercial sillimanite as a refractory material. III. Resistance to corrosion by glass. A. COUSEN, S. ENGLISH, and W. E. S. TURNER (J. Soc. Glass Tech., 1925, 9, 357-370). -Melting tests of soda-lime and potash-lead glasses in small sillimanite-ball clay and sillimanite-Stourbridge clay pots of compositions as studied in previous papers (cf. preceding abstracts), showed that the glass dissolved less iron oxide from the pots than it did from clay pots. The tests led to the choice of the mixture: fine sillimanite (through 100-mesh sieve) 5 pts., medium sillimanite (70- to 100-mesh) 1, coarse sillimanite (30- to 70-mesh) 1, ball clay (finely ground) 3 pts., for making larger pots for further work. These larger pots dried much more rapidly than did clay pots of the same size, the drying shrinkage being small, namely 1 in 39, and the firing shrinkage was extremely small. In the furnace, the pots were successfully used for relatively long periods at temperatures of 1400-1500° and with severe temperature drops at week ends, without signs of sagging or losing shape. Glass worked from the pots was of very varying types and composition, but there was a distinct absence of stones. Analysis of the glasses proved them to have abstracted, on the average, distinctly less iron oxide from the pots than would have been dissolved from clay pots. The alumina content of the glasses, however, was fairly large and indicated distinct general corrosion, although this corrosion was more uniform and less marked than with similar fireclay pots. A. COUSEN.

Mineralogy of clay. I. J. S. McDOWELL (J. Amer. Ceram. Soc., 1926, 9, 55-60).—In a review of the literature on the microscopical examination of clays, the constituents of clays are grouped and discussed under three headings, viz., the essential, the major accessory, and the minor accessory ingredients. F. SALT.

Rational analysis of clay. II. J. S. McDowell (J. Amer. Ceram. Soc., 1926, 9, 61—65).—A review of the literature. The methods of rational analysis are based on the inaccurate assumption that clays consist mainly of kaolinite, quartz, and undecomposed felspar, whereas microscopical examination shows that many clays contain hydromica, and not felspar. Greater accuracy in the calculation of rational analyses might be possible by the use of data supplied by time-temperature curves and microscopical investigation. F. SALT.

An eighteen months' high-temperature test on refractory test specimens. F. H. RIDDLE and A. B. PECK (J. Amer. Ceram. Soc., 1926, 9, 1-22).-The effect was studied of time, temperature, kiln atmosphere, etc. upon a number of standard bodies and experimental mixtures, which had been allowed to remain in various parts of a Dressler muffle kiln for a period of eighteen months. The kiln was fired to cone 17-18, and the atmosphere was mainly of an oxidising nature. The construction of the kiln is described. At about 600°, silicon carbide specimens expanded to nearly twice their original size,

much cristobalite being formed. At the highest temperature (above 1446°) swelling was not so apparent, but volatilisation was very pronounced and oxidation to cristobalite was almost complete. Mullite was found in varying amounts in most of the specimens. Porcelain sparking-plug bodies exhibited no change in microstructure up to 1200°, but at 1296° the mullite crystals had increased somewhat in size. At 1462° their average size was about four to five times larger than the original. No corundum was developed. At high temperatures the glaze had been absorbed or volatilised. Sections of pyrometer tubes were affected in a similar manner up to 1050°, but at the highest temperature crystal development was more marked. Pyrometer tubes were less refractory, and showed a tendency to blister and craze. The microstructure of silica bricks taken from various parts of the hot zone indicated definite variations in temperature, both vertically and horizontally, within that zone. Silicious bonding material proved more suitable than fireclay or chromite. A silicon carbide mortar was oxidised to cristobalite in the hot zone, the resulting expansion giving a very tight joint. The incidental formation of three artificial minerals was observed. Volatilised silica from the silicon carbide and silica bricks in the hot zone was deposited in the water-smoking zone as silica glass. Crystals composed of a mixture of  $Fe_3O_4$  and  $Fe_2O_3$  were found in the cooling zone under conditions indicating a dissociation temperature of Fe<sub>2</sub>O<sub>3</sub> lower than hitherto accepted. Lithophysæ composed of cristobalite were formed, by oxidation, on many silicon carbide specimens in the hot zone. F. SALT.

Deformation study of various aluminosilicates and borosilicates. K C. LU (J. Amer. Ceram. Soc., 1926, 9, 29—54).—The literature is briefly reviewed. Two aluminosilicate and three borosilicate eutectics were developed within the limits of 2.5 molecular per cent. differences of the various constituents. The molecular formulæ and approximate deformation temperatures are : PbO:  $0.254Al_2O_3.1.91SiO_2$ ,  $650^\circ$ ; ZnO: $0.225Al_2O_3.0.906$ SiO<sub>2</sub>, 1360°; PbO: $0.238B_2O_3.0.78SiO_2$ , 415°; Na<sub>2</sub>O:  $1.29B_2O_3.1.73SiO_2$ ,  $570^\circ$ ; K<sub>2</sub>O: $2.11B_2O_3.2.585SiO_2$ ,  $655^\circ$ . The relation between these eutectics was studied by plotting their positions, according to molecular percentages, on the triaxial diagram.

F. SALT.

Action of gases on porcelain glazes. A. PFAFF (Ber. Deuts. Keram. Ges., 1925, 6, 42-44; Chem. Zentr., 1926, I., 763).—The effects of the individual constituents of a normal mixture of furnace gases upon glazed hard porcelain during burning were investigated. Oxygen gave a rough, ugly effect, which was less pronounced with air and absent with nitrogen. Carbon dioxide caused no faults, but carbon monoxide produced small bubbles, and hydrogen foamy bubbles. A. COUSEN.

Thermal expansion of Jena glass 16<sup>111</sup>. VAN. AGT and ONNES.—See A., Mar., 231.

#### PATENTS.

Production of copper mirrors on glass. K. BAMBERGER and R. SCHWEIZER (G.P. 420,469, 6.2.24).—Copper mirrors may be produced on glass and other non-metallic substances by treating a suspension of cuprous hydroxide in a solution containing 0.5% of free alkali with hydrazine hydrate or sulphate in contact with the glass. The deposits adhere well and have a high lustre. A. R. POWELL.

Apparatus for washing clay. K. H. REICHAU (E.P. 245,608, 24.1.25).—A conduit formed of tubular members of different diameters increasing stepwise from bottom to top is suspended in a water container. At the top the conduit passes through the floor of a receptacle into which it discharges. The coarse particles separate from the clay suspension as it flows upwards through the conduit and fall through the gaps between the tubular members into the water container.

#### P. B. ROBINSON.

Preparing clay or body composition for ceramic articles. A. O. AUSTIN, ASST. to OHIO BRASS CO. (U.S.P. 1,569,251, 12.1.26. Appl., 1.3.23). —Material is prepared partly as a slip and partly dry, and mixed to provide a body mix having a slightly greater percentage of water than that at which the ceramic articles are formed, the excess water being removed by evaporation.

#### P. B. ROBINSON.

Manufacturing artificial abrasives. C. J. BROCKBANK, Assr. to ABRASIVE Co. (U.S.P. 1,568,873, 5.1.26. Appl., 11.9.20).—Substantially pure crystalline alumina is produced by fusing an aluminous ore with a reducing agent and boric acid.

#### F. SALT.

Method of making crucibles. A. J. JACKMAN, Assr. to VESUVIUS CRUCIBLE CO. (U.S.P. 1,570,064, 19.1.26. Appl., 18.3.25).—A crucible for metalmelting operations in which slag is formed has a graphite body adapted to be in contact with the bulk of the metal, whilst the portion which is to be in contact with the slag is made of material which is more resistant than graphite to the action of the slag. W. CLARK.

Manufacture of silica bricks. DR. NORTH A.-G. (G.P. 420,851, 30.10.21).—Powdered quartzite or sand is mixed with the usual binder and with powdered coal, which accelerates the transformation of the quartz into tridymite and cristobalite during firing. A. R. POWELL.

Glass. W. C. TAYLOR, ASST. to CORNING GLASS WORKS (U.S.P. 1,572,625, 9.2.26. Appl., 7.1.21).— See E.P. 192,919; B., 1923, 355 A.

Plastic [magnesia compositions]. O. FROSELL, Assr. to WILLIAMSPORT BUILDING PRODUCTS CO. (U.S.P. 1,572,714, 9.2.26. Appl., 25.3.21).—See E.P. 202,698; B., 1923, 1133 A.

#### Treating clay (U.S.P. 1,561,971).-See XIV.

#### IX.—BUILDING MATERIALS.

#### PATENTS.

Heat-insulating material. L. G. WILKENING (E.P. 226,522, 8.12.24. Conv., 22.12.23).—Light peat, e.g., peat moss or "fox peat," is coked at 400—600°, producing a fine porous material of low sp. gr. The product may be used alone as an insulating material, or mixed with cementitious or bituminous binding materials to form insulating blocks. B. W. CLARKE.

Heat-insulating material. W. T. IRVIN, Assr. to CELITE Co. (U.S.P. 1,569,755, 12.1.26. Appl., 24.12.24).—Heat-insulating material is prepared by mixing diatomaceous earth and an alkaline-earth, moulding, and then hardening and drying by exposure to large volumes of air under pressure. P. B. ROBINSON.

Kilns for use in cement manufacture. S. W. BURLEY (E.P. 245,866, 22.10.24).—Air is forced into the ash-pit of a non-rotary cement kiln and a draught over the slurry bed is induced by an extraction fan situated at the exit end of the kiln. The air supply is controlled so that it may be concentrated at one or more spots on the charge, thus producing more efficient working. The process renders the operation of the kiln independent of atmospheric conditions and increases the output of the kiln as well as enabling low-grade fuel to be burnt. B. W. CLARKE.

Cementitious material. N. V. S. KNIBBS (E.P. 245,935, 13.1.25).—Lime or magnesia or magnesian lime is subjected to carbonation at a high temperature in order to ensure a degree of carbonation or hydration between definite maximum and minimum limits, which vary with the composition of the raw material. Thus lime containing over 95% of calcium oxide is carbonated to a calcium carbonate content of 30—80%, using waste producergas etc. The product when ground possesses good cementitious properties, and may be used either alone or with hydrated lime etc. to form blocks, slabs, etc. B. W. CLARKE.

Portland cement. W. VERSHOFEN (G.P. 419,353, 16.1.23).—To the dry, powdered, clay-limestone mixture is added a water-soluble fluoride, capable of increasing the bonding power of the mixture when water is added, while also reducing the firing temperature necessary to produce clinkering in the kiln to about 1000°, owing to the chemical action of the fluoride on the mixture. A. COUSEN.

Wood preserving. F. RASCHIG (E.P. 246,010, 4.6.25).—Sodium silicate (water-glass) is added to a mixture of sodium fluoride and dinitrophenol or dinitrocresol, used as a wood preservative, to prevent the corrosion of iron or steel vessels by the woodpreserving liquid. B. W. CLARKE.

Dyeing wood. H. RENNER, Assr. to FOREST PRODUCTS RESEARCH CORP. (U.S.P. 1,570,575, 19.1.26. Appl., 26.10.23).—The sap in the cellular D 2 tissue of the wood is combined with a mixture of a colouring metal compound and a volatile or unstable chemical which forms unstable salts. The complex salts formed are decomposed when the wood is dried, so that permanent colouring substances are fixed in the cells of the wood. B. W. CLARKE.

Plaster of Paris. E. L. WILSON, ASST. to RUM-FORD CHEMICAL WORKS (U.S.P. 1,570,583, 19.1.26. Appl., 22.8.24).—Precipitated gypsum is subjected to pressure to cause coalescence of the crystals, which are converted into a non-compacted extended mass. This is uniformly calcined to produce plaster of Paris. B. W. CLARKE.

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

Some physical properties of steel and their determination. J. H. ANDREW, M. S. FISHER, and J. M. ROBERTSON (Proc. Roy. Soc., 1926, A 110, 391-422) .- The electrode potentials and specific resistances of carbon-, chromium-, nickel-, and nickel-chromium-steels, which have been subjected to various types of heat treatment, have been determined as functions of the carbon content of the steel. The variation of specific resistance with time during tempering at 240° has also been investigated for the same steels. The electrode potential (against a solution of ferric chloride as electrolyte) of quenched hypoeutectoid steels is raised, and that of hypereutectoid steels lowered, by raising the quenching temperature. The lowering in the latter case is very marked when the quenching temperature is raised from 1000° to 1100°. The potential of tempered steels rises with the temperature of tempering. The electrode potentials measured are those of a-iron, modified by the presence of carbon, nickel, and chromium in solu-tion; it is lowered, for example, by dissolution of carbon. The specific resistance of hypereutectoid steels is increased, and that of hypoeutectoid steels decreased by raising the quenching temperature. Corrected for the small amounts of silicon, phosphorus, and manganese present, the specific resistance, R, for the carbon steels is given by R=7.5+26.7×carbon content. In a nickel-chromium steel of given carbon content, the increase in resistance above that of the carbon steel is proportional to the amount of each element present. In the tempering experiments the specimen of steel was immersed in an oil bath at 240°, and its electrical resistance determined at intervals over a period of about 5 hrs. The tempering-resistance curves for all steels quenched at 900°, 1000°, or 1100° were similar, showing an initial rapid decrease in resistance, then a slow fall or an almost constant value. An analysis of these curves permits calculation of the rate at which carbon separates from solution in austenitic and martensitic steels respectively. The usual procedure for investigating the process of tempering, involving periodic cooling of the steel to ordinary temperature to make observations, may lead to erroneous results. During cooling a considerable amount of austenite tempers to martensite; the former can therefore temper in two ways, slowly at a constant temperature, and more rapidly when the temperature is falling. A. B. MANNING.

Determination of cerium in special steels. K. SWOBODA and R. HORNY (Z. anal. Chem., 1926, 67, 386-398).-Slightly low results are usually obtained by precipitation of a slightly acid solution of cerous nitrate with oxalic acid followed by ignition of the cerous oxalate to cerium dioxide. Precipitation as ceric peroxyacetate yields good results even in the presence of chromium and tungsten compounds if the operation is conducted as follows. The solution is treated with 1 g. of tartaric acid, diluted to 200 c.c., and neutralised with ammonia. Two drops of hydrochloric acid, 15 c.c. of 6% hydrogen peroxide, and 10 c.c. of 10% ammonium acetate solution are added and the mixture is boiled; a further 10 c.c. of acetate is added together with a little filter pulp and boiling is continued until the precipitate flocculates well, but care must be taken to avoid the complete destruction of the hydrogen peroxide. The brown precipitate is collected, washed with very dilute ammonium acetate solution, and ignited wet to the dioxide for weighing. In the presence of ammonium salts low results are obtained, in which case precipitation as fluoride is recommended. The procedure is similar to that described above except that precipitation is effected with 20 drops of hydrofluoric acid and a little ammonium fluoride in place of the peroxide and acetate. Ignition of the fluoride, without drying, leaves a residue of the dioxide. The latter method is applicable to the determination of cerium in special steels with the following modified procedure :---2 g. of the steel turnings are dissolved in 60 c.c. of 1:1 hydrochloric acid, and nitric acid is added drop by drop to oxidise the iron, avoiding an excess. The hot solution is treated successively with 60 c.c. of 25% tartaric acid solution, 30-35 c.c. of a 10% solution of stannous chloride to reduce the iron, and sodium hydroxide until slightly alkaline. The mixture is cooled and diluted to 500 c.c. in a graduated flask after adding 10 c.c. of alcohol, and filtered rapidly through pleated filters. The filtrate (250 c.c.), which is free from iron and tin, is acidified with hydrochloric acid, heated to boiling, and treated with 2 g. of solid ammonium fluoride. The solution is made just ammoniacal, again acidified with hydrochloric acid, and filtered, after 1 hr., with the addition of filter pulp. The precipitate is washed with a hot 0.3% solution of ammonium fluoride, ignited wet in a platinum crucible, and weighed as CeO<sub>2</sub>. A. R. POWELL.

Electroplating on non-rusting iron. J. HAAS and E. R. UNRUH (Metal Ind. N.Y., 1925, 23, 451-452; Chem. Zentr., 1926, I., 1278-1279).-Before electroplating on non-rusting iron the surface must be pickled in order to remove the thin layer of passive metal so as to obtain a good, strongly adherent deposit. This is best effected by a short electrolytic pickle in concentrated hydrochloric acid at  $60^{\circ}$ , or, somewhat less satisfactorily, by heating the article at  $60^{\circ}$  in 20% sulphuric acid. After this treatment the metal must be plated directly in an acid bath, which prevents the surface from again becoming passive better than an alkaline bath.

#### A. R. POWELL.

Protection of iron by cadmium. H. S. RAWDON (Trans. Amer. Electrochem. Soc., 1926, 49, 21-30. Advance copy).-Cadmium behaves similarly to zinc in preventing the corrosive attack of iron when specimens of iron and steel containing plugs of the protective metals are immersed in N/2-sodium chloride solution. The anodic behaviour of cadmium and zinc with respect to iron under the experimental conditions was clearly revealed by the etching action at the surface of the inserted plugs. Whilst the values assigned by most authorities to the electrode potentials of these metals place iron between the other two, the experiments show that for the conditions described, cadmium and zinc stand in the same relation to iron. The rate of etching indicated that the potential difference between zinc and iron when in contact and immersed in sodium chloride solution is considerably greater than that between cadmium and iron under the same conditions. M. COOK.

Nature of the protective film of iron. T FUJIHARA (Trans. Amer. Electrochem. Soc., 1926, 49, 1—8. Advance copy).—The uncorroded iron outside a drop of water on a polished iron surface is due to a protective film of ferrous hydroxide. The ferrous hydroxide is soluble, forming an alkaline solution, and the film would eventually stop corrosion if carbon dioxide were excluded from the air in which the iron is corroding. The protective film is destroyed by carbon dioxide neutralising the alkaline liquid. M. COOK.

Determination of silver, gold, and platinum in anode slimes. E. ECKERT (Metall u. Erz, 1925, 22, 595-598; Chem. Zentr., 1926, I., 987-988).-Ten g. of the slime are dissolved in nitric acid and, after removal of the insoluble matter, the silver is precipitated with hydrochloric acid and the chloride is weighed. Gold and platinum are determined by scorification and cupellation. The silver bead is dissolved in nitric acid, leaving a residue of gold and most of the platinum. The silver is precipitated as chloride from the solution and the filtrate evaporated to dryness on the water-bath. The residue is dissolved in hydrochloric acid and the solution transferred to a lead capsule, in which it is again evaporated. The capsule and residue are cupelled with the major portion of the platinum and gold and with sufficient silver for the subsequent parting. The bead is parted in sulphuric acid and the residue ignited and weighed. It is then re-alloyed with silver and the bead parted in nitric acid; this is repeated until the gold residue is of constant weight. Platinum is found by difference.

A. R. POWELL.

Reverberatory refining of copper. Influence of prolonging the blowing on the impurities in and properties of the metal. W. HEOKMANN (Metall u. Erz, 1925, 22, 527—546; Chem. Zentr., 1926, I., 1275).—An investigation of the mechanism of the refining of copper scrap in the reverberatory furnace by the addition of black copper, converter copper, and copper scale is described, in the course of which the composition and mechanical properties of samples taken from the bath at  $\frac{1}{2}$ -hr. intervals have been determined. When the blowing is prolonged so that the metal contains more than 0.8— 0.85% O, separation into two layers begins to take place, the upper layer of cuprous oxide then becoming further oxidised and combining with the fluxes on the surface. A. R. POWELL.

Influence of cuprous oxide on electrolytic and refined copper. H. ALTWICKER (Metall u. Erz, 1925, 22, 583-594; Chem. Zentr., 1926, I., 1027). -The mechanical properties of tests taken from charges of various kinds of copper during refining have been examined and correlated with the content of cuprous oxide. Less than 0.23% O has no effect on hot-rolled metal but 0.12% causes corrosion to take place readily in cold-rolled copper. The presence of cuprous oxide has little effect on the tensile strength, but it reduces the ductility very markedly; e.g., the elongation of copper containing 0.01% O is 360% greater than that of copper containing 0.387% O. The hardness of copper increases with the content of cuprous oxide, small proportions of which also improve the Erichsen number. The electrical conductivity decreases with increasing oxygen content, but the pliability is scarcely affected. A. R. POWELL.

Determination of copper, arsenic, and mercury. R. ROSENDAHL (Chem.-Ztg., 1926, 50, 73-74).—For the determination of arsenic in metals and ores the substance is dissolved in nitric acid and the solution evaporated with sulphuric acid to expel the nitric acid. After dilution an equal volume of hydrochloric acid is added, iron and copper are reduced by addition of stannous chloride to ferrous and cuprous chlorides, and the arsenic is separated by warming the solution at 80-90° with 5 g. of sodium hypophosphite. The washed precipitate is distilled with concentrated hydrochloric acid and a little ferric chloride, the residual solution is diluted to 750 c.c., manganese sulphate is added, and the ferrous chloride formed is titrated with permanganate (0.4475 Fe=As). Alternatively, the arsenic is dissolved in nitric acid and the solution evaporated with sulphuric acid and sulphur to reduce the arsenic acid. After dilution and expulsion of sulphur dioxide the arsenic may be determined as usual with iodine, or, after addition of a known weight of antimonious chloride and 12 c.c. of hydrochloric acid, by titration with permanganate. The addition of antimony ensures a good end-point. Copper may be determined in ores by dissolving in nitric and sulphuric acids a quantity containing not more than 0.2 g. of copper, evaporating to expel the nitric acid, diluting, adding ammonia until alkaline, 6-7 g. of tartaric acid, and 5 g. of sodium hypo-phosphite. After boiling for 20 min., the precipitated

copper is collected, washed, dissolved in hot acidified ferric chloride solution, and the ferrous chloride formed determined as usual. Mercury is determined by dissolving the metal in nitric acid, evaporating with sulphuric acid, adding 20 c.c. of water, boiling the solution with 50 c.c. of hydrochloric acid to expel arsenic, and finally precipitating the mercury as metal by prolonged boiling with 5 g. of sodium hypophosphite. The precipitate is collected into a globule, dried, and weighed.

#### A. R. POWELL.

Aluminium-lithium alloys. P. ASSMANN (Z. Metallk., 1926, 18, 51-54).-Aluminium and lithium form a eutectic with 7.8% Li melting at 598° and a solid solution containing 3.5% Li at  $598^{\circ}$  and only 2.2% Li at  $20^{\circ}$ . The Brinell hardness increases with the lithium content to a maximum of 130 with 12.1% Li, but the greatest increase occurs between 2 and 4% Li. On heating to 500°, quenching, and ageing at ordinary temperature all the alloys increase in hardness, especially those containing 0.3% and 2-4% Li; ageing at 200° entirely prevents this increase. The hardness of aluminium alloys containing 0.4-1.0% Li and 4-12% Li is appreciably increased by annealing at 300-400°, and still more so by quenching from 500° and ageing at 18°. Lithium-copper-aluminium alloys increase in hardness by nearly 70% after the latter treatment, the maximum hardness being obtained with 0.4% Li and 4% Cu or with 1.0% Li and 2% Cu. Alloys with 0.4% Li and 2-7% Cu are still further hardened by ageing at 100°, whereas those containing 1.0% Li and 2-4% Cu are slightly softened by this treatment. A. R. POWELL.

Metallic uranium. J. F. GOGGIN, J. J. CRONIN, H. C. FOGG, and C. JAMES (Ind. Eng. Chem., 1926, 18, 114-116).-Uranosouranic oxide is reduced to finely-divided metal on heating with metallic calcium but the yield is small. A good yield of very pure fused uranium is obtained by reduction of uranium chloride with a 10% excess of metallic calcium; the operation is carried out in an alundum crucible placed inside a nichrome-wound reduction bomb which is placed inside a steel cylinder from which the air is exhausted by means of Hyvac pumps. The product obtained by this method was a hard, brittle metal containing 0.57% Fe, 0.09% C, and 0.03% O when commercial calcium was used, but a much purer product, containing only 0.01% Fe, was obtained by the use of re-sublimed calcium. The ingot showed fine crystalline markings, had a very silvery lustre and convex surface, and appeared to be sound. Uranium chloride for the reduction may be obtained by heating the oxide in a current of chlorine and sulphur chloride to 900-1000°.

#### A. R. POWELL.

Preparation of antimony-regulus. F. BÖRNER (Metall u. Erz, 1925, 22, 559-564; Chem. Zentr., 1926, I., 1005).—A mixture of antimony ore and iron is roasted, sodium chloride or carbonate being added to render the slag (ferrous sulphide) liquid. By further treatment, with addition of antimony

sulphide, the iron is removed from the crude antimony. The author gives full details of the process and of the analysis of metal and slag. S. S. WOOLF.

Equilibrium between metals and salts in the molten state; a new form of law of mass action. R. LORENZ (Z. angew. Chem., 1926, 39, 88-90).-In the melt represented by the equation  $M'+M''X \rightleftharpoons M'X+M''$ , the metals and salts form two separate phases. The equilibrium state is defined by the relation [x/(1-x)].  $[(1-y)/y] = Ke^{y}$ , where x, 1-x, y, 1-y, represent the molecular proportions of the reacting and resulting substances, K is a constant, and u is defined by a somewhat complex equation. It is shown experimentally for the system  $Pb+CdCl_2 \Rightarrow PbCl_2+Cd$  that log K is an appreciably constant quantity. W. T. K. BRAUNHOLTZ.

Transformations of certain alloys of aluminium, and influence of deformation. L. GUILLET (Rev. Mét., 1926, 23, 48-52).-See B., 1925, 995.

Hydrogen-ion control of nickel-plating baths. PARKER and GREER.—See XI.

#### PATENTS.

Magnetic materials [iron-nickel alloys]. WESTERN ELECTRIC CO., LTD. From WESTERN ELECTRIC Co., INC. (E.P. 245,986, 22.4.25; cf. E.P. 189,410; B., 1923, 101 A).-Iron-nickel alloys containing nickel 80-83%, after suitable heat treatment, have certain electric and magnetic properties which are substantially independent of applied mechanical forces over a considerable range. Under tensions up to 5000 lb. per sq. in., the permeability, the hysteresis loss per cycle, and the resistivity remain practically unaffected. The alloys are particularly suitable as loading material for increasing the inductance of submarine cables. L. M. CLARK.

Annealing sheet steel. G. H. COLE, Assr. to WESTINGHOUSE ELECTRIC AND MANUF. Co. (U.S.P. 1,569,355, 12.1.26. Appl., 21.12.20).—Sheets of magnetic silicon alloy steel are annealed by heating to a temperature not exceeding 700° in an atmosphere containing approximately the same ratio of oxygen and nitrogen as in air. M. COOK.

Alloy [steel]. J. I. MEE (U.S.P. 1,569,996, 19.1.26. Appl., 6.2.25).-An alloy steel containing tungsten, vanadium, chromium, sulphur, copper, A. R. POWELL. and iridium is claimed.

Reduction of iron ores by means of methane or gases containing methane. GELSENKIRCHENER BERGWERKS-A.-G., ABT. SCHALKE (G.P. 421,462, 25.1.24).-In the presence of a small quantity of metallic iron, methane reduces ores containing iron oxide at temperatures below 700°. The metallic iron necessary to start the reduction may be added in powdered form to the ore or, preferably, produced in situ by giving the ore a short preliminary treatment with a gas containing hydrogen.

A. R. POWELL.

Cast-iron low in phosphorus. UNION DE CONSOMMATEURS DE PRODUITS METALLURGIQUES ET INDUSTRIELS (F.P. 577,661, 28.4.23; G.P. 421,991, 3.6.23).—Ordinary grey cast-iron is dephosphorised as usual in the converter and the requisite amount of carbonaceous material to regenerate cast iron is added, together with substances such as thermit, ferrosilicon, or ferromanganese which generate heat and raise the temperature of the molten metal so that it can dissolve a greater proportion of carbon. A. R. POWELL.

Manufacture of very thin wires. N. V. PHILIPS' GLOEHLAMPENFABRIEKEN (E.P. 235,893, 17.6.25. Conv., 18.6.24).—By using thin wires as the cathode in a glow discharge at such a current density that disintegration occurs, a very thin wire the diameter of which is practically uniform, and may be less than 10 microns, is obtained. The process may be applied to all single-crystal wires. L. M. CLARK.

Coating welding electrodes. SIEMENS-SCHUCKERTWERKE G.M.B.H. (E.P. 230,831, 11.3.25. Conv., 12.3.24).—In the preparation of papercovered electrodes, fluxes or materials for regulating the combustion of the covering may be introduced into the paper or pulp during the course of its manufacture (cf. following abstract). A. R. POWELL.

Jacketed welding electrodes. P. C. RUSHEN. From SIEMENS-SCHUCKERTWERKE G.M.B.H. (E.P. 245,635, 11.3.25).—A jacket or coating for welding electrodes consists of paper, paper pulp, or similar non-conductor which carbonises on heating. The combustion of the jacket during welding operations may be regulated by impregnating it with solutions of substances which aid its combustion (cf. preceding abstract) or by applying such substances either underneath or on the surface of the jacket.

A. R. POWELL.

Hardening copper. L. NEMITOF (U.S.P. 1,569,130, 12.1.26. Appl., 6.2.22).—Copper heated to bright redness is embedded in a mixture containing a silicious substance and calcium phosphate moistened with a hydrocarbon. M. COOK.

Refining copper-nickel matte. R. L. PEEK and T. F. TORELL, Assrs. to NATIONAL TRUST Co., LTD. (U.S.P. 1,569,137, 12.1.26. Appl., 26.8.20).— Bessemerised copper-nickel matte is utilised for precipitating copper from copper-nickel sulphate solution by the cementation process. The greater part of the copper is then removed from the partially exhausted matte and the residue is used for the preparation of fresh copper-nickel sulphate solution. L. M. CLARKE.

Disintegrating metals in a ball mill or the like. E. J. HALL, ASST. to METALS DISINTEGRATING Co. (U.S.P. 1,569,484, 12.1.26. Appl., 25.3.19. Renewed 3.6.25).—Metal dust is prepared by mechanically disintegrating the metal, while keeping it covered with a lubricating liquid which is inert to the metal, and protects it against the effects of the atmosphere. The lubricating liquid has a b.p. sufficiently high to prevent excessive loss by vaporisation during the disintegration, but not high enough to cause difficulty in its subsequent removal from the metallic powder by evaporation. A. B. MANNING.

Alloy for pen-points. M. M. GOLDSMITH (U.S.P. 1,569,921, 19.1.26. Appl., 16.11.23).—An alloy for tipping the ends of pen nibs comprises 10—20% Ru, 3—10% Ni, and 65—85% Os. A. R. POWELL.

Alloy [for soldering aluminium]. P. A. J. H, DEMEYER (F.P. 594,836, 6.3.25).—An alloy suitable for soldering aluminium to aluminium, iron, copper, lead, steel, brass, or bronze comprises 15—64% Zn and 85—36% Cd and is made by adding cadmium to molten zine. A. R. POWELL.

Aluminium alloys. G. KROLL (F.P. 594,851, 7.3.25. Conv., 8.3.24).—Alloys of aluminium with 20—35% Si and up to 5% of silver, titanium, zirconium, or boron or mixtures of these elements are claimed. The resulting alloys are much harder than those containing less silicon and also have a smaller coefficient of expansion. A. R. POWELL.

Hard [lead] alloys. MATERIEL TÉLÉPHONIQUE Soc. ANON. (F.P. 595,419, 3.2.25).—A mixture of lead with antimony, tin, lead, aluminium, or zinc is heated for a long time at a temperature below the m.p. of the mixture. The mass is then quickly cooled and aged at a temperature below 100°. For example, a mixture of 97.5 pts. of lead and 2.5 pts. of antimony is heated for 72 hrs. at 240°, cooled quickly, and aged ; the resulting mass has a tensile strength of 770 kg. per sq. dm. A. R. POWELL.

Treating sulphide ores [for recovery of sulphur, copper, and iron]. R. D. PIKE (U.S.P. 1,570,777, 26.1.26.Appl., 13.6.23).-Sulphide concentrates containing copper and iron are heated with a solution of ferric chloride or sulphate in an autoclave fitted with a steam injector, the temperature being maintained above the m.p. of sulphur (e.g., 140°). The residue is distilled for the recovery of sulphur and then worked up for the recovery of the precious metals. The solution is treated with pyrrhotite to reduce the excess of ferric salt used in leaching, then with sponge iron to precipitate copper and the precious metals. The residual ferrous chloride or sulphate solution is electrolysed to recover part of the iron and regenerate ferric chloride for use A. R. POWELL. again in the process.

Protective coatings for metallic surfaces. E. WEINTRAUB, and Soc. ALSACIENNE DE CON-STRUCTIONS MÉCANIQUES (F.P. 592,986, 22.4.24).— Metallic surfaces may be protected from oxidation and rendered non-porous by a coating of aluminium borate or other refractory borate. Metal treated in this way does not evolve gas when heated to high temperatures or when subjected to a high vacuum and is therefore suitable for electric vacuum apparatus. A. R. POWELL.

Electrolytic preparation of alkaline-earth metals. CHEM. FABR. KALK, H. OEHME, and E. HERRMUTH (G.P. 420,042, 24.11.23).—A contact cathode which is gradually raised is used to prevent re-melting of the deposited alkalineearth metal; the upper part of the rod, above the level of the fused electrolyte, is cooled from outside by means of a stream of gas, or by means of a cylinder, through which water flows. R. B. CLARKE.

Melting light metals, e.g., aluminium, magnesium, or their alloys in an induction furnace. RHEINISCHE METALLWAREN- U. MASCHINENFABR. (G.P. 421,281, 6.2.24).—Light metals and alloys are melted in an induction furnace in an atmosphere of an indifferent gas under such pressure that the combined pressure of gas and molten metal is sufficient to overcome the pinch effect in the secondary circuit. The furnace is enclosed in a gas-tight chamber provided with valves for the admittance of the compressed gas, the valves being coupled with the doors of the furnace so that the latter cannot be opened until the internal pressure has been relieved. A. R. POWELL.

Recovery of zinc from burnt pyrites after a chloridising roast. M. SCHMIDT (G.P. 422,044, 15.7.24).—The solutions obtained by leaching burnt pyrites that has been subjected to a chloridising roast are separated according to their content of sulphate and the solutions richest in sulphate are used for leaching fresh charges of roasted ore so as to enrich them in zinc and reduce the sulphate content. The enriched solutions are treated with calcium chloride to remove the sulphate as calcium sulphate and the weak zinc solutions are treated with milk of lime to obtain a mixture of zinc hydroxide and calcium sulphate. During the leaching process the ore is added to the solution as hot as possible.

A. R. POWELL.

Electrolytic deposition of chromium. H. Wolff (G.P. 422,461, 28.3.23).—An anode of pressed and sintered chromium powder is used in the electroplating of articles with chromium. This anode does not become passive and the bath is therefore not denuded of chromium and may be used for chromium-plating drawn wires.

A. R. POWELL.

Aluminium alloy. R. L. JOHNSTON, R. S. ARCHER, and Z. JEFFRIES, ASSTS. to ALUMINUM Co. OF AMERICA (U.S.P. 1,572,489-90, 9.2.26. Appl., 27.11.20 and 11.2.25).—See E.P. 172,018; B., 1923, 361 A. Special claim is made for an alloy containing Si 8, Zn 10, and Cu 2.5%.

Production of alloyed steel and iron with chromium, manganese, etc. T. G. and F. E. L. STIG (U.S.P. 1,571,382, 2.2.26. Appl., 21.3.24).— See E.P. 213,568; B., 1925, 13.

Metal-extraction process. L. VENN-BROWN (U.S.P. 1,571,502, 2.2.26. Appl., 23.9.24).—See E.P. 239,720; B., 1925, 887.

Method of starting electrolytic cells [for refining metals]. D. H. TILSON, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1.572,253, 9.2.26. Appl., 30.11.23).—See E.P 225,494; B., 1925, 362.

Smelting or heating furnace with firing of coal dust. E. VOGT and L. KIRCHHOF (U.S.P. 1,572,336, 9.2.26. Appl., 26.3.25).—See E.P. 231,513; B., 1925, 867.

Making castings of aluminium-silicon alloys. R. S. ARCHER and J. D. EDWARDS, Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,572,459, 9.2.26. Appl., 27.11.20).—See E.P. 171,997; B., 1923, 662 A.

Metallurgical furnaces. B. TALBOT (E.P. 246,627–8, 5.12.24).

Cupola furnaces with hot blast apparatus. E. VANDERSTEIN (E.P. 246,932, 11.11.24).

Annealing furnace (U.S.P. 1,569,356).—See I.

Treating alkali liquors from purification of lead (E.P. 245,479).—See VII.

Crucibles (U.S.P. 1,570,064).—See VIII.

#### XI.—ELECTROTECHNICS.

Hydrogen-ion control of nickel-plating baths with the quinhydrone electrode. H. C. PARKER and W. N. GREER (Trans. Amer. Electrochem. Soc., 1926, 49, 9-20. Advance copy).-The most satisfactory  $p_{\rm H}$  for nickel-plating solutions is between 5.7 and 6 and this range is most conveniently determined by the use of a quinhydrone electrode. The apparatus consists of a portable potentiometer, a Parker and Dannerth combination cell (B., 1925, 555), a beaker, and a burette. About 200 c.c. of the solution to be tested are placed in the beaker, 10 c.c. of a saturated quinhydrone solution are added, the combination cell is lowered into the liquid, a potentiometer measurement taken after 2–3 min., and the  $p_{\rm H}$  value read from a graph. A method is described for maintaining the acidity of the bath at the correct figure without weighing and without the use of standard solutions. The quinhydrone electrode method of determining the  $p_{\rm H}$  of nickel-plating baths is more accurate than any colorimetric method, is easier to manipulate, and requires less elaborate apparatus.

A. R. POWELL.

Determining silver, gold, and platinum in anode slimes. ECKERT.—See X.

#### PATENTS.

Electrolytic cells. BADISCHE ANILIN- U. SODA-FABR., Assees. of G. PFLEIDERER (G.P. 421,784, 13.11.23).—To avoid disturbances in the cell caused by leakage, it is enclosed in a container, inside which is a gas, *e.g.*, nitrogen, steam, or one of the gases produced by the electrolysis, under pressure. R. B. CLARKE. Producing lead oxide [from storage-battery plates]. S. M. EVANS (U.S.P. 1,570,438, 19.1.26. Appl., 5.3.23).—Finely divided lead compounds from storage-battery plates are treated with a hot solution of an alkali-metal base, washed, and oxidised by heat. J. GRANT.

Exposure of liquids to ultra-violet rays. QUARZLAMPEN-GES. M.B.H. (G.P. 421,568, 26.5.23). —By centrifugal force the liquid is drawn in a thin layer up the inner walls of a revolving drum inside which is a source of ultra-violet light and which provides for a circulation of the liquid. By tubes leading down into the bulk of the liquid, air or other gases may be introduced. Bleaching and polymerisation of oils, softening of water, sterilisation, and acceleration of fermentation are among the suggested uses. S. S. WOOLF.

Electrolysing fused baths. R. J. McNitt (E.P. 246,542, 30.10.24).—See U.S.P. 1,524,268; B., 1925, 249.

See also pages 224, Electrical precipitation (E.P. 246,046). 237, Didymium and cerium (F.P. 594,783). 245, Thin wires (E.P. 235,893); Welding electrodes (E.P. 230,831 and 245,635); Protective coatings for metals (F.P. 592,986).

#### XII.—FATS; OILS; WAXES.

Marine animal oils. Sperm whale oil and spermaceti. E. ANDRÉ and T. FRANÇOIS (Compt. rend., 1926, 182, 497—499).—Glycerol has been isolated from sperm whale oils, after saponification, in the following proportions :—Oil from the cranial cavity (sperm oil)  $8\cdot1\%$ , subcutaneous fat  $1\cdot3\%$ , oil from flesh  $5\cdot5\%$ . Alcohols of high molecular weight occur to the extent of  $36\cdot4$ ,  $40\cdot0$ , and  $17\cdot5\%$  respectively in the same oils. Solid commercial spermaceti contains  $0\cdot7\%$  of glycerol. L. F. HEWITT.

Fat from spent [hydrogenation] catalyst. C. STIEPEL (Seifensieder-Ztg., 1925, 52, 967-968; Chem. Zentr., 1926, I., 1486).—Considerable quantities (up to 1.8%) of a brownish viscous fatty mass were obtained when catalyst residues were saponified to recover the fat in them. The product thus obtained consisted of oxidised fatty acids with a low iodine value and high saponification value. As no such compounds were contained in the raw materials of the process, they must have been formed in the hydrogenation process. According to Normann, oxygen present in the hydrogen used could have this effect. S. BINNING.

See also A., 239, Interfacial tension and emulsification (HARKINS and ZOLLMAN). 241, Solutions of sodium behenate (LAING).

Drying of fatty oils. EiBNER and RASQUIN.— See XIII.

#### PATENT.

Exposing liquids to ultra-violet rays. (G.P. 421,568).—See XI.

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

Fastness of pigments to light. H. WAGNER (Farben-Ztg., 1926, 31, 1023-1026, 1073-1076).--The use of artificial light sources in the examination of pigments for permanency is discussed. Parallel tests on 75 coal tar colours and their lakes and 14 mineral pigments, negative the possibility of deducing the actual time of withstanding sunlight from the results of accelerated tests, the conversion factor of "ultra-violet lamp hours" into "sun hours " differing widely with the different pigments. With this limitation, however, exposure to the ultra-violet lamp is a test of great value, since the fading follows the same general course as on exposure to sunlight, and reaches the same end point where complete bleaching does not occur. For graphically representing the colour variations, the author uses ternary diagrams founded broadly on Ostwald's colour scheme (cf. B., 1919, 914 A). Factors affecting the fastness of a lake, e.g., method of precipitation, adsorption on to the base, nature of the base, reduction with other pigments, refractive index and general nature of the medium, are considered from the experimental results and the optimum conditions inferred. The colours studied are finally arranged in 10 groups in descending order of fastness to light. S. S. WOOLF.

Lead suboxide as pigment. A. V. BLOM (Farbe u. Lack, 1925, 504—505; Chem. Zentr., 1926, I., 1005).—The gradual combination of lead suboxide with the decomposition products of linseed oil produces finely dispersed lead soaps which assist the rapid solidification of the film by opposing the swelling of the linoxyn, and also diminish the tendency of linoxyn to crumble on prolonged keeping, *e.g.*, for a year. S. WOOLF.

Catalysis of the drying of fatty oils at ordinary temperatures; formation of varnish in the cold. A. EIBNER and H. RASQUIN (Chem. Umschau, 1926, 33, 29-38; cf. B., 1925, 930).-The authors stress the relativity of the widely accepted division of fatty oils into drying, semi-drying, and non-drying oils, and do not accept the statement that "the olein in linseed oil does not dry." The behaviour of a typical non-drying oil (olive oil) was observed alone and mixed with driers and pigments on glass, on lead, and over a red lead-poppyseed oil primer. In the last case a particularly marked acceleration of the drying resulted. The chemical reactions involved are identical with those of drying oils, and although the delay in the physical film-forming processes renders the oil unsuitable for paint and varnish, yet, owing to the gradual nature of the changes, the drying of such oils provides more valuable information than the drying of linseed oil, which has been investigated almost exclusively. A further possibility is opened up by the comparatively rapid air-drying of olive oil that has been exposed to ultra-violet light for about a week. A sample of Andalusian olive oil was found to have the composition : oleic acid 79.7, a-linoleic acid 6.7,

palmitic acid 5.5, stearic acid 3.4, glycerol residue 4.2, unsaponifiable matter 0.9%. The glycerides present were triolein, a-linoleodiolein, oleodipalmitin, and stearodiolein, the first-named being present in largest quantity and the sum of the first two being larger than the sum of the last two. S. S. WOOLF.

Composition of oils of Bucovinian fir trees. O. CZERNY (Bul. Soc. Chim. Romania, 1926, 7, 91-93; cf. A., 1924, i., 659; B., 1925, 179).-By the distillation of fir tree rosin a crude oil is obtained with a blue fluorescence and disagreeable smell. This contains abietic acid (m.p. 156°) which can be extracted by sodium carbonate solution. The purified oil gives the Storch-Morawski reaction (violet colour with benzene, acetic anhydride, and stannic chloride). After further purification with potassium hydroxide solution at 50-60°, a clear yellow odourless oil is obtained with no fluorescence. On fractional distillation under a pressure of 9 mm. the following substances are obtained :--(1) 195-198°, a colourless oil,  $d^{15}$  0.9647, of empirical composition C<sub>18</sub>H<sub>26</sub>, which on oxidation and saponification yields trimellitic acid; (2) 240-265°, a colourless, odourless oil,  $d^{15}$  0.960, consisting of styrene; (3) 310–340°, a light blue oil of composition  $C_5H_4$ ; (4) 340–348°, a greenish aromatic oil,  $d^{15}$  0.9686, which on extraction with sulphuric acid yields diterebenthyl (Renard, 1888, A., 161). The residue from the sulphuric acid extraction, when heated with sulphur as directed by Renard (loc. cit.), yields an oil, b.p. 392-394°, identified as retene. Corrections to an earlier paper (loc. cit.) are given.

W. HUME-ROTHERY.

Relation between the viscosity of resin solutions and the constitution of the solvent. E. KEYSSNER (Z. angew. Chem., 1926, 39, 99-105).-Using a given resin and different solvents in molecular proportions, the viscosity of the solutions is practically identical with solvents belonging to a homologous series. Almost identical viscosities are shown also by solutions in benzene homologues, in di-, tri-, and perchloroethylene, and in various petroleum products. The magnitude of the viscosity depends markedly in the constitution of the solvent, being much greater with saturated compounds, e.g., perand pentachloroethane, than with unsaturated compounds, e.g., dichloroethylene. Differences in the behaviour of isomerides, e.g., o- and m-xylene, are very slight. The presence of a hydroxyl group in the solvent appears to cause an increased viscosity. W. T. K. BRAUNHOLTZ.

Aeroplane dopes. M. DESCHIENS (Chim. et Ind., 1926, 15, 17—32).—The author traces the historical development of cellulose ester dopes and describes the functions of their constituents (base, light solvents, diluents, heavy solvents, and plasticisers). The acetylation of cellulose is briefly discussed. Various formulæ for pigmented and non-pigmented dopes are given, and the different methods for their manufacture, the nature of the surface to be coated, and methods of application, mechanically or by hand, are described. Determinations of the rate of evaporation of solvents from first, second, and third coats showed that the first coat dries relatively slowly and requires a greater weight of dope owing to penetration of the fibre; the dried film retains appreciable amounts of heavy solvent for a period exceeding three months. The elasticity, impermeability, and tensile strength of fabrics are increased and air friction is lessened by doping.

S. S. WOOLF.

#### PATENTS.

Phosphorescent or luminous masses. H. M. MINES (E.P. 245,612, 18.5.25).-Phosphorescent materials of good durability and intensity that may be sensitised by artificial light and that will withstand grinding to pigment size without loss of luminosity, consist of a base composed of mixed alkali and alkaline-earth carbonates, with which is incorporated sulphur, small amounts of carbonaceous matter, such as starch, and very small amounts of "phosphorogens " and " luminophores," the whole being subjected to heat. The phosphorogens-thallium, thorium, uranium, bismuth, silver, or nickel compounds-act as light-emission centres, whilst the luminophores-sodium, potassium, manganese, barium, or calcium compounds-act as fluxes and convey the phosphorogens throughout the entire mass during the heat treatment. Formulæ for products emitting phosphorescence of the seven main spectrum colours are given as well as suggestions for obtaining intermediate colours. S. S. WOOLF.

Coumarone resin composition. C. ELLIS, ASST. to ELLIS-FOSTER CO. (U.S.P. 1,570,584, 19.1.26. Appl., 12.11.17).—Coumarone resin containing acid substances is heated above its melting point, and simultaneously treated with a basic neutralising agent, in the absence of sufficient water to dissolve the basic material. B. W. CLARKE.

Preparation of solutions of organic substances such as cellulose esters, resins, dyes, caoutchouc, etc. FARBW. VORM. MEISTER, LUCIUS, U. BRÜNING, Assees. of R. LEOPOLD and A. MICHAEL (G.P. 419,223, 2.2.24).—The solvents consist of *cyclo*acetals, prepared from aldehydes and a- or  $\beta$ -glycols, either alone or mixed with additional substances, especially alcohols. The following examples are given : ethylene ethylidene ether, Me·CH O—CH<sub>2</sub>, from glycol and acetaldehyde ; ethylene butylidene ether,  $Pr^{\alpha} \cdot CH$ O—CH<sub>2</sub>, b.p. 130°, from butaldehyde O—CH<sub>2</sub> and glycol ; acetal of 1:3-butylene glycol,  $MeCH·CH_2 \cdot CH_2$ 

b.p. 180°; product from formaldehyde and 1:3-butylene glycol, b.p. 115—120°. A. COULTHARD.

Preparation of a solvent which increases the drying capacity of drying oils, varnishes, etc. M. LUGEON (F.P. 592,595, 3.2.25).—Oil of turpentine is treated (warm) with a mixture of hydrogen and carbon monoxide, preferably water-gas, in the presence of an acid, for example, oxalic acid. The product so obtained is then treated with compounds of manganese, lead, cobalt, or titanium, for example, manganese dioxide, and the resinates or linoleates of these metals. The solvent is freed from insoluble matter by decantation. A. COULTHARD.

Reclaiming asphalt with production of lampblack. (U.S.P. 1,569,462).—See II.

Dyestuff emulsions. (E.P. 245,678).-See IV.

Exposing liquids to ultra-violet rays. (G.P. 421,568.)—See XI.

Treating pulverulent material. (U.S.P. 1,561,971.)—See XIV.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### PATENTS.

Treating pulverulent material [clay]. J. G. COFFIN and A. W. KEEN, Assrs. to NAUGATUCK CHEMICAL Co. (U.S.P. 1,561,971, 17.11.25. Appl., 16.10.23.).-An aqueous suspension of clay is freed from coarse particles by settling, thickened by further settling, and then, while still fluid, is sprayed into the top of a chamber up which air mixed with hot furnace gases at a temperature of about 350° is passed. The clay is dried in its fall through the chamber and is obtained in the form of lightly cohering globular particles. The process is specially applicable to the preparation of clay for use as a filler for rubber goods and may also be applied to other fillers, pigments, etc., and to the preparation of clay mixtures in the ceramic industry, different clays being mixed together in the form of slips and the mixture dried as described. T. S. WHEELER.

Solutions of caoutchouc (G.P. 419,223).— See XIII.

#### XV.—LEATHER; GLUE.

Reversal of the double refraction of collagen fibres when treated with certain vegetable tannins. A. KUNTZEL (Collegium, 1925, 623-627; cf. Ebner, A., 1896, ii., 457).—The plane of polarisation in which light is polarised by collagen fibres is rotated through 90° (*i.e.*, the double refraction is changed from positive to negative) when the latter are treated with gallotannic acid, sumach, tea, divi-divi, algarobilla, or myrobalans. Those tannins which do not change the plane of polarisation, cause weakening of the original double refraction of the collagen fibres. The phenomenon can be used for ascertaining the tannage used on a leather. Synthetic tans and sulphite-cellulose have no effect on the double refraction of the collagen fibres.

D. WOODROFFE.

Behaviour of sharpened limes in unhairing. M. KAYE and R. H. MARRIOTT (J. Soc. Leather Trades Chem., 1925, 9, 591-620).—Pieces of dried goatskin, fresh ox hide, or calfskin were treated

with various solutions. Lime liquor attacked and partially dissolved the cells of the hair roots and follicles until the latter collapsed away from the hairs. The lowest layer of the epidermis was also attacked and the epidermis split off completely in 7-8 days. The unhairing power of the sulphides of sodium, potassium, barium, and calcium is practically the same. Sulphides of ammonium and magnesium do not unhair. Bivalent sulphide ions are necessary for unhairing. Hydrosulphide ions do not unhair. The presence of hydroxyl or hydrosulphide ions does not prevent unhairing if bivalent sulphur ions are present. Polysulphides unhair similarly to sulphides. Their unhairing effect increases with the concentration and with the addition of hydroxyl ions, and decreases with the complexity of the polysulphide. Previous soaking of skin in lime or caustic soda liquors retards unhairing with sulphides or polysulphides according to the time of soaking and the concentration of the soak liquor. Acid soaking does not affect the unhairing. The rotting of the hair shaft is not a true measure of the progress of unhairing. Sodium sulphide dissolves hair due to a reaction between its sulphur and the sulphur of the hair. The cystine of hair is removed when the hair is treated with caustic soda solutions, hence previous alkaline soaking of skin alters the hair and the sulphide cannot combine with it. In sulphide liquors there is a reaction between soluble nitrogen products, sulphide sulphur, and oxygen from the air, resulting in a rapid disappearance of the sulphide sulphur. The presence of a film of grease on the hair does not prevent combination with sulphide sulphur, but protects the hair from the solvent action of the sulphide liquor. The action of lime on hair leads to the formation of sulphides in the liquors.

D. WOODROFFE.

#### PATENT.

Tans comprising a reaction product of sulphite-cellulose. J. BREEDIS, ASST. to ROHM AND HAAS CO. (U.S.P. 1,563,010, 24.11.25. Appl. 23.2.24).—Natural tans, partially insoluble in water, are converted into water-soluble tans by combining them with sulphite-cellulose waste lye by heating in the presence of free alkali, and then slightly acidifying the mixture. R. B. CLARKE.

#### XVI.—AGRICULTURE.

Nitrogen recuperation in the soils of the Bombay Deccan. I. D. L. SAHASRABUDDHE and J. A. DAJI (Mem. Dep. Agric. India, 1925, 8, 53-68). —The influence of various conditions on fixation of nitrogen in a Deccan soil taken at the end of the hot weather has been studied. On addition of water, fixation of nitrogen begins rapidly and continues for about 35 days. The amount fixed increases with addition of water up to 30%. Drying and re-moistening after fixation has taken place still further increases the amount of nitrogen fixed. Both fixation and nitrification are greater at 40° than at lower temperatures. C. T. GIMINGHAM. Better utilisation of phosphoric acid present in soils. J. WITYN (Z. Pflanzenkrankh., 1925, A 6, 27-51; Chem. Zentr., 1926, I., 1019).--The influence of the reaction of the soil on the nature of the phosphoric acid compounds present is discussed. Plants suffer more from lack of phosphorus in acid soils than in similar soils when the acidity has been removed by liming. On strongly acid soils, an application of mineral phosphates before liming is recommended.

#### C. T. GIMINGHAM.

Neubauer's "seedling" method [for determining manurial requirements of soils]. E. GÜNTHER (Z. Pflanz. Düng., 1926, B 5, 32—36).— Critical experiments on Neubauer's method for determining the phosphoric acid and potassium requirements of soils show that considerable variations in the light intensity during growth of the seedlings affect the percentage of nutrients absorbed to a small extent only. Similarly, the absorption of nutrients by the seedlings is almost unaffected by changing the  $p_{\rm H}$  of a soil from an initial value of 5.0 to 6.5 and 8.0 by addition of calcium carbonate. Under normal conditions, both light intensity and soil reaction can be neglected in carrying out the test. C. T. GIMINGHAM.

Comparative fertilising activities of different forms of nitrogen in new urea fertilisers derived from cyanamide. C. BRIOUX and J. PIEN (Compt. rend., 1926, 182, 410-412).-Addition of ammonium sulphate to soils increases nitrification, guanylurea sulphate has a slight depressing action, whilst dicyanodiamide almost completely suppresses nitrification. The following proportions of the nitrogen administered were absorbed by maize and white mustard plants when treated with the fertilisers named : sodium nitrate 70.3%, ammonium sulphate 68%, cyanamide 64%, urea  $63\cdot1\%$ , urea sulphate and phosphate 64%, "phosphazote" (containing 25% of the nitrogen present as guanylurea sulphate) 45.9%, guanylurea sulphate 13.2%, dicyanodiamide 6%. Since guanylurea sulphate is practically inactive, and dicyanodiamide toxic to the plant, the proportions of these in artificial manures must be maintained at a minimum. L. F. HEWITT.

Determination of phosphoric acid as magnesium ammonium phosphate. G. JÖRGENSEN (Analyst, 1926, 51, 61-72).-Several methods that have been proposed for the determination of phosphoric acid in mineral phosphates and fertilisers have been examined and the sources of error are pointed out (cf. A., 1925, ii., 824). The following procedure, which has been tested over a period of several years, is recommended to give results correct to within 0.1%. Three stock solutions are prepared : (a) 100 g. of ammonium molybdate are dissolved in 280 c.c. of ammonia (d 0.97), 300 c.c. of this solution are poured into 700 c.c. of nitric acid  $(d \ 1.21)$ , and the mixture is filtered on the next day; (b) 80 c.c. of ammonia  $(d \ 0.91)$  are added to a mixture of 90 c.c. of nitric acid  $(d \ 1.4)$  and 1500 c.c. of water, and the solution is diluted to 2 litres; (c) 50 g. of magnesium

chloride and 150 g. of ammonium chloride are dissolved in water and the solution is diluted to 1 litre. For the analysis of mineral phosphates, 5 g. are dissolved in 25 c.c. of nitric acid (d 1.21) and 12.5 c.c. of hydrochloric acid (d 1.12), the solution is diluted to 250 c.c. and filtered and 50 c.c. of the filtrate are treated with the requisite amount of (a). The mixture is heated for 10 min. at 50° on a water-bath, cooled, and filtered, the precipitate is washed ten times with 20 c.c. of (b) and dissolved in 100 c.c. of 2.5% ammonia solution, and the solution is treated at 100° with 30-35 c.c. of (c) added drop by drop with stirring. After cooling for 4 hrs. the precipitate is collected and converted into magnesium pyrophosphate as usual. For the determination of the total phosphoric acid in Thomas slag, 5 g. are dissolved in 40 c.c. of hydrochloric acid (d 1.18) without addition of nitric acid. Evaporation to render the silica insoluble is unnecessary; it may in some cases result in loss of phosphoric acid. The remainder of the analysis is conducted as described above. The quantity of solution (a) to be used is computed roughly from the equations x=0.12y+345p for mineral phosphates and superphosphates and x =0.34y + 412p for Thomas slag, where x is the number of c.c. of (a), y is the volume of the phosphate solution, and p the content of phosphoric anhydride. Direct precipitation of magnesium ammonium phosphate from ammoniacal solutions of commercial phosphates in the presence of ammonium citrate yields high results if considerable quantities of sulphates, ferric, aluminium, or calcium salts, or any silica or arsenic acid are present. A. R. POWELL.

Determination of ammonia in fertilisers by the formaldehyde method. W. SELKE (Chem.-Ztg., 1926, 50, 83).—For the determination of ammonia in comparatively pure ammonium chloride or sulphate the formaldehyde method gives good results if the solution is made neutral to phenolphthalein before adding the formaldehyde. With ammonium superphosphate or crude preparations of the sulphate complications are introduced by the presence of acidic impurities, such as the salts of iron and aluminium. In the case of the phosphate these salts produce a turbidity on neutralising and the end-point is difficult to discern; addition of sodium citrate overcomes this difficulty but even then good results are obtained only when a small weight of salt (say 0.5 g.) is taken. The concentration of the solution after the addition of the formaldehyde has an appreciable effect on the results. To correct for this a solution of ammonium sulphate containing approximately the same amount of ammonia in the same volume as the solution tested is prepared and titrated with alkali until phenolphthalein is just coloured pink; a few drops of methyl orange are added and the solution is titrated with acid until pink. The amount of acid so found is added to the c.c. of alkali used in the analysis proper.

A. R. POWELL.

Action of calcium and magnesium on seedlings of yellow lupin [Lupinus luteus]. BURK (Z. Pflanz. Düng., 1926, B5, 1-23).-A detailed series of experiments in sand culture are recorded on the effect of calcium and magnesium chlorides, separately and together, and in presence and absence of potassium sulphate, on the early stages of development of yellow lupins. Calcium chloride was added at the rates of 1.58 g. and 4.74 g. per pot holding 1.5 kg. of sand, and magnesium chloride in equivalent amounts. In general, the presence of calcium chloride was harmful and of magnesium chloride beneficial; the difference in the effects of the two salts was particularly marked in regard to the development of roots and root-hairs. When magnesium chloride or potassium sulphate was added with calcium chloride, the injurious action of the latter was almost completely neutralised. The harmful effects of calcium chloride and the beneficial effects of magnesium chloride were due to the cations and not to the chlorine ions. The literature on the subject of the sensitivity of lupins to the presence of calcium is reviewed.

#### C. T. GIMINGHAM.

Mineral content of pasture grass and its effect on herbivora. W. ELLIOT, J. B. ORR, and T. B. WOOD. I. General. W. ELLIOT. II. Effect of addition of mineral salts on the ration of sheep. W. ELLIOT and A. CRICHTON. III. Analyses of samples of British pastures. W. GODDEN. IV. Seasonal variations in mineral content of pastures. (MISS) E. M. CRUIKSHANK. V. Effect of fertilisers on mineral content of soils. W. GODDEN (J. Agric. Sci., 1926, 16, 59-64, 65-77, 78-88, 89-97, 98-104).-I. There is no marked difference between the energy values of the herbage of good and poor pastures. Important differences occur in mineral composition, a high mineral content being associated with high nutritive value. Certain cases of excessive mortality in sheep are related to low mineral content of the pasture herbage. II. "Bent leg" in sheep is correlated with mineral deficiency in the food and with a general lower nutrition. Mineral deficiency in pasture herbage can be correlated with high mortality in sheep. III. The herbage of hill pastures is generally poorer than that of cultivated pastures in silica-free ash and in the individual constituents with the exception of sodium. It is also rather poorer in nitrogen. Herbage left ungrazed bears a similar relationship to grazed herbage. There are no marked differences in energy value as measured by calorimetry. Animals appear to choose herbage of high mineral content in grazing. IV. There is a definite seasonal variation, more marked in good than in inferior types, in the mineral content of pasture grasses. The variation is most pronounced in the case of calcium oxide, which attains a maximum and then decreases. Similar but smaller variations are shown by silica-free ash, sodium oxide, phosphoric oxide, chlorine, and nitrogen. The date at which the content of ash and of the individual constituents reach a maximum varies for different fields and is probably influenced by grazing. V. Considerable modifications are produced in the mineral composition of pasture herbage by the application of artificial fertilisers. The

constituents showing the biggest variations are calcium, potassium, and, to a less extent, phosphorus. Increase in calcium is generally correlated with increase in nitrogen. G. W. ROBINSON.

#### PATENTS.

Production of solid urea from solutions. J. Y. JOHNSON. From BADISCHE ANILIN- U. SODA-FABR. (E.P. 245,687, 14.8.25).—A hot highly concentrated solution of urea is distributed in the form of a moderately coarse spray (not atomised). The drops solidify and urea is obtained in the form of "sandy" grains suitable for use as a fertiliser. If a 95% solution is used, no subsequent drying is required. C. T. GIMINGHAM.

Decomposition of materials containing phosphoric acid. N. KRAUTZ and L. DE MOLTKE-HUITFELDT (F.P. 595,133, 10.3.25. Conv., 11.3 and 22.4.24).—Mineral phosphate, in large pieces, is treated with hydrochloric acid in towers. C. T. GIMINGHAM.

Manufacture of salts of urea. J. BRESLAUER and C. GOUDET, Assrs. to Soc. D'ETUDES CHIM. POUR L'IND. (U.S.P. 1,572,638, 9.2.26. Appl., 2.5.22).—See E.P. 179,544; B., 1923, 901 A.

#### XVII.—SUGARS; STARCHES; GUMS.

Polarimetric determination of sucrose and its mixtures with other sugars. FINCKE.—See XIX.

#### XVIII.—FERMENTATION INDUSTRIES.

Partial pressures of aqueous ethyl alcohol. Dobson.—See A., Mar., 235.

#### PATENTS.

**Producing wort.** AKTIEBOLAGET SEPARATOR (E.P. 233,321, 30.3.25. Conv., 5.5.24).—The slime separated from the primary wort is leached and the wort thus produced separated by centrifuging, this process being repeated on the slime from the secondary wort. The wort obtained at a subsequent centrifuging and freed from solid impurities is used as a leaching liquid for a slime obtained at a previous centrifuging. T. H. POPE.

Preparation and use of lactates [in brewing etc.]. H. N. MURPHY (E.P. 246,278, 18.12.24).—Dry preparations containing lactates, for adding to water to be used in the manufacture of, e.g., beer, vinegar, and malt extract, are obtained by adding suitable salts, such as calcium chloride, sodium sulphate, potassium chloride, and sodium phosphate, with or without the addition of soluble peptones, to lactic acid solutions, which may or may not be heated, and wholly or partially neutralising the acid by the addition of calcium oxide, hydroxide, or carbonate. On keeping, the solution sets to a homogeneous mass. For example, a product obtained by using 25% of lactic acid, 25% of water, 20% of calcium sulphate, 10% of potassium chloride, 5% of sodium chloride, 5% of magnesium sulphate, 3% of peptone, and 7% of calcium oxide, is added to brewing water at the rate of  $\frac{1}{4}$ —1 lb. per 36 gals. L. A. COLES.

Manufacture of lactic acid. S. E. FAITHFULL (U.S.P. 1,569,221, 12.1.26. Appl., 8.5.24).-In making lactic acid by the fermentation, in presence of lactic acid bacteria, at 44.5-45.5°, of an acid liquor which contains somewhat hydrolysed carbohydrate material and in which accumulation of excessive acidity is prevented, a gaseous oxidising material containing free oxygen is introduced in amount sufficient to retard considerably the formation of butyric acid. The liquor is afterwards made alkaline and heated to near the b.p. of water to convert the remaining carbohydrates into caramel-like products. The hydrogen-ion concentration is then adjusted and proteins and similar nitrogenous substances are removed. T. H. POPE.

Manufacture of aldehydes, oils, and organic oils from cacti. W. M. SINCLAIR (U.S.P. 1,569,339, 12.1.26. Appl., 25.2.25).—Cacti are mashed without water in conditions favourable to fermentation by their indigenous fungus; the fully fermented pulp is hydrolysed and then distilled, the aldehyde being condensed and the essential oil separated from the aldehyde. T. H. POPE.

Combustible materials from peat (G.P. 421,734).—See II.

Exposing liquids to ultra-violet rays (G.P. 421,568).—See XI.

#### XIX.—FOODS.

Effect of short periods of cold storage on beef and mutton. W. M. CLIFFORD (Biochem. J., 1925, 19, 998-1003).—Beef or mutton kept at  $-4^{\circ}$  and  $2^{\circ}$  are identical in appearance with freshly killed meat up to the third day of storage. The meat kept at  $-4^{\circ}$  shows ice spicules and the red colour characteristic of frozen meat on the sixth day of storage. In hot English weather beef and mutton will not keep for 6 days in a room at  $2^{\circ}$ . There is no change in total nitrogen, soluble nitrogen, amino-nitrogen, carnosine, or creatine in meat kept at  $2^{\circ}$  for 3 days or at  $-4^{\circ}$  for 13 days. S. S. ZILVA.

Milk-fat determination in foodstuffs. J. KUHLMANN and J. GROSSFELD (Z. Unters. Nahr. Genussm., 1925, 50, 329-346).- Experiments have shown that the Reichert-Meissl values determined on small quantities of milk-fat and calculated to 5 g. are too high. The effect is even more pronounced in the case of coconut oil, though the presence of other fats may partially compensate for it. The difference (saponification value - Reichert - Meissl value - 200) is approximately proportional to the content of coconut oil in a mixture, and may be used to calculate a correction to be applied to the Reichert-Meissl value when coconut oil is present. The application of the A- and B-values of Bertram, Bos, and Verhage to the determination of coconut oil and milk fats (cf. B., 1926, 140) is discussed, and tables are given showing their values for various fat-contents. Other tables show the extent of the Reichert-Meissl value error for various quantities of milk-fat and coconut oil, and the content of milk-fat in mixtures corresponding to different Reichert-Meissl values, taking an average figure of 27 for milk-fat. J. GRANT.

Examination of confectionery products containing sugar and milk or cream. J. KUHLMANN and J. GROSSFELD (Z. Unters. Nahr. Genussm., 1925, 50, 346—351; cf. preceding abstract).— Tables are given showing the compositions of a number of commercial products, and the procedure for the analysis of such products is discussed. Fat content is determined by treating an aqueous solution of the sample with copper sulphate and sodium hydroxide solution, filtering off the coagulum, drying it, and extracting with a fat solvent.

J. GRANT.

Polarimetric determination of sucrose and its mixtures with lactose and other sugars, in cacao products, condensed milk, and sugar products. H. FINCKE (Z. Unters. Nahr. Genussm., 1925, 50, 351-365).-In the polarimetric determination of sucrose in cacao products, a factor has to be introduced to correct the reading for the volume of the insoluble matter. The values of this factor (x) are given in tables for a number of readings; or the following formula may be used :—  $x=1-(0\cdot 1-0\cdot 0072d)r$ , where d is the observed reading and r the volume of 1 g. of insoluble matter (0.9 c.c. for chocolate, and 0.7 c.c. for cocoa-sugar mixtures). The solutions are prepared for the polarimeter by the usual precipitation with lead acetate, but for milk-chocolate and condensed milk a second polarimeter reading must be taken after treatment with lime water (cf. B., 1924, 489). Tables are given for the determination of lactose from the difference of these readings, and correcting factors for the presence of lævulose, dextrose, maltose, and invert sugar have also been determined. J. GRANT.

Amyl alcohol unsuitable for milk testing by Gerber's method. S. GOY and J. JANISCH (Z. Unters. Nahr. Genussm., 1925, 50, 373).—When used for Gerber's test on milk, two samples of amyl alcohol were found to give results 0.4 and 0.6% too high. Blank tests using water in place of milk resulted in the separation of an unsaponifiable oil resembling petroleum. J. GRANT.

Variations in mineral content of cabbage and sauerkraut. W. H. PETERSON, C. A. ELVEHJEM, and L. A. JAMISON (Soil Sci., 1925, 20, 451-457).—Analyses of samples of cabbage and of sauerkraut from different localities showed wide variations in the percentages of calcium, phosphorus, iron, sugar, and nitrogen. There is a loss of 10-20% of the mineral constituents in making sauerkraut, if the amounts present in the juice are neglected. C. T. GIMINGHAM. Mineral content of pasture grass. ELLIOT, ORB, and WOOD.—See XVI.

#### PATENTS.

Apparatus for drying casein curd. E. FOSTER, Assr. to VERMONT CASEIN PTY., LTD. (U.S.P. 1,571,518, 2.2.26. Appl., 12.12.22).—See E.P. 198,957; B., 1923, 852 A.

Preparation of aerated beverages. O. A. ELIAS (U.S.P. 1,572,111, 9.2.26. Appl., 20.3.23).— See E.P. 197,499; B., 1923, 676 A.

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

Determination of nicotine by the method of Ulex. F. MACH and F. SINDLINGER (Z. anal. Chem., 1926, 67, 369—386).—The method described by Ulex for the determination of nicotine (B., 1911, 237) is shown to be erroneous at every stage of the operations. The loss of nicotine during the various treatments may amount to 9-26% of the total, 4-5% of the ammonia present in the material fails to be removed, and a quantity of ammonia equal to 20-25% of that originally present is produced by decomposition of nitrogenous compounds during the analysis. The total amount of ammonia that is finally titrated with the nicotine exceeds by 1-2% the nicotine lost by evaporation and decomposition, so that by this process it is possible to find a value for nicotine in plants that do not contain this alkaloid. The only exact method for the determination of nicotine is that involving precipitation with silicotungstic acid of the distillate obtained by distilling the substance with alkali in a current of steam. A. R. POWELL.

Nopinene as a technical raw material. G. AUSTERWEIL (Chem.-Ztg., 1926, 50, 5-6, 33-35; cf. B., 1925, 613).—The physical properties of pinene (m.p. -57°, b.p. 154·5—155°, d<sup>15</sup> 0·8595,  $n_D^{12}$  1·166, dispersion Hg yellow / Hg green 1·29) are contrasted with those of nopinene (m.p.  $-61^{\circ}$ , b.p.  $162-163^{\circ}$ ,  $d^{15}$  0.8635,  $n_{\rm D}^{12}$  1.487, dispersion Hg yellow / Hg green 1.06). A method for the technical separation of these substances is based on the greater solubility of nopinene in organic solvents miscible with water, and a table showing the solubilities in alcohol containing varying amounts of water is given: 60% aqueous alcohol dissolves almost exclusively the nopinene from a mixture of the terpenes. A greater yield of bornyl chloride is obtained from nopinene than from rectified turpentine oil, by the action of hydrogen chloride, but further cooling is necessary. Bornyl esters are obtained more readily from nopinene than from pinene and, more important, with careful attention to temperature and acid concentration, contamination with monocyclic terpenes can be avoided. L. M. CLARK.

#### PATENTS.

Extraction of the essential principles of plants, flowers, fruit, and the like. E. FORAY

(E.P. 232,552, 9.1.25. Conv., 19.4.24).—Perfumes and similar essences are extracted by means of ether vapour, and are subsequently absorbed from the vapour by means of sago, flour, sugar, alcohol, or similar material. L. A. COLES.

Manufacture of compounds of alkaloids. A. G. BLOXAM. From HACO-GES. A.-G. (E.P. 245,838, 14.10.24).—Stable compounds insoluble in water, and of therapeutic value, are produced by bringing together a nuclein or a nucleic acid, or compounds containing them, and an alkaloid, and subsequently coagulating the product, *e.g.*, by heat. For example, a solution of 2 kg. of morphine hydrochloride is added to a suspension of 100 kg. of yeast in 500 litres of water, and, after maintaining the solution at 60—65° for  $\frac{1}{2}$  hr., it is brought to the boil by introducing steam, and the coagulated product is removed and dried. L. A. COLES.

Producing chloro-derivatives of methane. H. WADE. From S. KARPEN AND BROS. (E.P. 245,991, 27.4.25).-Mixtures of 10-30% by vol. of chlorine with 90-70% of methane and methyl chloride are passed through a reaction chamber maintained at 350-800°, but preferably at 400-500°, and the products pass successively through a scrubber for removing hydrogen chloride, a gas-holder, a compressor in which they are compressed to 10-15 atm., a condenser, and thence into apparatus for separating the condensed liquid, which consists of a mixture of methylene chloride, chloroform, and carbon tetrachloride, from residual gas. The liquid is withdrawn and separated into its constituents by fractional distillation, and the gas is mixed with fresh supplies of methane and chlorine and returned to the reaction chamber. In operating the process to obtain a yield of about 80% of methylene chloride, 15% of chloroform, and 5% of carbon tetrachloride, 253.75 vols. of residual gas, containing about 60% of methane and 40% of methyl chloride, are mixed with 100 vols. of chlorine and 46.25 vols. of methane, before re-entry into the reaction chamber.

L. A. COLES.

Lactates (E.P. 246,278).—See XVIII.

Lactic acid (U.S.P. 1,569,221).—See XVIII.

Aldehydes, oils, and acids from cacti (U.S.P. 1,569,339).—See XVIII.

#### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Fogging action of hydrogen peroxide. W. CLARK (Phot. J., 1926, 66, 78-84).—Examination of the fogging action of hydrogen peroxide on singlelayer plates prepared with a particular emulsion showed that the percentage of grains made developable increased with increasing time of treatment up to a maximum at about 60% and then decreased with further treatment. The larger grains in the emulsion were on the average more sensitive to peroxide than the smaller grains, the relative sensi-

tivities of different size classes being roughly parallel with their sensitivities to light. These facts are not in agreement with observations of Wightman, Trivelli, and Sheppard using another emulsion (J. Franklin Inst., 1925, 200, 335). In the case of a single-layer emulsion treated vertically in still hydrogen peroxide solution, the percentage of grains made developable in a given time falls off with increasing depth in the solution. The chemiluminescence theory of the action of peroxide was examined by studying the effect of bathing normal plates in catalase solution on the photographic action of peroxide vapour and solution. The effect was always less with the catalase-bathed plates than with those bathed only in plain water, which is contradictory to what would be expected if the chemi-luminescence theory held, and seems to indicate a purely chemical action. The author does not now hold the view (cf. B., 1924, 116) that the sensitive centres in emulsion grains consist of silver oxide or hydroxide. W. CLARK.

#### XXII.—EXPLOSIVES; MATCHES.

Modifying the brisance of explosives [azides]. L. BIRCKENBACH and W. Rörig (Preuss. Bergakad. Clausthal, Festschrift, 1925, 123-135; Chem. Zentr., 1926, I., 1492).—An investigation of the modification of the brisance of metal azides when they form mixed crystals or double salts. Double salt formation was detected in the systems lead azide-lead chloride and lead azide-lead bromide. The double salt with lead bromide was not exploded by a 10-kg. hammer falling through 100 cm., whereas the lead chloride double salt exploded when the same hammer fell 65 cm., and pure lead azide exploded when a 2-kg. hammer fell 35-40 cm. Both these double salts contain about 50% of the respective halide. The crystals of the chloride double salt are clear, transparent, long monoclinic needles with  $a:b:c=1\cdot 2347:1:1\cdot 7179$  and  $\beta=$ 101° 4'. S. BINNING.

Detection of carbon monoxide in after-damp. WEIN.—See II.

#### PATENTS.

Explosives [of the Sprengel type]. A. C. SCOTT and H. L. SULMAN (E.P. 246,352, 4.4.25. Addn. to 231,541; cf. B., 1925, 479).—Finely divided crystals of potassium chlorate are filled gradually into a cartridge case under pressure so that the volume of the voids between the crystals is less than 32% of the total volume occupied by the crystals in the cartridge. The cartridges are then impregnated with kerosene by partial immersion and upward percolation. S. BINNING.

Preparation of initiators [primers, caps, and detonators]. C. CLAESSEN (G.P. 419,556, 8.3.25).— Acetylacetone peroxide is used as either a main or a secondary charge or as a component. It is not particularly susceptible to shock or friction, but is easily caused to detonate by a spark or flame.

S. BINNING.

#### XXIII.—SANITATION; WATER PURIFICATION.

Rapid fine sand [water] filtration. H. W. BLAISDELL (J. Amer. Water Works' Assoc., 1925, 14, 581—597).—To operate a sand filter at high rates of flow the sand must be either dry when put into the filter or consolidated in place by agitation under water. By the combined use of fine sand, reverse flow, and filter washer, waters of medium turbidities, subject to occasional flood turbidities of 300 p.p.m., without settling, can be filtered at rates of flow of 25-50 million gals. per day, and waters of lowturbidity at a rate of flow of 50 million gals. per day. A high-percentage removal of bacteria, turbidity, and colour is effected. Efficiency depends upon the very fine surface sand, about  $\frac{1}{8}$  in. in thickness, left after washing. A suitable sand when screened to 20-mesh has a sufficient percentage of fines to accomplish the desired result. Reverse flow is permitted at the slow rate of 15 million gals. per day or §in. rise per min. for 5-15 min. daily, or oftener if required; no loss of filtered water is occasioned by this treatment. The quantity of raw water used for washing is 0.025% of the water filtered. All waters so far dealt with have been successfully treated without the use of alum. The cost of installation and operation of a rapid fine sand filter is less than one-half that of any other filter now in use. A filter-washer designed by the author is described. W. T. LOCKETT.

#### PATENTS.

Process for treating alumino-silicates [for use in softening water]. H. and H. REINBOLD (U.S.P. 1,570,006, 19.1.26. Appl., 21.1.24).— Material for softening water is produced from alumino-silicates, such as bentonite, by calcining the mineral after it has been ground and sized, and then treating it with a soluble aluminium compound, after which it is dried to remove excess water. L. A. COLES.

Oxygen from peroxides [for respirators]. HANSEATISCHE APPARATEBAU GES., VORM. L. VON BREMEN U. CO. (G.P. 419,610, 24.5.18).—Mixtures capable of suddenly liberating oxygen (e.g., for respirators) are obtained by mixing an anhydrous alkali peroxide with an approximately equivalent amount of an anhydrous, difficultly volatile, acid anhydride or material liberating water of crystallisation or hydration only above 150°. Suitable substances are silica, tungsten trioxide, molybdenum trioxide, manganese dioxide, technically anhydrous copper sulphate, and alkaline-earth hydroxides. Substances, such as catalysts or acid-producing agents, which react with the above with evolution of heat are further appropriately added.

#### A. COUSEN.

Filtering apparatus [for water]. W. PATERSON (U.S.P. 1,572,076, 9.2.26. Appl., 4.2.25).—See E.P. 233,031; B., 1925, 565.

Exposing liquids to ultra-violet rays (G.P. 421,568).—See XI.